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A STUDY AIMED AT DEVELOPING THE REQUIRED CONTAMINANT DISTRIBUTION INFORMATION WAS AUTHORIZED AND FUNDED BY USATHAMA. THE STUDY INCLUDED DEVELOPMENT OF SAMPLING PROTOCOL, SAMPLE COLLECTION, LEACH TESTING, ANALYSIS, AND REPORT PREPARATION. THIS REPORT SUMMARIZES THE WORK CONDUCTED BY THE VARIOUS CONTRACTORS, DOCUMENTS THE DATA OBTAINED, PRESENTS THE RESULTING CONCLUSIONS, AND PROVIDES CERTAIN RECOMMENDATIONS. BORING LOGS AND DATA REPORTS HAVE BEEN APPENDED.

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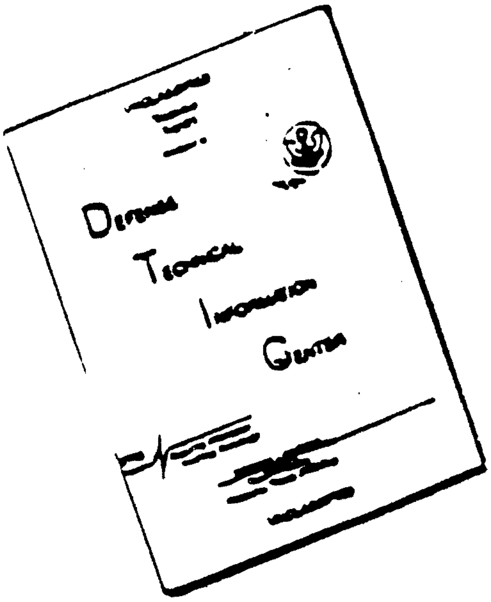

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BASIN F OVERBURDEN AND SOIL SAMPLING AND ANALYSIS STUDY
ROCKY MOUNTAIN ARSENAL

Environmental Laboratory
U. S. Army Engineer Waterways Experiment Station
P. O. Box 631, Vicksburg, Miss. 39180

by

Tommy E. Myers and Douglas W. Thompson

DRAFT

May 1982

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Prepared for US Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground, Maryland 21010

Under

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BASIN F OVERBURDEN AND SOIL SAMPLING AND ANALYSIS STUDY

PART I: INTRODUCTION

Background

1. Rocky Mountain Arsenal (RMA) located near Denver, Colorado, has been a chemical manufacturing and demilitarization facility since 1942. Military operations at the Arsenal have included the production of various chemical warfare agents, as well as the fabrication of munitions containing these agents and additional munitions containing White Phosphorous. Over the past decade, additional military missions at the Arsenal have included the demilitarization or destruction of various chemical agents and associated munitions and a Hydrazine blending operation. In addition to these military operations, private corporations have operated and continue to operate industrial facilities on the Arsenal under lease agreements for production of pesticides and other industrial chemicals.

2. Wastes from various chemical processes were discharged into unlined basins until an asphalt-lined evaporation basin designed for total waste retention was completed in 1956. This basin, designated Basin F, is located in the northwest part of the Arsenal in Section 26 (See Figure 1). At the maximum fluid level, Basin F had a surface area of 93 acres with a capacity of approximately 243 million gallons. The Basin, roughly oval in shape, was created in a natural depression by constructing a dike around the area. It measured approximately 2,900 feet across at the north end and 1,600 feet across at the south end. The average depth of the Basin was 10 feet. An asphaltic membrane (approximately 3/8 inches thick) was placed on the Basin bottom, extending to a projected high water elevation at the edge of the seal area. After the asphalt had been placed, a soil layer, one foot thick, was placed on top of the membrane to protect it. The asphaltic membrane was considered to have a design life of 15 years.

3. Through the years, wastes from the various Army operations and from the on-site production of pesticides by private corporations were disposed of in the Basin. These wastes included numerous inorganic and organic contaminants. Chemical analysis of the liquid in the Basin has been made periodically. A summary of an extensive chemical characterization analysis in 1977 is presented

in Table 1 (1). Waste disposal into Basin F was continued by various organizations through 1978. Since then and until recently, the primary flow into the Basin has been approximately 300,000 gallons per year from a Hydrazine blending facility and an undetermined amount of groundwater that infiltrates into the influent sewer line to the Basin. With the reduction in flow to the Basin, the volume of liquid contained has been steadily decreasing. The actual volume at any one time varies depending on climatological conditions, and the current estimated volume of liquid contained in the Basin is 30 million gallons. This reduced volume can be attributed primarily to loss through natural evaporation.

4. In early FY 82, an MCA program was initiated which includes the removal of the chemical sewer line back to the South Plants Area and the construction of a dike through the Basin to reduce the amount of surface water run-off into the existing liquid pool. Upon completion of the program, flow into the Basin will essentially be eliminated. The volume of liquid in the Basin should decrease due to evaporation to a point where 10 to 15 million gallons remain. Enhanced evaporation techniques under consideration for the liquid could reduce this volume even further.

Regulatory Setting

5. Basin F has been operated through the years essentially without restrictions on the amount or type of waste disposed in it. Clean up or control of the Basin was first addressed indirectly in 1975 by the "ceast and desist" orders issued by the Colorado Department of Health to the Shell Chemical Company and to RMA. Specifically, Shell and RMA were ordered to "take whatever steps are necessary to clean up all sources of the substances DIMP and DCPD and to perform all work necessary to ensure that ... DIMP and DCPD cannot enter the water of the state."

6. Then, in 1976, the Resource Conservation and Recovery Act (RCRA) was inacted. The resulting regulations were applicable to Basin F since it was an operating facility on 19 November 1980, and contained hazardous waste material. A notice of hazardous waste activity and Part A of the RCRA permit application was filed with EPA listing Basin F as a hazardous waste surface impoundment. As a result, the interim status standards as set forth in 40 CFR Part 265 are

Table 1

Chemical Characterization of Basin F Liquid (1977)

Compound or Parameter	Units	Concentration Range*
pH	-	6.9 - 7.2
Aldrin	ppm	50 - 400
Isodrin	ppb	2 - 15
Dieldrin	ppb	5 - 110
Endrin	ppb	5 - 40
Dithiane	ppb	30 - 100
DIMP	ppm	10 - 20
DMMP	ppm	500 - 2,000
Sulfoxide	ppm	4 - 10
Sulfone	ppm	25 - 60
Chloride	ppm	48,000 - 56,000
Sulfate	ppm	21,000 - 25,000
Copper	ppm	700 - 750
Iron	ppm	5 - 6
Nitrogen	ppm	120 - 145
Phosphorus (total)	ppm	2,050 - 2,150
Hardness	ppm	2,100 - 2,300
Fluoride	ppm	110 - 117
Arsenic	ppm	1.0 - 1.3
Magnesium	ppm	35 - 40
Mercury	ppb	26 - 29
Cyanide	ppm	1.45 - 1.55
COD	ppm	24,500 - 26,000
TOC	ppm	20,500 - 22,500

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* Based on the analysis of various samples from different locations and depths in the Basin.

applicable to Basin F. One of the major requirements under the interim status standards for such facilities is a written closure plan including a description of how and when the facility will be closed. These regulations are contained in 40 CFR Part 265, Subpart G. The closure plan was due 19 May 1981.

Data Requirements

7. In order to properly address the closure of Basin F, information is needed on the extent of contamination in the Basin area. Several field studies have been conducted in the Basin previously including sample collection and analysis (1, 2), but no information is available on the distribution of contamination below the liner. If contaminants have penetrated the liner and are contained in the underlying soils in high concentrations, the requirement for removal or clean up of the soils will have to be addressed in the final closure alternative. Additional information on the contaminant distribution in the sediment or overburden above the liner is also required.

Scope of Report

8. A study aimed at developing the required contaminant distribution information was authoized and funded by the US Army Toxic and Hazardous Materials Agency (USATHAMA). Computer Sciences Corporation (CSC) Inc., NSTL, MS was tasked to conduct the study with the support of RMA and US Army Engineer Waterways Experiment Station (WES) personnel. The study included development of a sampling protocol, sample collection, leach testing, analysis, and report preparation. RECRA Research, Inc., Amherst, N.Y., developed the sampling protocol; WES and CSC conducted the sample collection; Systems, Science, and Software (S-Cubed), Inc., LaJolla, CA performed the leach testing; Midwest Research (MRI), Inc., Kansas City, MO, conducted the sample analyses; and, WES prepared the final report. This report summarizes the work conducted by the various groups, documents the data obtained, presents the resulting conclusions, and provides certain recommendations. Boring logs and data reports have been appended.

PART II: METHODS AND PROCEDURES

Field Procedures

9. Proposed boring locations (a total of twenty) and recommended procedures for conducting the sample collection were developed by RECRA, Inc. and detailed in a sampling protocol which has been reproduced and is presented in Appendix A. Every attempt was made to follow the protocol during sample collection, however, certain minor modifications were made in the field as necessary to complete the work under the difficult conditions encountered (including severe cold weather, potential health hazards to personnel, and coordination with the construction activities on-going in the Basin). Four boring sites on the north end of the Basin were eliminated from the list of proposed sites due to the steep bank slopes encountered and the potential for personnel to come in contact with the liquid pool. The modified procedures used for sample collection and preparation are detailed below.

Establishment of boring locations

10. Based on the recommended site locations given in the protocol, a preliminary survey of the Basin was conducted and the proposed boring sites were located. A temporary bench mark (TBM) was established near the northeast gate of the Basin security fence from which the boring site locations were off-set. The surface elevation at each site was tied-in to the TBM. All locations were marked with a numbered, flagged stake for easy identification. Prior to initiation of sample collection, some of the sites had to be relocated to avoid interfering with the dike construction acitivities on-going in the Basin. The final sixteen boring locations are presented in Figure 2. No formal final survey of the completed boring sites was made, but such a survey can be made in the future if required.

Overburden removal and sampling

11. Overburden was removed from an area approximately 2.5 ft in diameter using shovels. Extreme care was exercised so as not to disturb the liner. Clean cloth rags were used to wipe the surface of the liner. A 2.0 ft diameter, steel caisson was placed in the hole and bentonite was poured around the outside of the bottom of the caisson. The outside of the caisson was then backfilled

to approximately 0.3 ft with overburden. The overburden and bentonite were mixed with a shovel in order to effect a seal between the liner and the bottom of the caisson. Overburden was then backfilled around the caisson to the original surface elevation. A plywood working surface was laid around the caisson, and the liner was re-wiped with clean cloth rags in order to remove excess bentonite. At this point, site preparation was complete.

12. At selected borings, the overburden was sampled by cutting down the face of the excavation after the liner had been wiped clean but before the caisson was placed. Each composite sample was placed in a 500 ml glass jar, sealed with a teflon lined lid, and labeled according to boring location and sample type. The liner was then re-cleaned with rags before the caisson was placed. This sampling procedure was used at borings No. 01, 14, 31, and 70. Two (2) 500 ml glass jars of overburden were collected for interlaboratory quality control purposes from stockpiled overburden at boring No. 11.

13. The overburden was field classified as to general appearance, color, and moisture. The liner condition was noted with regard to its presence (or absence), continuity, and general appearance. These observations were recorded for each hole on boring logs which are presented in Appendix B.

Soil boring and sampling

14. All soil sampling was done with a split-spoon sampler equipped with a basket shoe and a plastic sleeve (Figure 3). The sampler was manually advanced into the soil by driving with a 10 lb maul. Removal of the sampler from the hole was accomplished using pipe wrenches and a twisting/turning/pulling motion. Once out of the hole, great care was taken to avoid contacting the sampler with any contaminated surfaces. The sampler was broken down while supported in a cantilever fashion over a bucket. The plastic sleeve was removed, and the material lodged in the shoe of the sampler was pressed out and placed in the bottom of the sleeve. In this way, an undisturbed sample of the soil profile was obtained. The ends of the sleeve were then capped and labeled as to top or bottom and as to boring number and sample interval. The sleeves were transported to an on-site laboratory for classification and sampling for chemical analysis.

15. Between drives, the sampler was cleaned with a wire brush and wiped with clean cloth rags before a new plastic sleeve was inserted. The plastic sleeve prevented cross-contamination between drives. The drive rods were also

brushed and wiped clean. Each drive sampled a 1.5 ft interval. Three (3) drives were made on all but one hole, boring No. 23 where only two drives were made. After each drive, the depth of the hole was measured to confirm that the specified 1.5 ft interval had been sampled. Then using a 3 in. auger, the hole was cleaned out to the bottom of the completed drive. The auger was cleaned with a wire brush and wiped with clean cloth rags between drives. At this point the hole was ready for the next drive.

16. At borings No. 13, 14, 15, 21, 22, and 23 the liner was not trimmed prior to making the first drive with the sampler. For the remaining borings, the liner was trimmed away prior to the first drive using a flathead screwdriver. This technique was found to produce a better sample of the liner. The hole in the liner was cut so as not to contaminate the soils beneath the liner with liner material. The cut-out from the liner was tagged and placed in a glass jar for future reference. These samples (along with liner samples from the untrimmed holes) have been stored in Bldg. 802, RMA for future reference. At boring locations 01, 12, 14, 31, 33, and 70, a second hole was opened in order to obtain an extra soil sample from the 0.0-1.5 ft interval for bulk chemical analysis. The number and depth intervals of samples collected over the entire sampling effort are summarized in Table 2.

Closure of borings

17. Each boring was grouted with a mixture of Portland Type V cement and bentonite in ratio of 9:1 and 6-7 gal of water per bag of cement. (Type V Portland is a low tri-calcium aluminate cement that when compared to other cements has superior resistance to hydration and expansion caused by sulfate ion substitution). As grout was slowly poured into the hole, a wooden pole was used to vibrate voids to the surface. A permanent location marker and liner seal was established over the hole by filling a 12-in. ID sonotube mold with grout to an elevation of 0.4 ft above the surface of the overburden. In the case of borings No. 01, 12, 14, 31, 33, and 70 where a second hole was opened in order to obtain an extra soil sample, an 18-in. ID sonotube form was used. The larger ID was necessary in order to cover both holes. After the grout had set for a minimum of 24 hrs, the steel caisson was pulled and overburden was used to backfill around the marker. The measured height of the marker and overburden above the liner are presented in Table 3. Figure 4 provides details of a typically completed hole.

Table 2

Basin F Sediment and Soil Sampling Summary

BFB No.	Drives (6 ft) 0-1.5	1.5-3.0	3.0-4.5	Overburden
01	XX*	X	X	X
02	X	X	X	
11	X	X	X	X
12	XX	X	X	X
13	X	X	X	
14	XX	X	X	X
15	X	X	X	
21	X	X	X	
22	X	X	X	
23	X	X		
31	XX	X	X	X
32	X	X	X	
33	XX	X	X	
50	X	X	X	
60	X	X	X	
70	XX	X	X	X

---

* Double X's indicate sites where an extra drive was made for bulk analysis samples.

Table 3

Final Post and Overburden Elevations

Boring No.	Post Elev. Above Liner (ft)	Overburden Surface Elev. Above Liner (ft)
01	1.7	1.3
02	1.75	1.35
11	1.95	1.55
12	1.65	1.25
13	1.05	0.65
14	1.9	1.5
15	1.6	1.2
21	1.65	1.25
22	1.6	1.2
23	1.7	1.3
31	2.0	1.6
32	2.0	1.6
33	2.2	1.8
50	2.1	1.7
60	2.2	1.8
70	1.8	1.4

18. During the course of the sampling effort, severe weather conditions with sustained sub-freezing temperatures occurred forcing termination of the boring closure activity. Open holes were protected by taping a plastic sheet over the top of the steel caisson. Although the cold temperatures may have affected curing of the grout in some holes, an inspection made upon completion of the grouting revealed only minor sloughing from 1/2 to 1 in. of the marker top at some borings. The grout in immediate contact with the liner was not affected, thus insuring that each hole punched through the liner was securely sealed.

Photographic documentation

19. A color photograph was taken documenting the liner condition at each boring location either immediately before the caisson was placed or after site preparation for making the drives had been completed. Photographic documentation was also made of the boring procedures and of the grouting procedures. Complete sets of these photographs are on file at RMA and WES.

Safety procedures

20. Considerable efforts were made to insure the safe conduct of the work in the Basin F area. All personnel were required to wear disposable coveralls, rubber gloves and boots, safety galsses, and hard hats while in the Basin to prevent contact with any contaminated material. In addition, all personnel used respirators or air packs to prevent the inhalation of noxious or toxic fumes. All clothing, supplies, and materials other than respirators, air packs, and core samplers were disposed of in 55-gal drums which were left in the Basin. The equipment recovered was properly cleaned prior to removal from the basin area.

21. The safety procedures employed were reviewed by the RMA Safety Office and approved prior to initiation of work. These procedures complied with those specified by the RMA Safety Office and the CE, Omaha District for private contractors working in Basin F. Additional details are included in the sampling protocol or are available from the RMA Safety Office.

On-Site Laboratory Procedures

Sample preparation

22. The samples were processed and prepared for subsequent chemical analyses in an on-site laboratory located in Bldg. 741. Samples were removed from the plastic sleeves by tilting the sleeve and allowing the sample to slide out, top-end first, into lined core trays. Some samples were assisted by using a plunger to apply a slight force on the bottom end. Identification tags were placed on each core. Then color photographs of the individual cores were taken. Next, the cores were arranged and prepared for visual soil classification.

Soil classification

23. Field classification according to the Unified Soil Classification System (USCS) (3) was made by visual examination of the sample cores by an experienced geologist from WES who is familiar with the various types of soil found at RMA. Soil color was compared to standard soil color plates and appropriately noted along with the USCS calssification and soil texture. Texture was determined by visual examination. All classification information on the overburden and soils was noted on the boring logs. For reference, a summary table of the USCS has been reproduced and is included with the boring logs in Appendix B.

Subsample collection for chemical analysis

24. After the cores had been photographed and classified, the core from each boring was divided into four separate subsamples, each subsample consisting of all the soil in a particular one-foot interval. The intervals collected included 0.0-1.0 ft, 1.0-2.0 ft, 2.0-3.0 ft, and 3.0-4.0 ft, based on the depth below the liner. The subsamples were placed in 500 ml glass jars equipped with Teflon lined lids and labeled. The jars were sealed with tape, packed in an ice chest with blue-ice, and air freighted to S-Cubed for further testing. All samples arrived at S-Cubed on the day following collection. Those samples to be used for bulk analysis determinations were packaged in the same manner and air freighted to MRI. A list of the subsamples prepared is presented in Table 4. All residual soil and other materials from the cores were composited and returned to Basin F. Appropriate safety measures were taken in the laboratory to prevent immediate contact with the cores or inhalation of fumes by any personnel.

Table 4

Basin F Sediment and Soil Subsample Identification

BFB* No.	RMA No.	Sample Type	Interval (ft)
21	S20001	Core	0-1
21	S20002	Core	1-2
21	S20003	Core	2-3
21	S20004†	Core	3-4
22	S20005	Core	0-1
22	S20006	Core	1-2
22	S20007	Core	2-3
22	S20008†	Core	3-4
23	S20009	Core	0-1
23	S20010	Core	1-2
23	S20011	Core	2-3
13	S20012	Core	0-1
13	S20013	Core	1-2
13	S20014†	Core	2-3
13	S20015†	Core	3-4
14	S20016	Core	0-1
14	S20017	Core	1-2
14	S20018	Core	2-3
14	S20019†	Core	3-4
14	S20020	Surface	-
15	S20021	Core	0-1
15	S20022	Core	1-2
15	S20023	Core	2-3
15	S20024†	Core	3-4
70	S20025	Surface	-
70	S20026	Core	0-1
70	S20027	Core	1-2
70	S20028	Core	2-3
70	S20029†	Core	3-4

(Continued)

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* Basin F Boring.

† Indicates that the subsample was held and not extracted using the SWLP.

Table 4 (Continued)

BFB No.	RMA No.	Sample Type	Interval (ft)
60	S20030	Core	0-1
60	S20031	Core	1-2
60	S20032[†]	Core	2-3
60	S20033[†]	Core	3-4
32	S20034	Core	0-1
32	S20035	Core	1-2
32	S20036	Core	2-3
32	S20037[†]	Core	3-4
31	S20038	Surface	-
31	S20039	Core	0-1
31	S20040	Core	1-2
31	S20041	Core	2-3
31	S20042[†]	Core	3-4
33	S20043	Core	0-1
33	S20044	Core	1-2
33	S20045[†]	Core	2-3
33	S20046[†]	Core	3-4
12	S20047	Core	0-1
12	S20048	Core	1-2
12	S20049	Core	2-3
12	S20050	Core	3-4
01	S20051	Surface	-
01	S20052	Core	0-1
01	S20053	Core	1-2
01	S20054	Core	2-3
01	S20055	Core	3-4
11	S20056	Core	0-1
11	S20057	Core	1-2
11	S20058	Core	2-3
11	S20059	Core	3-4
50	S20060	Core	0-1
50	S20061	Core	1-2

(Continued)

Table 4 (Concluded)

BFB No.	RMA No.	Sample Type	Interval (ft)
50	S20062	Core	2-3
50	S20063	Core	3-4
02	S20064	Core	0-1
02	S20065	Core	1-2
02	S20066	Core	2-3
02	S20067	Core	3-4
11	S20068	Surface	-
14	S20069	Core (bulk)	0-1
70	S20070	Core (bulk)	0-1
33	S20071	Core (bulk)	0-1
12	S20072	Core (bulk)	0-1
12	S20073	Surface (bulk)	-
31	S20074	Core (bulk)	0-1
31	S20075	Surface (bulk)	-
01	S20076	Core (bulk)	0-1
11	S20077	Surface (bulk)	-

Leaching Procedure

25. Selected subsamples of the cores were leached using the Solid Waste Leaching Procedure (SWLP) as requested by USATHAMA. The procedure is detailed in a memorandum prepared by USATHAMA that has been reproduced and included in Appendix C. The SWLP involves a distilled water extraction of approximately 100 g of sample. The solution is mixed for 24 hours followed by filtration and analysis of the extract. S-Cubed performed the SWLP only on the fifty subsamples specified by RMA and WES personnel. After review of the results of the initial tests, five additional subsamples were extracted by S-Cubed using the SWLP (see Table 4). All remaining cores and excess soil not needed for testing were transferred to WES to be held for future testing if required. S-Cubed prepared a detailed summary of the extraction work conducted which has been reproduced and included in Appendix D.

Sample Analyses

Analysis of SWLP extracts

26. The SWLP extracts were air freighted to MRI for chemical analysis. A list of parameters for analysis of the samples was prepared by RMA and WES personnel. This list is as follows:

pH
Aldrin
Dieldrin
Endrin
Isodrin
DIMP
DMMP
Dithiane
Sulfone
Sulfoxide
DBCP
Mercury
Arsenic
Fluoride

MRI conducted these analyses using procedures approved by USATHAMA. Details of these procedures along with appropriate quality control data are on file at MRI and RMA.

Analysis of bulk samples

27. Bulk analyses were conducted on nine samples (as indicated in Table 4) for both organic and inorganic contaminants. Detailed written analytical methods for the bulk analyses were prepared by MRI which have been reproduced

and are included in Appendix E. In general, the organic analyses were conducted using standard GC/MS techniques while metal analyses were conducted using an inductively coupled plasma (ICP) emission spectrometer. Mercury and arsenic were analyzed using cold vapor and hydride generation AA spectrophotometry techniques, respectively. Fluoride analysis was conducted using an ion selective electrode method.

Control and distribution of analytical results

28. The completed analytical data were submitted by MRI to the analytical QA/QC group at RMA for review and concurrence. Copies of the data were then distributed to RMA and WES personnel for evaluation and use in this report.

PART III: RESULTS AND DISCUSSION

Depth of Overburden

29. The sediment or overburden as referred to in this report is the material located above the liner in Basin F. During construction of the Basin, a layer of sand approximately one foot in thickness was placed over the liner as a protective measure. Through the years of operation, additional material has been deposited in and on this sand layer probably due to precipitation of salts from the liquid, deposition of wind blown soil, and dumping of waste solids into the Basin. In certain areas of the basin where the overburden has been exposed (not covered with liquid) for long periods of time, it appears that some of the original cover sand has been lost, probably due to wind erosion.

30. In order to provide an overall picture of the depth of overburden in the Basin, a contour map was developed based on the measurements taken during the boring operations as presented in Table 3. This contour map is presented in Figure 5. The minimum depth found 0.65 ft, was at boring No. 13 which is in an area of the Basin exposed the longest time. The maximum depth found, 1.8 ft, was at borings No. 60 and 33, which are located in proximity to the two entrance gates in the fence on the east side of the Basin. The increased overburden depth in these areas may be the result of historic dumping of solid material into the Basin at these points due to their easy access. No information was obtained on sediment depths under the liquid, although past reports (1, 2) have indicated that the sediment is thicker in this area. This is probably true, since the various salts continue to precipitate from the liquid as additional water evaporates.

Liner Condition

31. During the conduct of the boring in the Basin, special attention was given to determining the condition of liner as the overburden was removed. The field personnel inspected the liner and noted its condition on the associated boring log. Overall, the liner was found to be in good condition with the exception of boring No. 2. In this area, the liner was liquified and had dispersed to some degree making it difficult to identify. Small holes, 1 to 2 inches in diameter, were found in the liner at boring sites No. 13 and 15.

The field personnel indicated that the liner irregularities at these two sites were probably the result of poor application technique.

Classification of Soils Beneath the Liner

32. As previously discussed, the cores taken in the Basin were field classified based on the USCS with the resulting information reported on the boring logs (Appendix B). This information was obtained in anticipation of future construction activities in the Basin where such information might be useful. In addition, certain contaminants can often be associated with specific soils with regard to adsorption or precipitation. Therefore, if certain intervals were found to be contaminated, it might be possible to relate contaminant distribution to soil type.

33. In order to illustrate the variations in soil types found with depth, a series of three horizontal cross-sections were prepared delineating the soil classifications in the intervals 0.1-1.0 ft, 1.0-2.0 ft, and 2.0-3.0 ft. Very little difference was found between the 2.0-3.0 ft and 3.0-4.0 ft intervals and therefore no cross-section was prepared for the deepest interval. The cross-section are presented in Figures 6, 7, and 8. Each specific soil group is identified using a standard symbol. The group symbols are described in Table 5.

34. The predominant soil groups identified include silty clays, inorganic silts, and inorganic clays. Inorganic clays become more predominant with increasing depth. The variation in soil types with depth over much of the Basin can be partially explained by considering the physical setting of the Basin. The Basin was constructed in a natural depression with the ground surface elevation decreasing from east to west and south to north. Therefore, dikes were constructed on the north and west sides of the Basin which entailed placement of fill. The soils found in the southeast section of the Basin probably represent the original, undisturbed surface.

35. All of the soil types identified provide some capacity for holding-up or retaining of contaminants since they are fine grained or contain clay or both. Generally, the higher the concentration of clay in a soil, the higher the capacity for retaining contaminants. In any case, if contaminants have passed through the liner in the Basin, sufficient amounts should have been retained in these soils to be evident.

Table 5

Descriptions of USCS Soil Groups (3)

Group Symbol	Typical group description	Example of corresponding USDA soil textural description
GW	Well-graded (poorly-sorted) gravels, gravel-sand mixtures, little or no fines	Gravel, gravelly sand
GP	Poorly-graded (well-sorted) gravels, or gravel-sand mixtures, little or no fines	Same as above
GM	Silty gravels, gravel-sand-silt mixtures	Very gravelly sand or silt loam
CC	Clayey gravels, gravel-sand-clay mixtures	Very gravelly clay loam
SW	Well-graded (poorly-sorted) sands, gravelly sands, little or no fines	Same as above
SP	Poorly-graded (well-sorted) sands, gravelly sands, little or no fines	Coarse to fine sand
SM	Silty sands, sand-silt mixtures	Loamy sand or sandy loam
SC	Clayey sands, sand-clay mixtures	Sandy clay loam or sandy clay

(Continued)

Table 5 (Concluded)

Group Symbol	Typical group description	Example of corresponding USDA soil textural description
ML	Inorganic silts, very fine sands, clayey silts, low plasticity	Silt or silt loam
CL	Inorganic clays, low to medium plasticity, lean clays	Silty clay loam or clay loam
OL	Organic silts and organic silty clays of low plasticity	Mucky silt loam
MH	Inorganic silts, micaceous or diatomaceous fine, sandy or silty soils, elastic silts	Micaceous or diatomaceous silt
CH	Inorganic clays or high plasticity, fat clays	Silty clay
OH	Organic clays of medium to high plasticity, organic silts	Mucky silty clay
Pt	Peat and other highly organic soils	Mucks and peats

Analytical Results

36. Due to the volume of the combined analytical data, the results of the analyses on the SWLP extracts and the bulk analyses are detailed in Appendix F. A separate list of tables for Appendix F has been prepared and is included in the front of the appendix to aid in locating particular data of interest. The analytical results are addressed only in a summary fashion in the following sections.

Results of analyses on the SWLP extracts

37. As indicated by the data tables in Appendix F, the concentration of many of the contaminants in the SWLP extracts were very low or below detectable limits. As a result, it was decided to purge the data base of these low values thus enabling the development of a less cluttered visible representation of the data. Action level concentrations for the contaminants were used as a reference for either eliminating or retaining a data point in the purged data base. An action level concentration is the EPA drinking water standard for a particular contaminant, if one exists, or a recommended maximum concentration established by the Army for those contaminants without regulated drinking water standards.

38. A list of action level concentrations has been prepared by USATHAMA for use at RMA. The action levels for the contaminants of interest in the SWLP extracts area as follows:

Parameter	Action Level
Aldrin	Hold to a minimum (assume 0.2 ppb)
Dieldrin	Hold to a minimum (assume 0.2 ppb)
Endrin	0.2 ppb
Isodrin	Hold to a minimum (assume 0.2 ppb)
DIMP	0.5 ppm
DMMP	No level set (assume 0.5 ppm)
Dithiane Sulfone Sulfoxide	100 ppb (total of all organo-sulfurs)
DBCP	0.2 ppb
Mercury	2.0 ppb
Arsenic	50.0 ppb
Fluoride	2.4 ppm

39. A plan map was developed for the purpose of summarizing the purged data base (Figure 9). All the contaminants found above their respective action levels in the SWLP extracts of the boring cores from the four intervals under the liner (0.0-1.0 ft, 1.0-2.0 ft, 2.0-3.0 ft, and 3.0-4.0 ft) are identified with respect to each boring site on the map. Those intervals from which either samples were not analyzed or no contaminants were found in the extracts above their action levels, are also identified.

40. The contaminants found in the SWLP extracts above their respective action level concentrations include Aldrin, Dieldrin, Endrin, Isodrin, organosulfurs, DBCP, arsenic, and fluoride. Some of the borings (No. 21, 22, 23, 60, and 70) had no associated extracts with contaminant concentrations above the action levels. Borings No. 1 and 2 (in "Little F") were found to have the greatest number of contaminants in the extracts for all intervals. They were the only borings in which the extracts of the cores from the 3.0-4.0 ft interval were found to contain organic contaminant concentrations above the action levels. The concentrations of the contaminants in the extracts associated with borings No. 1 and 2 were in general higher than those associated with the other borings. Overall, the results indicated that subliner soils associated with the borings outside of "Little F" are not highly contaminated. No particular correlation was found between contaminant distribution and soil type. This is probably due to the fact that all the soils identified contain significant amounts of clay and/or fine grained material.

41. The SWLP was conducted on overburden samples collected at five boring sites including Nos. 1, 11, 14, 31, and 70. The contaminants concentrations in these extracts were found to be much higher than in those associated with the below liner samples. In addition to the contaminants identified in the boring core extracts, concentrations of DIMP and DCPD were found in some of the overburden extracts.

Results of the bulk analyses

42. Bulk analyses were conducted on samples taken from the 0.0-1.0 ft core interval from borings No. 1, 12, 14, 31, 33, and 70, and on overburden samples from borings No. 11, 12, and 31. The results of these organic and metal analyses, as previously indicated, are presented in Appendix F. It should be noted that the concentrations of organics reported for the bulk analyses are

semi-quantitative in nature and should not be taken as absolute. The concentrations of contaminants in the bulk analyses range from 2 to 5 orders of magnitude greater than the concentrations in the SWLP extracts. This indicates that the SWLP extracted only a small fraction of the total amount of the contaminants present. The bulk analyses also indicated the presence of contaminants other than those analyzed for in the SWLP extracts. The reader is referred to the appropriate tables in Appendix F where these additional contaminants are identified. The additional contaminants identified in the core samples are not of particular concern due to their chemical natures and/or the generally low concentrations found. Of the overburden samples analyzed, the one from boring No. 11 was found to have the greatest number and highest concentration of organic contaminants other than those analyzed for in the SWLP extracts. A number of these contaminants could be of concern with respect to disposal of the overburden material, even though specific criteria or standards are not available.

Significance of Data with Respect to Basin F Closure

43. The RCRA regulations pertaining to the closure of a surface impoundment operating under interim status are contained in 40 CFR, Part 265, Subpart K, Section 265.228. The regulations specify two methods for closure of a surface impoundment. First, if the owner or operator elects to remove the following: (1) standing liquids, (2) waste and waste residues, (3) the liner, and (4) underlying and surrounding contaminated soil; or can demonstrate that none of these materials remaining at any stage of removal are hazardous: the surface impoundment can be closed without being subject to additional requirements of Part 265 regulations. Secondly, if the owner or operator does not remove all the impoundment materials or does not make the non-hazardous demonstration, the impoundment must be closed and post-closure care provided as for a landfill. The specific requirements for closure as a landfill are in general negotiable but at a minimum the remaining liquid must be removed by either treatment or drying. The specific requirements could vary from placement of a final cover along with a demonstration of positive control to prevent contaminant migration as detailed in Section 265.310, to construction of a liner (possibly including a leachate collection system) and placement of a final cover as detailed in Part 267, Subpart D, Sections 267.21, 267.22, and 267.23.

44. In order to evaluate the methods, a determination must be made of which impoundment materials are hazardous. A solid waste is defined as a hazardous waste under RCRA if it specifically listed in Section 261.31 (Hazardous Waste from Nonspecific Sources), Section 261.32 (Hazardous Waste from Specific Sources), or in 261.33 (Discarded Commercial Chemical Product, Off-Specification Species, Container Residues, and Spill Residues Thereof); if it is a mixture of solid waste and one or more hazardous wastes listed in Sections 261.31, 261.32, or 261.33; or if exhibits the characteristics of ignitability, corrosivity, reactivity, or EP toxicity. The liquid and sediment in the Basin are hazardous wastes since they are mixtures of solid waste and one or more hazardous wastes identified in the lists. The listed hazardous wastes disposed of in the Basin can in general be associated with lessee operations in the South Plants. In addition, the liquid and an EP extract of the overburden from the Basin have been shown to contain Endrin in excess of the 0.02 ppm criteria thus exhibiting the characteristic of EP toxicity. This information was developed as required for filing of Part A of the RCRA permit application.

45. Therefore, it is left to determine if the liner and the underlying and surrounding soils are contaminated. No specific tests have been conducted on the liner material and therefore no absolute determination can be made as to its hazardous or non-hazardous nature although, based on the RCRA criteria, the asphalt liner would probably not be classified as hazardous. However, in any anticipated construction activity in the Basin involving removal of the liquid and sediment, it would be impractical if not impossible to separate the liner material from the waste materials classified as hazardous.

