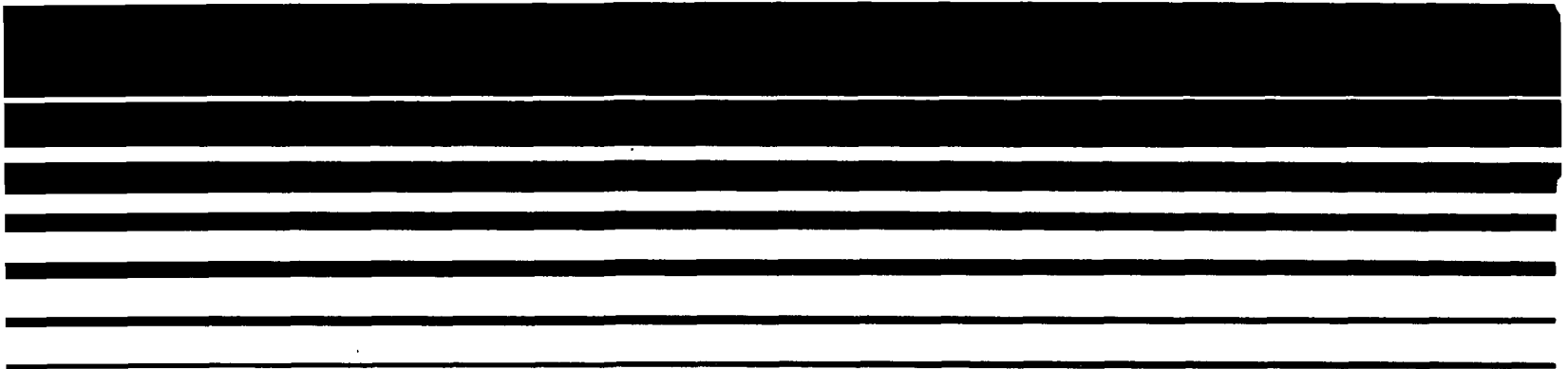


Air



Hazardous Waste Treatment, Storage, and Disposal Facilities

Site-Specific Test Report Burlington Northern Paradise, Montana



SITE-SPECIFIC TEST REPORT
BURLINGTON NORTHERN, PARADISE AND SOMERS, MONTANA
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1.0 INTRODUCTION

On September 21, 1985, Midwest Research Institute (MRI) observed process operations at Burlington Northern's facilities at Paradise and Somers, Montana. While on site, MRI took grab samples of the soil from two treatment, storage, and disposal related processes. It was originally intended that this site would be sampled in a manner similar to that of the seven other sites sampled as part of this study. However, due to (1) limited funds and (2) the availability of soil contamination data already obtained for this site, only limited grab sampling was conducted by MRI.

The purpose of visiting this site and the others in the 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 contaminated 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 contaminated fugitive particulate emissions from TSDF's.

The grab samples of soil collected by MRI at this facility were analyzed 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 μm) and the percent by weight of moisture in the soil.
- The degree of contamination of the soil silt fraction with metals, cyanide, and semivolatile organics.
- The percent by weight of soil silt that is less than 20 μm in diameter based on a sonic sieving technique.

At the Burlington Northern facilities, the two processes sampled were a soil storage pile at Somers, MT and a surface impoundment at Paradise, MT. No background samples were 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 cyanide and PEI and Associates performed the analyses for the semivolatile organics. Additional cleanup of semivolatile organic extracts was performed by Triangle Laboratories, Inc.

Field sampling was performed by Mr. Phillip Englehart of Midwest Research Institute (MRI). Mr. Lee Beck (EPA Lead Engineer) of the Industrial Studies Branch (ISB) observed the sampling program.

This report is organized into several chapters addressing 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 the processes sampled. The next chapter, "Sampling and Analysis," discusses the sample collection procedures, sample preparation, and sample analysis. The

appendices present the Analytical Data (Appendix A); detailed Analytical Procedures (Appendix B); Sampling Program Participants and Observers (Appendix C); and Process Operations Data (Appendix D).

2.0 SUMMARY AND DISCUSSION OF RESULTS

This chapter presents a summary of the analysis results and a brief discussion of the sampling and analysis procedures used. Since the detailed sampling and analytical procedures are not addressed in this chapter, it is recommended that the reader review Chapter 4, "Sampling and Analysis," prior to reading this chapter.

Soil samples were collected from two processes at Burlington Northern facilities. The processes included: (1) a soil storage pile at Somers, Montana and (2) a dry surface impoundment at Paradise, Montana. The analytical procedures used were those described in the Sampling and Analysis Protocol written specifically for this sampling program. These are briefly described in Chapter 4 and detailed in Appendix C. The sampling procedures deviated from the Sampling and Analysis Protocol in that the samples were collected using grab sampling techniques and therefore should not be considered random nor representative of the process from which they were taken.