46. With respect to the surrounding soils, a determination must be made as to the extent of contamination. The words "contaminated soils" as used in the RCRA regulations concerning surface impoundments have not been specifically defined in the regulations. However, based on the current philosophy being used by EPA in developing the regulations, any criteria developed concerning such contaminated soils will probably be based on the potential for the soils to act as a significant source of the contamination with respect to migration to underlying groundwater due to leaching. The extent to which a contaminated soil must be removed will probably have to be negotiated with the appropriate regulatory agency.

47. In the interim, until such time as the regulations are finalized and/or negotiations have been completed, USATHAMA has developed a criteria based

on the results of the SWLP and directed that it be used as a guideline in determining if contaminated soils represent a migration source. The criteria is detailed in the memorandum prepared by USATHAMA which has been included in Appendix C. In summary, the criteria is as follows: If the concentration of a particular contaminant in the SWLP extract of a soil exceeds 100 times the action level for that contaminant, then the soil is considered to have the ability to release the contaminant through leaching at a level requiring positive control or removal to prevent degradation of groundwater quality. Therefore, this criteria was applied to the soil samples collected from beneath the Basin F liner to determine which soil areas would require positive control or removal.

48. In applying the criteria, the action levels (as given in paragraph 38 of this report) were multiplied by 100 and the resulting values compared to the concentrations found in the SWLP extracts of the cores. Only the extracts from the cores collected at boring No. 2 from the 0.0-1.0 ft and 1.0-2.0 ft intervals exhibited concentrations exceeding the criteria (Figure 9). For the 0.0-1.0 ft interval, the concentrations of Aldrin, Dieldrin, Endrin, and Isodrin in the extract exceed the criteria. In the 1.0-2.0 ft interval, only the concentration of Dieldrin in the extract exceeds the criteria, although the concentration of Endrin is only slightly below the criteria. Thus, the soil beneath the liner around boring No. 2 was the only area found requiring positive control or removal based on the interim criteria.

49. As discussed previously, boring No. 2 was the only location in the study where the liner was found to be in poor condition. Contamination in the sediment in this area was probably able to migrate in high concentrations into the soil due to the deteriorated condition of the liner. In the other areas of the basin evaluated in this study, the liner appears to have maintained sufficient integrity to prevent the migration of large amounts of contaminants to the underlying soils.

50. Although the liquid and overburden in the Basin have been identified as hazardous wastes, the interim criteria were applied to the SWLP extracts conducted on the five overburden samples to determine if the contaminant concentrations found exceeded the criteria. Concentrations in the extracts from four of the five samples were found to exceed the criteria. Only the contaminant concentrations in the SWLP extract on the overburden sample from boring

No. 70 did not exceed the criteria. The contaminants identified whose concentrations in the extracts from the other samples exceeded the criteria are as follows:

Boring No.	Contaminants
1	Dieldrin, Endrin
11	Dieldrin, Endrin
14	Endrin, organo-sulfurs
31	Aldrin, Dieldrin

Therefore, based on the interim criteria, some positive control or removal action is required for the Basin F sediment regardless of the requirements imposed by the RCRA regulations.

PART IV: CONCLUSIONS AND RECOMMENDATIONS

Conclusions

51. The conclusions drawn from the evaluation of data obtained in this study are as follows:

a. The depth of overburden found above the liner in Basin F varies from 0.65 to 1.8 ft. This variation is probably due to wind erosion and deposition, precipitation of salt from the liquid, and dumping of waste solids into the Basin. The depth of sediment beneath the current liquid pool will probably be as great or greater than the maximum depth of overburden identified in this study due to continued deposition of material as the water portion of the liquid evaporates.

b. In general, the liner in Basin F in the study area is in good condition with the exception of the area around boring No. 2. It appears that the asphalt has been attacked and dissolved in this area. Therefore, the possibility exists that there may be other areas in the Basin not investigated in this study in which the liner has deteriorated.

c. The predominant soil groups identified as underlying the Basin F liner include silty sands, inorganic silts, and inorganic clays. Inorganic clays become more predominant with increasing depth. All of the soil types identified provide some capacity for retaining contaminants due to their clay content and fine grained characteristics. Therefore, of the contaminants passing through the liner, sufficient amounts should have been retained in the soil to be evident in the tests conducted.

d. Contaminants found in the SWLP extracts of the below liner soil cores in excess of their respective action level concentration include Aldrin, Dieldrin, Endrin, Isodrin, organo-sulfur, DBCP, arsenic, and fluoride. Borings No. 1 and 2 in "Little F" had the greatest number of contaminants in the extracts for all intervals. In general, the soils associated with the borings placed outside of "Little F' were not found to be highly contaminated.

e. No particular correlation was found between contaminant distribution and soil type.

f. The concentrations of contaminants in the bulk analyses were generally found to be several orders of magnitude greater than the concentrations in the SWLP extracts indicating that the SWLP extracted only a small percentage of the total amount of each contaminant present. Therefore, if the SWLP is assumed to simulate natural leaching conditions, the soils and overburden represented by the samples collected in this study could potentially continue to serve as sources of contaminants for a long period of time.

g. The liquid and overburden in the Basin are hazardous wastes since: (1) they are mixtures of solid waste and one or more hazardous wastes specifically identified in the RCRA regulations, and (2) the liquid and or EP extract of the overburden have been previously shown to contain Endrin in excess of the 0.02 ppm criteria and therefore exhibit the characteristics of EP toxicity.

h. Although under the current RCRA criteria, the asphalt liner would probably not be classified as hazardous, it would be impractical if not impossible to separate the liner material from the waste materials classified as hazardous.

i. Based on the criteria developed by USATHAMA, the soils associated with 0.0-1.0 ft and 1.0-2.0 ft intervals at boring No. 2 (in "Little F") were the only underlying soils evaluated in this study that were determined to represent a contaminant migration source. The contaminants whose concentrations in the SWLP extracts were found to exceed the criteria include Aldrin, Dieldrin, Endrin, and Isodrin.

j. Contamination in the overburden in proximity to boring site No. 2 was probably able to migrate in high concentrations into the underlying soil due to the deteriorated condition of the liner. In the other areas of the basin evaluated in this study, the liner appears to have maintained sufficient integrity to severly limit such migration.

k. Concentrations of certain contaminants in the SWLP extracts of the overburden in the Basin exceeded the USATHAMA criteria and thus the overburden was determined to represent a contaminant migration source in addition to being previously identified as a hazardous waste.

Recommendations

52. The following recommendations are made with regard to the eventual closure of Basin F:

a. Any final closure alternative considered for Basin F should address the removal and/or treatment of the liquid, overburden, and liner to the degree required under the RCRA regulation in effect at that time. In addition, positive control or removal actions should be included for the contaminated soil underlying the liner in "Little F." If removal is selected as the appropriate action in the "Little F" area, the soil should be excavated to a minimum depth of 2 ft.

b. As the liquid pool remaining in Basin F decreases in size, additional investigations should be conducted in that area to determine the extent of contamination in the soil underlying the liner at that point. The results of this study indicate that a simple physical inspection of the liner will provide a good indication of potential trouble spots without additional extensive boring, sampling, and analysis. Future investigations should be concentrated in areas where the liner is found to be deteriorated or breached.

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2. Mullen, J. C., Olsen, R. L., and Taylor, D. B. 1980. "Investigation of Basin F Solution Alternatives," D'appolonia Consulting Engineers, Inc., Denver, CO.

3. US Army Engineer Waterways Experiment Station. 1960. "The Unified Soil Classification System," Technical Memorandum No. 3-357, Vol. 1, Vicksburg, MS.

FIGURES

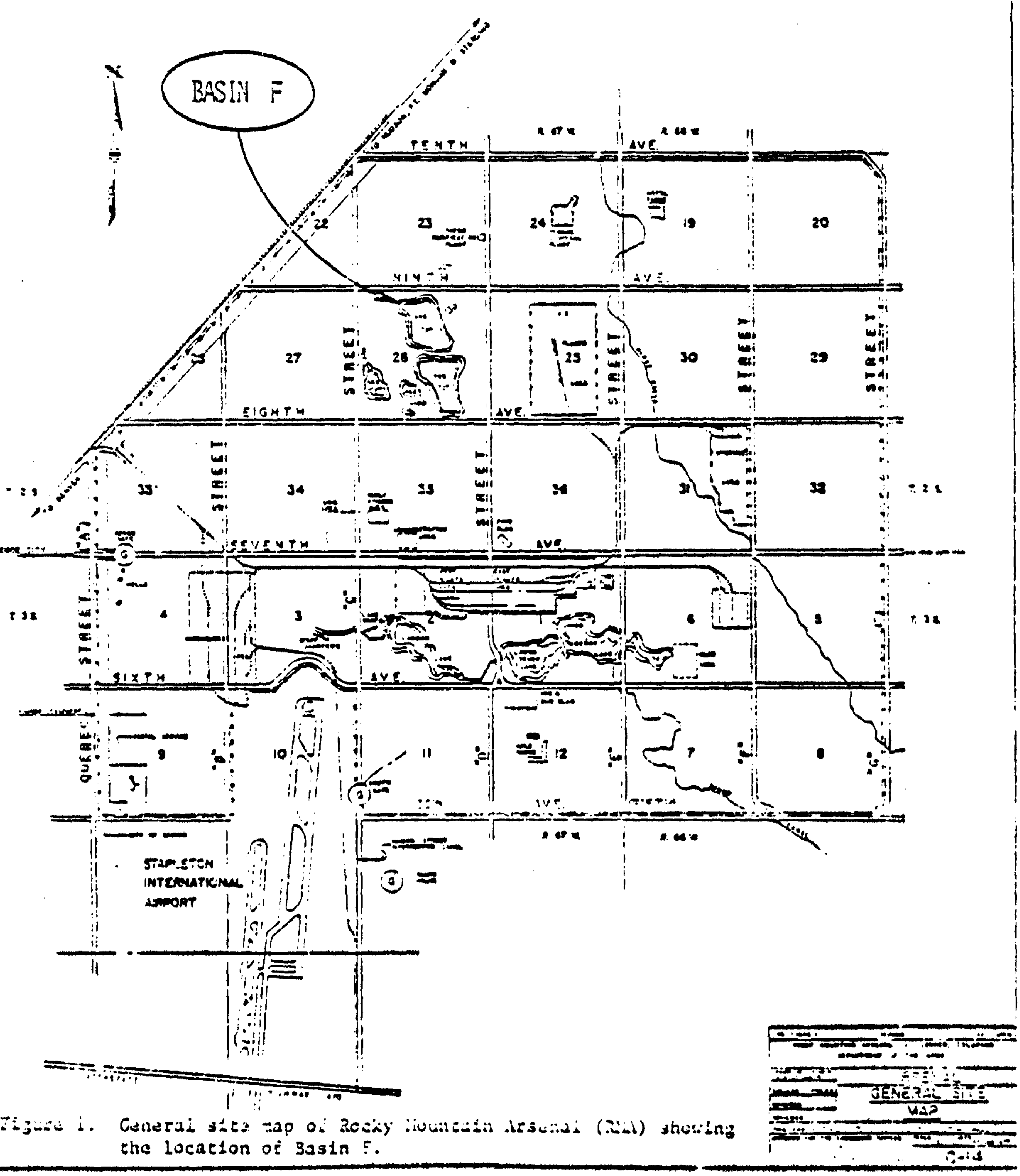


Figure 1. General site map of Rocky Mountain Arsenal (RMA) showing the location of Basin F.

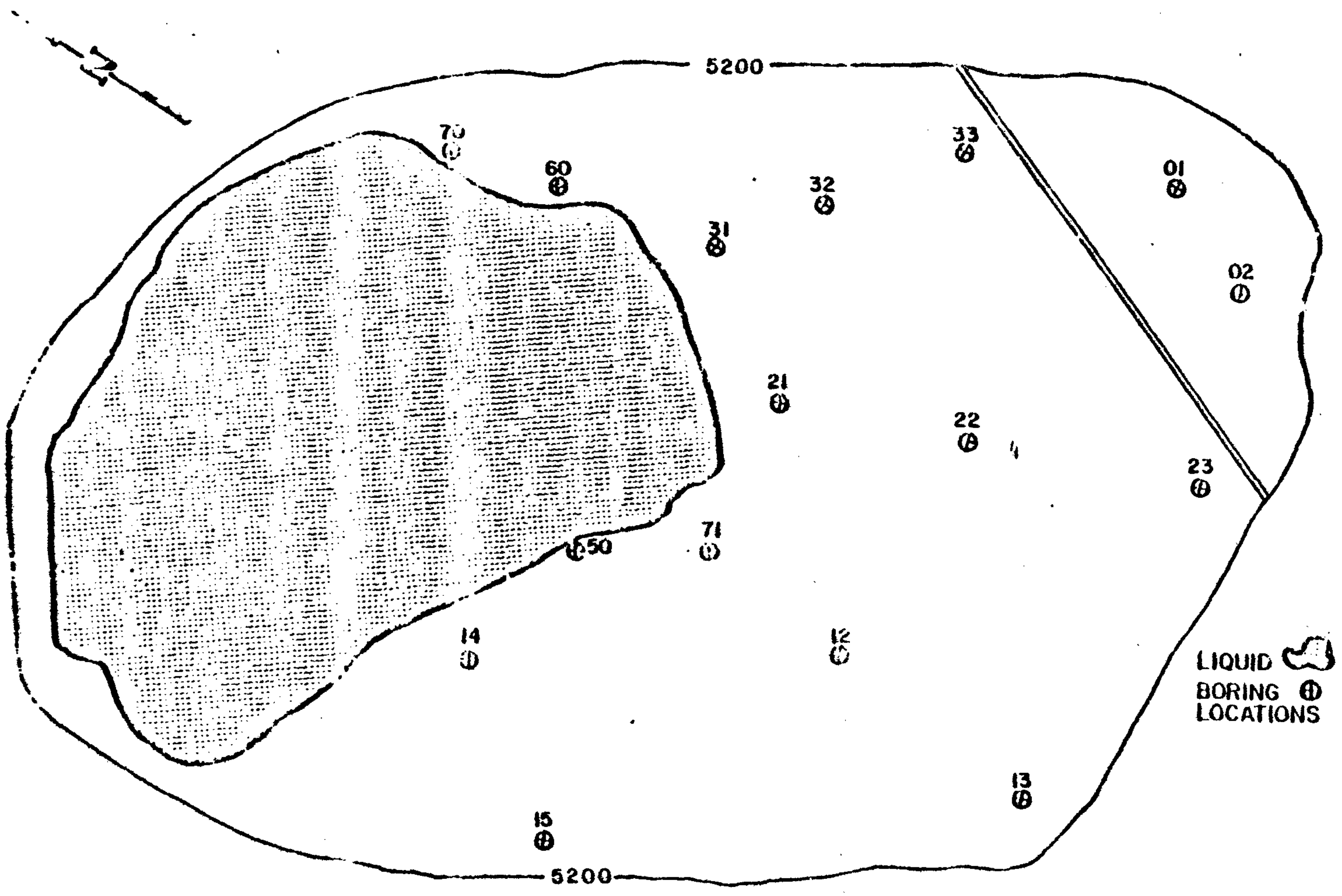


Figure 2. Location of boring sites within Basin F.

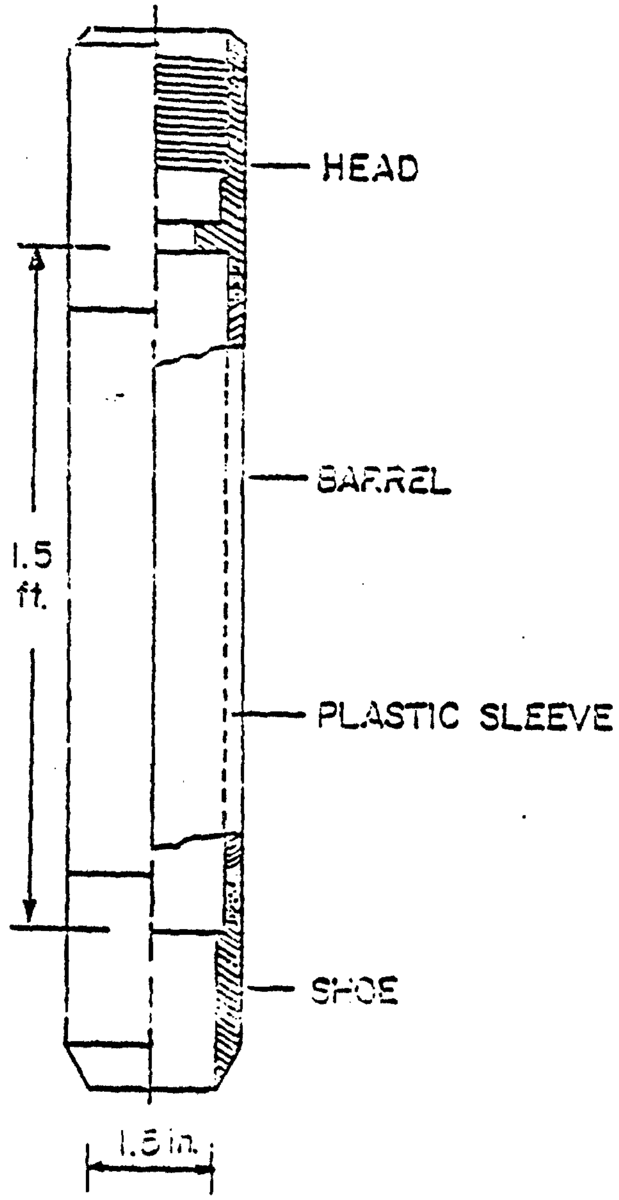


Figure 3. Cut-away view of split-spoon sampler

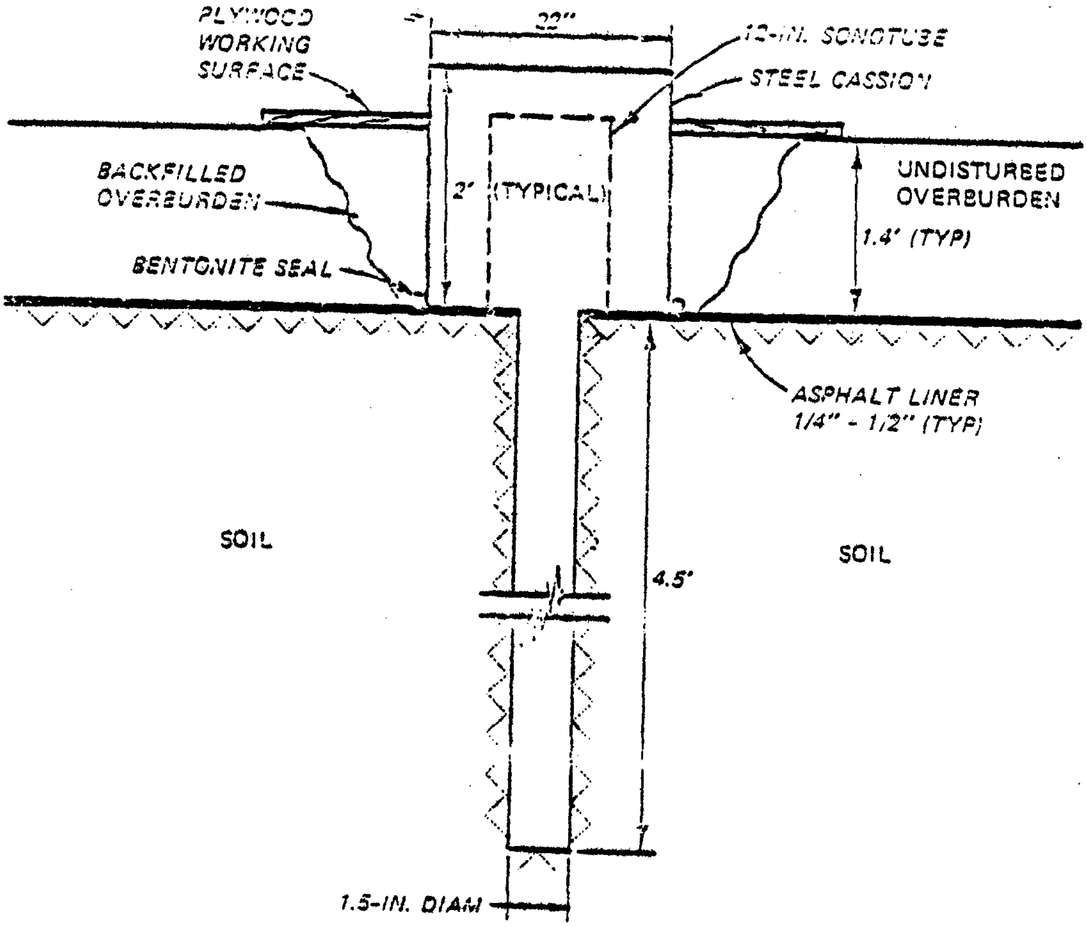


Figure 4. Typical detail of completed boring.

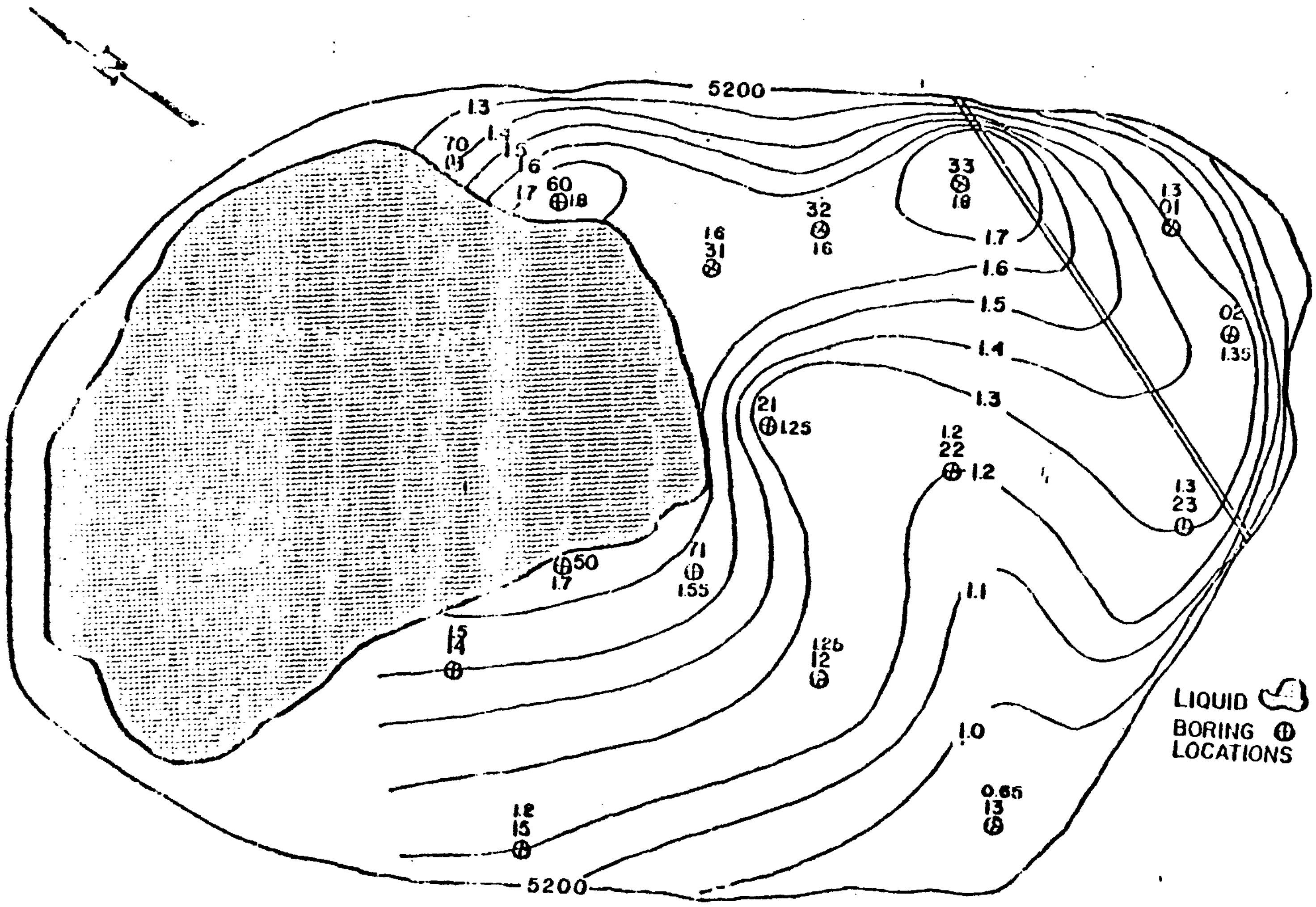
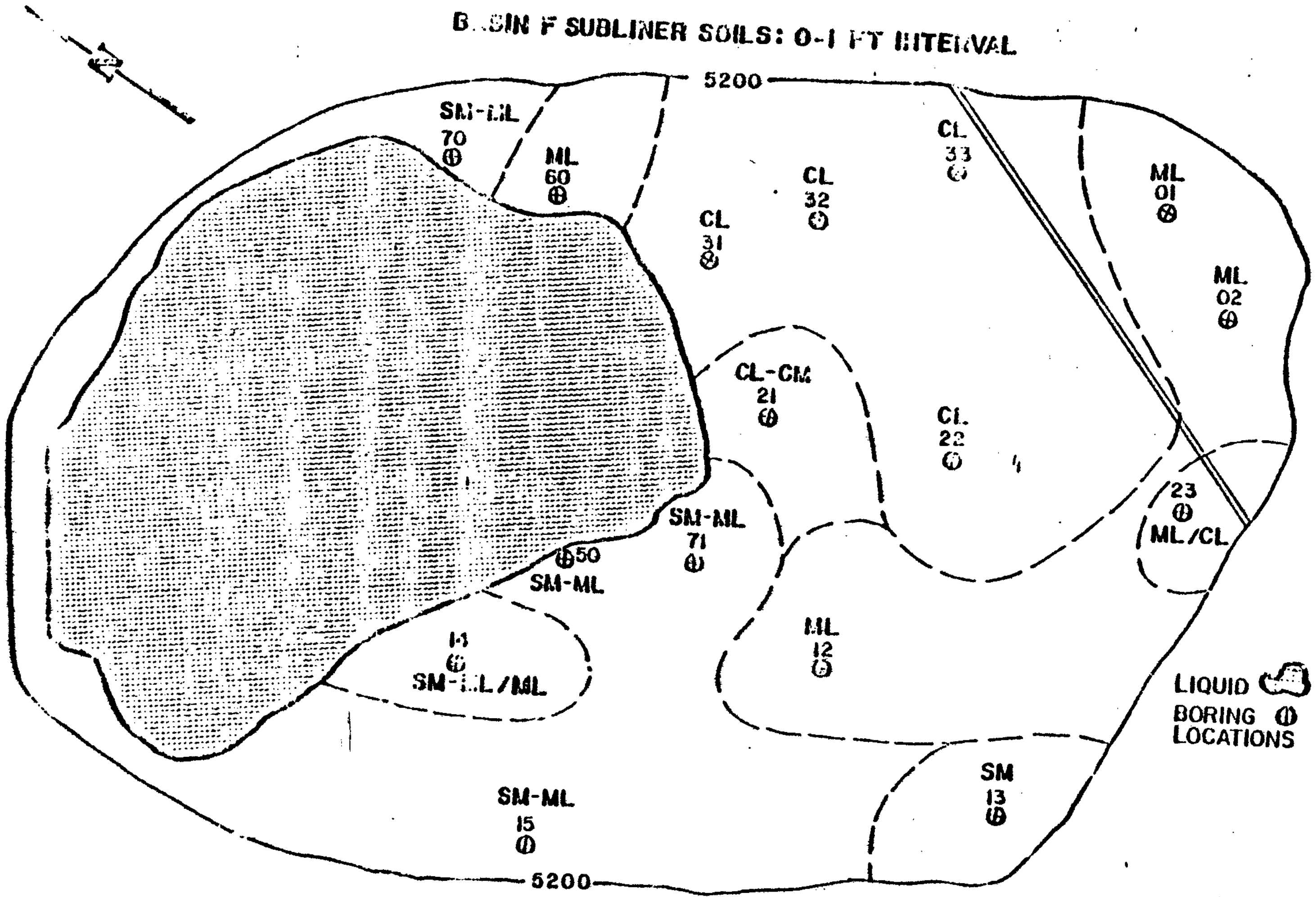


Figure 5 Basin F sediment depth (ft).



BASIN F SUBLINER SOILS: 0-1 FT INTERVAL

Figure 6. Distribution of soil types in Basin F, 0.0 - 1.0 ft. below the liner.

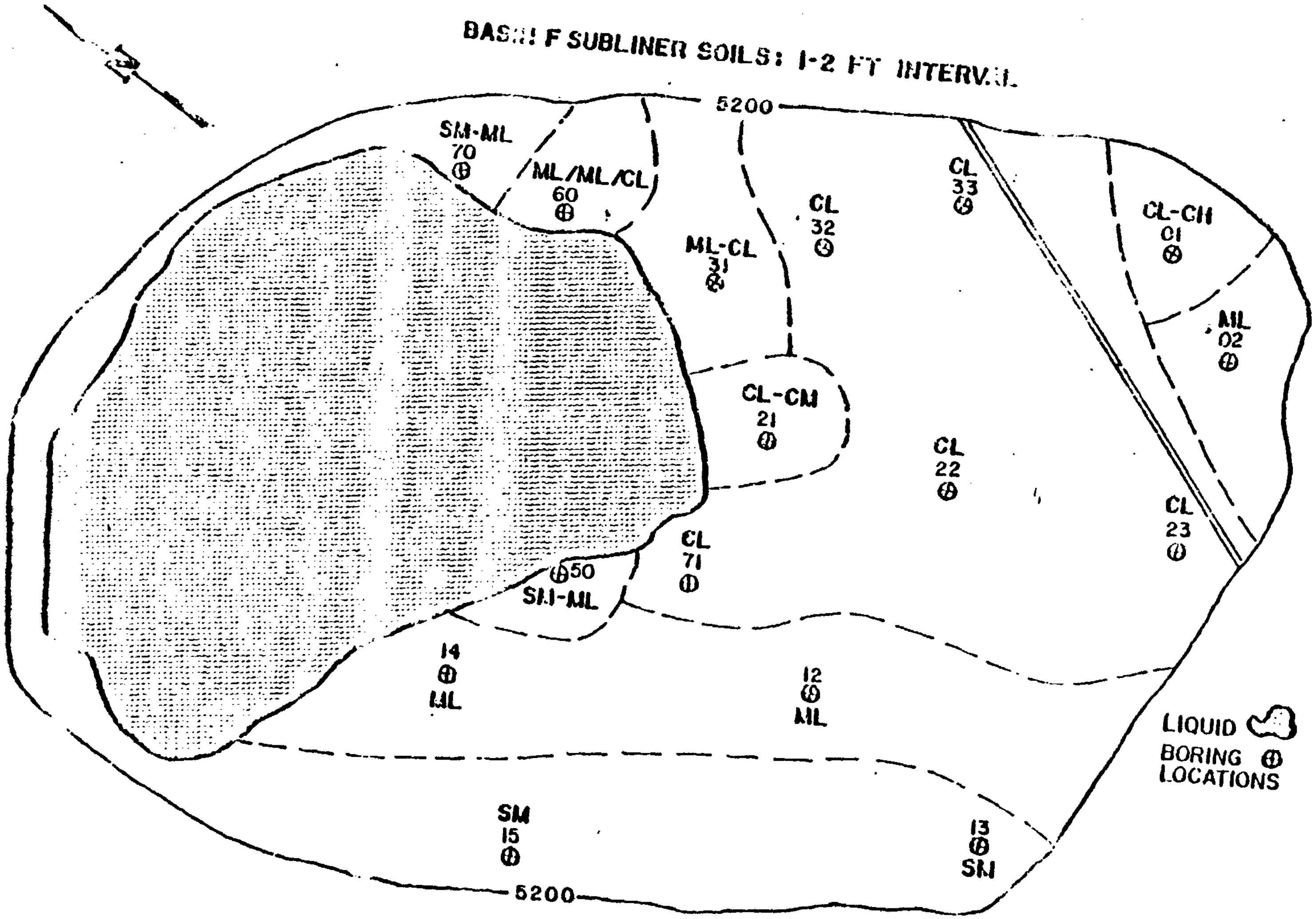


Figure 7. Distribution of soil types in basin F, 1.0 - 2.0 ft. below the liner.

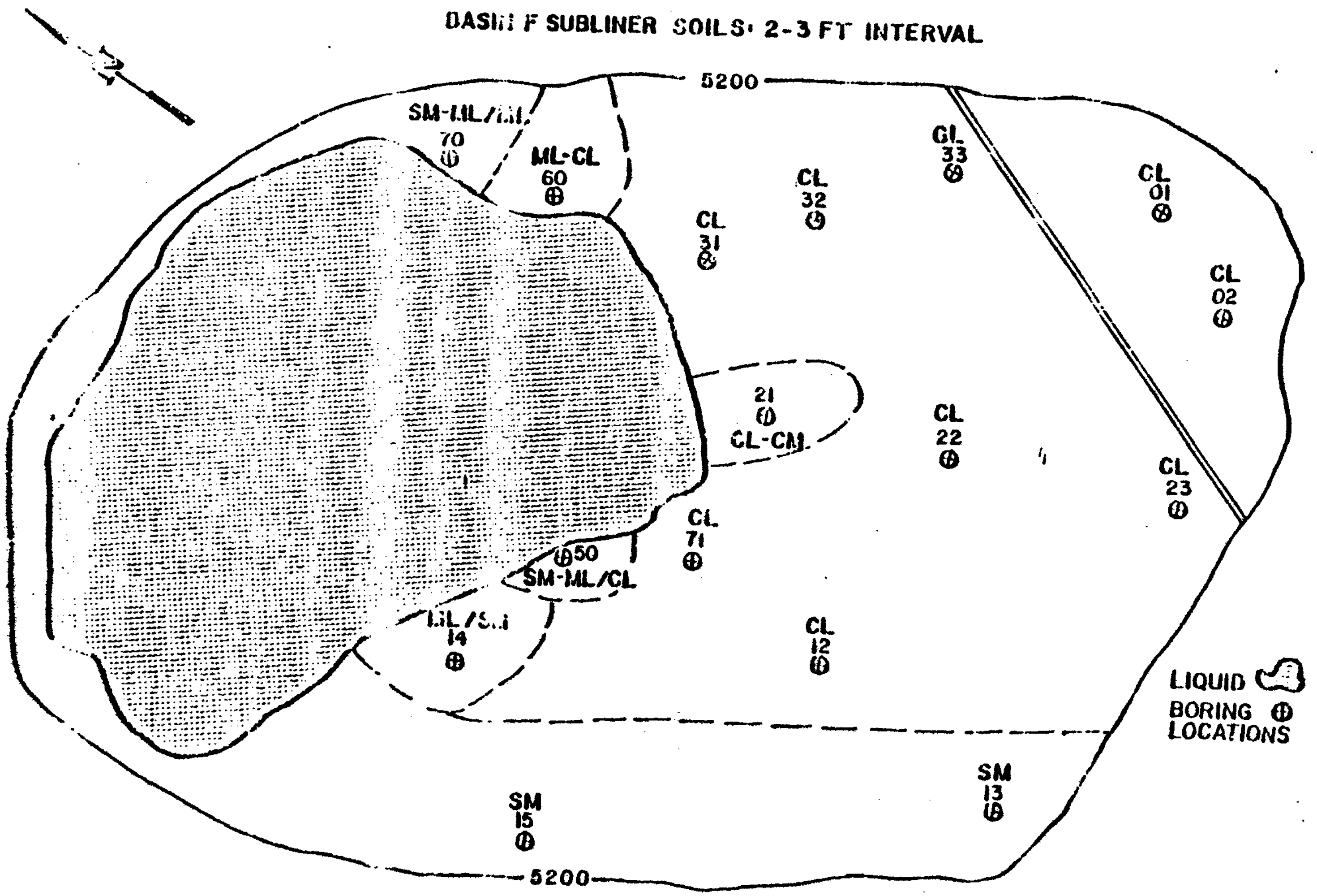


Figure 8. Distribution of soil types in Basin F, 2.0 - 3.0 ft. below the liner.

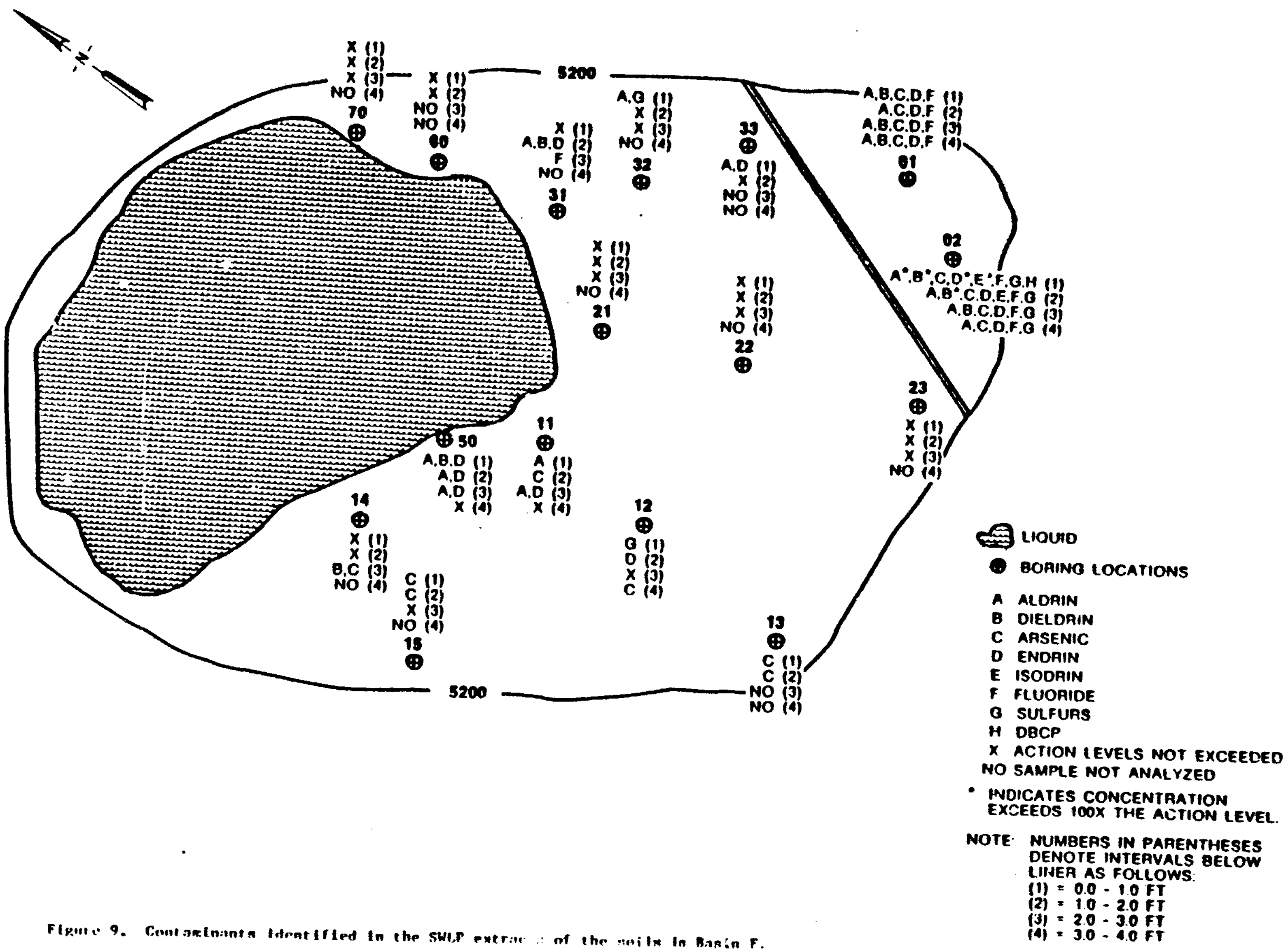


Figure 9. Contaminants identified in the SWLP extracts of the soils in Basin F.

APPENDIX A: BASIN F BORING PROTOCOL

SOIL BORING PROTOCOL WITHIN BASIN F

ROCKY MOUNTAIN ARSENAL
COMMERCE CITY, COLORADO

prepared for

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ROCKY MOUNTAIN ARSENAL

1.0 INTRODUCTION

1.1 Overview

Rocky Mountain Arsenal (RMA) is located in Commerce City, Colorado, north of Denver (Figure 1). Since its establishment in 1942, RMA has produced numerous munitions or has been involved in the demilitarization of chemical munitions. In 1946, portions of the manufacturing facilities at RMA were leased to private industry for production of various pesticide materials. Since 1952, the major lease holder has been the Shell Oil Company.

On-site industrial waste effluents have been discharged into storage basins/impoundments located on the arsenal. The capacity of these basins was, by necessity, increased over time in order to accomodate greater volumes of waste effluents from increased on-site production activities. On-site storage lagoons were identified as Basins A, B, C, D, E and F (Figure 2).

Basin F was constructed in 1955-1956. This basin is approximately 93 acres in size and has a capacity of greater than 243,000,000 gallons. Construction of Basin F included a low permeability liner to prevent chemical waste infiltration into the groundwater system. This liner consists of catalytically blown asphalt with an additional 12-inch thick sand layer.

Initally, waste input to the basin was pumped from Basin A. Problems associated with storage of liquid wastes in Basin F are basically as old as the basin itself. Initially, problems encountered were due to overflow of liquid as a function of wind induced wave activity and the lack of riprap. Tears in the liner were also found and after pumping some of the basin contents into Basin C, the liner was repaired and riprap installed.

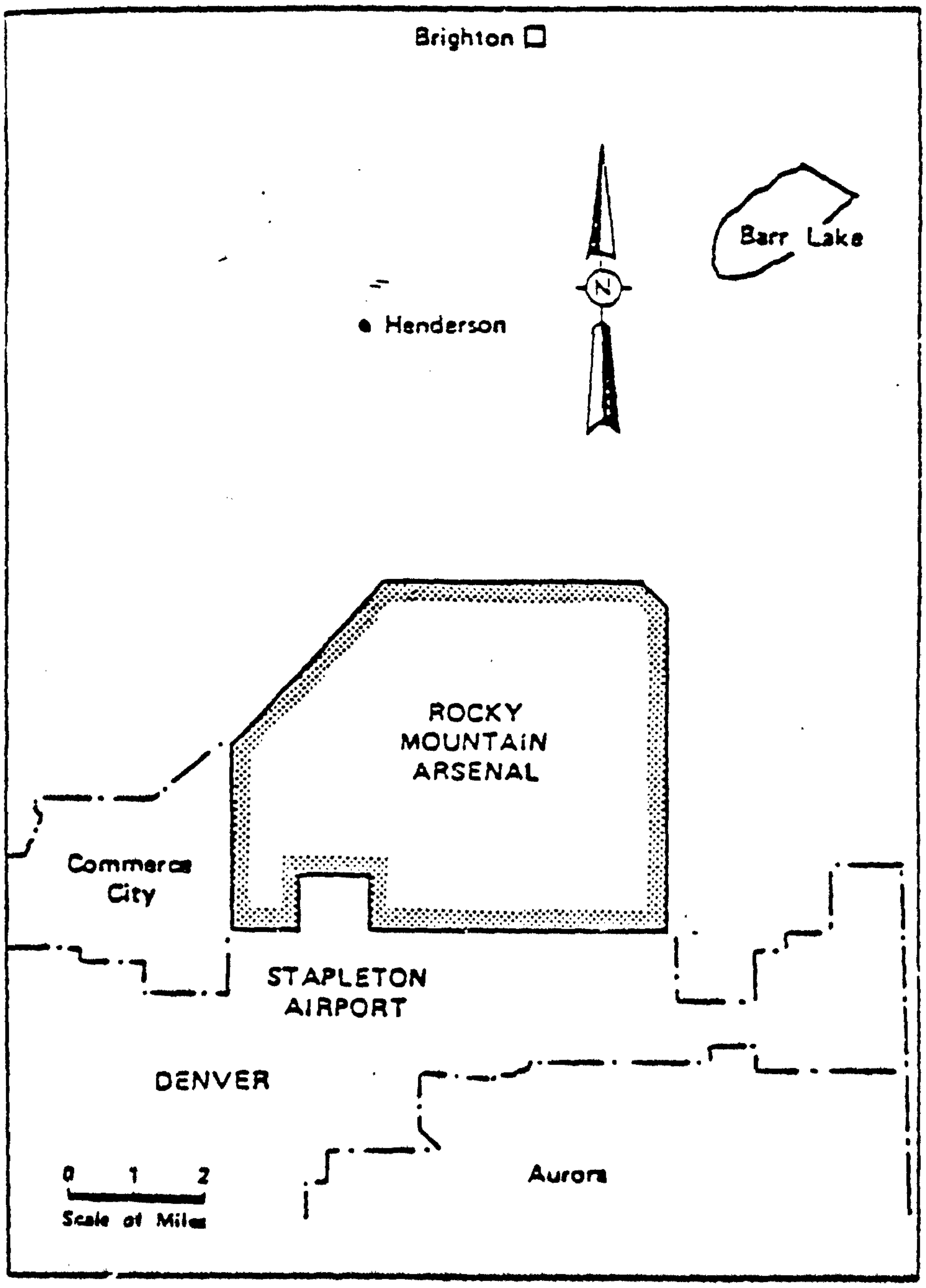


Figure 1. RMA-Denver vicinity (Kolmer and Anderson, 1977)

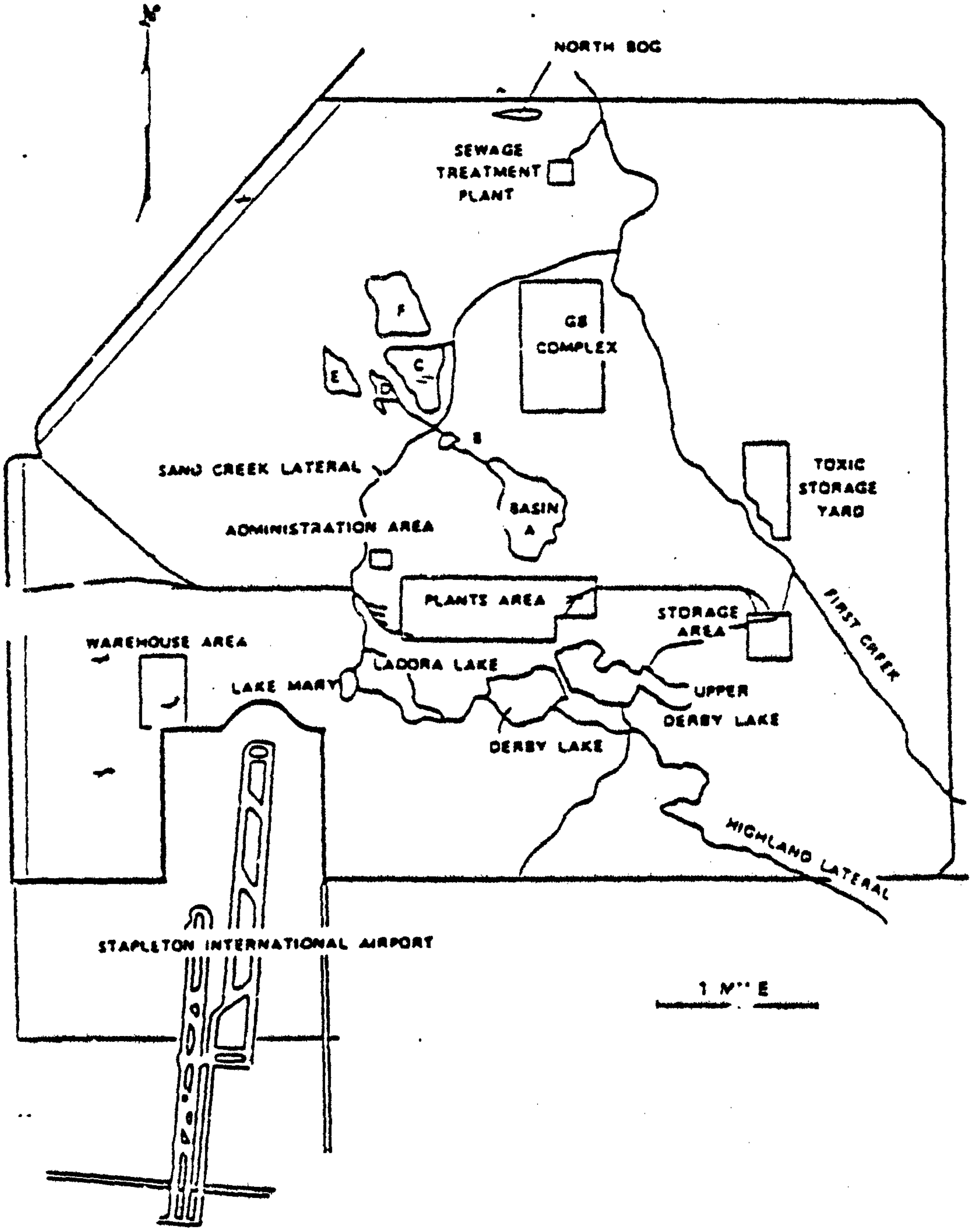


Figure 2. General map of RMA (Kolmer and Anderson, 1977)

Currently, no effluent waste streams are directed to Basin F. To date influent to the basin is limited to infiltration to the sewer line which originally fed the basin.

Studies of and in relation to Basin F have indicated that the torn liner has been exposed to liquid waste and that groundwater contamination has occurred as a result of this basin.

Numerous RMA and Basin F specific remediation scenarios have been proposed and studied. To date, the remediation efforts have been based on natural liquid evaporation of the basin contents. At this time, the liquid in the basin is estimate to be less than 50 million gallons.

The purpose of this effort is to develop a boring/sampling protocol in order to collect below-liner soils to quantify the degree of contamination, if any, and depth. This effort and the subsequent leachate analysis programs will be used to develop the design criteria for proper closure of Basin F.

1.2 Liquid Composition

The aqueous wastes present in Basin F are primarily the result of on-site munitions manufacturing and demilitarization operations, and pesticide manufacturing by Shell Oil.

Major components in the Basin F liquid are presented in Table 1 and sedimented/solid waste constituents are presented in Table 2. These data are based upon analytical results reported in August, 1978. Concentrations of these constituents in the aqueous phase may be higher than listed in Table 1 as a function of the volume reduction/evaporation processes currently underway. Additionally, other organic constituents have also been qualitatively confirmed as being present in Basin F liquid. These constituents include p-chlorophenylmethylsulfide

TABLE 1

WASTE BASIN LIQUID ANALYSIS

COMPONENT	COMPONENT ANALYSIS RANGE
	PARTS PER BILLION
Aldrin	20 - 480
Isodrin	<1 - 17
Dieldrin	5 - 110
Endrin	<20 - 123
	PARTS PER MILLION
Diisopropylmethylphosphonate	6 - 55
Dimethylmethylphosphonate	320 - 3,750
p-Chlorophenylmethylsulfoxide	4 - 10
p-Chlorophenylmethylsulfone	19 - 76
Chloride	47,500 - 57,500
Sulfate	20,500 - 32,500
Copper	709 - 760
Iron	5 - 13
Nitrogen	112 - 150
Orthophosphate	99 - 131
Hardness (as $CaCO_3$)	2,090 - 2,850
Total Solids	140,000 - 174,000
Fluoride	110 - 117
Total phosphorus	2,060 - 2,170
Arsenic	1.0 - 1.3
Magnesium	35.6 - 41.2
Mercury	0.026 - 1.53
Cyanide	1.44 - 1.53
COD	24,400 - 26,000
TOC	20,200 - 22,800

(Source: Asselin and Hildebrandt, 1978)

TABLE 2

WASTE BASIN SEDIMENT ANALYSIS

COMPONENT	COMPONENT ANALYSIS RANGE PARTS PER MILLION
Aldrin	16 - 10,700
Isodrin	2 - 870
Dieldrin	4 - 3,600
Endrin	2 - 1,100
DDT	<2 - 198
Diisopropylmethylphosphonate	1 - 10
Dimethylmethylphosphonate	<1 - 82
p-Chlorophenylmethylsulfone	14 - 290
Copper	230 - 21,000
Iron	190 - 11,000
Total Phosphate	<1 - 34,300

(Source: Asselin and Hildebrandt, 1978)

and oxathione. .

Varying concentrations of some of these same constituents have been found in surrounding groundwater monitoring well samples indicating Basin F as a contaminant source.

1.3 Geologic Character

The general subsurface conditions in the vicinity of Basin F consist of a surface fine to medium grained sand that varies in thickness from less than one foot to as much as fifteen feet. Underlying this surface layer is a clay-like silt to clay-like sandy silt to clay-like sand that may be as much as twenty feet thick. Underlying this sediment is coarse to very coarse sand that, in some places, is quite gravelly. This is the unit that makes up much of the near-surface aquifer over the Arsenal and, in the vicinity of Basin F, it is saturated in the lower portions. The underlying bedrock is predominantly a mudstone of the Denver-Arapahoe formation that varies in depth from about thirty to sixty feet.

This underlying bedrock surface is the subcrop of the Paleocene Denver formation. The Denver formation contains clays (or clay shales), sands, siltstone and sandstone layers or lenses, and a variable thickness (described as being up to 100 feet) basal shale (but also described as containing sandy materials). The shale strata is part of the Denver formation and is considered by personnel of the State of Colorado Division of Water Resources to be a "buffer zone" forming the basal Denver formation which overlies the Cretacaous Arapahoe formation.

In the vicinity of the basin, the bedrock surface, on the basis of the borings around the reservoir, appears to have little relief on it and the general slope on that surface is northward. The highest bedrock in the vicinity of Basin F occurs in the southeast corner where depth to bedrock is less than 40 feet.

1.4 Hydrologic Setting

The general hydrogeologic conditions at Basin F are schematically illustrated in Figure 3. This illustration assumes direct leakage from the basin and presents an oversimplified description of the existing subsurface soil conditions.

In order to understand the groundwater conditions in the vicinity of Basin F, 27 monitoring wells were installed around its perimeter. These wells are used for both water quality, as referenced in section 1.2, and water level determinations.

The groundwater pattern in the vicinity of Basin F based upon the perimeter monitoring wells is illustrated in Figure 4. Additional water table (alluvial aquifer) contours for the region north of the basin and for the majority of the arsenal as a whole are presented in Figure 5 and 6 respectively.

The principal flow component underneath Basin F is in a northerly direction. Along the north side of the basin a groundwater divide occurs and results in two principal flow components, one in a north-westerly direction towards the northwest boundary, and the other in a northeast direction towards the north boundary. Along the east side of the basin, a minor northeast flow component occurs. Minor west and northwest flow components also occur along south and southwest areas of Basin F, respectively. The gradients on the water table vary between a high of about 0.04 to less than 0.002. The average gradient is about 0.01. The steepest gradient occurs in the vicinity of the southeast corner of Basin F and may relate to the fact that this is the area in which the fine to medium grained bedrock sand occurs.

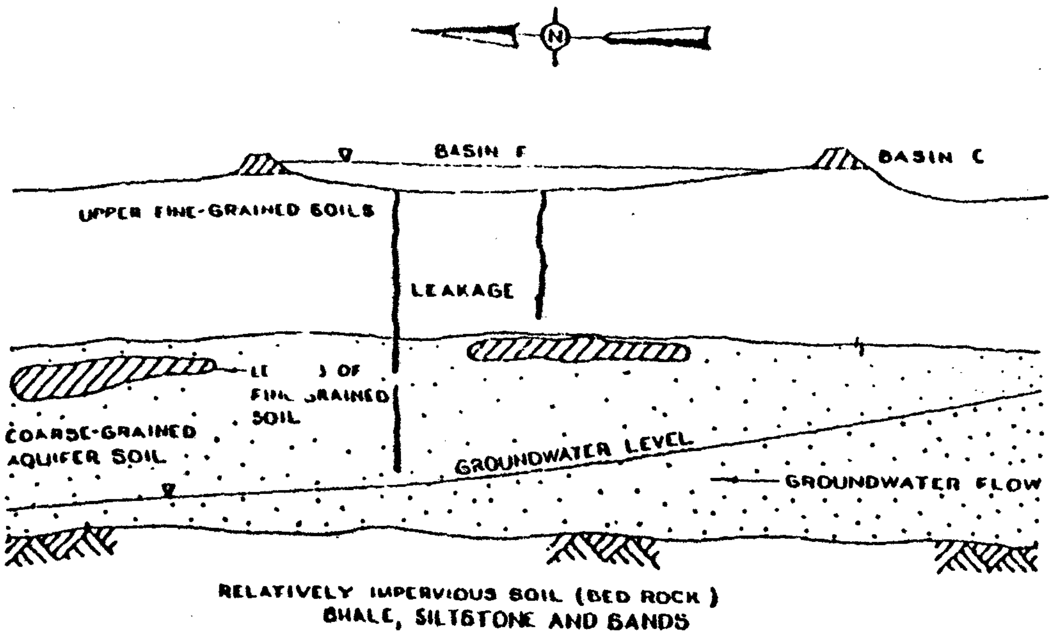
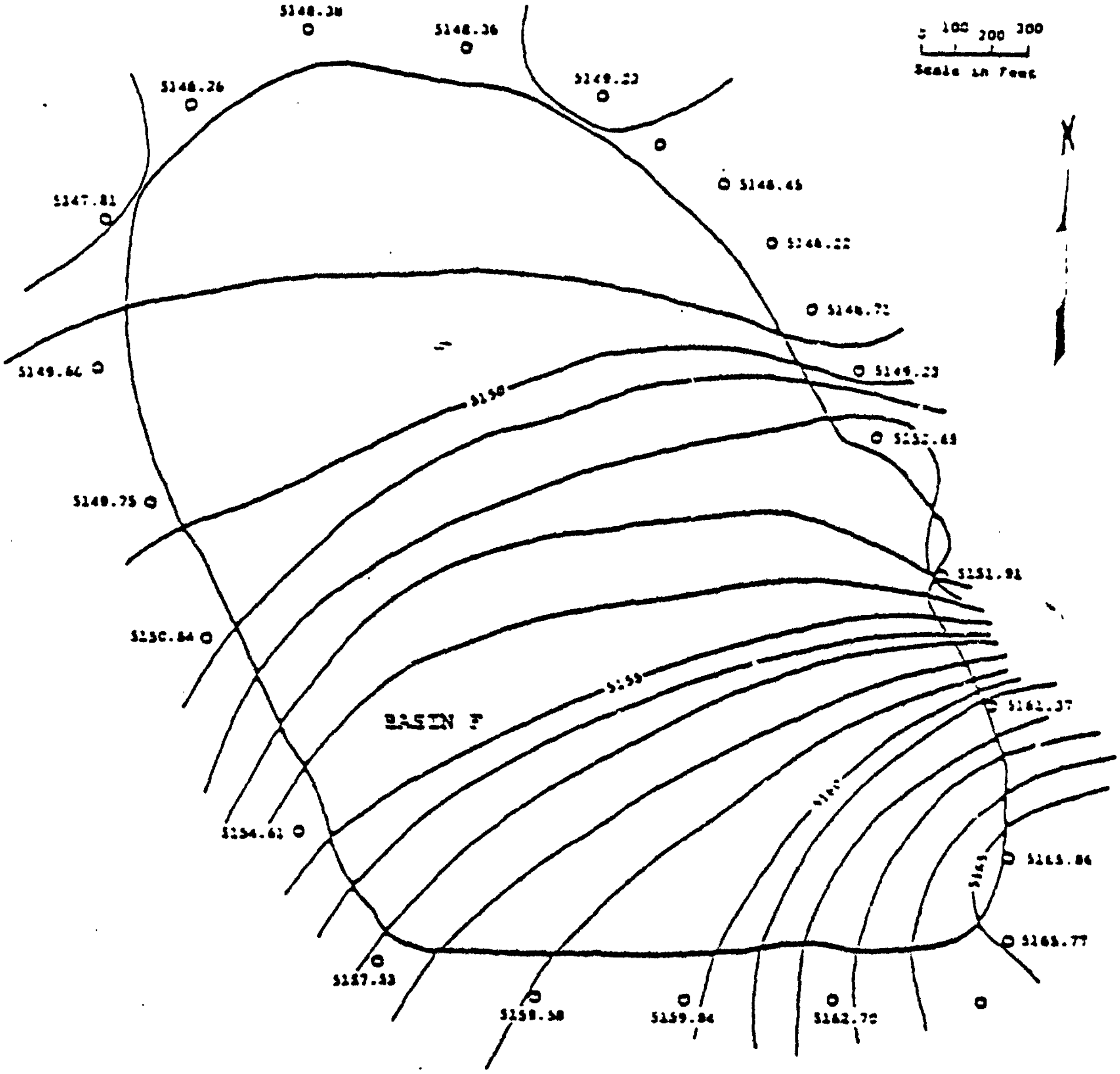


Figure 3. Basin F and underlying soils

(Source: Miller, 1979)



Note
Contour interval of 1 foot

Figure 4. Water Table Elevations

(Source: Interim Report on Basin F, 1978)

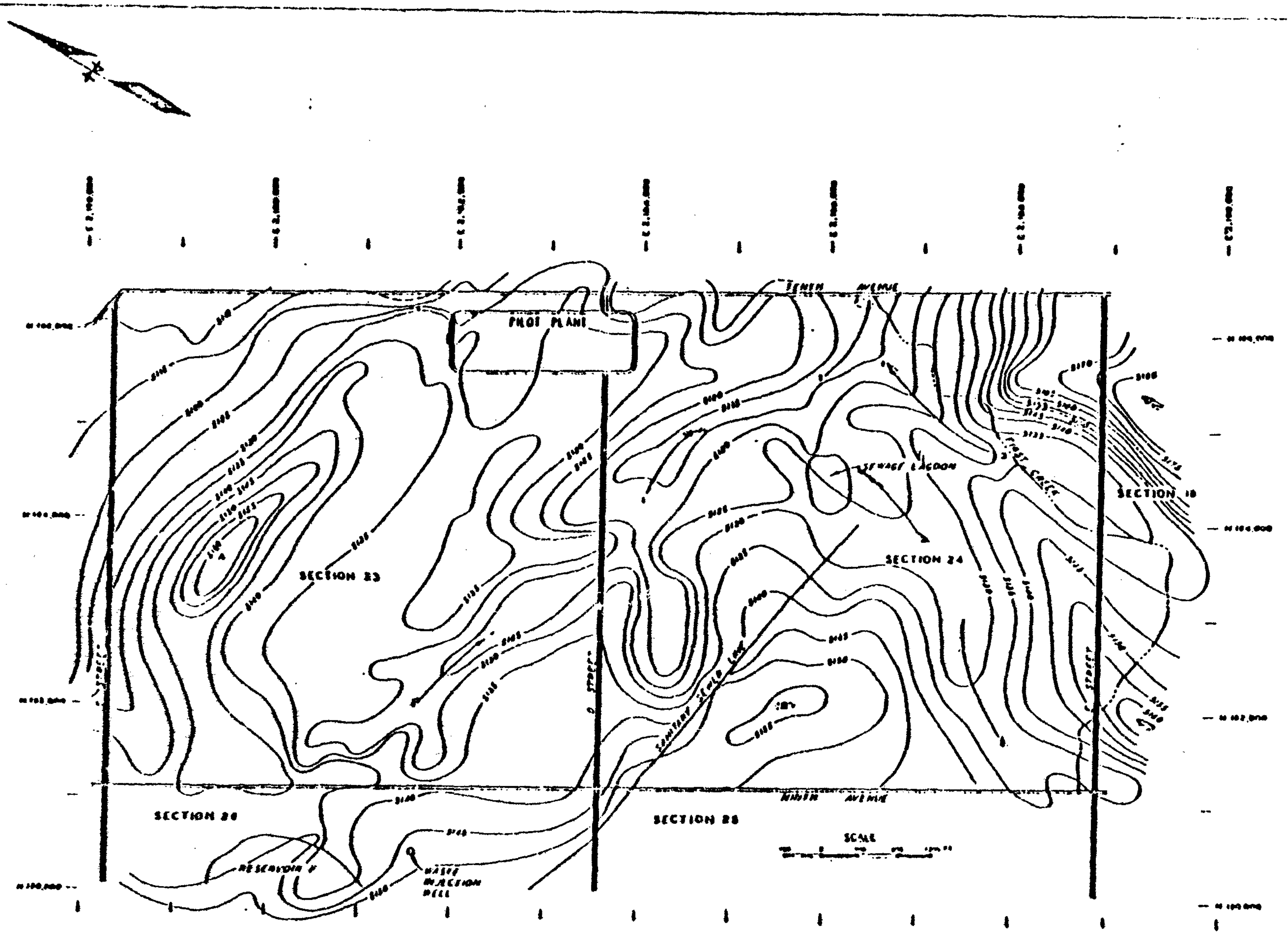


Figure 5. Base of the Alluvial Aquifer Contours
(Source: Zebell, 1979)

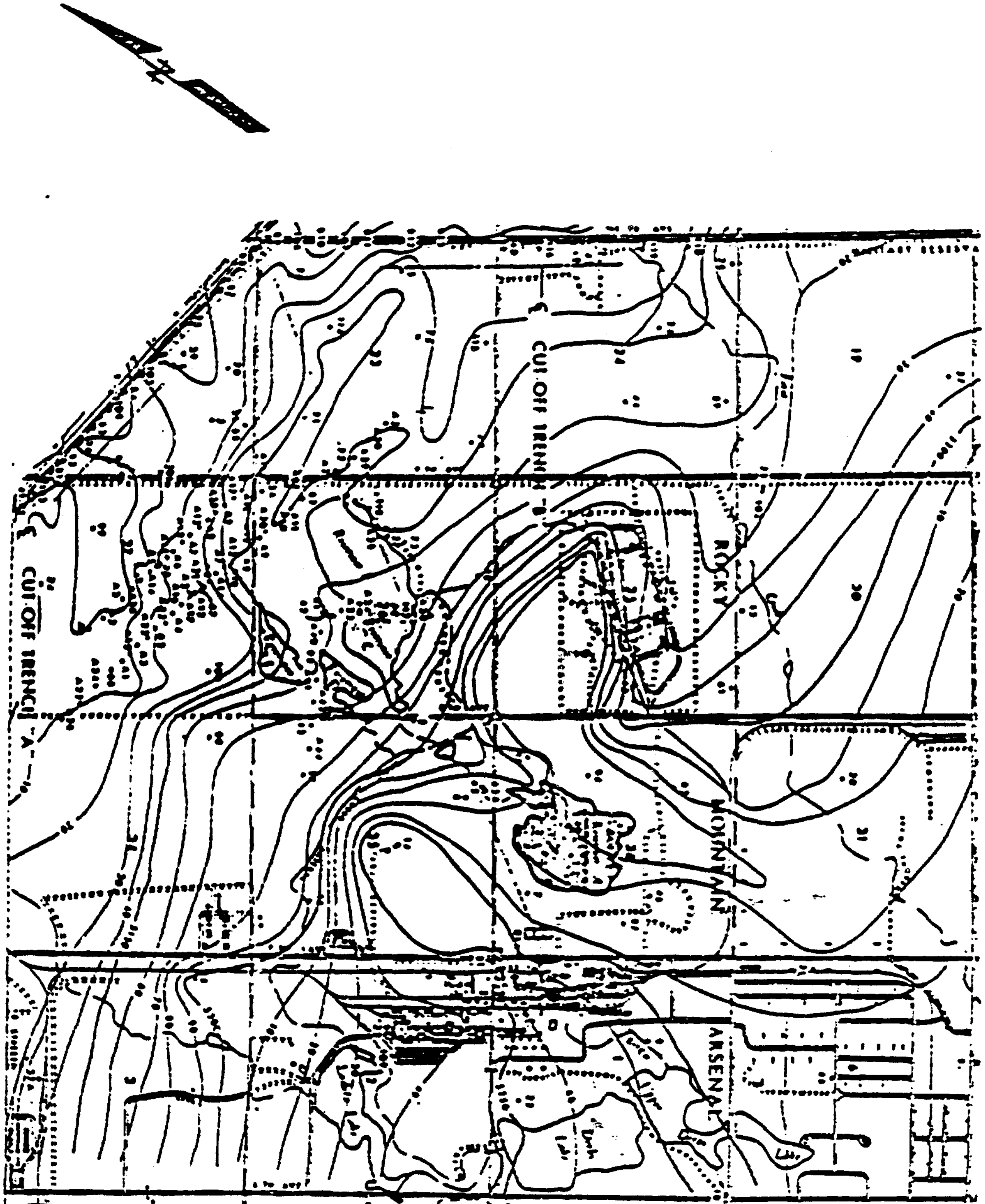


Figure 6. Alluvium Water Levels (1959-60)

(Source: Zebell, 1979)

2.0 BORING/SAMPLING PROGRAM DESIGN

The boring and sampling program has been designed to obtain a sufficient number of soil samples such that the ultimate goal of developing a design criteria for closure of Basin F can be accomplished. Therefore, the boring/sampling program must be a cost effective and expediate study.

The boring/sampling program consists of twenty (20) boring sites located within Basin F and "Little F" (Figure 7). This program, as presented in greater detail in a later section of this report, will be based on split spoon sampling to a depth of four and one half feet. During this field effort, it is estimated that approximately 100 samples will be generated.

3.0 FIELD PROCEDURES

3.1 Establishment of Staging Area

Prior to any work being undertaken within the fenced area of the basin, a staging area immediately adjacent to Basin F should be established. At a minimum, the staging area should include the necessary equipment and materials to be used for one day's activities. This area should also be located in close proximity to the boring sites but outside the fenced area. Possibly, the buildings located adjacent to the basin could serve as this staging area. This area will also serve as the personnel decontamination zone prior to any personnel involved in the boring program leaving the vicinity of the basin for any purpose.

It is anticipated that this staging area could house all equipment and supplies. Additionally, it is proposed that a truck towing a small water tank will accompany the personnel responsible for the actual boring work, into the basin area and will be located on the perimeter road as close to the working area/bore hole as possible.

The actual equipment to be stored in the staging area is presented in a later section of this document.