This site-specific report is intended to present the data relevant to the samples obtained at one facility in this study and the procedures used to obtain these samples. Some statistical analyses may be performed on the data concerning this site; however, the majority of statistical analyses may involve the data collected over the entire study and will be included in a summary report to be completed at the conclusion of the program.

The sampling plan for the Burlington Northern facilities is shown in Table 2.1. In this case, the sample collection techniques involved using a scooping technique to obtain near surface samples. The analyses of the collected samples were conducted to measure the concentration of the most

TABLE 2.1. SAMPLING PLAN FOR BURLINGTON NORTHERN

Process Sampled	Process Designation	Number of Samples	Collection Method	Analyses
Soil Storage Pile Somers, MT	--	4	Grab Sample	Loss on drying Silt and PM ₁₀ content Metals and cyanide Semivolatile organics
Surface Impoundment Paradise, MT	--	2	Grab Sample	Loss on drying Silt and PM ₁₀ content Metals and cyanide Semivolatile organics

likely compounds or elements that could be soil contaminants (metals, cyanide, semivolatile organics, and pesticides).

According to the Sampling and Analysis Protocol, the collected samples were to be analyzed for the metals, cyanide, semivolatile organics, and pesticides. If significant quantities of cyanide, semivolatile organics, or pesticides were not expected to be present in samples from a particular process, the analysis of those corresponding compounds was not performed. MRI decided that at this particular site, pesticides were not likely to be present in significant quantities and therefore, pesticides analyses were deleted. All samples were analyzed for metals, cyanide and semivolatile organics. 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). Organic compounds in some samples caused the detection limits to be higher than desired for the semivolatile organic analyses. An alternative cleanup method was developed to minimize this problem, and the samples were analyzed at a lower detection limit.

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

2.1 SOIL STORAGE PILE (SOMERS, MONTANA)

Four grab samples were collected from the soil storage pile. The storage pile precluded the use of a sampling grid. The percent weight loss on drying (LOD) determined on a ten-gram aliquot of each sample averaged 11.7 percent. The storage pile samples were oven dried at 105^oC for 1.5 hours and then stored in a desiccator for 67.5 hours prior to being screened for silt content. The silt content of the four jars constituting the storage pile samples (sample identification numbers 11 through 14) averaged 10.2 percent silt by

weight (see Table 2.2). The composite silt material (sample identification by number 52) separated from the soil samples was sonic sieved. Material passing through a 20 um sieve constituted the PM_{10} content. The PM_{10} content averaged 10.84 percent by weight of the silt material. The silt screening did not produce a sufficient quantity of silt to allow the production of PM_{10} or greater than PM_{10} material (silt not passing through a 20 um sieve, referred to as $>PM_{10}$) for the chemical analyses.

Results of the analyses for metals, cyanide, and semivolatile organic compounds are shown in Table 2.3. The analytical results for the metals and cyanide in the storage pile silt sample (sample ID 51) are in terms of micrograms of the metal or cyanide per gram of silt sample (dry basis). The storage pile silt sample (sample ID 50) was also analyzed for semivolatile organic compounds. The analysis was on the storage pile silt composite sample extract prepared by following the low-level procedures in 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 screened by gas chromatography/flame ionization detection (GC/FID) as specified in the CLP and found to be at the medium level concentration (organic compound content greater than 19.8 ug/g) and cleaned by adsorption chromatography on Sephadex LH-20. The cleaned extract was analyzed after a 9.1-fold dilution resulting from the cleanup procedure and a second 10-fold dilution necessary to protect the gas chromatograph/mass spectrometer (GC/MS). At a detection limit of 29.7 ug/g, sixteen semivolatile compounds found on the CLP hazardous substance list (HSL) were detected in the soil storage sample (see Table 2.3). Four compounds (acenaphthylene, benzo(k)fluoranthene, indeno(1,2,3-cd)pyrene, and benzo(g,h,i)perylene) were found in the sample at concentrations below the quantifiable detection limit; they met the mass spectral criteria, however, and are reported as estimated values.

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

Site and Process	Sample ID	Percent Silt*	Percent Loss on Drying	Sample ID	Percent PM ₁₀
Somers, MT Soil Storage Pile	11	7.7	16.65		
	12	8.8	8.40		
	13	14.8	9.53	52	11.26
	14	9.3	12.21	52	10.41
		Average 10.2	11.7		10.84
		Std. Dev.	3.2	3.7	0.60
Paradise, MT Surface Impoundment	21	1.6	9.26	62	0.81
	22	0.4	11.98	62	1.05
		Average 1.0	10.62		0.93
		Std. Dev.	0.9	1.92	0.17

*All silt values determined using a full stack of sieves.