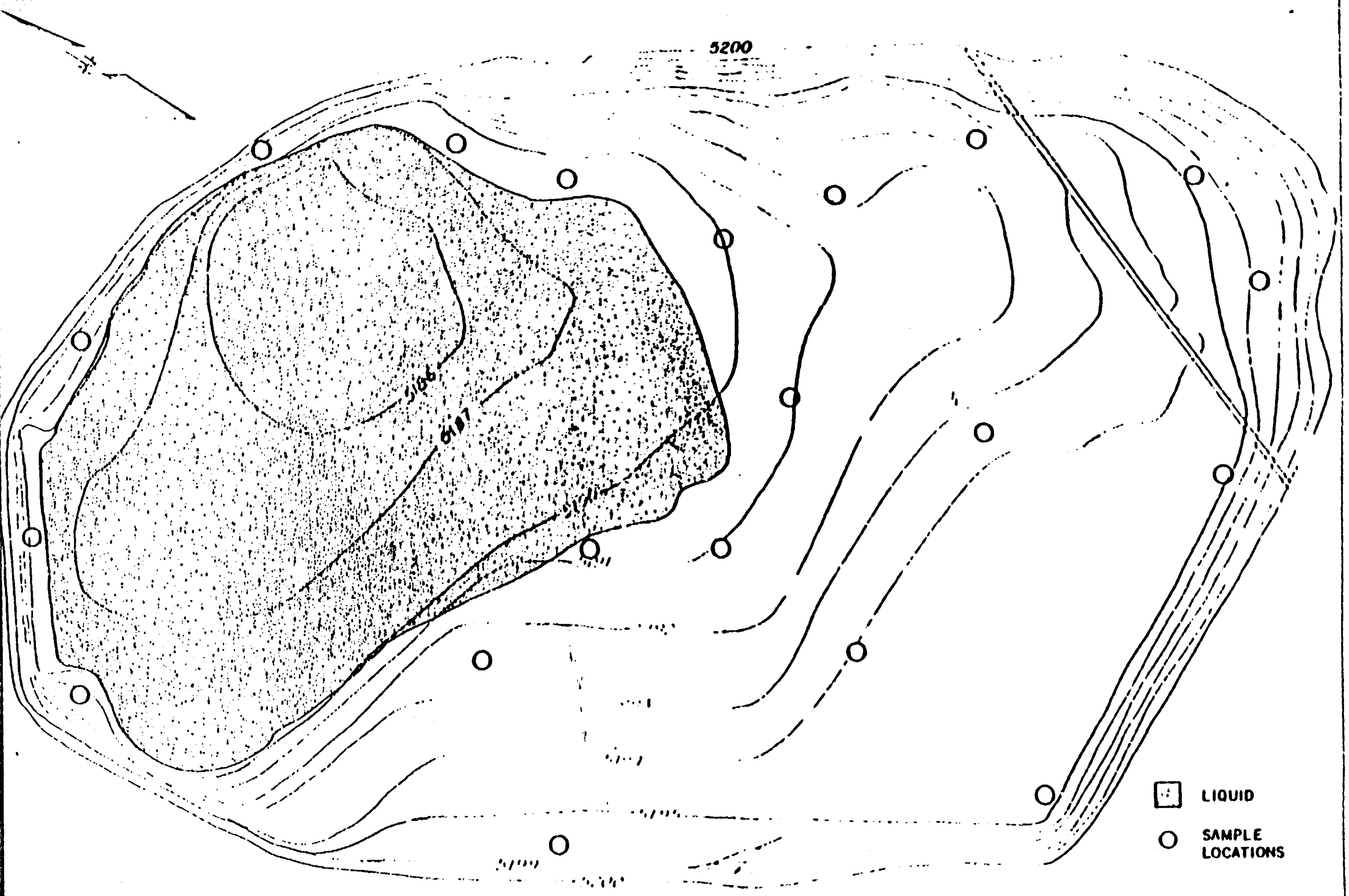


Figure 7. Basin F Boring Location Map

3.2 Establishment of Boring/Sample Locations

Prior to any boring activities, stakes/markers will be placed at the approximate location of the proposed boring sites as indicated in Figure 7. Location of the boring sites within Basin F can be accomplished using a surveying tape and a Brunton compass. Special attention will have to be paid to the sites located around the liquid boundary. These boring sites must be located as near to the liquid boundary as possible.

Upon completion of all boring activities, the exact location and elevation of the boring sites will be established via standard land surveying procedures.

3.3 Boring/Sampling Procedures

Upon staking/marking of the bore holes, the following step-by-step procedure should be followed to collect the sub-liner soil samples. The following program must be strictly adhered to in order to avoid any cross contamination of samples from sludge materials above the liner and/or from different depths below the liner. Failure to follow these procedures could produce results that would indicate a greater contamination depth than actually exists. This inaccuracy due to sampling error, could easily result in millions of dollars of additional remedial expenses if excavation and disposal of soils beneath the basin are eventually undertaken.

STEP 1 Locate boring position and pre-label all sample bottles/boxes prior to actual boring activity.

STEP 2 Remove all overburden (sludge) down to the asphalt liner within an area of approximately 3' in diameter. Use extreme care so as not to break the liner material.

STEP 3 Seat a 24" ID x 1 1/2' tall galvanized steel casing into but not through the asphalt, sealing the outside contact between the casing and asphalt with bentonite

STEP 4 Place the 5' x 5' plywood working platform with its 30" center opening, around the galvanized steel collar.

STEP 5 Carefully clean out any remaining sludge or debris within the collar.

STEP 6 If necessary, apply absorbent material inside the collar to remove any liquid. Throughout the boring/sampling operation, careful attention must be given to any fluids entering the collar. If this occurs, add additional absorbent and remove with a small shovel or similar device.

STEP 7 Construct a hole in the center of the asphalt, enclosed by the collar. This hole should be constructed with a hard auger through the asphalt but should not be allowed to penetrate the underlying soils. The auger used for this purpose shall not be used for purposes of sample collection.

STEP 8 Set a 4" ID x 2' SCH 40 PVC flush threaded joint casing through the asphalt and into the soil. This step and later steps referencing PVC casing may not be necessary if the subsurface soils are capable of maintaining the integrity of the hole without support

STEP 9 Using a split spoon sampler equipped with a plastic liner and attached to an A or AW rod, advance the split spoon 1 1/2'. Advancement of the split spoon will be accomplished by striking a coupling with a solid top attached to the end of the A or AW rod with a sledge hammer.

STEP 10 Retrieve the split spoon with the use of pipe wrenches and twisting/turning/pulling the split spoon and A or AW rod out of the boring. If retrieval of the split spoon and A or AW rod cannot be accomplished with the use of pipe wrenches, it can be retrieved by using a tripod and pulley set up.

STEP 11 Place the portion of the sample retained in the shoe of the split spoon into a wide mouth jar. Remove the plastic liner from the split spoon and cap. Label both the jar and plastic liner as to bore hole #, sample #, sample interval and date. Place all pertinent information including sample interval, field description and sample number on the boring log for this location/sampling point.

STEP 12 With a wire brush, clean off all soil from the split spoon and any other equipment or utensils used in obtaining the sample.

STEP 13 Place split spoon and/or other equipment over a bucket and thoroughly rinse with water and then acetone. A second clean split spoon can be used for sampling while the other is being cleaned in order to hasten the sampling process.

STEP 14 Advance the bore hole with a hand auger to the base of the split spoon sample interval depth.

STEP 15 Attach another 2' section of PVC casing onto the first section. Throughout the boring process always attach the next section of PVC before the threaded portion of the casing is advanced below the top of the collar. This procedure is necessary in case the use of strap wrenches is needed to tighten the casing sections.

STEP 16 Advance PVC casing to bottom of hole. Note that casing is settling at the proper depth. If not, remove enough soil to set the casing properly and remove any soil debris from the hole.

STEP 17 Advance split spoon 1 1/2 feet and remove. Place sample within the shoe of the split spoon into sample jar and cap plastic liner. Follow labelling and logging procedures as outlined above.

STEP 18 Follow cleaning procedures presented in Steps 12 and 13.

STEP 19 Advance PVC casing as described in steps 15 and 16.

STEP 20 Continue sampling procedure as presented in step 17 until field analytical results indicate sampling can be terminated or at a maximum depth of 4 1/2 feet.

STEP 21 Upon completion of the bore hole, move samples to the staging area.

STEP 22 At the staging area, photograph each split spoon sample using a 35 mm camera attached to a tripod. Describe the physical characteristics of the sample recording this description on the boring log.

STEP 23 Obtain samples, at least 100 grams, from 0, 1', 2', 3', 4' depths and at any depth where a noticeable lithological transition occurs. Place each sample into side mouth jars. Label each jar as to bore hole #, sample #, sample interval and date. Seal sample jar with custody tape and initiate chain of custody sheet. Wrap remaining split spoon sample and label as to bore hole #, sample interval and date.

STEP 24 Place sample jars and remaining split spoon sample in labelled box and in an ice chest. Maintain samples under refrigerated conditions.

3.4 Sample Custody

Upon collection of subsurface Basin F soil samples each subsample will be placed in pre-cleaned and labelled glass jar. All jars should be sealed with custody tape and immediately placed within an ice chest for eventual sample shipment.

After sample collection, all pertinent information should be immediately logged on the chain of custody record sheet. An example chain of custody form, which in this or a modified form would be useable for this purpose, is appendiced. It is anticipated that each boring location would utilize a separate custody sheet. Upon completion of each boring the sheet must be signed by the sampler and maintained with the samples. Upon relinquishing the samples, the shipper should sign/date the first "received by" block on the form and the sampler should also sign the first "relinquished by" block. If as is often the case, the shipper does not agree to sign the custody form, this should be so noted in the "comments" block. The form would then be placed inside the ice chest and the chest itself should then also be sealed with custody tape.

If the shipper agrees to signing the form, the form should be affixed to the outside of the ice chest where the receiving party could retrieve and sign upon receipt. It is also necessary at this time for the shipper to re-sign the form in the second "relinquished by" block.

If the shipper did not agree to sign the document, the receiving party should:

1) examine the custody tape on the chest
2) remove the custody tape on the chest
3) note in the comment block the condition of receipt (ice chest and individual sample)

At this time, with or without the signature of the shipper, the third copy of the form should be returned to the sampler/firm responsible for sampling. The original should accompany the samples until disposal of or archives with the second copy being maintained by the sample custodian receiving the sample shipment

3.5 Grouting/Boring Closure Procedure

Upon completion of the boring/sample acquisition to the 8' depth or depth indicated by field testing procedures outlined in section 3.5, each boring must be closed via the following grouting procedure. Failure to close the bore holes properly willlead to sub-surface contamination if such contamination has not already occurred.

STEP 1 At the staging area, prepare a grout consisting of Portland cement and bentonite. Use 10% bentonite and no more than 7 gallons of water per bag of cement.

STEP 2 Grout shall be placed by slowly pouring the above material down the casing and slowly removing the casing simultaneously. The casing will not be reused.

STEP 3 If removal of the casing by hand is not possible, a tripod and pulley should be set up on the working platform. By wrapping a rope around the casing and using the pulle, the casing can be removed.

STEP 4 Grout materials should be added until completely filling the hole up to the top of the galvanized steel casing. During the period of the first day after placement of the grout, inspection of the closed bore hole must be made in order to evaluate any subsidence of the grout material within the bore hole. If subsidence has occurred, additional grouting material should be added.

STEP 5 Upon completion of the grouting, the numbered stake should be re-established into the cement/bentonite grout. Upon completion of all bore hole closures, the exact location and elevation will be determined via standard surveying techniques.

STEP 6 Clean all equipment according to the procedures outlined in Section 3.4. Remove used PVC casing and store separately within the confines of the fenced area of Basin F. Empty waste solvent/wastewater into a waste solvent container. Collect and inventory all equipment.

4.0 SAFETY PROCEDURES

From both a safety and operations point of view the above program will involve a full time commitment of at least three (3) people for the duration of the boring/sampling program. Only personnel well trained and experienced in decontamination/safety procedures should be employed for purposes of completing this work effort.

All personnel will wear the following equipment during all times within the fenced area of Basin F:

1) steel toe boots
2) disposable all purpose coveralls
3) knee high rubber boots
4) cotton gloves
5) plastic/rubber overgloves
6) hard hat
7) full face cartridge type respirator

If for any reason personnel must leave the area, standard personnel decontamination procedures including removal of and/or disposal of protective equipment, washing etc. will be accomplished. All respirator cartridges will be replaced on a daily basin or whenever odors are detected. No eating, smoking or chewing will be allowed when personnel are within the fenced area of Basin F.

All disposable supplies and equipment should be deposited in a 55 gallon drum for eventual disposal either on- or off-site based upon the current RMA policy.

Additionally, prior to beginning any work at the basin, an emergency communication network should be established with the arsenal's security staff.

The staging area will hold the supply of those safety articles previously mentioned and will also house ancillary safety items included in the equipment lis provided in section 5. Specifically the required ancillary equipment will include Scott air packs, first aid kit, eye wash bottles, fire extinguisher(s) and spray attachment for water tank to be used in the case of emergency.

Special attention must be paid to the control of contaminant materials on personal clothing upon daily/final departure of the site area and regularly worn clothing and/or articles such as watchbands, and hard hat liners.

5.0 EQUIPMENT LIST

The following equipment list assures a suitable staging area and a truck/vehicle capable of pulling a portable water tank and holding necessary ancillary supplies.

5.1 Survey/Marker Equipment

a.) Brunton compass

b.) 100' steel surveying tape

c.) Survey equipment including tripod, transit and stadia rod

d.) Stakes (40) and flags

5.2 Boring/Sampling Equipment

a.) 5' x 5' x 3/4" plywood with centered 2-1/2' diameter hole

b.) 2' sections of 4" I.D. SCH 40 PVC flush joint casing (70)*

c.) 3-1/4" cylinder diameter regular hand auger head (3)

d.) Cross handle for auger (2)

e.) Auger extensions

- 4' extension (1)
- 3' extension (1)
- 2' extension (1)

f.) 1-1/2' sections of 2' I.D. galvanized steel casing (23)

g.) Portable water tank (1)

h.) Absorbent materials (50 lbs.)

i.) Ice chests

j.) Reagent grade acetone (5 gallons)

k.) Waste solvent container (1)

l.) Tripod (1)

m.) Pulley and rope with snap hook (1)

n.) Split spoon sampler with basket shoe and coupling to attach to A or AW rod (2)

o.) Plastic tube inserts for center section of split spoon (150') and caps (120)

p.) A or AW rods

- 5' section (1)
- 3' section (1)
- 2' section (2)

q.) Threaded couplings for A or AW rods (4)

r.) Coupling for A or AW rods with solid top (3)

s.) Coupling for A or AW rods with ring attachment

t.) 1' wire or rope with snap hooks at each end (1)

u.) Wash bottles (4)

v.) Miscellaneous tools and supplies

- boring logs
- chain of custody record and sealing tape
- pens, pencils, magic marker, chalk and/or crayons
- wide mouth bottles (2 gross; precleaned)
- bottle labels
- 14" pipe wrenches (2)
- machinist files (2)
- 1 lb. sledge hammer (2)
- 6 lb. sledge hammer (2)
- 8" screw driver (2)
- wire brushes (2)
- cleaning brushes (2)
- hammer (2)
- hack saw (1) with blades
- putty knife (1)
- strap wrenches (2)
- coal shovels (2)
- wisk brooms (2)
- hand spade (1)
- 5 gallon buckets (3)
- 16' retractable carpenter tape (2)

w.) Photographic equipment

- 35mm camera with flash attachment
- engineer scale or equivalent
- tripod
- close-up extension device

* Note: If during the advancement of a hole the use of casing is not necessary to maintain the integrity of the hole, the PVC casing may be eliminated.

5.3 Closure Supplies and Equipment

In addition to equipment and supplies already available as a function of the boring/sampling procedure, the following is required:

a.) Portland Cement

b.) Bentonite

c.) Trough for mixing of cement, bentonite and water

d.) 1" diameter wood dowel (6')

5.4 Safety Equipment

a.) Disposable all purpose coveralls

b.) Knee high rubber boots

c.) Cotton gloves

d.) Plastic/rubber overgloves

e.) Hard hats

f.) Hard hat liners

g.) Full face respirators with supply of replacement cartridges

h.) Open top 55 gallon drum

i.) Scott or equivalent air packs

j.) First aid kit

k.) Eye wash (2)

l.) Fire extinguisher(s)

m.) Emergency shower/spray attachment for water tank

6.0 REFERENCES

"An Analyses of the Subsurface Investigations Conducted Around Basin F"; Rocky Mountain Arsenal; March 1978.

"Basin F Containment Hydrogeology Assessment"; Geotechnical Laboratory; August 1979.

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Buhts, Robert E., Norman Francingues and Andrew Green; "Basin F Investigative Studies"; Historical Review; June 1979.

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Department of the Army; "Installation Restoration at Rocky Mountain Arsenal - Part II"; August 1980.

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Kolmer, Joseph and Gerald A. Anderson; "Installation Restoration of Rocky Mountain Arsenal, Part I - Pilot Containment Operations"; Department of the Army; July , 19

"Laboratory Evaporation Studies of Rocky Mountain Arsenal Basin F Fluid"; Test Summary; December 1979.

McKown, Dr. Gary L. and Dr. Lee G. Taft; "Alternatives for Reducing the Liquid Portion of the Contents of Basin F, Rocky Mountain Arsenal"; Report #DRXTH-IS-CR-80059; November, 1980.

Miller, Samuel P.; "Geotechnical Containment Alternatives for Industrial Waste Basin F"; Technical report GL-79-23; September, 1979.

Thompson, Douglas W. and Paul Law; "Basin F North Boundary Area"; Volume II; Draft Report; October, 1979.

Zebell, Robert; "Basin F North Boundary Area"; Volume I; Geotechnical Definition; 1979.

APPENDIX B: BASIN F BORING LOGS

Table B1

Descriptions of USCS Soil Groups (3)

Group Symbol	Typical group description	Example of corresponding USDA soil textural description
GW	Well-graded (poorly-sorted) gravels, gravel-sand mixtures, little or no fines	Gravel, gravelly sand
GP	Poorly-graded (well-sorted) gravels, or gravel-sand mixtures, little or no fines	Same as above
GM	Silty gravels, gravel-sand-silt mixtures	Very gravelly sand or silt loam
GC	Clayey gravels, gravel-sand-clay mixtures	Very gravelly clay loam
SW	Well-graded (poorly-sorted) sands, gravelly sands, little or no fines	Same as above
SP	Poorly-graded (well-sorted) sands, gravelly sands, little or no fines	Coarse to fine sand
SM	Silty sands, sand-silt mixtures	Loamy sand or sandy loam
SC	Clayey sands, sand-clay mixtures	Sandy clay loam or sandy clay

(Continued)

Table B1 (Concluded)

Group Symbol	Typical group description	Example of corresponding USDA soil textural description
ML	Inorganic silts, very fine sands, clayey silts, low plasticity	Silt or silt loam
CL	Inorganic clays, low to medium plasticity, lean clays	Silty clay loam or clay loam
OL	Organic silts and organic silty clays of low plasticity	Mucky silt loam
MH	Inorganic silts, micaceous or diatomaceous fine, sandy or silty soils, elastic silts	Micaceous or diatomaceous silt
CH	Inorganic clays or high plasticity, fat clays	Silty clay
OH	Organic clays of medium to high plasticity, organic silts	Mucky silty clay
Pt	Peat and other highly organic soils	Mucks and peats

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 30 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig Inspector T.M. & R.H. Operator Dan Taylor Surface El Boring No. 01

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/30	(-) 1.3						shovel			brown, moist sludge from surface to
											liner, lt. seepage, liner in good
			0.0								shape.
		0.0						split-spoon			ML, sdy. silt, containing mica
											flakes, firm (probably reworked)
			0.7								fill, moist, dk. gray br. 2.5Y 4/2
		0.7									CL-CH, clay w/minor silt & sd.
											containing mica flakes, firm-hard,
											moist, very dk gray br. 2.5Y 3/2
											becoming somewhat lighter toward
			1.9								bottom
		1.9									CL, silty, clay mottled w/caliche,
											firm-hard, moist, dk grayish br.
											2.5Y 4/2. caliche is lt. gray
											(2.5Y 7/2) rather than normal white
			4.5								to pale yel. bor at 4.5'

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Sheet 1 of 1 Sheets

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 1 Feb 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 02

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	2/1	(-) 1.35						shovel			dk, br. wet sludge, liner very
											soft with locally emulsified
											pockets, impossible to remove over
			0.0								burden W/O disturbing liner.
		0.0						split-spoon			ML, sandy-clayey silt, w/mica
											flakes & minor sd, firm, very moist
											very dk gray br. in upper 0.2',
											2.5Y 3/2; below 0.2' spl is olive
											br. 2.5Y 4/4, gradually changing to
											lt. olive br. (2.5Y 5/4) w/depth
			1.7								(possibly reworked fill)

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Sheet 1 of 2 Sheets

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 1 Feb 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M & R.H. Operator Dan Taylor Surface El ______ Boring No. 02

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	2/1	1.7						split-spoon			CL, silty clay w/minor sd and mica
											flakes, firm, very moist, occ.
											caliche veinlets, lt. olive br
											(2.5Y 5/4) gradually changing to
			3.3								yel. br. (10YR 5/6) with depth.
		3.3									CL, silty clay, calcareous w/minor
											sd, soft very moist, very pale br.
			4.3								10YR 7/4
		4.3									CL, silty clay w/minor sd, w/mica
											flakes, soft-firm, very moist
			4.5								yel br. 10YR 5/4 bot. at 4.5

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED Sheet 2 of 2 Sheets

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 30 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ____ Inspector T.M. & R.H. Operator Dan Taylor Surface El ____ Boring No. 11

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM (-)	TO	FROM	TO	FROM	TO				
	1/30	1.55						shovel			lt. br. sandy sludge to 0.2', br
											wet sludge to liner, mod. seepage
			0.0								liner in good shape.
		0.0						split-spoon			ML-SM, sdy, silt-silty very fine
											sand containing mica flakes, firm
											moist-slightly moist. (Probably
											reworked fill), dk. gray br.
											2.5Y 4/2 in top 0.3' changing to
			1.7								lt. olive br. in bottom (2.5Y 5/4).
		1.7									CL, silty clay, w/minor sd,
											containing mica flakes, occ. calich
											inclus. firm-hard, moist dk. yel.
			2.4								br. 10YR 4/6.

WES FORM JAN 74 819 EDITION OF NOV 1971 MAY BE USED

Sheet 1 of 2 Sheets

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 30 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 11

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		2.4						split-spoon			CL, silty clay, w/minor sd & occ.
											small pea grav. contains mica
											flakes, firm, moist, very pale br.
			4.5								to yel. 10YR 7/4-7/6 bot. at 4.5

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Sheet 2 of 2 Sheets

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 29 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 12

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/29	1.25						shovel			lt. br. sandy to 0.7', black wet
											sludge to liner, moderate seepage,
			0.0								liner in good shape
		0.0						split-spoon			ML, clayer, sandy, silt containing
											mica flakes, firm, moist, dk. yel.
			2.1								br. 10YR 4/4
		2.1									CL, silty clay, w/minor sd.
											containing mica flakes, hard, moist
											dk. yel. br. 10YR 4/4, numerous
			4.0								small caliche veinlets & inclus.
		4.0									CL, silty clay, soft-firm,
											calcareous, very pale br.
			4.5								10YR 7/4, bot. at 4.5

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Sheet 1 of 1 Sheets

BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 27 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 13

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/27	0.65						shovel			brown sandy over burden, moist, no
											seepage, liner in fair condition
											surface very
											rough. (probably due to poor prep.
			0.0								of base during construction).
		0.0						split-spoon			3/8" plastic asphalt at top.
											SM, very silty fine sand soft
											(slightly to non-cohesive) very
											moist, yel. br. 10YR 5/4 to 10YR
			2.8								5/6, contains mica flakes.
		2.8									CL, silty clay w/minor sand, firm,
											moist, numerous small caliche
											vainlets and inclusions in bottom
											ft. (color as above), contains mica
			4.5								flakes, bot.at 4.5'

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 27 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.H. & R.H. Operator Dan Taylor Surface El ______ Boring No. 14

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/27	1.5						shovel			brown, sandy to 0.2', then black
											sludge to 0.7" then light br. to
											1.0', then black, very wet sludge
											to 1.3', br. sandy, very wet to
											liner. heavy seepage, liner in
											good shape 3/8" plastic asphalt at
			0.0								top.
		0.0						split-spoon			SM-ML, very fine silty sand-sandy
											silt, soft, (non-cohesive) moist,
											very moist, contains mica flakes
			0.5								lt. yel. br. 2.5Y 16/4
		0.5									ML, clayey-sdy silt, firm, moist,
											very moist, contains mica flakes,
			2.5								lt olive br 2.5Y 5/4.

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 27 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 14

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/27	2.5						split-spoon			SM, silty, very fine sand, w/occ.
											small grav. at base. Soft, (non-
											cohesive), moist-very moist, yel.
			2.9								br. 10YR 5/4.
		2.9									CL, silty-sdy clay, mottled w/
											caliche, firm-very firm, moist, dk.
			4.5								yel. br. 10YR 4/6 bot. at 4.5'

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 27 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.H. & R.H. Operator Dan Taylor Surface El ______ Boring No. 15

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		1.2						shovel			over burden tan w/soil-like
											texture, moist, no-seepage, one-
											hole (1"x2") in liner, (probably
											caused by construction mishap),
											otherwise liner in good shape.
			0.0								1/2" plastic asphalt at top.
		0.0						split-spoon			SM-ML, very fine silty sandy-sdy
											silt, firm, yel. br. 10YR 5/4-5/6.
											moist-very moist, contains mica
			1.0								flakes.
		1.0									SM, very silty fine sand, soft,
											(slightly cohesive) moist-very
											moist, yel. br. 10YR 5/4-5/6,
			4.5								contains mica flakes. bot. at 4.5'

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 26 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ____ Inspector T.M. & R.H. Operator Dan Taylor Surface El ____ Boring No. 21

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM (-)	TO	FROM	TO	FROM	TO				
	1/25	1.25						shovel			lt. br. crust, bk, wet sludge to
											liner, mod. seepage, liner in good
			0.0								shape 1/4" plastic asphalt at top.
	1/26	0.0						split-spoon			CL-CM, silty clay-clayey silt, firm
											minor fine sand, moist, very dk.
											gray brown, 2.5Y 3/2 (lighter with
											depth), changing to dk. gray brown
			1.7								(2.5Y 4/2 in bottom)
		1.7									CL-ML, (as above) but color changes
											to yellow br. 10YR 5/4 becomes more
											silty w/depth caliche nodules in
			4.4								bottom 0.5', bot. at 4.4'.

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 26 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ____ Inspector T.H. & R.H. Operator Dan Taylor Surface El ____ Boring No. 22

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/25	(-) 1.2						shovel			br. wet sludge to 1.0', br. very
											wet sludge to liner, heavy seepage,
											liner in good shape. 1/4" plastic
			0.0								(pliable) asphalt at top.
	1/26	0.0						split-spoon			CL, clay, silty, w/minor sand, firm
											moist, very dk gray br. 2.5Y 3/2,
											gradual color change at 1.3 to dk
											gray, br, 2.5Y 4/2, gradually
			3.7								becoming lighter in color w/depth.
		3.7									CL, silty clay mottled w/caliche,
			4.1								2.5Y 7/4, firm, moist
		4.1									CL, silty clay, firm-hard, pale
			4.4								yellow, 2.5Y 7/4 bot. at 4.4'

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 26 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ____ Inspector T.H. & R.H. Operator Dan Taylor Surface El ____ Boring No. 23

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM (-)	TO	FROM	TO	FROM	TO				
	1/26	1.3						shovel			lt. br. sandy, wet to 1.0', black,
											wet sludge to liner, moderate
			0.0								seepage, liner in good shape
		0.0	0.05					split-spoon			asphalt
		0.05									ML, silt, firm, moist, very dk.
			0.5								gray br. 2.5Y 3/2.
		0.5									CL, sandy clay, approx. 15% fine
											grain sand, firm, moist very dk.
			1.3								gray br. 2.5Y 3/2.
		1.3									CL, sandy, clay, approx. 25% fine
											sand, firm-hard, moist, br-dk br
			2.5								10YR 4/3.
		2.5									CL, clay w/minor fine sand, caliche
											nodules, 3/4" rock fragment, firm
											moist, yel. br. 10YR 5/4, bot. at
			3.0								3.0

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 29 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 31

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		(-) 1.6						shovel			green crust, bk, very wet sludge
											to 0.2', dk. br. wet sludge to 0.7'
											bk. very wet sludge to liner, heavy
			0.0								seepage, liner in good shape
		0.0						split-spoon			CL, silty clay, w/occ. caliche
											inclusions, hard, contains mica
											flakes, dry to very slightly moist,
			1.3								lt. olive br. 2.5Y 5/4.
		1.3									ML-CL, clayey silt-silty clay, firm
											dry, minor sd, occ. caliche vein-
											lets, contains mica flakes, yel.
			2.5								br. 10YR 5/6
		2.5									CL, silty clay w/minor sd, mottled
											w/caliche, hard, dry-very slightly
											moist, yel. br. to dr. yel. br.
											10YR 5/6-4/6, contains mica flakes,
			4.5								bor. at 4.5'

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 28 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El. ______ Boring No. 32

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
	1/28	(-) 1.6						shovel			br. wet sludge to 0.2', then dk
											br. grading grad. to lt. br. wet
											sludge at 1.0', bk, very wet sludge
											to liner, heavy seepage, liner in
			0.0								good shape.
		0.0						split-spoon			CL, silty clay, hard, moist, very
											dk gray br. 2.5Y 3/2 becomes
											lighter and sandy in bot 0.3' (dk
			1.6								gray br.) 2.5Y 9/2.
		1.6									CL, silty sandy clay, hard,
											slightly moist, numerous caliche
			2.7								veinlets dk. yel br. 10YR 4/4.
		2.7									CL, silty-sdy clay, hard slightly,
											moist, mottled w/caliche, hard, dr,
			4.5								yel. br. 10YR 4/4. bot. at 4.5'

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 29 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 33

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
	1/29	(-) 1.8						shovel			lt. br., moist, sandy to 1.0'
											black wet sludge to liner, lt
			0.0								seepage liner in good shape.
		0.0						split-spoon			CL, silty clay w/minor sand,
											contains mica flakes, occ. small
											caliche inclus. & veinlet hard,
											moist from top to 1', gradual color
											change from very dk gray br. to
											dk gray br. (2.5Y 3/2-2.5Y 4/2).
											below 1.0' color varies from yel
											br. to dk. yel. br. 10YR 5/4-
			4.5								10YR 4/4. bot. at 4.5

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 30 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 50

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/30	(-) 1.7						shovel			green crust, bk wet sludge to 0.2'
											br wet sludge to 0.7', bk very wet
											sludge to liner, mod. seepage,
			0.0								liner in good shape.
		0.0						split-spoon			SM-ML, silty fine sand-sandy silt,
											soft, non to slightly cohesive,
											(probably reworked fill) slightly
											moist, contains mica flakes, dk
											gray br. 2.5Y 4/2 in top 0.2-0.3'
											dk yel br. (10YR 4/6), below 0.3'
											gradually changing to yel. br.
			2.3								10YR 5/4 with depth

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 30 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.M. & R.H. Operator Dan Taylor Surface El ______ Boring No. 50

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		2.3						split-spoon			CL, silty clay w/minor sd & occ.
											small pea grav., occ. caliche
											veinlets (increasing in bot. 0.5)
											firm, hard, moist, yel. br. 10YR
			4.5								5/6, contains mica flakes, bot. 4.5

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BORING LOG
FIELD DATA

Project ~~Sub-Liner Contamination Survey~~ Site Basin F Date 28 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ____ Inspector T.H. & R.H. Operator Dan Taylor Surface El ____ Boring No. 60

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/28	(-) 1.8						shovel			br., moist, sandy sludge to 1.0',
											bk, wet sludge to liner, mod.
			0.0								seepage, liner in good shape.
		0.0						split-spoon			ML, sandy-clayey silt, firm
											contains mica flakes, dry to
											slightly moist near top. (probably
											reworked & compacted fill) dk br.
											10YR 3/3 in top 0.2', br. to dk
			1.1								br. below 0.2'.
		1.1									CL-ML, silty clay-clayey silt, firm
											dry, numerous caliche veinlets,
											yel. br. 10YR 5/4 contains mica
			3.1								flakes.
		3.1									SM-ML, silty very fine sand to
											sandy silt, non-cohesive, contains
											mica flakes, dry dk. yel. br.,
			4.0								10YR 4/4.

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 28 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ______ Inspector T.H. & R.H. Operator Dan Taylor Surface El ______ Boring No. 60

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/28	4.0						split-spoon			CL-ML, (as above) but less caliche,
			4.4								bot. at 4.4'.

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BORING LOG
FIELD DATA

Project Sub-Liner Contamination Survey Site Basin F Date 28 Jan 82
Location Rocky Mountain Arsenal Job No. E708
Drill Rig ____ Inspector T.N. & R.H. Operator Dan Taylor Surface El ____ Boring No. 70

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/28	(-) 1.4									
								shovel			br. moist, sandy sludge to 0.3'
											bk, wet sludge to liner, mod.
			0.0								seepage, liner in good shape.
		0.0						split-spoon			SM-ML, very fine silty sand-sandy
				(material		from 0.0 to		2.0 is probably			silt, slightly cohesive, dry,
				reworked		& compacted		fill placed			contains mica flakes, dk. yel. br.
			2.5	during construction)							10YR 4/4.
		2.5									ML, sandy, silt, slightly cohesive
											occ. caliche veinlets, dry, yel.
			3.0								br. 10YR 5/4, contains mica flakes.
		3.0									CL, silty clay, w/minor sand, occ.
											caliche veinlets, firm-hard dry,
			4.3								br. 10YR 5/3.
		4.3	4.5								ML (as above)

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APPENDIX C: USATHAMA MEMORANDUM ON SOIL LEACHING PROTOCOLS

DRXTH S

18 November 1981

MEMORANDUM TO: ALL RMA IR PROGRAM PARTICIPANTS INVOLVED IN SOIL CONTAMINATION DETERMINATIONS

SUBJECT: Protocol to Determine Migration Potential of Contaminated Soils (Solid Waste Leaching Procedure)

1. Objective: To establish program policy for the determination of migration potential of contaminated soils at RMA.

2. Discussion:

a. Problem definition studies performed to date at RMA have been oriented to (1) define the extent of groundwater pollution beneath the Arsenal and (2) locate its primary sources. Through guidance from the State of Colorado, the "action level" for these studies have been set to be drinking water criteria established by EPA or the State, whichever is more stringent. Development of Government control strategies for the migration pathways is initiated when groundwater contamination reaches the aforementioned "action level."

b. Difficulty arises when one examines the source areas to determine the extent (area and volume) of contaminated material that should be controlled. Historical records are often imprecise. Groundwater surveys typically are not detailed enough to locate accurately the point of pollutant introduction into the aquifer. Lastly, due to the lack of corresponding "action levels" for pollutants in soil, any previous soil sampling has been only exploratory at best.

c. Various regulatory agencies have wrestled with the preceeding lack of soil criteria for several years. To date no state or federal guidance has been promulgated addressing this specific issue. The closest regulatory attempt has been the batch leaching acceptability protocol (EP Toxicity Test) for hazardous wastes in the implementation guidelines to the Resource Conservation Recovery Act (inclosures 1 and 2). Discussions with EPA reveal that the EP toxicity test may be the closest soil contamination criteria industry and Government will receive for some time. Severe research and development funding cut backs in EPA has delayed ongoing research in this area.

d. Formulation of FY82 program plans for the RMA IR project has resulted in the immediate need to apply a protocol to investigate if select contaminated soils on the Arsenal represent a migration source. Similar requirements at other USATHAMA IR sites necessitated a rapid review of current regulatory statutes to establish interim USATHAMA policy. The USATHAMA Technology Division performed the assessment with support from Field Systems, Industrial Systems and Environmental/Safety Divisions. Inclosure 3 represents their guidance on the subject issue until such time regulatory agencies promulgate appropriate criteria.

e. To place the inclosed Battelle/EPA soil leaching protocol into its proper frame of reference on the RMA IR project, the following categorization of available extraction techniques and their use is provided:

TABLE 1

EXTRACTION TECHNIQUE	USE
1. Solvent Extraction	Laboratory analytical technique to qualitatively determine types of organics within a solid waste*
2. EP Toxicity	Protocol to determine if a waste is hazardous under RCRA. Alternately, a technique to assess whether a treated RCRA waste still is considered hazardous. Assumes disposal in a municipal landfill.
3. Solid Waste* Leaching Procedure (SWLP)	Protocol to determine whether a solid media has the ability to leach a contaminant at a level requiring control strategies to be employed. Assumes waste material remains contained under in-situ conditions.