TABLE 2.3. ANALYTICAL RESULTS FOR METALS, CYANIDE, AND SEMIVOLATILE ORGANIC HSL COMPOUNDS, FUGITIVE PARTICULATE FROM TSDF (85/12)

Metals Analysis	Soil Storage, RCRA Pond	Pond Bottoms
Sample Identity	51 Silt	61 Silt
Element	(ug/g)	(ug/g)
Aluminum (Al)	23,736	16,461
Antimony (Sb)	<0.5	<0.5
Arsenic (As)	7.4	2.1
Barium (Ba)	222	176
Beryllium (Be)	1.00	0.40
Cadmium (Cd)	16.3	<5
Chromium (Cr)	31.5	21.5
Cobalt (Co)	6.2	4.4
Copper (Cu)	132	362
Iron (Fe)	18,405	12,412
Lead (Pb)	95.7	41.8
Manganese (Mn)	219	126
Mercury (Hg)	<0.03	0.12
Molybdenum (Mo)	<6	<6
Nickel (Ni)	12.8	12.1
Osmium (Os)	<2	<2
Selenium (Se)	<0.5	<0.5
Silver (Ag)	<9	<9
Thallium (Tl)	0.6	<0.5
Vanadium (V)	39.4	26.7
Zinc (Zn)	4,157	298
cyanide	<0.5	<0.5
Organic Analysis	Soil Storage, RCRA Pond	Pond Bottoms
Sample Identity	50 Silt	60 Silt
Compound	(ug/g)	(ug/g)
Napthalene	120	240
2-Methylnapthalene	300	670
Acenapthylene	8.4 J	38.0 J
Acenapthene	680	2,800
Dibenzofuran	420	1,500
Fluorene	650	2,600
Phenanthrene	710	4,800
Anthracene	480	2,300
Fluoranthene	370	2,600
Pyrene	290	2,100
Benzo(a)anthracene	170	790
Chrysene	160	850
Benzo(k)fluoranthene	6.1 J	480
Benzo(a)pyrene	59.0	280
Indeno(1,2,3-cd)pyrene	21.0 J	120
Dibenz(a,h)anthracene	N.D.	30 J
Benzo(g,h,i)perylene	15.0 J	89 J
Sample Detection Limit (ug/g)	29.7	94.0

N.D. = less than the samples detection limit.

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

With the exception of (1) the sampling procedures, (2) diluting the semivolatile organic sample extract prior to the GC/MS analysis, and (3) the use of the LH-20 cleanup method for the second analysis, all procedures for the soil storage pile samples followed the Sampling and Analysis Protocol.

2.2 SURFACE IMPOUNDMENT (PARADISE, MT)

The surface impoundment at Paradise, MT was also not sampled using a grid layout; instead two grab samples were collected from this process. The scoop sampling technique was employed to obtain the near-surface samples. The LOD for the samples (ID numbers 21 and 22) averaged 10.62 percent by weight. The samples were oven dried at 105⁰C for 1.5 hours followed by desiccation for 18.5 hours prior to silt screening. The two samples were screened for silt content which averaged 1.0 percent silt by weight (see Table 2.2). The silt composite (sample identification number 62), resulting from screening samples 21 and 22, was then sonic sieved for PM₁₀ content which averaged 0.93 percent by weight. The screening did not produce a sufficient quantity of silt to allow the production of PM₁₀ and >PM₁₀ fractions for chemical analyses. The results for the metals and cyanide are expressed in micrograms (ug) of the metal per gram of sample on a dry basis.

The silt fraction from the surface impoundment samples was also analyzed for semivolatile organic HSL compounds at two different detection limits. The analysis was conducted on the sample extract prepared by the low-level procedure. The sample extracts were screened by GC/FID and found to be at the medium concentration level. The extracts were cleaned by adsorption chromatography on Sephadex LH-20. The cleaned extracts were analyzed after a 20-fold dilution, in addition to the 14.3-fold dilution resulting from the

cleanup procedure. In the silt sample, seventeen semivolatile HSL compounds were detected. Three compounds (acenaphthylene, dibenz(a,h)anthracene, and benzo(g,h,i)perylene) were found at concentrations below the sample's quantifiable detection limit of 94.0 ug/g (see Table 2.3).

With the exception of (1) the grab sampling, (2) diluting the semivolatile organic sample extracts prior to the GC/MS analysis, and (3) the use of the LH-20 cleanup method, all procedures used for the surface impoundment samples followed the Sampling and Analysis Protocol.

2.3 CONCLUSIONS

No major problems were encountered collecting the grab samples; for sampling, the Sampling and Analysis Protocol was not followed and the samples should therefore not be considered representative of their respective processes as specified by this protocol.

The LOD measurement was intended to measure the moisture content of the soil samples. However, the LOD procedure is an indirect measure of moisture along with volatile components. A high bias can occur when volatile compounds are lost from the sample during the procedure. The LOD values were used to select the drying procedures for the samples (e.g., desiccation or oven drying). In the analyses of the samples, no problems were encountered in obtaining silt content or determining PM₁₀ content. The results of the metals analyses are also believed to be accurate.

The only significant problem encountered during the organic analyses was the fact that the samples contained a significant amount of non-HSL organic compounds. This prevented the semivolatile organics analyses from being conducted at the level described in the analytical protocol. Because of the high concentrations of organics, the samples had to be diluted to protect the

analytical equipment. An alternative sample clean-up procedure was used on the sample extracts in an attempt to remove these organics. The clean-up procedure used on the semivolatile organic sample extracts allowed the samples to be analyzed at lower quantifiable detection limits.

3.0 PROCESS DESCRIPTION

The primary rationale for conducting a site survey at this facility, was different than that for the remaining sites visited as part of the TSD particulate emissions study. Whereas the other facilities were chosen to represent more or less "permanent" TSDs that might be expected to continue operations indefinitely, this facility was chosen for a site visit in order to observe a set of activities with finite duration. Specifically, this facility was visited in order to observe the excavation of residual hazardous material from a surface impoundment (SI), and subsequent transfer of this material to an acceptable storage unit. Based on conversations with regional EPA personnel it appears that this sequence of operations--excavation and transfer--is an option that many facilities will pursue in response to the November 1984 Hazardous and Solid Waste Amendments to RCRA.¹ These amendments require that operating SIs have a double liner system; as a result residual material must be removed in order to install the liner system or alternatively to "close" the SI.

Because construction was not completed on the storage unit, no actual excavation and transfer operations were observed during the site visit. The following description represents the anticipated excavation and transfer procedures as supplied by facility personnel.

Figure 3.1 provides a schematic of the expected operations. The existing surface impoundment (A) contains approximately 14,500 yards³ of residual creosote-contaminated material (EPA hazardous waste No. K001). The existing storage pile (B) contains approximately 4,000 yards³ of K001 material. Note that B is located at a separate facility about 80 miles from A and C. After completion, the new storage pile (C) will contain about 18,500 yards³ of K001 material. C will have a double lined system with a bottom liner of 40 mil HDPE and an upper liner of 100 mil HDPE. The pile surface will be covered with plastic to prevent moisture infiltration and to control wind erosion.

According to facility personnel it is anticipated that the material in storage will eventually be used in a land treatment operation (D). At the time of survey, two pilot plots (200 ft² each) had been established and experiments were underway to determine environmentally acceptable loading rates, application frequencies, etc. About 20 acres are available for development of a full-scale land treatment unit. It is anticipated that the unit will consist of four, equal area (~ 5 acre) plots.

¹ Resource Conservation and Recovery Act (RCRA) Section 3004 Part (o)(1)(A) RCRA Section 3005 Part (j)(1) and (9).