*NOTE: "Solid Waste" refers to a solid media containing or having a potential to contain process wastes from Arsenal operations.

f. Three tasks within the FY82 RMA IR project are anticipated to require use of a solid extraction technique. A listing of those tasks keyed against the probable extraction protocol follows:

TABLE 2

TASK	SOLVENT EXTRACTION	EP TOXICITY	SWLP
Basin F Soil Contamination Survey	X		X
Basin F Solidification Study		X	X
Potential Source Area Definition	X		X

G. The rationale used by EPA for selecting a 100 fold attenuation factor, between the point at which the leachate leaves the waste media and the point of human or environmental exposure, is well documented at inclosure 1. EPA recognized that choosing an attenuation factor which reasonably represents the amount of attenuation likely to occur in the real world was one of the most difficult problems faced in formulating any extraction protocol. Specific site parameters greatly control the degree of natural forces acting on the leachate. All things considered, however, EPA decided, pending the completion of further studies, to adopt an "across-the-board" attenuation factor of 100.

h. USATHAMA concurs with EPA's approach of adopting an interim attenuation factor of 100 until RMA site specific data can be reviewed to establish a more accurate factor. Upon updating of the general attenuation factor, the State of Colorado will be approached for concurrence. As long as the leaching procedure itself does not change, a re-assessment of the leachate data can always be undertaken at a later time with the new attenuation factor. The above 100 fold factor would be applied against all drinking water standards now in effect (inclosure 4).

i. Modification to the SWLP to focus on-site specific conditions has been permitted by allowing tayloring of the leaching medium pH and number of sequential extractions.

(1) pH of the leaching medium may be altered from in-situ conditions if it is felt that anomallus natural phenomena (e.g., acid rains) would be encountered at the site. For the case of RMA, this phenomena is a real occurrence and should be incorporated into the leaching procedure for near surface soils.

(2) Sequential extraction steps simulate repeated perculation events at a site. Its use at a recent contamination spill/landfill site would be a valuable tool in providing a qualitative estimate of the degree (increasing, decreasing, or steady state) of future leaching. However, its use at a historic site would appear to be marginal. Each task manager should consider the usefulness and cost effectiveness of sequential extractions within their tasks.

j. If there are any questions regarding this policy during implementation, please contact the undersigned at ext 2041.

Donald Campbell

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DONALD L. CAMPBELL
Senior Project Engineer

E. Section 281.24 (Characteristic of EP Toxicity)

There is persuasive evidence that the contamination of groundwater through the leaching of waste contaminants from land disposed wastes is one of the most prevalent pathways by which toxic waste constituents migrate to the environment. EPA's damage files contain numerous incidents of groundwater pollution resulting from the indiscriminate dumping and improper landfilling of wastes. Additionally, the legislative history of RCRA is replete with indications that such groundwater contamination was one of Congress' primary areas of concern. In the proposed regulation, EPA addressed this problem by developing a test procedure called the Extraction Procedure (EP) designed to identify wastes likely to leach hazardous concentrations of particular toxic constituents into the groundwater under conditions of improper management. Under this procedure, constituents were extracted from the waste in a manner designed to simulate the leaching action that occurs in landfills. This extract was then analyzed to determine whether it possessed any of the toxic contaminants identified in the National Interim Primary Drinking Water Standards (NIPDWS). If the extract contained any of the contaminants in concentrations 100 times greater than that specified in the National Interim Primary Drinking Water Standards, the waste was considered to be hazardous.

Like other test procedures employed to identify hazardous characteristics, the EP was intended to serve as a quick test for identifying wastes which are capable of posing a substantial present or potential hazard when improperly managed. Consequently, in devising the test, EPA necessarily had to make certain assumptions about the improper management to which toxic wastes capable of contaminating groundwater are likely to be subjected. In making such assumptions, EPA believed it important to employ a reasonably conservative mismanagement scenario—in view of the statutory mandate to protect human health and the environment, the broad statutory definition of hazardous waste and also because the phenomenon of long term leaching is only incompletely understood. On the other hand, EPA considered it important not to utilize a wholly implausible mismanagement scenario, since by doing so it would end up regulating as hazardous those wastes which were quite unlikely to ever cause a problem.

The result of these deliberations was a decision to model the EP upon a mismanagement scenario for toxic wastes which constitutes a prevalent form of improper management—namely, the co-disposal of toxic wastes in an actively decomposing municipal landfill which overlies a groundwater aquifer. EPA realized in making its co-disposal assumption that actively decomposing municipal waste landfills generate more aggressive leachate media than other landfills and thus, that its assumption was a relatively conservative one. It

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nevertheless believed the co-disposal assumption to be reasonable. first, because wastes are customarily landfilled, second, because most categories of waste have the potential to be disposed of in municipal waste landfills, third, because the predicted degree of contaminant concentration in leachate could occur with respect to wastes which are not likely to be disposed of in municipal landfills and fourth, because Congress expressed particular concern about the disposal of toxic wastes in municipal landfills. EPA also realized its assumption that the landfill overlies a groundwater aquifer was a relatively conservative one. It believed, however, that this assumption was consistent with its concern for the disposal of wastes in environmentally sensitive areas and with the fact that a groundwater body, once contaminated, may remain contaminated for a number of years. Furthermore, it believed this assumption to be somewhat mitigated by its further assumption that there would be some attenuation in the concentration of toxicants in the leachate between the point the leachate leaves the disposal site and the point the toxicants reach environmental receptors.

Taking these assumptions as its framework, EPA developed the EP test to simulate the physical processes which would occur in an actual landfill characterized by these assumptions. To simulate the acidic leaching medium which occurs in actively decomposing municipal landfills, EPA chose to employ an acetic acid leaching medium with a pH of 5.0 (±0.2). To simulate the leaching process, EPA specified a procedure requiring mixing of the solid component of the waste with the acidic leaching medium for a period of 24 hours. To duplicate the attenuation in concentration expected to occur between the point of leachate generation and the point of human or environmental exposure, EPA applied a dilution factor of 10 to the concentration of toxic constituents observed in the test extract.

EPA was convinced that the proposed EP represented a valid and acceptable test for identifying wastes likely to leach toxic constituents into groundwater. Because, however, this test was innovative in character and reflected a fair amount of groundbreaking inquiry, it drew the greatest response from the public of all the test protocols utilized in identifying the characteristics. The most important of these comments are discussed below.

A number of commenters expressed disagreement with EPA's proposed use of a 10-fold dilution factor to calculate the attenuation in toxicant concentration expected to occur between the point at which the leachate leaves the waste and the point of human or environmental exposure. Some commenters thought that the 10-fold dilution factor was too liberal and that no dilution factor would be more appropriate. The majority felt that the 10-fold dilution factor was too conservative and that a higher dilution factor would be more appropriate.

Choosing an attenuation factor which reasonably represents the amount of attenuation likely to occur in the real world was one of the most difficult problems EPA faced in formulating the EP—a problem which reflects in microcosm many of the difficulties of modeling complex physical processes with a short term test. As leachate migrates vertically from the landfill site towards the groundwater strata, a number of attenuating processes can occur—including adsorption, absorption, ion exchange, filtration, and dilution. When the leachate enters the groundwater zone its movement changes from vertical to horizontal and it will tend to form a slug or plume of contaminated water rather than mix generally with the groundwater flow. This plume of contaminants may experience some dilution, depending on the local geology, the groundwater flow, and the nature of the contaminants. Once the plume of contaminated water is drawn into a pumping well, some further dilution tends to take place, depending upon the amount of water withdrawn and the rate at which it is withdrawn. Unfortunately, all these attenuation mechanisms are dependent upon site specific conditions. While some sites may exhibit attenuation of 500-fold, others will exhibit very little attenuation at all. Moreover over time, a site that originally exhibits 500-fold attenuation may become so saturated that the attenuation mechanisms no longer work and the site begins to flush at the same rate at which it is charged.

In order to formulate a reasonable dilution factor, EPA assumed in the proposed regulations that leachate from the landfill passed unattenuated through the soil underlying the landfill to the groundwater zone and that drinking water wells were situated 500 feet down gradient from the landfill site. Relying on projections from a mathematical model which incorporated these assumptions and on empirical data from field analyses, EPA concluded that a dilution factor of 10 was a conservative, but reasonable, figure.

EPA has had an opportunity to carefully re-evaluate its original choice of a dilution factor and is now of the opinion that the 10-fold dilution factor was inappropriate. A number of considerations have prompted it to come to this conclusion. In the first place, EPA is concerned that, while the dilution factor plays a critically important role in determining the scope of coverage of the EP, there is relatively little empirical data upon which to base such an attenuation factor. It is consequently somewhat troubled by its assumption that the soil underlying the landfill is a delay mechanism only and that there is no attenuation in the concentration of toxic contaminants between the point of actual leachate generation and arrival at the groundwater aquifer. Second, in view of this uncertainty, EPA attaches some importance to the fact that there is no variance or "delisting" procedure for wastes which fail the EP. This absence of a variance procedure, while perfectly permissible, tends to magnify the consequences of a wastes being anomalously brought into the system by the EP. Third, EPA believes the EP to be a somewhat less precise instrument than the listing mechanism for determining hazard, inasmuch as the EP fails to take into account factors such as the concentration of toxicants in the waste itself and the quantity of waste generated which could have a bearing on the hazardousness of the waste. EPA consequently prefers to entrust determinations of marginal hazard to the listing mechanism rather than to the EP.

On the basis of these considerations, EPA has decided, pending the completion of further studies, to alter the proposed dilution factor by adopting an attenuation factor of 100. EPA is adopting a 100-fold attenuation factor because it is confident that anything which fails the EP at this factor has the potential to present a substantial hazard regardless of the attentuation mechanisms at play. If forthcoming studies demonstrate that another attenuation factor is more appropriate EPA will adjust the dilution factor accordingly.

EPA does not intend this alteration in the dilution factor to constitute what may be perceived as an untoward relaxation of the EP. It is simply electing to exercise a degree of caution in the face of the lack of empirical substantiation for its EP leaching test to ensure that the EP only captures wastes which are certain to present a substantial hazard. Since this alteration of the attenuation factor is based as much on EPA's desire to engage in cautionary rulemaking as on an environmental re-evaluation of the attenuative processes which influence concentrations in leachate. EPA has listed and intends to continue to list wastes which have extract concentrations of less than 100-times drinking water standards. This listing will to a significant degree compensate for the alteration in the attenuation factor and will prevent the overall coverage of the Subtitle C regulations from being measurably reduced.

A number of commenters argued that EPA improperly based the EP on a mismanagement scenario which assumed co-disposal in the acidic environment of a municipal waste landfill. These commenters generally argued that the co-disposal assumption is inapplicable to numerous classes of waste which are never co-disposed with municipal wastes and which do not leach at the aggressive rates characteristic of co-disposal situations. These commenters suggested that EPA employ an alternative leachate medium, such as distilled water, for those wastes which are unlikely to be co-disposed with municipal wastes.

EPA disagrees with these commenters. EPA believes that the level of leachate concentration predicted by the EP is reasonably in keeping with the concentrations which could realistically occur in most waste management situations and that employment of an acidic leaching medium is therefore appropriate. Most wastes, even those which are unlikely to be disposed of in a municipal landfill, are likely to come into contact with some form of acidic leaching media during their management histories or could otherwise encounter environments which could cause them to leach comparable levels of toxic constitutents. Furthermore, inasmuch as the phenomenon of long term leaching is not well understood and there is no consensus within the scientific community on a short term leaching test, EPA believes it has the power to employ a leaching model which fails to take into account the physical processes affecting particular generators even if this model errs on the side of caution. See, *Ethyl Corp.* v. *EPA*, 541 F.2d 1, 24–29 (D.C. Cir. 1976 *en banc*); *Hercules, Inc.* v. *EPA*, 598 F.2d 91, 104–106 (D.C. Cir. 1978).

In any event, the change to an attenuation factor of 100 lays to rest the concerns of those who argued that the acidic leaching medium was too aggressive to apply to them. EPA is quite convinced that any waste which fails the EP at the 100-times standard presents the potential for substantial hazard if improperly managed no matter what leaching media it is actually exposed to.

A number of commenters argued that the EP is not sufficiently reproducible for use in defining hazardous waste. Some commenters, basing their argument on studies which have been conducted on the reproducibility of the EP, argued that these studies demonstrate an unacceptable variability in the results obtained by the EP. Other commenters, who did not base their arguments on these studies, argued simply that EPA has not shown the EP to be reproducible and therefore may not appropriately employ the EP in a regulatory framework.

EPA disagrees. Sensitive throughout the process of developing the EP to the issue of ensuring reproducibility, EPA commissioned a number of studies to evaluate the EP, including a study by the NUS Corporation, a study by the American Electroplaters' Society, and an ongoing study being conducted by the Oak Ridge National Laboratory. In addition, a study commissioned by the Electric Power Research Institute (EPRI) has been completed. None of these studies present enough data to draw any hard and fast conclusions. However, data from the EPRI report—the only report which was able to separate out the reproducibility of the EP from the reproducibility of the analytical procedures—suggests that the reproducibility of the EP itself is of the same order of magnitude as the analytical procedures used to analyze the toxic constituents in the extract. Since these analytical procedures have proven to be widely acceptable to private industry, EPA believes that the EP should also prove acceptable.

EPA concedes that the preliminary data indicate some variability in the results obtained by the EP. This, however, is true of all analytical procedures and test methods, especially those which are novel in character. Furthermore, variability can be easily corrected by running further replicates of the test to achieve greater certainty in the results. To accommodate any problems with variability, EPA intends to provide generators with guidance on the number of extractions which they can perform if they want to ensure confidence in the result. In addition, EPA is engaged in research studies which will enable it to further isolate and get a handle on the causes of this variability.

A number of commenters argued that extract from the EP should be tested for toxic contaminants other than those specified in the National Interim Primary Drinking Water Standards.

EPA originally intended the extraction procedure to identify toxic contaminants other than those specified in the National Interim Primary Drinking Water Standards. EPA has been unable to do this, however, because no other chronic exposure threshold levels relating to drinking water consumption have been established for other contaminants. This should not cause a problem, because EPA is regulating wastes containing non-drinking water standard contaminants through the listing process. EPA will reassess its position on this issue, when thresholds are developed for additional contaminants or when the Clean Water Act Water Quality Criteria are adopted in final form.

The proposed EP required generators to separate the liquid and solid portions of their waste as the first step of the procedure, based on the assumption that the liquid portion of the waste would flow out of the landfill independent of any leaching action. Generators were then required to mix the separated solid portion with the acidic leaching medium and, after a further separation, combine the resulting extract with the originally separated liquid portion for analysis. EPA gave generators the option of using either centrifugation or filtration to perform the initial solid-liquid separation and to perform the subsequent separation of solid from leaching solution. However, information obtained since publication of the proposed regulation indicates that use of centrifugation alone is not as efficient as filtration and can lead to carryover of particles larger than 0.45 um. Since a filter the size of 0.45 um was originally selected because particles larger than 0.45 um are expected to be filtered out by the soil prior to reaching the groundwater, EPA has revised the EP to require filtration of both the liquid portion and the extract prior to analysis.

A number of commenters said they encountered severe operational problems when performing the EP on liquids containing very small percentages of solids. To accommodate this problem, EPA is amending the proposed regulation so generators need not perform the EP on liquids containing less than 0.5% solids. Instead, the liquid itself, after filtration, should be considered the extract and directly analyzed for its toxic constituents.

Appendix I—Representative Sampling Method[1]

The methods and equipment used for sampling waste materials will vary with the form and consistency of the waste materials to be sampled. Samples collected using the sampling protocols listed below, for sampling waste with properties similar to the indicated materials, will be considered by the Agency to be representative of the waste.

Extremely viscous liquid—ASTM Standard D140-70 Crushed or powdered material—ASTM Standard D346-75 Soil or rock-like material—ASTM Standard D420-69 Soil-like material—ASTM Standard D1452-65

Fly Ash-like material—ASTM Standard D2234-76 [ASTM Standards are available from ASTM, 1916 Race St., Philadelphia, PA 19103]

Containerized liquid wastes—"COLIWASA" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," [1] U.S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C. 20460. [Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair St., Cincinnati, Ohio 45268]

Liquid waste in pits, ponds, lagoons, and similar reservoirs.—"Pond Sampler" described in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods." [1]

This manual also contains additional information on application of these protocols.

CL II

Appendix II—EP Toxicity Test Procedure

A. Extraction Procedure (EP)

1. A representative sample of the waste to be tested (minimum size 100 grams) should be obtained using the methods specified in Appendix I or any other methods capable of yielding a representative sample within the meaning of Part 260. [For detailed guidance on conducting the various aspects of the EP see "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods," SW-846, U.S. Environmental Protection Agency Office of Solid Waste, Washington, D.C. 20460.[1]]

2. The sample should be separated into its component liquid and solid phases using the method described in "Separation Procedure" below. If the solid residue[2] obtained using this method totals less than 0.5% of the original weight of the waste, the residue can be discarded and the operator should treat the liquid phase as the extract and proceed immediately to Step 8.

3. The solid material obtained from the Separation Procedure should be evaluated for its particle size. If the solid material has a surface area per gram of material equal to, or greater than, 3.1 cm^2 or passes through a 9.5 mm (0.375 inch) standard sieve, the operator should proceed to Step 4. If the surface area is smaller or the particle size larger than specified above, the solid material should be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5 mm (0.375 inch) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described below.

4. The solid material obtained in Step 3 should be weighed and placed in an extractor with 16 times its weight of deionized water. Do not allow the material to dry prior to weighing. For purposes of this test, an acceptable extractor is one which will impart sufficient agitation to the mixture to not only prevent stratification of the sample and extraction fluid but also insure that all sample surfaces are continously brought into contact with well mixed extraction fluid.

5. After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and if the pH rises above 5.2, 0.5N acetic acid should be added to bring the pH down to 5.0 ± 0.2. However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hours and maintained at 20°–40° C (68°–104° F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5N acetic acid. If such a system is not available, the following manual procedure shall be employed:

(a) A pH meter should be calibrated in accordance with the manufacturer's specifications.

(b) The pH of the solution should be checked and, if necessary, 0.5N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2. The pH of the solution should be adjusted at 15, 30 and 60 minute intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5N pH units.

(c) The adjustment procedure should be continued for at least 6 hours.

(d) If at the end of the 24-hour extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional four hours, during which the pH should be adjusted at one hour intervals.

6. At the end of the 24 hour extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

V= (20)(W) − 16(W) − A
V= ml deionized water to be added
W= weight in grams of solid charged to extractor
A= ml of 0.5N acetic acid added during extraction

7. The material in the extractor should be separated into its component liquid and solid phases as described under "Separation Procedure."

8. The liquids resulting from Steps 2 and 7 should be combined. This

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[1] These methods are also described in "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA 600/2-80-018, January 1980.

[1] Copies may be obtained from Solid Waste Information, U.S. Environmental Protection Agency, 26 W. St. Clair Street, Cincinnati, Ohio 45268.

[2] The percent solids is determined by drying the filter pad at 80° C until it reaches constant weight and then calculating the percent solids using the following equation:

$$\frac{(\text{weight of pad} + \text{solid}) - (\text{tare weight of pad})}{\text{initial weight of sample}} \times 100 = \%\ \text{solids}$$

combined liquid (or the waste itself if it has less than ½ percent solids, as noted in Step 2) is the extract and should be analyzed for the presence of any of the contaminants specified in Table I of § 261.24 using the Analytical Procedures designated below.

Separation Procedure

Equipment: A filter holder, designed for filtration media having a nominal pore size of 0.45 micrometers and capable of applying a 5.3 kg/cm² (75 psi) hydrostatic pressure to the solution being filtered shall be used. For mixtures containing nonabsorptive solids, where separation can be affected without imposing a 5.3 kg/cm² pressure differential, vacuum filters employing a 0.45 micrometers filter media can be used. (For further guidance on filtration equipment or procedures see "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.")

Procedure:[3]

(i) Following manufacturer's directions, the filter unit should be assembled with a filter bed consisting of a 0.45 micrometer filter membrane. For difficult or slow to filter mixtures a prefilter bed consisting of the following prefilters in increasing pore size (0.65 micrometer membrane, fine glass fiber prefilter, and coarse glass fiber prefilter) can be used.

(ii) The waste should be poured into the filtration unit.

(iii) The reservoir should be slowly pressurized until liquid begins to flow from the filtrate outlet at which point the pressure in the filter should be immediately lowered to 10–15 psig. Filtration should be continued until liquid flow ceases.

(iv) The pressure should be increased stepwise in 10 psi increments to 75 psig and filtration continued until flow ceases or the pressurizing gas begins to exit from the filtrate outlet.

(v) The filter unit should be depressurized, the solid material removed and weighed and then transferred to the extraction apparatus, or, in the case of final filtration prior to analysis, discarded. Do not allow the material retained on the filter pad to dry prior to weighing.

(vi) The liquid phase should be stored at 4°C for subsequent use in Step 8.

B. Structural Integrity Procedure.

Equipment: A Structural Integrity Tester having a 3.18 cm (1.25 in.) diameter hammer weighing 0.33 kg (0.73 lbs.) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA., 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 1.

Procedure:

1. The sample holder should be filled with the material to be tested. If the sample of waste is a large monolithic block, a portion should be cut from the block having the dimensions of a 3.3 cm (1.3 in.) diameter x 7.1 cm (2.8 in.) cylinder. For a fixated waste, samples may be cast in the form of a 3.3 cm (1.3 in.) diameter x 7.1 cm (2.8 in.) cylinder for purposes of conducting this test. In such cases, the waste may be allowed to cure for 30 days prior to further testing.

2. The sample holder should be placed into the Structural Integrity Tester, then the hammer should be raised to its maximum height and dropped. This should be repeated fifteen times.

3. The material should be removed from the sample holder, weighed, and transferred to the extraction apparatus for extraction.

Analytical Procedures for Analyzing Extract Contaminants

The test methods for analyzing the extract are as follows:

(1) For arsenic, barium, cadmium, chromium, lead, mercury, selenium or silver: "Methods for Analysis of Water and Wastes," Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268 (EPA–600/4–79–020, March 1979).

(2) For Endrin; Lindane; Methoxychlor; Toxaphene; 2,4-D; 2,4,5-TP Silver: in "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," September 1978, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268,

as standardized in "Test Methods for the Evaluation of Solid Waste, Physical/Chemical Methods."

For all analyses, the method of standard addition shall be used for the quantification of species concentration.

This method is described in "Test Methods for the Evaluation of Solid Waste." (It is also described in "Methods for Analysis of Water and Wastes.")

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[3] This procedure is intended to result in separation of the "free" liquid portion of the waste from any solid matter having a particle size >0.45um. If the sample will not filter, various other separation techniques can be used to aid in the filtration. As described above, pressure filtration is employed to speed up the filtration process. This does not alter the nature of the separation. If liquid does not separate during filtration, the waste can be centrifuged. If separation occurs during centrifugation the liquid portion (centrifugate) is filtered through the 0.45um filter prior to becoming mixed with the liquid portion of the waste obtained from the initial filtration. Any material that will not pass through the filter after centrifugation is considered a solid and is extracted.

US ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
INTERIM POSITION PAPER
SOLID WASTE LEACHING PROCEDURE

Contamination of groundwater through the leaching of waste contaminants from land disposed wastes is one of the most prevalent pathways by which toxic waste constituents migrate to the environment. Land disposal includes both landfilling of solid and liquid wastes and lagoon storage/disposal of liquid waste. For many years the Army has disposed of explosives contaminated waste water, organic solvents, pesticides and other chemicals in unlined lagoons that have failed and are proving to be a source of groundwater contamination. This groundwater contaminating leachate arises from the liquid present in the waste and from infiltration of rainwater, surface water or groundwater into the waste.

Obtaining permission from regulatory agencies (EPA/State) to close lagoons/landfills contaminated with explosives wastes and other chemicals that are the source (potential source) of groundwater contamination is a current problem for Army installations and USATHAMA. In the absence of soil standards for these explosive wastes, their degradation products and other chemicals, the recurring question becomes "to what level (concentration) must these soils/sediments be removed and/or treated to permit closure of the lagoon/landfill (what levels of explosive/chemicals are/are not acceptable in the soil)?"

Under the Resource Conservation and Recovery Act (RCRA), EPA developed a test procedure called the Extraction Procedure (EP) designed to identify wastes likely to leach hazardous concentrations of toxic constituents into the groundwater under conditions of improper management. This improper management is based on the co-disposal of toxic wastes in an actively decomposing municipal landfill which overlies a groundwater aquifer. EPA assumes tht this landfill will produce an aggressive acidic leaching media that will migrate to the groundwater.

To simulate the acidic leaching medium, EPA chose to employ an acetic acid leaching medium with pH of 5.0 (± 0.2). To simulate the leaching process, the solid component of the waste will be mixed with the acidic leaching medium for 24 hrs. To simulate the dilution expected to occur in the groundwater, a 100-fold attenuation factor is applied.

Currently, the EP is used to determine the concentration of 8 heavy metals and 6 insecticides/herbicides identified in the National Interim Primary Drinking Water Standards. If the extract contains any of the contaminants in concentrations 100 times greater than that specified in the drinking water standards, the waste is considered to be hazardous.

A small minority of the landfills and none of the lagoons at Army Installations would fit the "improper management scenario" of the EP test. Therefore, the acidic leaching medium may not be representative of the conditions at many installations.

Leaching procedures utilize either columns or batch/shake tests. Column tests require 6 months to 2 or 3 years of time for completion and a considerable cost outlay in manpower and laboratory set-up. The batch/shake test can be accomplished (several tests) in a 2 week period with a considerable savings in cost and time as compared to the column test.

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A USATHAMA team composed of a member from Technology Division, Field Systems Division, Industrial Systems Division and Environmental and Safety Division has reviewed the EP test method, the column leaching method, and the Battelle Solid Waste Leaching Procedure (SWLP), developed under contract to EPA, to determine which procedure would best meet the requirements of problems peculiar to Army Installations. The column leaching test was ruled out by time and economic considerations. The Battelle method which permits other than an acidic leaching medium was chosen by the team because the leaching medium could be tailored to site specific needs and because the method tracks closely the EP test methods. The multiple extractions of the same sample of solid waste will not be performed as given in the Battelle method.

Since no soil standards exist for explosives and other chemicals of interest, standards/criteria for specific waste leachate constituents will have to be negotiated with the regulatory agency (EPA/State) on a site by site basis. In keeping with the philosophy and procedures of the EP test, the same 100-fold attenuation factor (groundwater dilution) will be applied (attenuation factor part of negotiations with regulatory agency), i.e. a leachate constituent concentration greater than 100 times the applicable standard/criteria will be considered hazardous.

This protocol (Battelle Method attached) will be used on an interim basis by USATHAMA and its sub-performers to determine the extent of polluted soil underlying landfills/lagoons that is or has the potential to result in contaminant migration. When EPA or state regulators pass appropriate soil criteria or superceeding soil contamination protocols, the above USATHAMA policy will be so revised.

SOLID WASTE LEACHING PROCEDURE

Battelle Columbus Laboratories

1981

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SOLID WASTE LEACHING PROCEDURE (SWLP)

1.0 EXPERIMENTAL PARAMETERS

1.0 Contact Area/Particle Size

The contact area of the sample should be 3.1 cm^2/g or sized to pass through a 9.5mm standard sieve, unless the solid waste is monolithic. The requirement for contact area and particle size is designed to approach the conditions likely to be encountered in the field disposal environment due to mechanical filling operations and weathering. Some wastes are naturally monolithic. These wastes will not have their particle size reduced as this would cause them to be more leachable than under field conditions. Any waste passing the Structural Integrity Procedure (as given in EPA manuel, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods) will be considered to be monolithic and will be tested as a whole rather than at a reduced particle size.

1.2 Leaching Medium

Laboratory reagent water is suggested for use as the leaching medium. This water should be free from interferences that might interact with the sample and should conform to one of the grades of Reagent Water consistent with Federal Test Method Standard No. 7916.

Where environmental conditions warrant, the use of an alternative medium, such as one to duplicate acid rain, might be justified. However, in the case of acid rain, the acidity of the medium must reflect any changes brought about by passage of the rain through overlying layers of waste and soil, which serve to neutralize both sulfur-based acid precipitation from such causes as the burning of sulfur-containing fuel and naturally occurring acids that arise from biological activity.

1.3 Temperature

The temperature should be normal room/laboratory temperature. The temperature has a decided effect upon the solubility, rate of reaction, and, perhaps leaching of most species. Although ambient temperatures to be expected at land disposal sites range form extremely cold (-40 C) to very high (45 C), the tempertures for the leachates associated with these sites are likely to be less varied. The overlying soil and waste layers with which the leachate is associated have a dampening effect on variations in temperature. Consequently, the temperature for the leachate emerging from the bottom of a disposal site is likely to be that of the soil at the same depth. The limits on seasonal fluctuations in soil temperature at various depths are probably obtainable from disposal site data or can be measured during preliminary site investigations. If the expected temperatures differ substantially from the range of normal laboratory temperatures, then the use of other temperatures is justified.

1.4 Method of Mixing

Any mixing device can be used that will impart sufficient agitation to the mixture such that stratification of the leaching medium-sample mixture is avoided and sample surfaces are continuously brought into contact with the leaching medium.

The specification given in the SWLP follows that contained in EPA's Extraction Procedure (EP) Toxicity Test (40 CFR261.24). Currently only the rotary mixer meets these criteria for preventing stratification and ensuring continuous liquid-solid contact. Examples of rotary extractors are shown in Figures 1 and 2.

1.5 Time of Mixing

The approximate time of mixing is 24 hours. The time specified for each leaching ideally should be sufficient to allow equilibrium to be attained. However, due to the diversity of constituents and effects, no reasonable time per leaching is likely to be satisfactory for all situations. Therefore, the specification of leaching time has to be made out of consideration of factors other than attainment of equilibrium. A time of approximately 24 hours is normally convenient for laboratory scheduling and is consistent with the time specified for other related leaching procedures.

1.6 Solid to Liquid Ratio

The ratio of solid to liquid used for each leaching is calculated to incorporate both an amount sufficient to wet the sample and an excess amount to allow sufficient liquid for proper mixing and subsequent analyses.

The amount of liquid necessary to wet the sample can be determined by packing the sample into a column having some sort of drain, such as a stopcock, at the bottom. A known mass of sample is packed in the column to the density specified for the solid waste and soil cover at the land disposal site. If land disposal site density is not known, the textbook density value for specific soil types can be used. A measured column of liquid is added stepwise to the packed column to avoid edge effects and channeling. Liquid is added until it begins to flow from the column. The volume of liquid added equals the amount needed to wet the sample. For some samples, the waste contains sufficient free liquid that very little or possibly no additional liquid will be needed to wet the sample.

The amount of liquid in excess of that sufficient to wet the sample is added in the ratio of ten volumes per unit weight of sample; that is, a liquid to solid ration of ten to one.

Examples below illustrate the calculation of the proper volume of leaching medium, where the amount of sample to be tested by the SWLP is 100 g.

Example 1. A.

$$\text{Volume to wet sample} = V_{wet} = \frac{\text{Volume liquid added to column}}{\text{Mass sample in column}}$$

$$V_{wet} = \frac{450 ml}{300\ g} = 1.5\ ml/g$$

or

150 ml/100-g sample

B. Excess Volume = Vex = (10ml/g) amount sample

Vex = (10ml/g)(100 g) = 1000ml

C. Total Volume = Vtot = (Volume to wet sample) + (Excess Volume)

Vtot = Vwet + Vex = 150 ml + 1000 ml = 1150 ml or 1.15 l

Example 2. A. Volume to wet sample = Vwet = 0 (that is, sample contained sufficient liquid such that any added to the packed column freely drained out.)

B. Excess Volume = Vex = (10ml/g) amount sample

Vex = (10 ml/g)(100 g) = 1000 ml

C. Total volume = Vtot = Vwet + Vex

Vtot = 0 + 1000 ml = 1000 ml or 1.0 l

The true solid to liquid ratio that a solid waste will experience is highly site dependent and very difficult to forecast precisely. In most cases the ratio will be one of a large amount of solid per unit volume of leachate. The specified ration does not truly reflect the likely field conditions; rather it is a workable amount that will allow sufficient liquid for proper mixing and constituent analysis.

2.0 EXPERIMENTAL

2.1 Apparatus and Materials

In general the apparatus and materials used in the solid waste leaching procedure must be demonstrated to be free from species that might interfere with the analysis of the leachates at the minimum levels of detection. In practice, the apparatus and materials chosen for use in the procedure must be selected with concern for potential interactions between the laboratory equipment and the waste-leachate solution. Examples of interactions to be avoided by careful selection of equipment are:

Dissolution of the solution container by the leachate, as might happen with a hydrofluoric acid containing waste contained in a glass vessel.

Preferential sorption of constituents out of the leachate by the sample container or filtration unit, as might happen with some organic compounds when contained in polypropylene vessels.

Contamination of the leachate by constituents of the sample container, as might happen with leachates when nickel and chromium levels are of interest in wastes that are extracted in stainless steel vessels.

The following discussion is designed to guide the investigator in selecting various items for use in the procedure. Specification of a particular manufacturer or model is for purposes for guidance only. Addresses of suppliers referenced in this section can be found in Appendix A.

2.1.1 Extraction Apparatus

An extraction apparatus must avoid stratification of the sample of solid waste and soil and the leaching medium which would inhibit adequate contact between the sample and leachate. The type of extraction apparatus deemed acceptable for this procedure is the rotary extractor or tumbler (see Figure 1: NBS-design Extractor and Figure 2: EPRI/Acurex Extractor). The extractor consists of a rack or box device to hold the sample containers, which are rotated through 360 at about 30 revolutions per minute.

A four-place tumbler extractor derived from a design by the National Bureau of Standards is illustrated in Figure 1. This equipment may be fabricated by the investigator or obtained commercially (Associated Design and Manufacturing Company, model #3740-40-BRE (four-place tumbler) or model #3740-6-BRE (six-place tumbler)). A six-place tumbler which may be fabricated by the investigator or obtained commercially (Acurex Corporation, no model number available) is shown in Figure 2.