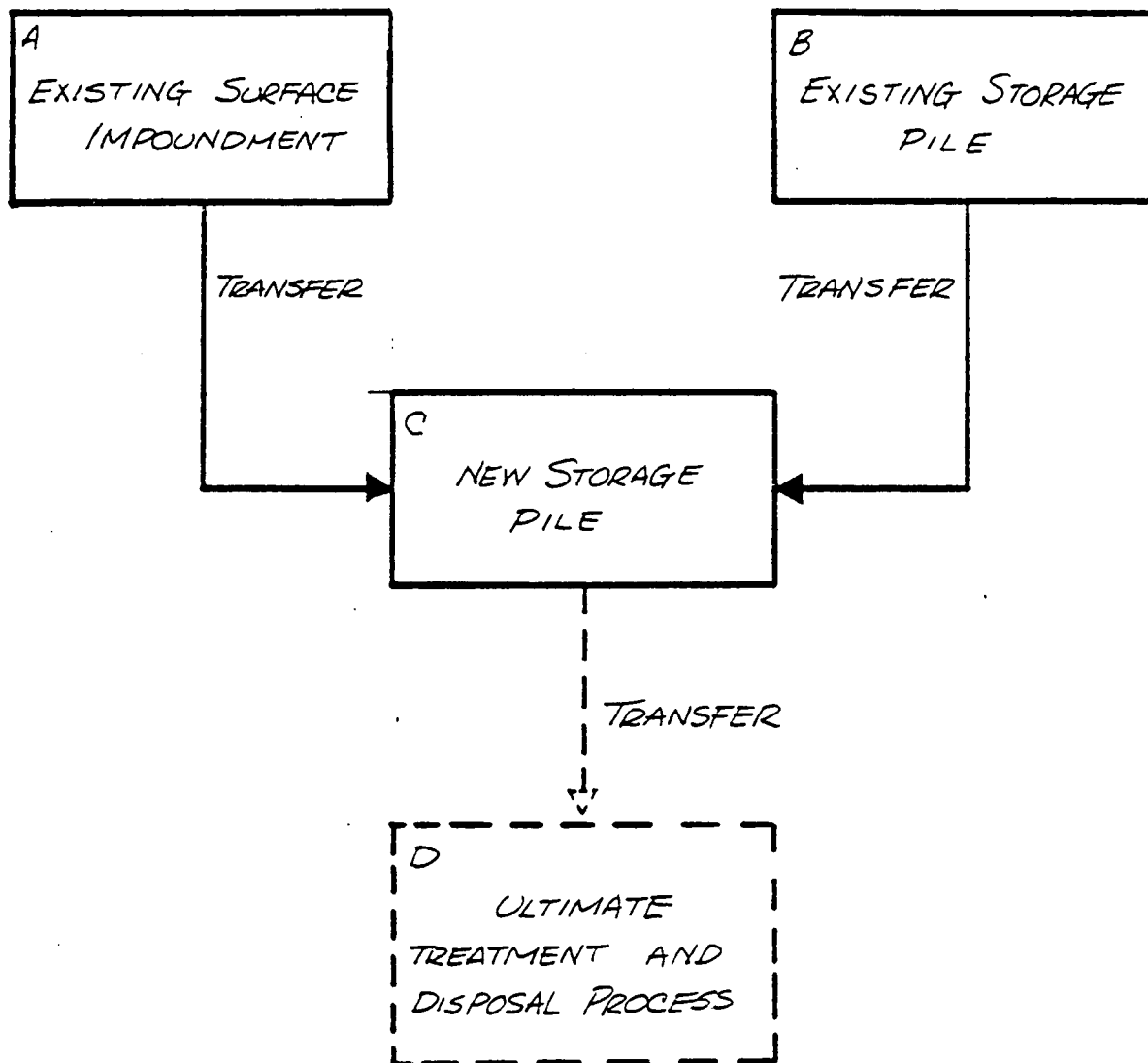


Figure 3.1. Schematic of expected operations for management of K001 wastes at the Paradise and Somers, MT facilities.

The principal equipment types, functions, and approximate level of activity expected for the excavation and transfer operations, are summarized below.

Equipment (commercial designation if available)	Function	Activity units
Excavator (JD-690A)	Excavation of material remaining in slough at Paradise, MT (A). Will be transferred to dump trucks and move to storage pile (C).	Bucket capacity--1 yd ³ ; short duration project with activity exceeding 8 hr/day.
Front-end loader (CAT 645-E or CAT 745-HB)	Anticipated function will be removal of material from existing waste pile (C) and transfer to dump trucks/trailers.	Short duration project. Activity will probably exceed 8 hr/day.
5 or 6 dump trucks-- 3 axle, 10 wheel; 3 tag-along trailers.	Transfer of waste pile material from C to A.	Dump truck capacity--12 yd ³ ; tag-along trailer capacity--10 yd ³ .

In removal of material from the existing waste pile, the front-end loader will be the only equipment operating in the waste pile area. An HDPE liner will be located adjacent to the pile and will be used as the load-in area. This represents an operational/control measure to prevent spreading of the contaminated material. After completion of the operation, the liner will be steam-cleaned.

4.0 SAMPLING AND ANALYSIS

This section outlines the procedures used for the sampling and analysis of the samples collected at the Burlington Northern facilities. Sample collection procedures did not follow those presented in the Sampling and Analysis Protocol developed for this sampling program. Rather, they consisted only of grab sampling the soil in specific areas and piles selected by the tester. The analytical procedures used to analyze these soil grab samples did follow the Sampling and Analysis Protocol. Analyses specific to this site are described in this chapter; any deviations from the standard analysis procedures (see Appendix C) are discussed.

Two processes were sampled: a soil storage pile and a surface impoundment. The samples from each of these processes were analyzed for silt and PM_{10} content, metals, cyanide, and semivolatile organics. A tabular presentation of the sampling plan for the Burlington Northern facilities 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 applicable analytical procedures.

4.1 SOIL STORAGE PILE (SOMERS, MT)

The soil storage pile was located at Somers, Montana. MRI determined that four samples would be collected from this pile. A simple grab sampling technique with no procedures to ensure representativeness was used to collect the samples. The four samples collected were numbered 11 through 14.

A ten-gram aliquot of each sample from this site was first analyzed for

weight loss on drying (LOD) by drying for 12 to 16 hours in a 105°C oven. Because the LOD value was greater than ten percent, all samples were oven dried at 105°C for 1.5 hours and stored for 67.5 hours in a desiccator (see Table 4.1). Following drying, the samples were screened to determine percent silt content and were sonic sieved to determine percent PM₁₀ content (see Appendix C for specifics of sample handling during each of these analyses). The part of the silt sample that did not pass through the 20 um sonic sieve was referred to as the "greater than PM₁₀" (>PM₁₀) fraction.

TABLE 4.1. SAMPLE DRYING PROCEDURE SUMMARY

Sample ID	Process Description	Drying Procedure
11	Soil Storage Pile Somers, MT	Oven dried at 105°C for 1.5 hours and stored for 67.5 hours in a desiccator
21	Surface Impoundment Paradise, MT	Oven dried at 105°C for 1.5 hours followed by 18.5 hours of desiccation

Using the screening techniques described in Appendix C, all the samples from this process were utilized to make composite samples of the silt. A portion of this fraction was then sent to RTI for metals and cyanide analysis. The silt screening did not produce a sufficient quantity of silt to allow for the production of PM₁₀ and >PM₁₀ material for chemical 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 limit of 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

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

Element	ICAP***	Detection Limits (ug/g)*	
		GFAA***	Cold Vapor AA***
Aluminum (Al)	40	-----	
Antimony (Sb)	-----	1.0	
Arsenic** (As)	-----	1.0	
Barium** (Ba)	0.7	-----	
Beryllium (Be)	0.1	-----	
Bismuth (Bi)	10.0	-----	
Cadmium** (Cd)	0.4	-----	
Chromium** (Cr)	0.7	-----	
Cobalt (Co)	0.7	-----	
Copper (Cu)	7.3	-----	
Iron (Fe)	100	-----	
Lead** (Pb)	10.0	-----	
Manganese (Mn)	5.9	-----	
Mercury** (Hg)	-----	-----	0.25
Molybdenum (Mo)	9.0	-----	
Nickel (Ni)	2.2	-----	
Osmium (Os)	4.0	-----	
Selenium** (Se)	-----	1.0	
Silver** (Ag)	10	-----	
Thallium (Tl)	-----	1.0	
Vanadium (V)	3.9	-----	
Zinc (Zn)	0.2	-----	

* 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

(Hg) samples were prepared and analyzed by the cold-vapor atomic absorption procedure following EPA Method 7471. 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 by EPA Method 7841 were diluted to achieve a final concentration of 0.5% nitric acid.

Cyanide determinations were done by colorimetric measurement following EPA Method 335.3 found in "Methods for the Evaluation of Water and Wastewater," EPA-600/4-79-020. The analyses for metals and cyanide were performed without any problems.

A portion of the composite sample of the silt was also sent to PEI; this was analyzed for the semivolatile organic compounds listed in Table 4.3. The silt from the soil storage pile was prepared for analysis of semivolatile organics following the low concentration level extraction method detailed in EPA Contract Laboratory Program, Statement of Work for Organic Analysis, 7/85 Revision (referred to as the CLP in this report). The sample extracts were screened by gas chromatography/flame ionization detection (GC/FID) to determine the concentration level of the organic compounds in the sample extract. The extracts were found to be at the medium level (i.e., containing any organic compound over 19.8 ug/g). An alternative cleanup procedure for the sample extracts using adsorption chromatography was developed to reduce the amount of sample dilution necessary to protect the GC/MS, which correspondingly allowed the GC/MS analyses to be conducted at a lower detection limit. The

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
 2-NITROANILINE

(Continued)

TABLE 4.3. (continued)

3-NITROANILINE
 4-NITROANILINE
 NITROBENZENE
 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

extract from the soil storage pile sample was concentrated and subjected to an adsorption chromatography cleanup procedure using Sephadex LH-20 (described in Appendix C). The clean up procedure resulted in a 9.1-fold dilution of the sample extract. The extracts were diluted another 10-fold prior to analysis on a capillary-column gas chromatograph/mass spectrometer (GC/MS). The dilutions resulted in a detection limit of 29.7 ug/g for the semivolatile organic HSL compounds. The dilutions resulted in a higher detection limit than the originally intended level of 0.330 ug/g, but the dilutions were necessary to protect the GC/MS.