The tumbler bottles should be sized to fit the particular tumbler, such as Wheaton model #348522 roller culture vessels or equivalent, 1.8 to 2.5 L capacity, with an inert cap liner.

2.1.2 Separation Apparatus

Separation of the solid and liquid layers following the extraction of the waste sample will be accomplished by a combination of settling and filtering. Details on performing these manipulations can be found in Section 3.4, which gives stepwise leachate generation instructions.

(a) Filter holder. The filter holder must be capable of supporting a 0.45 micrometer membrane filter and withstanding the pressure needed to accomplish separation. These units may be simple vacuum units (Millipore model #XX10-047-00; Nuclepore model #410400; or equivalent). However, the units capable of being pressurized up to 75 psi are more likely to be needed for the majority of solid wastes (Millipore model #YT30-142-HW; Nuclepore model #420800; or equivalent).

(b) Filter pads. Three sized of filter pads are suggested for use for all filtrations.

(1) Coarse glass fiber prefilter pad (Millipore model #AP25-042-00 or #AP25-127-50 or equivalent).

(2) Fine glass fiber prefilter pad (Millipore model #AP15-042-00 or #AP15-124-50 or equivalent).

(3) 0.45 micrometer nitrocellulose membrane filter (Millipore model #HAWP-047-00 or #HAWP-142-50 or equivalent).

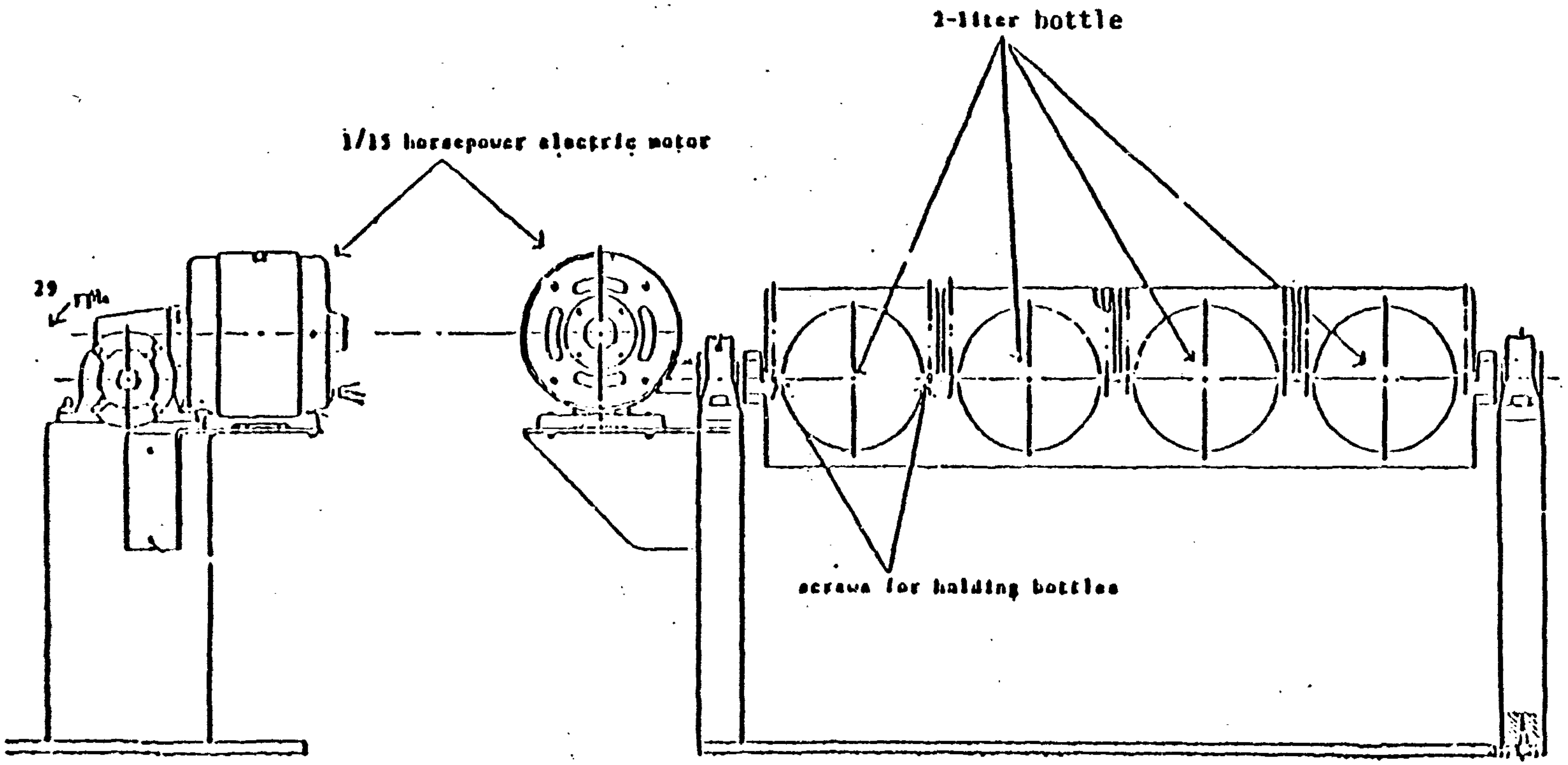

Figure 1 NBS-design Rotary Extractor

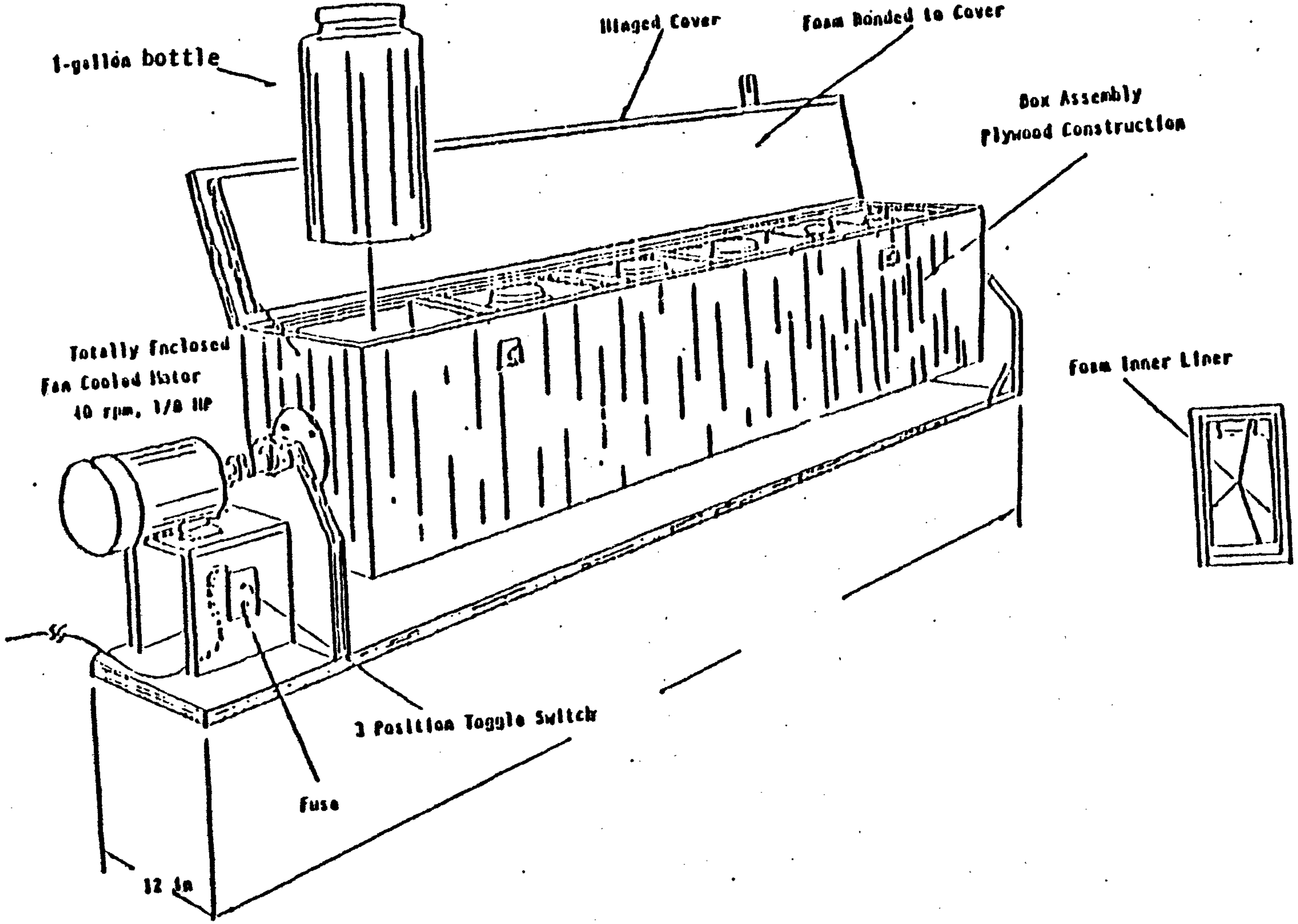

Figure 2 EPRI/Acurex Rotary Extractor

2.1.3 General Labware

(a) Analysis sample bottles. The sample bottles used for containing large amounts of waste or leachate should be of suitable materials, such as glass for organic analysis or polypropylene for inorganic analysis; and they should have screw caps with an inert liner, such as TeflonR.

(b) Sample vials. The container for samples for analysis of volatile organic constituents should have about 40 mL capacity (Pierce Chemical Company model #13075 or equivalent) and have a screw cap with a TeflonR-face silicone septum (Pierce model #12722).

(c) Syringe. The syringe for use in withdrawing a sample of any water-immiscible liquid in the leachate should be a 50 mL glass hypodermic syringe with Luer-Lok tip and a 20-cm 16 guge stainless steel wide-bore needle (Bolab Incorporated model #BB829 or equivalent).

2.2 REAGENTS

2.2.1 Leaching Medium

The leaching medium selected for general use in the SWLP is reagent water. This water must be of sufficient quality that it is free of organic and inorganic interferences at the minimum levels of interest in the subsequent leaching and leachate analyses that will be performed. Water is the recommended leaching medium because it is deemed the best general leaching medium for simulating natural conditions. An acidic leaching medium or a synthetic leaching medium having a multi-component mixture is not recommended for use with this procedure unless justified on the basis of site-specific information. In certain situations, such as the siting of the potential landfill in an area known to have acid rain, a different medium may be justified. In such cases, the investigator must recognize that the use of a different leaching medium alters the comparability of results with those of other investigators using reagent water.

2.2.2 Nitric Acid

A 50% (v/v) mixture trace metals analysis grade concentrated nitric acid (such as J. T. Baker product #9598 or equivalent) and distilled water is recommended for use with this procedure for preservation of leachate samples after collection for analysis of inorganic constituents.

2.3 SAMPLING

2.3.1 Sample Collection and Handling

A representative sample of the solid waste to be tested should be collected using an ASTM standard method that can be applied satisfactorily (such as D140-70, D346-75, D420-69, D1452-65, D223476) or by using one of the methods described in EPA Manuel SW-846. It is particularly important that the solid waste sample be representative of the solid waste.

A minimum sample of 5 kg should be collected and sent to the laboratory in a sealed container or containers. The container must be of suitable material such that it will not react with the waste. In many cases a polyproplyene container will be inert to the waste and, hence, adequate for use. However, the suitability of the container should be assessed in light of the likely composition of the waste.

2.3.2 Sample Preservation

Samples that are stabilized with regard to biological or chemical change may be shipped and stored at room temperature. Samples that are not stabilized and might undergo significant biological or chemical change at room temperture must be maintained at 0-5 C, during shipping and storage. If the stability of the waste or soil is uncertain or unknown, shipping and storage of the waste at 0-5 C is recommended.

Leaching of samples must be initiated within one week of sample collection to preclude gross changes in sample composition with storage time, unless the sample is known to be stable to potential changes in composition.

2.4 LEACHATE GENERATION

2.4.1 Extraction

Step 1 (Mixture Preparation) Take a 100-g representative sample of the solid waste and soil that has been prepared for testing and place it in an extraction vessel (tumbler bottle or equivalent container). Add the calculated volume of leaching medium (Section 1.7) to the extraction vessel.

Step 2 (Tumbling) Tighten the cap on the vessel and mix by tumbling, using the rotary extractor. Tumble for 24 (± 2) hours at room temperature. Stop the rotary extractor, remove the extraction vessel, and allow the mixture to settle for 15 minutes.

Step 3 (Sampling for Volatile Organic Constituents) If a sample of the leachate is needed for analysis of volatile organic constituents, the aliquot should be withdrawn prior to filtration. Obtain a sample for volatile organic constituent analysis by completely filling a 40 mL sample vial with the leachate. Fill the sample vial in such a manner that no air bubbles pass through the sample as the vial is being filled and no air space remains in the vial. Seal the vial with a TeflonR-faced septum and screw-cap. Store it at 0-5 C in an inverted position until the time of analysis. Be certain the sample container is labelled properly to include the date, extraction sequence number, and an appropriate sample identification number.

If a discrete water-immiscible layer is present, withdraw the layer using a syringe with a wide-bore needle. Transfer the layer to a tared sample container of suitable material, such as glass. Determine the mass of the layer and analyze it separately.

Step 4 (Separation) Assemble the filter holder and filter pads following the manufacturer's instructions. Place the 0.45 micrometer nitrocellulose membrane filter pad on the support screen of the filter holder. Add first the fine glass fiber prefilter pad and place the coarse glass fiber prefilter pad on top of the membrane pad, so that the coarse pad will be the one closest to the filter cake.

After assembling the filter apparatus, wet the uppermost filter pad with a small portion of the liquid phase of the extraction mixture. Transfer the remainder of the extraction liquid layer to the filtration unit. Take care to avoid transferring much of the solid from the extraction vessel, because substantial amounts of solid can clog the filter pads. Apply vacuum or gentle pressure (10 to 15 psi) until all liquid passes through the filter.

Stop the filtration when all the liquid has passed throught he filter pads. If this point is not reached under vacuum or using gentle pressure, then increase the pressure stepwise in 10 psi increments to a final maximum pressure of 75 psi.

If liquid remains above the filter pads after 30 minutes of filtration at 75 psi, halt the filtration by slowly venting the pressurizing gas. Be certain to follow the manufacturer's instructions for venting a pressurized filtration apparatus. Some liquid may be trapped in the vent port and may be released. Care must be taken to direct the vent port away from laboratory personnel. After venting, decant the liquid above the filter pads into a suitable container. Place the top-most (coarse) prefilter pad plus any solid/filter cake in a suitable container, such as the extraction vessel for use in the next extraction. Replace the filter pads, placing the fresh pads on the unit in the correct order, and resume filtering.

Repeat the process of replacing the filter pad as often as necessary until all the liquid has been filtered. In each process, retain the topmost (coarse) prefilter pad along with any solid/filter cake.

After halting the filtration, return the unit to atmospheric pressure by either carefully breaking the vacuum or slowly venting the filtration apparatus.

Step 5 (Liquid for pH and Inorganic Constituents Analyses) Transfer an aliquot (usually 25 to 50 mL) of the liquid/filtrate from Step 4 to a suitable container, such as a beaker. Determine the pH. If an analysis for inorganic constituents is needed, add a minimum volume of nitric acid (see Section 2.2.2) to lower the pH to less than 2. Transfer the acidified sample to a suitable container, such as a screw-cap polypropylene bottle. Store at room temperature prior to analysis. The sample container must be labelled properly, to include the date, extraction sequence number, and an appropriate sample identification number.

Step 6 (Liquid for Semivolatile Organic Constituents Analysis) Transfer an aliquot (usually 200 mL) of the Liquid filtrate from Step 4 to be used for semivolatile organic constituents analysis to a suitable container, such as a glass bottled with an inert-lined screw-cap. Store the sample at 0-5 C prior to analysis. The sample container must be labelled properly, to include the date, extraction sequence number, and an appropriate sample identification number.

Step 7 (Remainder of Filtrate) Discard any remaining filtrate after samples have been removed for analysis and dispose of it in accordance with approved laboratory procedures for disposal of potentially hazardous liquids. Dispose of the solid/filter cake in accordance with approved laboratory procedures for disposal of potentially hazardous solid waste.

2.4.2 Further Extractions

The need for further extractions is determined based on the interpretation of results. Depending on the amount of solid waste that is dissolved on each extraction, the repeated extraction of the same solid waste sample with fresh leaching medium can be carried on indefinitely. Repeated extractions of the same solid waste sample will suggest trends in a leachate constituent level (increasing, decreasing, or no change as the waste sample is subjected to repeated extractions).

3.0 QUALITY CONTROL

3.1 Introduction

Quality control for the solid waste leaching procedure involves two aspects. One aspect ensures that the steps to be taken in carrying out the procedure both are free of interferences and meet the needs of the investigator regarding the reliability of the results. The other aspect monitors the procedure while it is underway to determine whether the desired level of quality is being achieved.

The guidelines given in this section are designed to help the investigator fulfill these two aspects of quality control. The basic method used is to process procedure blanks through the various steps in the procedure. These blanks are analyzed to determine whether interferences do appear. The analytical results are used to either modify the procedure to eliminate the source of the interferences or correct the solid waste sample results for background levels routinely and unavoidably picked up. Replicate samples are processed to monitor the precision and accuracy of the procedure.

At present no solid waste reference material or simulant is available. Consequently, interlaboratory comparisons of results from using the procedure on such a material are not possible.

Analytical procedures shall conform to the Quality Assurance Program for US Army Toxic and Hazardous Materials Agency (USATHAMA) and shall not be performed until method/laboratory certification is issued by USATHAMA.

3.2 Leachate Generation

3.2.1 Preliminary

Before any solid waste sample is tested using the solid waste leaching procedure, demonstrate that the procedure is free from any analytical interferences by processing procedure blanks through the various steps.

Step QC (1) (Mixture Preparation) Using a graduate cylinder, add 1.0 L of the leaching medium to an extraction vessel containing no solid waste sample.

Step QC (2) (Tumbling) Tighten the cap on the vessel and mix by the rotary extractor or selected alternative method. Mix for 24 (± 2) hours at room temperature. Stop the rotary extractor, remove the extraction vessel, and allow the mixture to settle for 15 minutes.

Step QC (3) (Separation) Prepare the filtration apparatus by the method of Step 4, Section 2.4.1. Filter the extraction mixture in the same manner as that to be used with the solid waste samples.

Step QC (4) (Sampling for Analysis) Remove aliquots of the procedure blank solution for each type of analysis to be run on the solid waste sample (analysis for pH, inorganics, and volatile and semivolatile organic constituents). If sampling for volatile organic analysis is done, follow the method of Step 3, Section 2.4.1 for filling the sample vial. Label all sample containers, to include date and appropriate sample identification number.

Step QC (5) (Results) Examine the results of the analyses and determine whether any interferences are present. Identify the likely sources of the interferences and modify the procedure accordingly. Repeat the processing of a procedure blank on the modified procedure until the interferences have been eliminated.

3.2.2 Sample Testing

Step QC (6) (Procedure Blank) The procedure blank consists of the leaching medium with no waste added. Process one procedure blank for every batch or every ten solid waste samples tested. Carry the procedure blank through the same steps as the solid waste sample. Ensure that the procedure blank is treated identically to the solid waste sample.

4.0 EXAMINATION OF DATA

4.1 Calculation of Concentration

The data accumulated using the solid waste leaching procedure can be used directly in terms of the concentration of the constituent that was found on analysis of the leachate solution. The general method of calculating this concentration is given in Equation 1.

$$C(x)i = C(anal)i \times DF \qquad \text{Equation 1}$$

In this equation, C(x)i is the concentration x in the leachate solution from extraction sequence number i and has the dimensions of mass of x per unit volume of leachate. C(anal)i is the concentration of x that was found on analysis. DF is the dilution factor or concentration factor for the analysis. The DF gives the extent to which the leachate solution was diluted or concentrated prior to analysis.

4.2 Calculation of Mass Released Per Extraction

The data accumulated can be used to calculate the mass of the constituent released from the solid waste sample for each extraction. The general method for calculating this mass released is given in Equation 2.

$$M(x)_i = \frac{C(x)_i}{S:L} \qquad \text{Equation 2}$$

In this equation, M(x)i is the mass of constituent x that was released from the solid waste sample during extraction sequence number i. C(x)i, the concentration of x in the leachate solution for the extraction i, is calculated using Equation 1. S:L is the solid to liquid ratio used in the initial extraction, in terms of the mass of solid waste sample used to the volume of leaching solution. The volume of leachate in the denominators of both C(x)i and S:L must be in the same units, such as liters or milliliters, so that they will cancel. The term M(x)i will then have the dimensions of mass of x released per unit mass of solid waste, such as mg of x per g of waste.

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APPENDIX A

SUPPLIERS

Acurex Corporation
485 Clyde Avenue
Mountain View, CA 94042
(415) 964-3200

Associated Design and Manufacturing Company
814 North Henry Street
Alexandria, VA 22314
(703) 549-5999

J. T. Baker Chemical Company
222 Red School Lane
Phillipsburg, NJ 08865
(201) 859-2151

Bolab Incorporated, Div. of Water W. Platt Industries
6 Tinkam Avenue
Derry, NH 03038
(604) 434-4941

Millipore Corporation
Ashby Road
Bedford, MA 01730
(800) 225-1380

Nuclepore Corporation
7035 Commerce Circle
Pleasanton, CA 94566
(415) 462-2230

Pierce Chemical Company
P. O. Box 117
Rockford, IL 61105
(815) 968-0747

Wheaton Scientific
1000 North Tenth Street
Millville, NJ 08332
(609) 825-1400

WATER QUALITY CRITERIA

PARAMETER	APPLICABLE CRITERIA	REFERENCE
Aldrin	Hold exposure to a minimum	"Quality Criteria for Water" EPA, 1976.
DBCP	0.0002 mg/l	State of Colorado Department of Health limit per letter to Commander, RMA, 26 Jun 79.
DCPD	1.3 mg/l (toxicity) 0.024 mg/l (odor)	These guidelines are recommended by the US Army Medical Bioengineering Research & Development Lab (26 Aug 76) and are based on toxicology studies conducted by the Army. The National Academy of Sciences Committee on Military Environmental Research has reviewed the procedures and results of the toxicology studies and concurred in the drinking water levels (1 Feb 77). The State of Colorado has requested the Army to meet a lower limit of 0.024 mg/l for DCPD based on an odor threshold value.
DIMP	0.5 mg/l	
Dieldrin	Hold exposure to a minimum	"Quality Criteria for Water" EPA. 1976.
Endrin	0.0002 mg/l	EPA National Interim Primary Drinking Water Regulation.
Fluoride	2.4 mg/l	State of Colorado Department of Health limit per letter to Commander, RMA, 2 Aug 79.
Priority Pollutants	See Federal Register for specific guidelines.	Federal Register Vol 45, No. 231, Friday Nov 23, 1980, pp 79318.
All other organics	No available limits. Removal to detectable limits.	Guidance from OTSG.

FIGURE II-I

-cl 4

APPENDIX D: S-CUBED FINAL REPORT ON EXTRACTION OF BASIN F SOILS

Berry

SYSTEMS, SCIENCE AND SOFTWARE

Final Report

Extraction of Basin F Soils Using the Solid Waste Leachate Procedure

SYSTEMS, SCIENCE AND SOFTWARE

SSS-R-82-5421

EXTRACTION OF BASIN F SOILS USING THE SOLID WASTE LEACHATE PROCEDURE

Final Report

By:
Tobias R. Acciani, Ph.D.
S-Cubed
La Jolla, California 92038

Performed for:

COMPUTER SCIENCES CORPORATION
NSTL- ENGINEERING LABORATORY

Contract No. CSC/ATD-82-C-503

February 19, 1982

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APPENDIX A

SOIL BORING LOG FIELD DATA

1.0 INTRODUCTION

This report provides results of extractions performed by S-Cubed under Contract CSC/ATD-82-C-503 from Computer Science Corporation during the period January 27 through February 15, 1982. The extractions were performed in accordance with the USATHAMA Solid Waste Leaching Procedure (SWLP), prepared by Battelle Columbus, 1981 (Exhibit B, RFP CSC/ATD-82-R-503), on Basin F soil samples supplied by Computer Science Corporation (CSC). Fifty of the total sixty-eight samples received were extracted in accordance with directions from the CSC Project Officers.

This report describes the specific activities undertaken in the execution of the extraction effort. The report is organized in five sections: initial sample handling, liquid-to-solid ratio, leaching, separation of the solid and liquid layer, and pH determination. Raw data are provided in three appendices: Appendix A - copies of data sheets from CSC field team, Appendix B - copies of data sheets for the determination of liquid/solid ratio, and Appendix C - data record of SWLP activities. The S-Cubed SWLP project was successful in that all samples were extracted and shipped to the Midwest Research Institute for chemical analysis.

2.0 INITIAL SAMPLE HANDLING

Samples received by S-Cubed from the CSC field team were stored at 4°C and their receipt documented by filling out a log sheet. Table 1 summarizes the time and date of receipt of samples by S-Cubed. The samples remained in cold storage until they were ready for processing. Appendix A contains the Boring Log Field Data documentation which S-Cubed received along with the samples.

The first step in the SWLP was to grind up the sample to obtain triplicate representative 100-gram samples for extraction. The samples shipped to S-Cubed were between 400- to 500-grams each; only Sample S20011 was less than 300 grams. In this case, two 100-gram replicates were made up and the third only contained 57 grams. The total sample was placed into a mortar which was located in a hood and ground up using a pestle until a particle size of less than a quarter of an inch was obtained. All ground samples were sized with a standard ASTM 1/4-inch sieve. If a sample had an odor, care was exercized by minimizing time of exposure to ambient conditions. After grinding, a 5-gram sample was removed for the liquid/solid ratio determination, the remainder of the sample was returned to the sample container and stored at 4°C.

When the leaching experiment was ready, the sample was taken from storage and poured onto a clean, flat surface. The sample was spread out into a flat disk shape and divided into quarters. Three 100-gram samples were taken, each one was taken from a separate quarter section and the remaining quarter was returned for storage. The 100-gram sample was place into a one-gallon polyethylene container, deionized-distilled water was added, and the sample was leached for 24 hours using a rotating leaching device, e.g., Acurex design.

Table 1

RECEIVING OF SAMPLES

Sample Number	Receiving Time	Sample Date
S20001	12:10 pm	1/27/82
S20002	12.10 pm	1/27/82
S20003	12:10 pm	1/27/82
S20004	12.10 pm	1/27/82
S20005	12:10 pm	1/27/82
S20006	12.10 pm	1/27/82
S20007	12:10 pm	1/27/82
S20008	12.10 pm	1/27/82
S20009	12:10 pm	1/27/82
S20010	12.10 pm	1/27/82
S20011	12:10 pm	1/27/82
S20012	11:30 am	1/28/82
S20013	11:30 am	1/28/82
S20014	11:30 am	1/28/82
S20015	11:30 am	1/28/82
S20016	11:30 am	1/28/82
S20017	11:30 am	1/28/82
S20018	11:30 am	1/28/82
S20019	11:30 am	1/28/82
S20020	11:30 am	1/28/82
S20021	11:30 am	1/28/82
S20022	11:30 am	1/28/82
S20023	11:30 am	1/28/82
S20024	11:30 am	1/28/82
S20025	11:45 am	1/29/82
S20026	11:45 am	1/29/82
S20027	11:45 am	1/29/82
S20028	11:45 am	1/29/82
S20029	11:45 am	1/29/82
S20030	11:45 am	1/29/82
S20031	11:45 am	1/29/82
S20032	11:45 am	1/29/82
S20033	11:45 am	1/29/82
S20034	11:45 am	1/29/82
S20035	11:45 am	1/29/82
S20036	11:45 am	1/29/82

Table 1 (Continued)

RECEIVING OF SAMPLES

Sample Number	Receiving Time	Sample Date
S20037	9:30 am	1/30/82
S20038	9:30 am	1/30/82
S20039	9:30 am	1/30/82
S20040	9:30 am	1/30/82
S20041	9:30 am	1/30/82
S20042	9:30 am	1/30/82
S20043	9:30 am	1/30/82
S20044	9:30 am	1/30/82
S20045	9:30 am	1/30/82
S20046	9:30 am	1/30/82
S20047	9:30 am	1/30/82
S20048	9:30 am	1/30/82
S20049	9:30 am	1/30/82
S20050	9:30 am	1/30/82
S20051	3:30 pm	2/2/82
S20052	3:30 pm	2/2/82
S20053	3:30 pm	2/2/82
S20054	3:30 pm	2/2/82
S20055	3:30 pm	2/2/82
S20056	3:30 pm	2/2/82
S20057	3:30 pm	2/2/82
S20058	3:30 pm	2/2/82
S20059	3:30 pm	2/2/82
S20060	3:30 pm	2/2/82
S20061	3:30 pm	2/2/82
S20062	3:30 pm	2/2/82
S20063	3:30 pm	2/2/82
S20065	3:30 pm	2/2/82
S20066	3:30 pm	2/2/82
S20067	3:30 pm	2/2/82
S20068	3:30 pm	2/2/82

3.0 LIQUID-TO-SOLID RATIO

The Liquid-to-Solid Ratio (LSR) was determined to be the amount of water that naturally sorbs onto the soil extracted. This amount of water must be corrected for in making calculations of extraction efficiencies in accordance with SWLP.

S-Cubed took 5 grams of Basin F sample and placed it into a glass column fitted with a stopcock. The dimensions of this column were 15-cm long by 1-cm internal diameter. The 5-gram sample initially filled the column to about 10 cm. The soil was compressed to 5 cm. S-Cubed was never given any information about soil density, but the packing procedure was consistent for all samples. After the column was packed, 5 ml of deionized-distilled water was added to the column with the stopcock closed. The water was allowed to wet the soil. When the soil was wet, the stopcock was opened and the excess water was allowed to drain into a graduated cylinder. The volume of water retained by the soil was calculated and this number was entered into the data records.

The calculations for the volume of excess water to be added for leaching were:

$$\frac{V}{W} = \frac{\text{liquid}}{\text{solid}} \text{ ratio} = \text{LSR}$$

where

V = volume of water retained by oil, mL

W = weight of soil tested, g

LSR x 100 g = volume of water to be added to leaching solution, mL

In addition to the volume of excess water to be added from the above calculation, one liter of deionized-distilled water was employed as the bulk extraction medium.

When the sample was a sludge, the sludge was initially filtered using an 0.45-micrometer membrane filter. The resulting filtrate was added to the final extracted solution. For these samples, a 100-gram sample was taken from the moist soil sludge sample which remained after filtering without adding excess water.

Table 2 contains the results of the liquid/solid ratio determination.

Table 2

RESULTS OF LIQUID/SOLID RATIO DETERMINATION

Sample Number	Sample Weight (g)	Volume of Water Retained (mL)	Ratio	Volume of Water Added (mL)	Total Water Volume for Extraction (mL)
S20001	5	1.4	0.28	28	1028
S20002	5	2.0	0.40	40	1040
S20003	5	1.5	0.30	30	1030
S20005	5	1.2	0.24	24	1024
S20006	5	1.2	0.24	24	1024
S20007	5	1.5	0.30	30	1030
S20009	5	0.8	0.16	16	1016
S20010	5	1.2	0.24	24	1024
S20011*	5	1.3	0.26	26*	1026*
S20012	5	1.4	0.28	28	1028
S20013	5	1.1	0.22	22	1022
S20016	5	0.8	0.16	16	1016
S20017	5	1.5	0.30	30	1030
S20018	5	1.3	0.26	26	1026
S20020	sludge				1000
S20021	5	1.0	0.20	20	1020
S20022	5	1.6	0.32	32	1032

Table 2 (Continued)

RESULTS OF LIQUID/SOLID RATIO DETERMINATION

Sample No.	Sample Weight (g)	Volume of Water Retained (mL)	Ratio	Volume of Water Added (mL)	Total Water Volume for Extraction (mL)
[illegible]	Sludge				1000
[illegible]	5	1.5	0.30	30	1030
[illegible]	5	1.2	0.24	24	1024
[illegible]	5	2.0	0.40	40	1040
[illegible]	5	1.0	0.20	20	1020
[illegible]	5	1.2	0.24	24	1024
[illegible]	5	0.6	0.12	12	1012
[illegible]	5	1.1	0.22	22	1022
[illegible]	5	1.6	0.32	32	1032
[illegible]	Sludge				1000
[illegible]	5	1.9	0.38	38	1038
[illegible]	5	2.0	0.40	40	1040
[illegible]	5	2.3	0.46	46	1046
[illegible]	5	0.8	0.16	16	1016
[illegible]	5	0.4	0.08	08	1008
[illegible]	5	1.5	0.30	30	1030
[illegible]	5	2.0	0.40	40	1040
[illegible]	Sludge				1000
[illegible]	5	0.8	0.16	16	1016
[illegible]	5	1.6	0.32	32	1032
[illegible]	5	1.9	0.38	38	1038
[illegible]	5	2.7	0.54	54	1054
[illegible]	5	1.0	0.20	20	1020
[illegible]	5	1.2	0.24	24	1024
[illegible]	5	1.1	0.22	22	1022
[illegible]	5	0.7	0.14	14	1014
[illegible]	5	1.3	0.26	26	1026
[illegible]	5	0.5	0.10	10	1010
[illegible]	5	0.8	0.16	16	1016
[illegible]	5	0.6	0.12	12	1012
[illegible]	5	0.5	0.10	10	1010
[illegible]	5	0.5	0.10	10	1010
[illegible]	Sludge				1000

[illegible] 2011c only used 57 grams, volume for extraction was 585 ml.

4.0 LEACHING EXPERIMENT

S-Cubed employed two Acurex rotary extractors which had the capability to extract twelve 100-gram samples per 24-hour period. After the liquid/solid ratio was determined, three 100-gram replicates of the soil samples were placed into three 1-gallon polyethylene containers. One liter of deionized-distilled water was added to each container, plus the volume determined from the liquid/solid ratio. The container was then placed into the extractor and leached for the 24-hour period. Table 3 lists the total time for each leaching.

Since the rotary extractor was operating continuously for 13 days, the extractions usually ran for 23 hours so that the equipment would have a rest; thus preserving the integrity of the equipment.

Because of an electric power failure and a breakdown of one extractor during the course of the leaching experiment, Samples S20057, S20058, S20060, S20061, S20067, and S20068 were extracted on the eighth day after sample collection and Sample S20062 was extracted on the ninth. The SWLP procedure called for seven days to preclude gross change in sample composition, but the time could be extended if the samples are stable. In order to obtain greater resolution on the stability of these samples, Samples S20053 through S20056 were collected the same day as Samples S20057 through S20061 and the latter series were extracted within the timeframe stipulated by the SWLP. Upon analysis by MRI, it may be estimated whether or not the extra one or two days of storage affected the stability of these samples.