4.2 SURFACE IMPOUNDMENT (PARADISE, MT)

The surface impoundment was located at Paradise, MT. MRI determined that two samples would be collected by the simple grab sampling technique described earlier. The two samples taken from the surface impoundment were numbered 21 and 22.

Because the LOD determination on a 10-gram portion of sample yielded a value greater than 10 percent, the samples from this process were oven-dried at

105°C for 1.5 hours followed by desiccation for 18.5 hours prior to screening (see Table 4.1). They were then screened to determine percent silt content and sonic sieved to determine PM₁₀ content (see Appendix C for a complete explanation of sample handling during these analyses).

The same screening and sieving techniques used for the soil storage pile samples were used to make a composite sample of the silt from the surface impoundment. Portions of the silt composite were sent to RTI for metals and cyanide analyses and to PEI for semivolatile organics analysis. All samples were analyzed for metals, cyanide, and semivolatile organic compounds as described previously for the composite sample from the soil storage pile.

Like the soil storage pile sample, the semivolatile organic extract from the surface impoundment sample was prepared by the low-level procedure, concentrated, and subjected to the LH-20 cleanup procedure. The cleaned extracts were analyzed by GC/MS after an additional 20-fold dilution following a 14.3-fold dilution resulting from the LH-20 cleanup procedure. The detection limit for the surface impoundment silt sample was 94.0 ug/g (see Table 2.4) after a total of a 285-fold dilution.

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. An NBS fly ash sample (1633 A) was used as a QA sample to check the overall accuracy of the digestion and analysis procedures. One sample was spiked with eight elements and their percent recoveries calculated to assess matrix effects. Another sample was analyzed in duplicate to demonstrate analytical precision. Quality assurance results for the metals analysis are presented in Table 5.1.

For the QA on the analysis of the semivolatile organics and pesticides (Table 5.2), PEI used a sample (ID number 50) 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 were calculated. The percent recoveries of the matrix spike compounds were outside the QA limits for all compounds except 2-chlorophenol in the MS and MSD samples and phenol in the MS sample. For acenaphthene and pyrene, the concentration of these compounds originally in the unspiked sample were 205 and 86.7 times higher than their matrix spike concentrations. For all the spike compounds, the spike concentrations were 5-to-10 fold less than the sample's quantifiable detection limit due to the sample dilutions necessary to protect the GC/MS. Six semivolatile compounds were detected in the MS and/or MSD samples, but not in the unspiked sample (see Table 5.2). The dilutions of the samples may also have been the reason that the compounds were found in the MS and/or MSD but not the unspiked sample.

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 Recovery			Duplicates	
	Expected	Found	Expected	Found	Expected	Found	Expected	Found	Percent	Silt	Silt
Elements (ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)	(ug/g)		(ug/g)	(ug/g)
Aluminum (Al)	-	-	140,000	18,000	58,000	14,000	30,859	31,436	-	89,102	83,695
Antimony (Sb)	8.2	9.0	7.0	3.5	0.73	0.73	-	-	-	1.5	1.3
Arsenic (As)	43.0	43.6	145	136	10.6	10.3	25.50	28.30	111%	12.0	5.7
Barium (Ba)	-	-	1500	743	-	46.0	1,713	1,300	76%	94.4	88.6
Beryllium (Be)	29.0	30.5	12.0	3.9	1.9	0.9	482	422	88%	3.7	3.6
Cadmium (Cd)	9.1	7.7	1.0	3.0	0.6	0.1	481	412	86%	<5	<5
Chromium (Cr)	7.1	6.8	196	41.4	71.0	31.3	173	142	82%	4,278	4,103
Cobalt (Co)	43.0	40.1	46.0	15.9	10.8	10.5	10.1	11.5	-	250	240
Copper (Cu)	8.9	12.3	118	43.3	25.1	23.3	594	541	91%	248	239
Iron (Fe)	-	-	94,000	35,000	36,500	23,000	15,285	14,718	-	173,248	172,113
Lead (Pb)	43.0	43.0	72.4	64.5	34.0	53.2	521	446	86%	97.3	94.5
Manganese (Mn)	13.0	12.9	190	78.0	513	322	619	550	89%	192	187
Mercury (Hg)	-	-	0.17	0.18	-	-	0.45	0.46	103%	<0.03	<0.03
Molybdenum (Mo)	-	-	29	66	-	25.4	109	84	77%	89.3	92.0
Nickel (Ni)	-	-	127	40.0	29.5	22.8	136	119	88%	528	483
Osmium (Os)	-	-	-	-	-	-	-	-	-	<2	<2
Selenium (Se)	7.6	6.9	10.3	7.6	0.4	0.4	20.0	19.7	99%	<0.5	<0.5
Silver (Ag)	-	-	-	-	-	-	494	437	88%	52.3	116
Thallium (Tl)	25.2	26.7	5.7	2.7	0.7	0.3	19.9	17.8	89%	0.5	<0.5
Vanadium (V)	130	123	300	121	72.4	42.9	158	147	93%	694	663
Zinc (Zn)	10.0	10.0	200	94.2	191	247	703	599	85%	963	912
cyanide	-	-	-	-	-	-	-	-	-	<0.5	<0.5