Table 3

TIME OF SAMPLE LEACHING

Sample Number	Extraction Start Time	 Date	 Finish Time	 Date	Total Time in Hours/Minutes
S20001	3:53 pm	1/27/82	3:53 pm	1/28/82	24
S20002	3:53 pm	1/27/82	3:53 pm	1/28/82	24
S20003	4:05 pm	1/28/82	4:15 pm	1/29/82	24
S20005	4:05 pm	1/28/82	4:15 pm	1/29/82	24/10
S20006	4:05 pm	1/28/82	4:15 pm	1/29/82	24/10
S20007	4:05 pm	1/28/82	4:15 pm	1/29/82	24/10
S20009	4:48 pm	1/29/82	4:00 pm	1/30/82	23/22
S20010	4:48 pm	1/29/82	4:00 pm	1/30/82	23/22
S20011	4:48 pm	1/28/82	9:15 am	1/31/82*	24/33
S20012	4:48 pm	1/28/82	9:15 am	1/31/82*	24/33
S20013	4:15 pm	1/30/82	4:20 pm	1/31/82	24/05
S20016	4:15 pm	1/30/82	4:20 pm	1/31/82	24/05
S20017	5:22 pm	1/31/82	4:35 pm	2/1/82	23/23
S20018	5:22 pm	1/31/82	4:35 pm	2/1/82	23/23
S20020	5:00 pm	2/1/82	4:00 pm	2/2/82	23
S20021	5:22 pm	1/31/82	4:35 pm	2/1/82	23/23
S20022	5:22 pm	1/31/82	4:35 pm	2/1/82	23/23
S20025	5:00 pm	2/1/82	4:00 pm	2/2/82	23
S20026	5:00 pm	2/1/82	4:00 pm	2/2/82	23
S20027	5:00 pm	2/1/82	4:00 pm	2/2/82	23
S20028	5:00 pm	2/2/82	4:00 pm	2/3/82	23
S20030	5:00 pm	2/2/82	4:00 pm	2/3/82	23
S20031	5:00 pm	2/2/82	4:00 pm	2/3/82	23
S20034	5:00 pm	2/2/82	4:00 pm	2/13/82	23
S20035	5:00 pm	2/3/82	3:55 pm	2/4/82	22/55
S20036	5:00 pm	2/3/82	3:55 pm	2/4/82	22/55
S20038	5:00 pm	2/3/82	3:55 pm	2/4/82	22/55
S20039	5:00 pm	2/3/82	3:55 pm	2/4/82	22/55
S20040	4:30 pm	2/4/82	8:30 pm	2/5/82**	23
S20041	4:30 pm	2/4/82	8:30 pm	2/5/82**	23
S20043	4:30 pm	2/4/82	8:30 pm	2/5/82**	23
S20044	4:30 pm	2/4/82	8:30 pm	2/5/82**	23
S20047	8:30 pm	2/5/82	6:30 pm	2/6/82	22
S20048	8:30 pm	2/5/82	6:30 pm	2/6/82	22
S20051	8:30 pm	2/5/82	6:30 pm	2/6/82	22
S20052	8:30 pm	2/5/82	6:30 pm	2/6/82	22

Table 3 (Continued)

TIME OF SAMPLE LEACHING

	Extraction				
	Start		Finish		
Sample Number	Time	Date	Time	Date	Total Time in Hours/Minutes
S20053	6:30 pm	2/6/82	4:30 pm	2/7/82	22
S20054	6:30 pm	2/6/82	4:30 pm	2/7/82	22
S20055	6:30 pm	2/6/82	4:30 pm	2/7/82	22
S20056	6:30 pm	2/6/82	4:30 pm	2/7/82	22
S20057	4:35 pm	2/7/82	4:00 pm	2/8/82	23/25
S20058	4:35 pm	2/7/82	4:00 pm	2/8/82	23/25
S20060	4:35 pm	2/7/82	4:00 pm	2/8/82	23/25
S20061	4:35 pm	2/7/82	4:00 pm	2/8/82	23/25
S20062	5:45 pm	2/8/82	4:30 pm	2/9/82	22/45
S20064	5:45 pm	2/8/82	4:30 pm	2/9/82	22/45
S20065	5:45 pm	2/8/82	4:30 pm	2/9/82	22/45
S20066	5:45 pm	2/8/82	4:30 pm	2/9/82	22/45
S20067	5:30 pm	2/9/82	5:00 pm	2/10/82	23/20
S20068	5:30 pm	2/9/82	5:00 pm	2/10/82	23/20

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*Samples S20011 and S20012 - rotary extractor breakdown at night, instrument was not repaired until 12:00 pm.

**Samples S20040, S20041, S20043, and S20044 - S-Cubed had a five-hour power failure at night.

5.0 SEPARATION OF THE SOLID AND LIQUID LAYERS

S-Cubed employed six Millipore Hazardous Waste Sample Filtration System devices (Millipore Model No. YT30142HW). The filter pads were the fine glass fiber prefilter pad (Millipore Model AP1512450) and the 0.45 micrometer nitrocellulose membrane filter (Millipore Model HAWP14250). Because the Basin F samples contained clay which clogged the filters continuously, S-Cubed had to modify SWLP filtration procedure. The modification consisted of filtering the sample twice, first with the fine glass fiber prefilter pad, then with the 0.45 micrometer membrane filter. This procedure was employed because placing three filters on top of each other was too slow and the uppermost filter would clog and no sample would pass through the other filters. The following paragraph describes how S-Cubed filtered the Basin F samples. Table 4 contains the times and dates for filtering samples.

After the sample was mixed for 24 hours, it was either filtered immediately or placed into storage at 4°C. A Millipore filtration system was set up with a fine glass fiber prefilter pad, and the sample was poured into the device. Usually, about half of the approximately one-liter sample was filtered at a time. If the filter pad clogged, the remaining unfiltered material was transferred to a beaker and the filter was replaced. The filter device was then reassembled and filtering continued. The second half of the sample contained most of the solids and they were quantitatively transferred to the filter device and filtered. After the sample was filtered, the filter system was taken apart, cleaned with deionized-distilled water, and reassembled with an 0.45-micrometer filter. Again, if clogging of the filter occurred, the filter was replaced and the process continued. The pressure employed for filtration was 75 psi.

When the filtration was complete, the sample extract filtrate was transferred into a one-gallon glass container and stored at 4°C. When the other two replicates were completed, their filtrates were added to the gallon container; the volume of sample extract filtrate totaled three liters. The only exception to this was for Sample S20011, where the volume was 2585 ml because of a small soil sample size.

A 500-mL aliquot of the filtrate was taken from the 3-liter sample and placed into a polyethylene container for pH measurement and preservation for metal analyses (see Section 6.0). The remaining sample was stored at 4°C, waiting for shipment to MRI.

Table 4

SCHEDULE FOR FILTERING

	Filtering		Storage	
Sample Number	Time	Date	Time	Date
S20001	4:30 pm	1/28/82	2:00 pm	1/29/82
S20002	4:30 pm	1/28/82	2:05 pm	1/29/82
S20003	8:00 am	2/1/82	12:00 pm	2/1/82
S20005	1:00 pm	2/1/82	4:30 pm	2/1/82
S20006	8:00 am	2/2/82	1:15 pm	2/2/82
S20007	8:00 am	2/2/82	2:15 pm	2/2/82
S20009	1:20 pm	2/2/82	2:15 pm	2/3/82
S20010	2:20 pm	2/2/82	6:30 pm	2/2/82
S20011	9:00 am	2/3/82	1:00 pm	2/3/82
S20012	1:00 pm	2/3/82	2:00 pm	2/3/82
S20013	1:00 pm	2/3/82	3:30 pm	2/3/82
S20016	8:15 am	2/4/82	10:00 am	2/8/82
S20017	3:30 pm	2/3/82	10:15 am	2/4/82
S20018	3:30 pm	2/3/82	10:00 am	2/4/82
S20020	10:00 am	2/4/82	11:30 am	2/8/82
S20021	10:00 am	2/4/82	12:00 pm	2/8/82
S20022	11:00 am	2/5/82	12:30 pm	2/8/82
S20025	2:30 pm	2/9/82	5:50 pm	2/9/82
S20026	2:30 pm	2/9/82	9:00 am	2/10/82
S20027	2:30 pm	2/9/82	11:30 am	2/10/82

Table 4 (Continued)

SCHEDULE FOR FILTERING

Sample Number	Filtering Time	Filtering Date	Storage Time	Storage Date
S20028	8:30 am	2/10/82	9:30 am	2/10/82
S20030	8:30 am	2/10/82	11:30 am	2/10/82
S20031	9:30 am	2/10/82	12:30 pm	2/10/82
S20034	1:30 pm	2/9/82	9:00 am	2/11/82
S20035	8:00 am	2/9/82	11:30 am	2/9/82
S20036	8:30 am	2/9/82	12:00 pm	2/9/82
S20038	2:30 pm	2/10/82	9:00 am	2/11/82
S20039	8:30 am	2/10/82	1:00 pm	2/10/82
S20040	8:30 am	2/10/82	1:30 pm	2/10/82
S20041	9:30 am	2/10/82	11:00 am	2/10/82
S20043	9:00 am	2/11/82	2:00 pm	2/11/82
S20044	1:00 pm	2/11/82	9:00 am	2/12/82
S20047	11:00 am	2/9/82	3:00 pm	2/9/82
S20048	10:30 am	2/9/82	3:00 pm	2/9/82
S20051	1:30 pm	2/11/82	5:50 pm	2/11/82
S20052	8:30 am	2/12/82	3:30 pm	2/12/82
S20053	9:30 am	2/14/82	5:15 pm	2/12/82
S20054	2:55 pm	2/12/82	11:00 am	2/16/82
S20055	10:00am	2/16/82	3:00 pm	2/16/82
S20056	9:00 am	2/11/82	12:30 pm	2/11/82
S20057	10:30 am	2/10/82	2:30 pm	2/10/82
S20058	11:00 am	2/10/82	4:50 pm	2/10/82
S20060	3:30 am	2/16/82	8:30 pm	2/16/82
S20061	9:00 am	2/13/82	4:50 pm	2/13/82
S20062	11:30 am	2/13/82	1:30 pm	2/13/82
S20064	10:00 am	2/14/82	2:00 pm	2/14/82
S20065	12:00 pm	2/13/82	1:30 pm	2/13/82
S20066	1:30 pm	2/14/82	2:00 pm	2/14/82
S20067	4:30 pm	2/16/82	8:30 am	2/16/82
S20068	10:30 pm	2/13/82	12:00 pm	2/13/82

6.0 pH DETERMINATION

After filtration, the sample was split into a 500-mL sample (polyethylene container) for inorganic analysis and the remainder of the sample (amber glass, Teflon cap) for organic analysis. The pH of the sample was obtained with an Orion pH meter (Model 407A) and an Orion combination pH electrode (Model 91-05). The Orion pH meter and electrode were calibrated after each determination with two buffer solutions (pH4, pH7). The results of the pH determinations are listed in Table 5.

After determination of the pH, the inorganic aliquot was adjusted to a pH value of less than two, with Ultrex nitric acid (volume of added acid was between one and two milliliters). S-Cubed also provided to MRI a one-liter water blank sample for analysis of organic compounds and a 500-mL (with the added Ultrex nitric acid) inorganic blank.

Table 5

pH VALUES FOR THE LEACHATE SAMPLES

Sample Number	pH Value
S20001	6.9
S20002	4.8
S20003	4.9
S20005	5.8
S20006	4.8
S20007	4.4
S20009	4.8
S20010	5.4
S20011	5.9
S20012	8.4
S20013	8.2
S20016	5.6
S20017	5.8
S20018	5.6
S20020	8.1
S20021	5.3

Table 5 (Continued)

pH VALUES FOR THE LEACHATE SAMPLES

Sample Number	pH Value
S20022	5.6
S20025	8.5
S20026	4.7
S20027	5.6
S20028	5.5
S20030	5.2
S20031	5.6
S20034	5.5
S20035	5.8
S20036	5.9
S20038	8.5
S20039	5.7
S20040	5.5
S20041	5.6
S20043	5.5
S20044	5.9
S20047	5.0
S20048	5.4
S20051	7.2
S20052	6.5
S20053	8.2
S20054	8.2
S20055	8.7
S20056	5.3
S20057	4.6
S20058	6.5
S20060	6.2
S20061	5.0
S20062	5.2
S20064	9.1
S20065	9.1
S20066	9.1
S20067	9.1
S20068	8.6

BASIN F SUB-SOIL CONTAMINATION SURVEY

Date 26 Jan 72

RMA SAMPLE #	TYPE SAMPLE	LOCATION	Depth From (ft)	Depth To (ft)	REMARKS
S20001	Soil Boring	RMA-Basin F	0.0	1.0	Boring 21
S20002	"	"	1.0	2.0	
S20003	"	"	2.0	3.0	
S20004	"	"	3.0	4.0	
S20005	"	"	0.0	1.0	Boring 22
S20006	"	"	1.0	2.0	
S20007	"	"	2.0	3.0	
S20008	"	"	3.0	4.0	
S20009	"	"	0.0	1.0	Boring 23
S20010	"	"	1.0	2.0	
S20011	"	"	2.0	3.0	< 300 grams provided

BORING LOG
FIELD DATA

Project Basin F Sub-soil contamination survey Site Basin F Date 1/26/82
Location RMA Job No.
Drill Rig – Inspector [illegible] Operator – Surface El Boring No. 21

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER		Core	CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
								Split Spoon			
–	1/25/82	surface	liner	↓(−) 1.25	0						driven, [illegible] [illegible] to liner / liner in good shape
	1/26/82	liner	down	0	1.5			"		1.35	very difficult to [illegible] for [illegible]
	1/26/82		—	1.5	2.9			"		1.2	[illegible]
	"		—	2.9	4.4			"		1.2	
											1/4" plastic asphalt at
S20001						0.0	1.0				top.
S20002		0.0				1.0	2.0				CL-ML silty clay, - clayey
S20003						2.0	3.0				silt firm, minor fine sand,
S20004						3.0	4.0				moist, very dk gray br.
											2.5Y 3/2. (lighter w/depth)
											changing to dk gray br.
			1.7								(2.5Y 4/2 in bottom)
		1.7									CL-ML (as above) but
											color changes to yel. br.
											10YR 5/4. becomes more
			4.4								silty w/depth, Caliche nodules

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in bot. 0.5'
bot. depth 4.4'

BORING LOG
FIELD DATA

Project Basin F Sub-soil Contamination Survey Site Basin F Date 26 Jan 82
Location RMA Job No.
Drill Rig Inspector T. Myers Operator — Surface El Boring No. 22

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE ft		TYPE OF SAMPLER		Core length	CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
								split spoon			
—	1/26/82	surface	liner	[illegible]	0	—					brown ~ , then black [illegible] [illegible]
[illegible] 1.6	1/26/82	liner	—	0	1.6			"		1.1	Liner ~ 1" thick, steel
	"	—	—	1.6	3.1			"		1.35	
	"			3.1	4.4			"		1.2	Turned from grey to tan at ~ [illegible]
S20005						0.0	1.0				
S20006						1.0	2.0				
S20007						2.0	3.0				
S20008						3.0	4.0				

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BORING LOG
FIELD DATA

Project Basin F Sub-Soil Contamination Survey Site Basin F Date
Location RMA Job No.:
Drill Rig — Inspector Operator Surface El Boring No. BFB-22

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
											¼" plastic (pliable)
											at top.
		0.0									CL, clay, silty, w/minor
											sand, firm, moist, v.
											dk gray br. 2.5Y3/2,
											gradual color change at
											1.3(±) to dk gray br, 2.5 Y4/2,
											gradually becoming lighter
			3.7								in Color w/depth.
		3.7									CL, silty clay mottled w/
											caliche. (above color + pale
			4.1								yellow, 2.5Y7/4. firm moist
		4.1									CL, silty clay, firm-moist,
			4.4								pale yellow, 2.5Y7/4
											BOH 4.4

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Sheet ______ of ______ Sheets

BORING LOG
FIELD DATA

Project Basin F sub-soil contamination Survey Site Basin F Date 1/26/92
Location RMA Job No.
Drill Rig — Inspector T. myers Operator Surface El Boring No. 23

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER		core length	CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	1/26/92	Surface	liner	(1.5)	∞			—			sandy, tan on top
	"	liner	down.	0	1.5			split spoon		1.2	at 1' [illegible] grade
	"	—	—	1.5	3.0			split spoon		1.1	
		0.0	0.05								Asphalt
520009		0.05				0.0	1.0				ML, silt, firm, moist,
520010			0.5			1.0	2.0				very dk. gray br. 2.5Y 3/2
520011		0.5				2.0	3.0				CL, sandy clay, Approx. 15%
											fine grain sand, firm, moist
			1.3								very dk gray br, 2.5Y 3/2
		1.3									CL, sandy clay, approx 25% fine
											sand firm-hard, moist, br-
			2.5								dk br. 10YR 4/3
		2.5									CL, clay w/minor fine sand
											caliche nodules, 1-3/4" rock
											fragment, firm, moist, yel. br.
			3.0								10YR 5/4 - tot. depth 3.0

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BASIN F SUB-SOIL CONTAMINATION SURVEY

T Hill

Date 27 JAN 9

RMA SAMPLE #	TYPE SAMPLE	LOCATION	DEPTH From	DEPTH To	REMARKS
S20012	Boring	Basin F RMA Site 13	0'	1'	
S20013	Boring	Basin F Site 13	1'	2'	
S20014	Boring	Basin F Site 13	2'	3'	
S20015	Boring	Basin F Site 13	3'	4+	
S20016	Boring	Basin F Site 14	0'	1'	
S20017	Boring	Basin F Site 14	1'	2'	
S20018	Boring	Basin F Site 14	2'	3'	
S20019	Boring	Basin F Site 14	3'	4'	
S20020	Boring/slu	Basin F Site 14	0	0	Surface Sludge Sample- Grab
S20021	Boring	Basin F Site 15	0	~~0~~ 1'	
S20022	Boring	Basin F Site 15	1'	2'	
S20023	Boring	Basin F Site 15	2'	3'	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	

BORING LOG
FIELD DATA

Project Basin F Sub-soil Contamination Survey Site Basin F
Location RMA Date 1/27/82
Drill Rig — Inspector T[illegible] Operator Job No. 2.0[illegible]
Surface El Boring No. [illegible] 13

SAMPLE NUMBER	DATE TAKEN	STRATUM FROM	STRATUM TO	DRIVE FROM	DRIVE TO	SAMPLE FROM	SAMPLE TO	TYPE OF SAMPLER		[illegible]	CLASSIFICATION AND REMARKS
		imp.	lin	-0.65							
					0.0						
		~~[illegible]~~	~~[illegible]~~								overburden [illegible] Tan, Sandy
				0.0	1.5			Split Spoon		1.4	no seepage, no caving [illegible]
				1.5	3.0			"		1.5	
				3.0	4.55			"		1.0	
1											3/4" plastic asphalt at
		0.0									top.
i											SM, very silty fine sand
											soft (slightly to non-cohesive),
			2.8								very moist, yel. br. 10YR 5/4
		2.8									to 10YR 5/6, micaceous.
											CL, silty clay w/minor sand,
											firm, moist, numerous
											small caliche veinlets and
			4.5								inclusions in bottom foot.
											(Color as above), micaceous.

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Sheet 1 of 1 Sheets

BORING LOG
FIELD DATA

Project Basin F Sub-liner Contamination Survey Site Basin F Date 29 Jan.
Location RMA Job No.
Drill Rig — Inspector Tyson Operator Surface El Boring No. 31

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER		Single Lgth	CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		Surface	Liner	-1.6	0.0						[illegible] 2" Black Green surface; then Tan for 6", Black I [illegible] to liner.
		Beneath liner	down	0.0	1.55					1.0	Liner looked good.
				1.55	2.9						
				2.9	4.5						

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BORING LOG
FIELD DATA

Project Basin F Sub-Soil Contamination Survey Site Basin F Date 27 Jan 82
Location ________________ Job No. ________
Drill Rig — Inspector Tingen Operator ________ Surface El ________ Boring No. 32

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		-1.6	0.0								Black 1st 1-2"; then dark brown
	1/27	Bollen flim	Down	0.0	1.5		very	light			to a lighter brown ~6-8" above
	1/27			1.5	3.0						liner; liner looked good
	1/27			3.0	4.5						
		0.0									CL, silty clay, hard,
											moist, (wet color) very
											dk gray br. 2.5Y 3/2
											becomes lighter and sdy
											in bot 0.3' (dk gray br.
			1.6								2.5Y 4/2.
		1.6									CL, silty sandy clay,
											hard, slightly moist
											numerous caliche veinlets.
			2.7								(wet color) dk yel. br. 10YR 4/4
		2.7									CL, silty-sdy clay, hard, slightly
			4.5								moist-moist, mottled w/caliche,
											hard, (wet color) dk yel. br. 10YR 4/4

wet
[illegible]

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BORING LOG
FIELD DATA

Project Basin F Sub-Plan Contamination Survey Site Basin F Date 27 Jan 82
Location RMA Job No.
Drill Rig — Inspector T. Myers Operator Surface El Boring No. 33

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		Surface	line	-1.8	0.0						Tan or light brown 1st 1"
		0.1 in	Down	0.0	1.5						Black from there to line,
		[illegible]		3.0	4.5	Hi	[illegible]	H$_2$O in [illegible]			cel. dry,
						[illegible]	[illegible]	carbonate	[illegible]	[illegible]	
		0.0									CL, silty clay w/minor
											sand, siliceous, occ.
											small caliche inclus. &
											veinlet hard, moist
											(wet color) from top to
											1.0' - gradual color change
											from very dk gray br to
											dk gray br. (2.5Y 3/2 -
											2.5Y 4/2). Below 1.0' to
											4.5' color varies from yel.
											br. to dk yel. br. 10YR 5/4
			4.5								10YR 4/4

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BORING LOG

FIELD DATA

Project Basin F Sub-Liner Contamination Survey Site Basin F Date 28 Jan 82
Location RMA
Drill Rig — Inspector T. Myers Operator ____ Surface El ____ Job No. ____ Boring No. 12

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		surface	liner	-175	0.0			- —			
				0.0	1.55						tan for 1st 8-10"; then black
				1.55	3.0						to liner, relatively dry, soil like

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BFB 31 Soil Classifications
Borings 31 + 12

0.0 – 1.3 – CL silty clay w/occ
caliche inclus., hard, micaceous
dry to very slightly moist (wet
color) lt olive br. 2.5Y5/4

1.3 – 2.5 ML-CL clayey silt -silty clay, firm, dry,
(minor sd) occ. caliche veinlet, micaceous,
(wet color), yell. br. 10YR5/6

2.5 – 4.5 CL, silty clay w/minor sand,
mottled w/ caliche, hard, dry to
very slightly moist, (wet color)
yel. br. to dk yel. br. 10YR 5/6 –
4/6 micaceous

BFB 12

0.0 – 2.1 – ML, clayey sandy silt micaceous,
firm, moist (wet color)
dk yel. br. 10YR 4/4

2.1 – CL, silty clay w/minor sand,
micaceous, hard, moist (wet color)
4.0' dk yel. br. 10YR 4/4, numerous
small caliche veinlets + inclus.

4.0' – 4.5 CL, silty clay, soft-firm, (calcareous) very moist
(wet color) very pale br. 10YR 7/4

11 samples
3 extractions

BASIN F SUB-SOIL CONTAMINATION SURVEY

Date 30JAN82

RMA SAMPLE #	TYPE SAMPLE	LOCATION	DEPTH FROM	DEPTH TO	REMARKS
S20051 X	Boring/Sludge	Site 01	0	0	Surface Sludge Composite Grab
S20052 X	Boring	Site 01	0'	1'	
S20053 X	Boring	Site 01	1	2'	
S20054 X	Boring	Site 01	2'	3'	
S20055 X	Boring	Site 01	3'	4'	
S20056 X	Boring	Site 11	0'	1'	
S20057 X	Boring	Site 11	1'	2'	
S20058 X	Boring	Site 11	2'	3'	
S20059	Boring	Site 11	3'	4'	
S20060 X	Boring	Site 50	0	1	
S20061 X	Boring	Site 50	1'	2'	
S20062 X	Boring	Site 50	2'	3'	
S20063	Boring	Site 50	3'	4'	

BORING LOG
FIELD DATA

Project Basin F Sub-liner Survey Site Basin F-RMA Date 30 Jan 82
Location
Job No.
Drill Rig Inspector Operator Surface El Boring No. 50

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
1		-1.7	0.0	surface to bin							Green grass, 2" black, 5-6" Tan Black to bottom
		0.0									Sm-ML, silty fine sand-sandy
											silt, soft (non to slightly
											cohesive, (Probably reworked
											fill) slightly moist, micaceous,
											dk gray br, 2.5Y 4/2 in top
											0.2-0.3', dk yel. brown
											(10 YR 4/6) below 0.3', gradually
											changing to yel. br. 10YR 5/4
			2.3								with depth.
		2.3									CL, silty clay w/minor sd
											+ occ small pea grav., occ.
											caliche nodules (increasing
											in bot 0.5') firm-hard, moist,
											yel. br., 10YR 5/6, contains
											mica flakes.

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BORING LOG
FIELD DATA

Project Basin F Sub-line Survey Site RMA Date 30 Jan '82
Location "
Job No.
Drill Rig Inspector Operator Surface El Boring No. 01

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	—	Surface	[illegible]	-1.5	0.0						Brown, moist (not wet) soil-like
		1.0									ML, sdy silt, micaceous,
											firm, (Probably reworked
											fill) moist, dk. gray br.
			0.7								2.5Y 4/2
		0.7									CL-CH, Clay w/minor silt
											& sd, (micaceous), firm-hard,
											moist, very dk gray br. 2.5Y 3/2
			"								becoming somewhat lighter color
			1.9								toward bot.
		1.9									CL, silty clay mottled w/
											caliche, firm-hard, moist
											dk grayish br. 2.5Y 4/2. Caliche
			4.5								is lt. gray (2.5Y 7/2) - (Rather than

normal white to pale yel.)

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BORING LOG
FIELD DATA

Project [illegible] E [illegible] Survey Site [illegible] F-[illegible] Date 30 Jan 82
Location –
Drill Rig Inspector Operator Surface El Job No.
Boring No. 4

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	–	Surface	[illegible]	-1.55	0.0						
											Tar & Sandy first 2-3", [illegible]
											Black to [illegible], [illegible] dry,
											[illegible] moist with [illegible]
		0.0									
											ML-SM, sdy silt - silty very
											fine sand, micaceous, firm
											moist - slightly moist. (Probably
											reworked fill), dk gray br.
											2.5Y 4/2 in top 0.3' changing
											to lt olive br. in bottom
			1.7								(2.5Y 5/4).
		1.7									CL, silty clay w/minor sd,
											micaceous, occ. caliche incl.,
											firm - hard, moist, dk yel. br.
			2.4								10YR 4/6.
		2.1									CL, silty clay w/minor sd sizes, small gra.
											grav. inclusions, calcareous, firm, moist,

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4.5

Sheet ___ of ___ Sheets

Very pale br. to yellow, 10YR 7/4 - 7/6

BASIN F SUB-SOIL
CONTAMINATION SURVEY

Date 1 Feb 82

PRA SAMPLE #	TYPE SAMPLE	LOCATION	DEPTH FROM	DEPTH TO	REMARKS
S20064 X	Boring	Site 02	0	1'	
S20065 X	Boring	Site 02	1	2	
S20066 X	Boring	Site 02	2	3	
S20067 X	Boring	Site 02	3	4	
S20068 X	Boring/sludge	Site 11	0	0	Surface Sludge Samposite-Compos. Grab

BORING LOG
FIELD DATA

Project Basin E - Soil Liner Survey Site Basin RMA Date 1 Feb 72
Location --
Drill Rig ______ Inspector ______ Operator ______ Surface El ______ Job No. ______ Boring No. 02

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
	--	surface	liner	-1.35	0.0						overburden - Brown col. dry
											sludge, liner very dark
											with local pockets of [illegible]?
											~~[illegible]~~
											~~[illegible]~~
		0,0									dk. sandy clayey silt
											w/mica flakes & minor sd
											Firm, very moist, very dk
											Grey br. in upper 0.2'; 2.5Y3/2;
											below 0.2' spl is olive br
											2.5Y 4/4 gradually changing
			1.7								to lt. olive br (2.5Y 5/4) w/depth.
		1.7									(Possibly reworked Fill)
											lt. silty clay w/minor sd
											& mica flakes, firm, very moist,
											occ. caliche veinlets. lt olive br.
			3.3								(2.5Y 5/4) gradually changing to

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Sheet 1 of 2 Sheets

yel. br (10YR 5/6) with depth.

BORING LOG
FIELD DATA

Project ______ Site ______ Date ______
Location ______ Job No. ______
Drill Rig ______ Inspector ______ Operator ______ Surface El ______ Boring No. 07 (Continued)

SAMPLE NUMBER	DATE TAKEN	STRATUM		DRIVE		SAMPLE		TYPE OF SAMPLER			CLASSIFICATION AND REMARKS
		FROM	TO	FROM	TO	FROM	TO				
		3,3									CL, silty clay, calcareous
											w/minor sand, soft, very
			4.3								moist, very pale br., 10YR 7/4
		4.3									CL, silty clay w/minor sd.,
											mica flakes, soft-firm,
			4.5								very moist, yel. br. 10YR 5/4

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Sheet 2 of 2 Sheets

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BASIN F Sub-liner Soils Analysis

ple #	Sample Type	Site #	Depth (ft)	Shipping Date	EXTRACTION	
001	Boring	21	0-1	26 JAN	X	
02	"	"	1-2	"	X	
03	"	"	2-3	"	X	
04	"	"	3-4	"	NO	
05	"	22	0-1	"	X	
06	"	"	1-2	"	X	
07	"	"	2-3	"	X	
08	"	"	3-4	"	NO	
09	"	23	0-1	"	X	
10	"	"	1-2	"	X	
11	"	"	2-3	"	X	
12	"	13	0-1	27 Jan	X	
13	"	"	1-2	"	X	
14	"	"	2-3	"	NO	
15	"	"	3-4	"	NO	
16	"	14	0-1	"	X	
17	"	"	1-2	"	X	
18		"	2-3	"	X	
19		"	3-4	"	NO	
20	Surface Sample	"	--	"	X	
21	Boring	15	0-1	"	X	
22	"	"	1-2	"	X	
23	"	"	2-3	"	NO	
24	"	"	3-4	"	NO	
25	Surface Sample	70	—	28 Jan	X	

Basin F Sub-liner Soils Analysis

mple #	Sample Type	Site #	Depth	Shipping Date	EXTRACTION
0 26	Boring	70	0-1	28JAN	X
27	"	"	1-2	"	X
28	"	"	2-3	"	X
29	"	"	3-4	"	NO
30	"	60	0-1	"	X
31	"	"	1-2	"	X
32	"	"	2-3	"	NO
33	"	"	3-4	"	NO
34	"	32	0-1	"	X
35	"	"	1-2	"	X
36	"	"	2-3	"	X
37	"	"	3-4	29JAN	NO
38	SURFACE SAMPLE	31	—	"	X
39	Boring	"	0-1	"	X
40	"	"	1-2	"	X
41	"	"	2-3	"	X
42	"	"	3-4	"	NO
43	"	33	0-1	"	X
44	"	"	1-2	"	X
45	"	"	2-3	"	NO
46	"	"	3-4	"	NO
47	"	12	0-1	"	X
48	"	"	1-2	"	X
49	"	"	2-3	"	NO
50	"	"	3-4	"	NO
					16

Basin F Sub-liner Soils Analysis

le #	Sample Type	Site #	Depth	Shipping Date	Extraction	
51	Surface SAMPLE	01	—	2 FEB	X	Sampled 30
52	BORING	"	0-1	"	X	
53	"	"	1-2	"	X	
54	"	"	2-3	"	X	
55	"	"	3-4	"	X	
56	"	11	0-1	"	X	
57	"	"	1-2	"	X	
58	"	"	2-3	"	X	
59	"	"	3-4	"	NO	
60	"	50	0-1	"	X	
61	"	"	1-2	"	X	
62	"	"	2-3	"	X	
63	"	"	3-4	"	NO	
64	"	02	0-1	"	X	Sampled 1 Feb
65	"	02	1-2	"	X	
66	"	02	2-3	"	X	
67	"	02	3-4	"	X	
68	Surface Sample	11	—	"	X	
					16	
					Total = 50	

APPENDIX B

LIQUID/SOLID RATIO (LSR) DETERMINATION DATA SHEETS

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20001A	5.0	1.4 ml	.28	28	1000	1028	100.0	6.9	<2	500
S20001B							100.0	~~4.8~~	2	
S20001C							100.0	~~4~~		
S20002A	5.0	2.0 ml	.40	40	1000	1040	100.0	4.8	<2	500
S20002B							100.0			
S20002C							100.0			
S20003A	5.0	1.5 ml	.30	30	1000	1030	100.0 ✓	4.9	<2	500
S20003B							100.0 ✓			
S20003C							100.0 ✓			
S20005A	5.0	1.2 ml	.24	24	1000	1024	100.0 ✓	5.8	<2	500
S20005B							100.0 ✓			
S20005C							100.0 ✓			
S20006A	5.0	1.2 ml	.24	24	1000	1024	100.0 ✓	4.8	<2	500
S20006B							100.0 ✓			
S20006C							100.0 ✓			
S20007A	5.0	1.5 ml	.30	30	1000	1030	100.0 ✓	4.4	<2	500
S20007B						?	100.0 ✓			
S2000[illegible]							100.0 ✓			

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20009A	5.0	0.8	.16	16		1016		4.8	<2	500
S20009B										
S20009C										
S20010A	5.0	1.2	.24	24		1024		5.4	<2	500
S20010B										
S20010C										
S20011A	5.0	1.3	.26	26		1026	100.0	5.9	<2	500
S20011B							100.0			
S20011C*	5.0	1.3	.26	14.8		584	57.0			
S20012A	5.0	1.4	.28	28		1028		8.4	<2	500
S20012B										
S20012C										
S20013A	5.0	1.1	.22	22		1022		8.2	<2	500
S20013B										
S20013C										
S20016A	5.0	0.8	.16	16		1016		5.6	<2	500
S20016B										
S20016C										

* less than 100g sample

Sample No.	Wt. for L/S.	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S200 [illegible]	5.0	1.5	.30	30		1030		5.8	<2	500
S200 [illegible]										
S200 12										
S200 [illegible]	5.0g	1.3	.26	26		1026		5.6	<2	500
S200 13										
S200 [illegible]										
S200 21	5.0	1.0	.2	20		1020		5.3	2	500
S200 [illegible]										
S200 [illegible]										
S200 [illegible]	5.0	1.6	.32	32		1032		5.6	<2	500
S200 [illegible]										
S200 [illegible]										
S200 [illegible]						1000		8.1	<2	500
S200 [illegible]	sludge				1000	1000				
S200 [illegible]	"					1000				
S200 [illegible]	"					1010		8.5	<2	500
S200 [illegible]	"					1000				
S200 [illegible]	"					1000				

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20026A	5.0	1.5	.3	30	1000	1030		4.7	<2.0	500
S20026B										
S20026C										
S20027A	5.0	1.2	.24	24	1000	1024		5.6	<2	500
S20027B										
S20027C										
S20025A	5.0	2.0	.4	40		1040		5.5	<2	500
S20025B										
S20025C										
S20030A	5.0	1.0	.2	20		1020		5.2	<2	500
S20030B										
S20030C										
S20031A	6.0	1.2	.24	24		1024		5.6	<2	500
S20031B										
S20031C										
S20034A	5.0	0.6	.12	12		1012		5.5	<2	500
S20034B										
S20034C										