TABLE 5.2. QUALITY ASSURANCE RESULTS FOR SEMIVOLATILE ORGANICS ANALYSIS

SOIL SURROGATE PERCENT RECOVERY SUMMARY

Sample Identity	Silt 50	Silt 60	Sample Blank	Matrix Spike	Matrix Spike Duplicate
Surrogate Compounds					
Nitrobenzene-d5	72%	0%	0%	63%	72%
2-Fluorobiphenyl	108%	256%	1%	144%	126%
Terphenyl-d14	171%	456%	120%	180%	154%
Phenol-d5	0%	114%	0%	68%	63%
2-Fluorophenol	0%	0%	0%	23%	0%
2,4,6-Tribromophenol	72%	0%	72%	54%	90%

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY SUMMARY

Sample Identity 50	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	3.33	0.0	1.1	34%	0.0	0%	0%
Acenaphthene	3.33	680	682	60%	633	-1397%	-27.2%
2,4-Dinitrotoluene	3.33	0.0	6.6 J	198%	9.3 J	280%	-4.3%
Pyrene	3.33	290	661	11147%	569	8364%	3.6%
N-Nitrosodi-n-Propylamine	3.33	0.0	1.1	34%	0.7 J	21%	0%
1,4-Dichlorobenzene	3.33	0.0	0.0	0%	0.0	0%	0%
Pentachlorophenol	6.66	0.0	0.0	0%	0.0	0%	0%
Phenol	6.66	0.0	3.0	45%	0.0	0%	0%
2-Chlorophenol	6.66	0.0	2.7	41%	2.4 J	36%	0%
4-Chloro-3-methylphenol	6.66	0.0	0.0	0%	0.0	0%	0%
4-Nitrophenol	6.66	0.0	0.0	0%	8.5 J	128%	0%
N-Nitrosodiphenylamine *	0.0	0.0	15.0 J	-	16.0 J	-	-
4-Nitroaniline *	0.0	0.0	0.0	-	21.0 J	-	-
3,3'-Dichlorobenzidine *	0.0	0.0	15.0 J	-	6.6 J	-	-
Benzo(b)fluoranthene *	0.0	0.0	44.0	-	140	-	-
Dibenz(a,h)anthracene *	0.0	0.0	24.0 J	-	0.0	-	-
Benzo(g,h,i)perylene *	0.0	0.0	14.0 J	-	0.0	-	-
Sample Detection Limit (ug/g)		29.7	29.7		29.8		

* = 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	Di-n-butylphthalate	0.59 ug/g

All samples received were also spiked with surrogate compounds and then the percent recoveries of these compounds were determined. For nitrobenzene-d₅, the recoveries were within the QC limits for samples 50, 50 MS, and 50 MSD and the compound was not detected in the method blank or sample 60. For 2-fluorobiphenyl, the recoveries were within the QA limits for sample 50, above the limit for samples 60, 50 MS, and 50 MSD, and below the limit for the method blank. For terphenyl-d₁₄, the recovery for the method blank was within the QA limits and for samples 50, 60, 50 MS and 50 MSD the recoveries were above the QA limit. For phenol-d₅, the recoveries for samples 50 MS and 50 MSD were within the QA limits and for sample 60 the recovery was above the QA limit. The compound was not detected in the method blank or sample 50. For 2-fluorophenol, the recoveries were below the QA limit for all five samples with the compound only being detected in sample 50 MS. For 2,4,6-tribromophenol, the recoveries were within the QA limits except for sample 60 where the compound was not detected. Because the sample extracts were diluted, the surrogate compounds were present only in trace quantities. Surrogate compound recovery data are less accurate when the surrogates are at trace levels.

An analysis was conducted on a method blank sample consisting of a purified solid matrix spiked with surrogate compounds and carried through extraction, clean up, and concentration. The CLP specifies surrogate recovery limits for the blanks as well as limits on the levels of common phthalate esters and Hazardous Substances List (HSL) compounds. The blank results for di-n-butylphthalate were below the specified limit.