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20035A	5.0	1.1	.22	22	1022	→		5.8	<2	500
S20035B										
S20035C										
S20036A	5.0	1.6	.32	32	1032	→		5.9	<2	500
S20036B										
S20036C										
S20038A	5.0							8.5	<2	500
S20038B										
S20038C										
S20039A	5.0	1.9	.38	38	1038	→		5.7	<2	500
S20039B										
S20039C										
S20040A	5.0	2.0	.40	40	1040	→		5.5	<2	500
S20040B										
S20040C										
S20041A	5.0	2.3	.46	46	1046	→		5.6	<2	500
S20041B										
S20041C										

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20043A	5.0	.8	.16	16	1016	→		5.5	<2	500
S20043B										
S20043C										
S20044A	5.0	0.4	.08	8	1008	→		5.9	<2	500
S20044B										
S20044C										
S20047A	5.0	1.5	.3	30		1030		5.8	<2	500
S20047B										
S20047C										
S20048A	5.0	2.0	.40	40		1040		5.4	<2	500
S20048B										
S20048C										
S20051A	sludge							7.2	<2	500
S20051B										
S20051C										
S20052A	5.0	0.8	.16	16		1016		6.5	<2	500
S20052B										
S20052C										

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20053A	5.0	1.6	.32	32		1032		8.2	<2	500
S20053b										
S20053c										
S20054A	5.0	1.9	.38	38		1038		8.2	<2	500
S20054b										
S20054c										
S20055A	5.0	2.7	.54	54		1054		8.7	<2	500
S20055b										
S20055c										
S20056A	5.0	1.0	.2	20		1020		5.3	<2	500
S20056b										
S20056c										
S20057A	5.0	1.2	.24	24		1024		4.6	<2	500
S20057b										
S20057c										
S20058A	5.0	1.1	.22	22		1022		6.5	<2	500
S20058b										
S20058c										

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S20060A	5.0	0.7	.14	14		1014		6.2	<2	500
S20060b										
S20060C										
S20061A	5.0	1.3	.26	26		1026		5.0	<2	500
S20061b										
S20061C										
S20062A	5.0	0.5	.10	10	✓	1010		5.2	<2	500
S20062b										
S20062C										
S20064A	5.0	0.8	.16	16	✓	1016		9.1	<2	500
S20064b										
S20064C										
S20065A	5.0	0.6	.12	12	✓	1012		9.1	<2	500
S20065b										
S20066C										
S20066A	5.0	0.5	.10	10	✓	1010		9.1	<2	500
S20066b										
S20066c										

Sample No.	Wt. for L/S	Vol. for L/S	Ratio L/S	Vwet	Vex 1000	Total Volume	Wt. for Extraction 100 g	Sample pH	Adjusted pH	Volume 500
S10067A	5.0	0.5	.10	10		1010		9.1	<2	500
S10067B										
S10067C										
S10068A	Sludge					1000		8.6	<2	500
S10068B										
S10068C										
[illegible]	5.0	2.5	.5	50		1050		5.7	<2	500
[illegible]										
[illegible]										
[illegible]	5.0	1.7	.34	34		1034		5.5	<2	500
[illegible]	5.0	1.5	[illegible]	30		1030		6.0	<2	500
[illegible]	5.0	2.3	.46	46		1046		6.4	<2	500
[illegible]	5.0	1.5	.3	30		1030		8.0	<2	500

APPENDIX C

SWLP ACTIVITIES DATA SHEETS

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S2000 1A	12:10PM	1/27/82	3:53PM	1/27/82	3:53PM	1/28/82	4:30PM	1/28/82	6:30PM	1/28/82	4:30PM	2/8/82
S2000 1B	12:10PM	1/27/82	3:53PM	1/27/82	3:52PM	1/28/82	8:10AM	1/29/82	11:30AM	1/29/82	4:30PM	2/8/82
S2000 1C	12:10PM	1/27/82	3:53PM	1/27/82	3:53PM	1/28/82	12:15PM	1/29/82	2:00PM	1/29/82	4:30PM	2/8/82
S2000 2A	12:10PM	1/27/82	3:53PM	1/27/82	3:53PM	1/28/82	4:30PM	1/28/82	6:30PM	1/28/82	4:30PM	2/8/82
S2000 2B	12:10PM	1/27/82	3:53PM	1/27/82	3:53PM	1/28/82	8:10AM	1/29/82	11:30AM	1/29/82	4:30PM	2/8/82
S2000 2C	12:10PM	1/27/82	3:53PM	1/27/82	3:53PM	1/28/82	12:15PM	1/29/82	2:15PM	1/29/82	4:30PM	2/8/82
S2000 3A	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	8:00AM	2/1/82	12:00PM	2/1/82	4:30PM	2/8/82
S2000 3B	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	8:00AM	2/1/82	12:00PM	2/1/82	4:30PM	2/8/82
S2000 3C	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	8:00AM	2/1/82	12:00PM	2/1/82	4:30PM	2/8/82
S2000 4	12:10PM	1/27/82							1:00PM	1/27/82		
S2000 5A	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	1:00PM	2/1/82	4:30PM	2/1/82	4:30PM	2/8/82
S2000 5B	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	1:00PM	2/1/82	4:30PM	2/1/82	4:30PM	2/8/82
S2000 5C	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	1:00PM	2/1/82	4:30PM	2/1/82	4:30PM	2/8/82
S2000 6A	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	8:00AM	2/2/82	1:15PM	2/2/82	4:30PM	2/8/82
S2000 6B	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	8:00AM	2/2/82	1:15PM	2/2/82	4:30PM	2/8/82
S2000 6C	12:10PM	1/27/82	4:05PM	1/28/82	4:15PM	1/29/82	8:00AM	2/2/82	1:15PM	2/2/82	4:30PM	2/8/82

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20007A	12:10 PM	1/27/82	1:05 PM	1/28/82	4:15 PM	1/29/82	8:10 AM	2/2/82	2:15 PM	2/2/82	4:30 PM	2/8/82
S20007B	12:10 PM	1/27/82	1:05 PM	1/28/82	4:15 PM	1/29/82	8:10 AM	2/2/82	2:15 PM	2/2/82	4:30 PM	2/8/82
S20007C	12:10 PM	1/27/82	1:05 PM	1/28/82	4:15 PM	1/29/82	8:10 AM	2/2/82	2:15 PM	2/2/82	4:30 PM	2/8/82
S20005B	12:10 PM	1/27/82							1:00 PM	1/27/82		
S20009A	12:10 PM	1/27/82	1:48 PM	1/29/82	4:00 PM	1/30/82	1:20 PM ~~4:20 PM~~	2/2/82	12:00 PM	2/3/82	4:30 PM	2/8/82
S20009B	12:10 PM	1/27/82	4:48 PM	1/29/82	4:00 PM	1/30/82	1:20 PM	2/2/82	12:00 PM	2/3/82	4:30 PM	2/8/82
S20009C	12:10 PM	1/27/82	4:48 PM	1/29/82	4:00 PM	1/30/82	1:20 PM	2/2/82	12:00 PM	2/3/82	4:30 PM	2/8/82
S20010A	12:10 PM	1/27/82	4:48 PM	1/29/82	4:00 PM	1/30/82	2:20 PM	2/2/82	6:30 PM	2/2/82	4:30 PM	2/8/82
S20010B	12:10 PM	1/27/82	4:48 PM	1/29/82	4:00 PM	1/30/82	2:20 PM	2/2/82	6:30 PM	2/2/82	4:30 PM	2/8/82
S20010C	12:10 PM	1/27/82	4:48 PM	1/29/82	4:00 PM	1/30/82	2:20 PM	2/2/82	6:30 PM	2/2/82	4:30 PM	2/8/82
S20011A	12:10 PM	1/27/82	4:48 PM	1/29/82	9:15 AM	1/31/82	9:00 AM	2/3/82	1:00 PM	2/3/82	4:30 PM	2/8/82
S20011B	12:10 PM	1/27/82	4:48 PM	1/29/82	9:15 AM	1/31/82	9:00 AM	2/3/82	1:00 PM	2/3/82	4:30 PM	2/8/82
S20011C	12:10 PM	1/27/82	4:48 PM	1/29/82	9:15 AM	1/31/82	9:00 AM	2/3/82	1:00 PM	2/3/82	4:30 PM	2/8/82
S20012A	11:30 AM	1/28/82	4:48 PM	1/29/82	9:15 AM	1/31/82	1:00 PM	2/3/82	2:00 PM	2/3/82	4:30 PM	2/8/82
S20012B	11:30 AM	1/28/82	4:48 PM	1/29/82	9:15 AM	1/31/82	1:00 PM	2/3/82	2:00 PM	2/3/82	4:30 PM	2/8/82
S20012C	11:30 AM	1/28/82	4:48 PM	1/29/82	9:15 AM	1/31/82	1:00 PM	2/3/82	2:00 PM	2/3/82	4:30 PM	2/8/82

* instrument broke down, extraction had to be stopped

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20013A	11:30 AM	1/25/82	4:15 PM	1/30/82	4:20 PM	1/31/82	1:00 PM	2/3/82	3:30 PM	2/3/82	4:30 PM	2/5/82
S20013B	11:30 AM	1/28/82	4:15 PM	1/30/82	4:20 PM	1/31/82	1:00 PM	2/3/82	3:30 PM	2/3/82	4:30 PM	2/5/82
S20013C	11:30 AM	1/28/82	4:15 PM	1/30/82	4:20 PM	1/31/82	1:00 PM	2/3/82	3:30 PM	2/3/82	4:30 PM	2/5/82
S20014	11:30 AM	1/28/82							12:30 PM	1/28/82		
~~[illegible]~~	~~[illegible]~~	~~[illegible]~~										
~~[illegible]~~	~~[illegible]~~	~~[illegible]~~										
S20015A	11:30 AM	1/28/82							12:30 PM	1/28/82		
S20016A	11:30 AM	1/28/82	4:15 PM	1/30/82	4:20 PM	1/31/82	8:15 AM	2/4/82	10:00 AM 2/8/82	2/8/82	3:00 PM	2/9/82
S20016B	11:30 AM	1/28/82	4:15 PM	1/30/82	4:20 PM	1/31/82	8:15 AM	2/4/82	10 AM 2/8/82	2/8/82	3:00 PM	2/9/82
S20016C	11:30 AM	1/28/82	4:15 PM	1/30/82	4:20 PM	1/31/82	8:15 AM	2/4/82	10 AM 2/8/82	2/8/82	3:00 PM	2/9/82
S20017A	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	3:30 PM	2/3/82	10:15 AM	2/4/82	3:00 PM	2/9/82
S20017B	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	3:30 PM	2/3/82	10:15 AM	2/4/82	3:00 PM	2/9/82
S20017C	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	3:30 PM	2/3/82	10:15 AM	2/4/82	3:00 PM	2/9/82
S20018A	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	3:30 PM	2/3/82	10:00 AM	2/4/82	3:00 PM	2/9/82
S20018B	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	3:30 PM	2/3/82	10:00 AM	2/4/82	3:00 PM	2/9/82
S20018C	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	3:30 PM	2/3/82	10:00 AM	2/4/82	3:00 PM	2/9/82
S2001[illegible]	11:30 AM	1/28/82							12:30 PM	1/28/82		

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20020A	11:30 AM	1/28/82	5:00PM	2/1/82	4:00PM	2/2/82	10:00AM	2/4/82	(1.8) 11:30 AM	2/8/82	3:00 PM	2/9/82
S20020B	11:30 AM	1/28/82	5:00 PM	2/1/82	4:00PM	2/2/82	10:00AM	2/4/82	11:30 AM	2/8/82	3:00 PM	2/9/82
S20020C	11:30 AM	1/28/82	5:00 PM	2/1/82	4:00PM	2/2/82	10:00AM	2/4/82	11:30 AM	2/8/82	3:00 PM	2/9/82
S20021A	11:30 AM	1/28/82	5:22PM	1/31/82	4:35 PM	2/1/82	10:00AM	2/4/82	12:00	2/8/82	3:00 PM	2/9/82
S20021B	11:30 AM	1/28/82	5:22 PM	1/31/82	4:35 PM	2/1/82	10:00AM	2/4/82	12:00	2/8/82	3:00 PM	2/9/82
S20021C	11:30 AM	1/28/82	5:22PM	1/31/82	4:35PM	2/1/82	10:00AM	2/4/82	2:00	2/8/82	3:00 PM	2/9/82
S20022A	11:30 AM	1/28/82	5:22PM	1/31/82	4:35 PM	2/1/82	11:00AM	2/5/82	12:30PM	2/8/82	3:00PM	2/9/82
S20022B	11:30 AM	1/28/82	5:22PM	1/31/82	4:35 PM	2/1/82	11:00AM	2/5/82	12:30PM	2/8/82	3:00PM	2/9/82
S20022C	11:30 AM	1/28/82	5:22PM	1/31/82	4:35 PM	2/1/82	11:00AM	2/5/82	12:30PM	2/8/82	3:00PM	2/9/82
S20023A	11:30 AM	1/28/82							12:30PM	1/28/82		
~~[illegible]~~	~~[illegible]~~	~~[illegible]~~										
~~[illegible]~~	~~[illegible]~~	~~[illegible]~~										
S20023	11:30 AM	1/28/82							12:30PM	1/28/82		
S20024A	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30PM	2/9/82	5:50PM	2/9/82	4:30PM	2/10/82
S20024B	11:45 AM	1/29/82	5:00PM	2/1/82	4:00PM	2/2/82	2:30PM	2/9/82	5:50PM	2/9/82	4:30PM	2/11/82
S20024C	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00PM	2/2/82	2:30PM	2/9/82	5:50PM	2/9/82	4:30PM	2/11/82

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20026[illegible]	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30 PM	2/9/82	9:00 AM	2/10/82	4:30 PM	2/12/82
S200[illegible]	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30 PM	2/9/82	9:00 AM	2/10/82	4:30 PM	2/12/82
S200[illegible]	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30 PM	2/9/82	9:00 AM	2/10/82	4:30 PM	2/12/82
S200 27A	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30 PM	2/9/82	5:30 PM	2/9/82	4:30 PM	2/15/82
S200 27[illegible]	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30 PM	2/9/82	5:30 PM	2/9/82	4:30 PM	2/15/82
S20028	11:45 AM	1/29/82	5:00 PM	2/1/82	4:00 PM	2/2/82	2:30 PM	2/9/82	5:30 PM	2/9/82	4:30 PM	2/15/82
S20028[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	8:30 AM	2/10/82	9:30 AM	2/10/82	1:30 PM	2/15/82
S20028[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	8:30 AM	2/10/82	9:30 AM	2/10/82	1:30 PM	2/15/82
S200 [illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	8:30 AM	2/10/82	9:30 AM	2/10/82	1:30 PM	2/15/82
S20029	11:45 AM	1/29/82							12:30 AM	1/29/82		
S200 30[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	8:30 AM	2/10/82	11:30 AM	2/10/82	1:30 PM	2/15/82
S200 30[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	8:30 AM	2/10/82	11:30 AM	2/10/82	4:30 PM	2/15/82
S200 30[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	8:30 AM	2/10/82	11:30 AM	2/10/82	4:30 PM	2/15/82
S200 31[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	9:30 AM	2/10/82	12:30 PM	2/10/82	4:30 PM	2/15/82
S200 31[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	9:30 AM	2/10/82	12:30 PM	2/10/82	4:30 PM	2/15/82
S200 31[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	9:30 AM	2/10/82	12:30 PM	2/10/82	1:30 PM	2/15/82
S200 32	11:45 AM	1/29/82							12:30 AM	1/29/82		
S200 3[illegible]	11:45 AM	1/29/82							12:30 AM	1/29/82		

Sample No.	Receiving Sample		Extraction Start		Extraction Finish		Filtering		Storage		Shipping	
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	1:30 PM	2/9/82	9:00AM	2/11/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	1:30 PM	2/9/82	9:00AM	2/11/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/2/82	4:00 PM	2/3/82	1:30 PM	2/9/82	9:00AM	2/11/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:00 AM	2/9/82	11:30AM	2/9/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:00 AM	2/9/82	11:30AM	2/9/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:00AM	2/9/82	11:30AM	2/9/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:30 AM	2/9/82	12:00PM	2/9/82	4:30 AM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:30 AM	2/9/82	12:00PM	2/9/82	4:30 PM	2/15/82
200 3[illegible]	11:45 AM	1/29/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:30AM	2/9/82	12:00PM	2/9/82	4:30 PM	2/15/82
200 3[illegible]	9:30 AM	1/30/82							10:00AM	1/30/82		
200 3[illegible]	9:30 AM	1/30/82	5:00 PM	2/3/82	3:55 AM	2/4/82	15:30	2/10/82	9:00AM	2/11/82	4:30 PM	2/15/82
200 3[illegible]	9:30 AM	1/30/82	5:00 PM	2/3/82	3:55 AM	2/4/82	15:30PM	2/10/82	9:00AM	2/11/82	4:30 PM	2/15/82
200 3[illegible]	9:30 AM	1/30/82	5:00PM	2/3/82	3:55 PM	2/4/82	15:30PM	2/10/82	9:00AM	2/11/82	4:30 AM	2/15/82
200 3[illegible]	9:30 AM	1/30/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:30AM	2/10/82	1:00PM	2/10/82	4:30 AM	2/15/82
200 3[illegible]	9:30 AM	1/30/82	5:00 PM	2/3/82	3:55 AM	2/4/82	8:30AM	2/10/82	1:00PM	2/10/82	4:30 AM	2/15/82
200 3[illegible]	9:30 AM	1/30/82	5:00 PM	2/3/82	3:55 PM	2/4/82	8:30AM	2/10/82	1:00PM	2/10/82	4:30 AM	2/15/82

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S200401	9:30 AM	1/30/82	4:30 PM *	2/4/82	8:30 PM *	2/5/82	8:30	2/10/82	1:30 PM	2/10/82	4:30 PM	2/15/82
S200 41	9:30 AM	1/30/82	4:30 PM *	2/4/82	8:30 PM *	2/5/82	8:30	2/10/82	1:30 PM	2/10/82	4:30 PM	2/15/82
S200 41	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	8:30	2/10/82	1:30 PM	2/10/82	4:30 PM	2/15/82
S200 41	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	9:30 AM	2/10/82	11:00 AM	2/10/82	4:30 PM	2/15/82
S200 41	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	9:30 AM	2/10/82	11:00 AM	2/10/82	4:30 PM	2/15/82
S20041	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	9:30 AM	2/10/82	11:00 AM	2/10/82	4:30 PM	2/15/82
S20042	9:30 AM	1/30/82							10 AM	1/30/82		
S20043	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	9:00 AM	2/11/82	2:00 PM	2/11/82	4:30 PM	2/15/82
S20043	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	9:00 AM	2/11/82	2:00 PM	2/11/82	4:30 PM	2/15/82
S20043	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	9:00 AM	2/11/82	2:00 PM	2/11/82	4:30 PM	2/15/82
S20044	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	11:00 PM	2/11/82	9:00 AM	2/12/82	4:30 PM	2/17/82
S20044	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	14:00 PM	2/11/82	9:00 AM	2/12/82	4:30 PM	2/17/82
S20044	9:30 AM	1/30/82	4:30 PM	2/4/82	8:30 PM *	2/5/82	14:00 PM	2/11/82	9:00 AM	2/12/82	4:30 PM	2/17/82
S20045	9:30 AM	1/30/82							10 AM	1/30/82		
~~[illegible]~~	~~[illegible] AM~~	~~[illegible] 82~~										
~~45~~	~~9:30 AM~~	~~[illegible] 82~~										
S200[illegible]	9:30 AM	1/30/82							10 AM	1/30/82		

[illegible] power outa for 5 hrs.

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20047A	9:30 AM	1/30/82	8:30 PM	2/5/82	6:30 PM	2/6/82	11:00 AM	2/9/82	3:00 PM	2/9/82	4:30 PM	2/17/82
S20047B	9:30 AM	1/30/82	8:30 PM	2/5/82	6:30 PM	2/6/82	11:00 AM	2/9/82	3:00 PM	2/9/82	4:30 PM	2/17/82
S20047C	9:30 AM	1/30/82	8:30 PM	2/5/82	6:30 PM	2/6/82	11:00 AM	2/9/82	3:00 PM	2/9/82	4:30 PM	2/17/82
S20048A	9:30 AM	1/30/82	8:30 PM	2/5/82	6:30 PM	2/6/82	10:30 AM	2/9/82	3:20 PM	2/9/82	4:30 AM	2/17/82
S20048B	9:30 AM	1/30/82	8:30 PM	2/5/82	6:30 PM	2/6/82	10:30 AM	2/9/82	3:20 PM	2/9/82	4:30 PM	2/17/82
S20048C	9:30 AM	1/30/82	8:30 PM	2/5/82	6:30 PM	2/6/82	10:30 AM	2/9/82	3:20 PM	2/9/82	4:30 PM	2/17/82
S20049	9:30 AM	1/30/82							10:30 AM	1/30/82		
8 ~~[illegible]~~	~~[illegible]~~	~~[illegible]~~										
~~[illegible]~~	~~[illegible]~~	~~[illegible]~~										
S20050	9:30 AM	1/30/82							10:00 AM	1/30/82		
S20051A	3:30 PM	2/2/82	8:30 PM	2/5/82	6:30 PM	2/6/82	1:30 PM	2/11/82	5:50 PM	2/11/82	4:30 PM	2/17/82
S20051B	3:30 PM	2/2/82	8:30 PM	2/5/82	6:30 PM	2/6/82	1:30 PM	2/11/82	5:50 PM	2/11/82	4:30 AM	2/17/82
S20051C	3:30 PM	2/2/82	8:30 PM	2/5/82	6:30 PM	2/6/82	1:30 PM	2/11/82	5:50 PM	2/11/82	4:30 AM	2/17/82
S20052A	3:30 PM	2/2/82	8:30 PM	2/5/82	6:30 PM	2/6/82	8:30 AM	2/12/82	3:20 PM	2/12/82	4:30 PM	2/17/82
S20052B	3:30 PM	2/2/82	8:30 PM	2/5/82	6:30 PM	2/6/82	8:30 AM	2/12/82	3:20 PM	2/12/82	4:30 PM	2/17/82
S20052C	3:30 PM	2/2/82	8:30 PM	2/5/82	6:30 PM	2/6/82	8:30 AM	2/12/82	3:20 PM	2/12/82	4:30 PM	2/17/82

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20053A	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	9:30 AM	2/12/82	5:15 PM	2/12/82	4:30 PM	2/17/82
S20053B	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	9:30 AM	2/12/82	5:15 PM	2/12/82	4:30 PM	2/17/82
S20053C	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	9:35 AM	2/12/82	5:15 PM	2/12/82	4:30 PM	2/17/82
S20054A	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	2:55 PM	4/12/82	11:00 AM	2/16/82	4:30 PM	2/17/82
S20054B	3:30 PM	2/2/82	6:30 AM	2/6/82	4:30 PM	2/7/82	2:55 PM	2/12/82	11:00 AM	2/16/82	4:30 PM	2/17/82
S20054C	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	2:55 PM	4/12/82	11:00 AM	2/16/82	4:30 PM	2/17/82
S20055A	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	10:10 AM	2/16/82	3:00 PM	2/16/82	4:30 PM	2/17/82
S20055B	3:30 PM	2/2/82	6:30 AM	2/6/82	4:30 PM	2/7/82	10:10 AM	2/10/82	5:00 PM	2/16/82	4:30 PM	2/17/82
S20055C	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 AM	2/7/82	10:10 AM	2/16/82	3:00 PM	2/16/82	4:30 PM	2/17/82
S20056A	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 AM	2/7/82	9:00 AM	2/11/82	12:30 PM	2/11/82	4:30 PM	2/17/82
S20056B	3:30 PM	2/2/82	6:30 PM	2/6/82	4:30 PM	2/7/82	9:00 AM	2/11/82	12:30 PM	2/11/82	4:30 PM	2/17/82
S20056C	3:30 PM	2/2/82	6:30 AM	2/6/82	4:30 PM	2/7/82	9:00 AM	2/11/82	12:30 PM	2/11/82	4:30 PM	2/17/82
S20057A	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	10:30 AM	2/10/82	2:30 PM	2/10/82	4:30 PM	2/17/82
S20057B	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	10:30 AM	2/10/82	2:30 PM	2/10/82	4:30 PM	2/17/82
S20057C	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	10:30 AM	2/10/82	2:30 PM	2/10/82	4:30 PM	2/17/82
S20058A	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	11:00 AM	2/10/82	4:50 PM	2/10/82	4:30 PM	2/17/82
S20058B	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	11:00 AM	2/10/82	4:50 PM	2/10/82	4:30 PM	2/17/82
S20058C	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	11:00 AM	2/10/82	4:50 PM	2/10/82	4:30 PM	2/17/82

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S200 59	3:30 PM	2/2/82							4:00 PM	2/2/82		
S200 60A	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	3:00 PM	2/16/82	4:30 PM	2/16/82	4:30 PM	2/17/82
S200 60B	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	3:00 PM	2/16/82	4:30 PM	2/16/82	4:30 PM	2/17/82
S200 60C	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	3:00 PM	2/16/82	4:30 PM	2/16/82	4:30 PM	2/17/82
S200 61A	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	9:00 AM	2/13/82	10:30 AM	2/13/82	4:30 PM	2/17/82
S200 61B	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	9:00 AM	2/13/82	10:30 AM	2/13/82	4:30 PM	2/17/82
S200 61C	3:30 PM	2/2/82	4:35 PM	2/7/82	4:00 PM	2/8/82	9:00 AM	2/13/82	10:30 AM	2/13/82	4:30 PM	2/17/82
S200 62A	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	11:30 AM	2/13/82	1:30 PM	2/13/82	4:30 PM	2/17/82
S200 62B	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	11:30 AM	2/13/82	1:30 PM	2/13/82	4:30 PM	2/17/82
S200 62C	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	11:30 AM	2/13/82	1:30 PM	2/13/82	4:30 PM	2/17/82
S200 63	3:31 PM	2/2/82	-						4:00 PM	2/2/82		
☆ S200 64A	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	10:00 AM	2/14/82	2:00 PM	2/14/82	4:30 PM	2/17/82
☆ S200 64B	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	10:00 AM	2/14/82	2:00 PM	2/14/82	4:30 PM	2/17/82
☆ S200 64C	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	10:00 AM	2/14/82	2:00 PM	2/14/82	4:30 PM	2/17/82
☆ S200 65A	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	12:00 PM	2/13/82	1:30 PM	2/13/82	4:30 PM	2/17/82
☆ S200 65B	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	12:00 PM	2/13/82	1:30 PM	2/13/82	4:30 PM	2/17/82
☆ S200 65C	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	12:00 PM	2/13/82	1:30 PM	2/13/82	4:30 PM	2/17/82

✓ samples had [illegible]

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S200 66A	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	1:30 PM	2/14/82	3:00 PM	2/14/82	4:30 PM	2/17/82
S200 66B	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	1:30 PM	2/14/82	3:00 PM	2/14/82	4:30 PM	2/17/82
S200 66C	3:30 PM	2/2/82	5:45 PM	2/8/82	4:30 PM	2/9/82	1:30 PM	2/14/82	3:00 PM	2/14/82	4:30 PM	2/17/82
S200 67A	3:30 PM	2/2/82	5:30 PM	2/9/82	5:00 PM	2/10/82	1:30 PM	2/16/82	8:30 AM	2/17/82	4:30 PM	2/17/82
S200 67B	3:30 PM	2/2/82	5:30 PM	2/9/82	5:00 PM	2/10/82	4:30 PM	2/16/82	8:30 AM	2/17/82	4:30 PM	2/17/82
S200 67C	3:30 PM	2/2/82	5:30 PM	2/9/82	5:00 PM	2/10/82	4:30 PM	2/16/82	8:30 AM	2/17/82	4:30 PM	2/17/82
S200 68A	3:30 PM	2/2/82	5:30 PM	2/9/82	5:00 PM	2/10/82	10:30 AM	2/13/82	12:00 PM	2/13/82	4:30 PM	2/17/82
S200 68B	3:30 PM	2/2/82	5:30 PM	2/9/82	5:00 PM	2/10/82	10:30 AM	2/13/82	12:00 PM	2/13/82	4:30 PM	2/17/82
S200 68C	3:30 PM	2/2/82	5:30 PM	2/9/82	5:00 PM	2/10/82	10:30 AM	2/13/82	12:00 PM	2/13/82	4:30 PM	2/17/82

[illegible]

Sample No.	Receiving Sample		Extraction				Filtering		Storage		Shipping	
			Start		Finish							
	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date	Time	Date
S20023A	11:30 AM	1/28/82	10:33 AM	3/6/82	9:30 AM	3/7/82	11:00 AM	3/8/82	12:30 PM	1/28/82	4:00 PM	3/9/82
S20023B	11:30 AM	1/28/82	10:33 AM	3/6/82	9:30 AM	3/7/82	11:00 AM	3/8/82	12:30 PM	1/28/82	4:00 PM	3/9/82
S20023C	11:30 AM	1/28/82	10:33 AM	3/6/82	9:30 AM	3/7/82	11:00 AM	3/8/82	12:30 PM	1/28/82	4:00 PM	3/9/82
S20049A	9:30 AM	1/30/82	10:33 AM	3/6/82	9:30 AM	3/7/82	1:00 PM	3/8/82	10:00 AM	1/30/82	4:00 PM	3/9/82
S20049B	9:30 AM	1/30/82	10:33 AM	3/6/82	9:30 AM	3/7/82	1:00 PM	3/8/82	10:00 AM	1/30/82	4:00 PM	3/9/82
S20049C	9:30 AM	1/30/82	10:33 AM	3/6/82	9:30 AM	3/7/82	1:00 PM	3/8/82	10:00 AM	1/30/82	4:00 PM	3/9/82
S20050A	9:30 AM	1/30/82	10:33 AM	3/6/82	9:30 AM	3/7/82	9:45 AM	3/8/82	10:00 AM	1/30/82	4:00 PM	3/9/82
S20050B	9:30 AM	1/30/82	10:33 AM	3/6/82	9:30 AM	3/7/82	9:45 AM	3/8/82	10:00 AM	1/30/82	4:00 PM	3/9/82
S20050C	9:30 AM	1/30/82	10:33 AM	3/6/82	9:30 AM	3/7/82	9:45 AM	3/8/82	10:00 AM	1/30/82	4:00 PM	3/9/82
S20059A	3:30 PM	2/2/82	10:33 AM	3/6/82	9:30 AM	3/7/82	2:00 PM	3/8/82	4:00 PM	2/2/82	4:00 PM	3/9/82
S20059B	3:30 PM	2/2/82	10:33 AM	3/6/82	9:30 AM	3/7/82	2:00 PM	3/8/82	4:00 PM	2/2/82	4:00 PM	3/9/82
S20059C	3:30 PM	2/2/82	10:33 AM	3/6/82	9:30 AM	3/7/82	2:00 PM	3/8/82	4:00 PM	2/2/82	4:00 PM	3/9/82
S20065A	3:30 PM	2/2/82	9:35 AM	3/7/82	9:35 AM	3/8/82	3:30 PM	3/8/82	4:00 PM	2/2/82	4:00 PM	3/9/82
S20065B	3:30 PM	2/2/82	9:35 AM	3/7/82	9:35 AM	3/8/82	3:30 PM	3/8/82	4:00 PM	2/2/82	4:00 PM	3/9/82
S20065C	3:30 PM	2/2/82	9:35 AM	3/7/82	9:35 AM	3/8/82	3:30 PM	3/8/82	4:10 PM	2/2/82	4:10 PM	3/9/82

APPENDIX E: METHODS FOR BULK ANALYSIS OF BASIN F SAMPLES

METHODS FOR BULK ANALYSIS OF ROCKY MOUNTAIN ARSENAL (RMA) SURFACE SOIL SAMPLES FOR SEMIVOLATILE ORGANICS

I. Introduction

MRI personnel are currently validating a proposed method for the analysis of hazardous waste samples for organic compounds. Some of the samples have included contaminated soil and sediment samples. We believe the method described below will work very well to determine the identity and amounts of semivolatile organic compounds, including the 10 designated compounds, in the surface soil samples from RMA.

II. Analytical Method

A. Scope and Application

1. This method covers the determination of semivolatile organic compounds in nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, tars, mosses, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

2. The method is applicable to the determination of most neutral, acidic, and basic compounds that are soluble in methylene chloride and are capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused silica capillary column coated with a slightly polar silicone. Such compounds include polycyclic aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols including nitrophenols.

3. The detection limit of the method for determining an individual compound is approximately 1 ppm (μg/g, wet weight). For samples which contain more than 1 mg/g of total solvent extractable material, the detection limit is proportionately higher.

4. This method is based upon a solvent extraction, gas chromatographic/mass spectrometric (GC/MS) procedure.

5. This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

B. Summary of the Method

A measured weight of sample, 3.0 g wet weight, is adjusted to pH 7.0 and sonified with 150 ml of methylene chloride. Anhydrous sodium sulfate is added to bind the water present. A portion of the methylene chloride supernatant is concentrated and analyzed by GC/MS using a fused silica capillary column. Qualitative identification is performed using the retention time of the compound and the relative abundance of three or more characteristic ions. Quantitative analysis is performed using an internal standard technique with a single characteristic ion.

C. Safety

The toxicity or carcinogenicity of each sample, reagent, and calibration compound cannot be precisely defined. Thus, each sample and each chemical compound is treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be minimized by whatever means available. All operations involving the use of methylene chloride and the samples will be performed in a hood. Care will be taken to avoid contact of skin with methylene chloride. All work with the sample will be performed in a limited access laboratory. Laboratory coats, safety glasses, and gloves will be worn by all personnel working with the contaminated soil samples.

D. Sample Storage, Preservation, and Handling

The contaminated soil samples will be contained in glass jars having Teflon-lined screw caps. The samples will be refrigerated at 4°C from the time of collection until extraction. All samples will be extracted within 10 days of receipt and completely analyzed within 21 days of receipt.

E. Details of the Analytical Method

1. An aliquot of the sample is first extracted to determine the pH.

   a. Thoroughly mix sample.

   b. Weight 3.0 g (wet weight) into 200-ml centrifuge tube.

   c. Add 15 ml methylene chloride and 15 ml of water.

   d. Sonify mixture for 2 min.

   e. Transfer mixture to 400-ml beaker using 50 ml methylene chloride and 150 ml water as rinses.

   f. Adjust pH of mixture to 7.0 ± 0.2 by titration with 0.4 M H_3PO_4 or 0.4 M K_3PO_4 using a pH meter to measure pH. Record volume of acid on base required.

2. The extraction with methylene chloride is then performed using a fresh portion of the sample.

   a. Weigh 3.0 g (wet weight) of sample into 200-ml centrifuge tube.

   b. Add 15 ml methylene chloride.

   c. Add 1.0 ml of 4 M phosphate buffer.

d. Add an amount of 4 $\underline{M}$ H_3PO_4 or 4 $\underline{M}$ H_3PO_4 equal to one-tenth of the pH 7 acid or base requirement from above.

e. Sonify mixture for 1 min (cool if necessary to maintain 20 to 30°C).

f. Add 135 ml of methylene chloride.

g. Sonify mixture for 1 min.

h. Add an amount of anhydrous sodium sulfate powder equal to 15.0 g plus 3.0 g/ml of the 4 $\underline{M}$ H_3PO_4 or 4 $\underline{M}$ K_3PO_4 used above.

i. Cap centrifuge tube and shake for 1 min.

j. Sonify mixture for 2 min.

k. Obtain clear supernatant by letting sample stand on centrifuging if necessary.

l. Filter a portion (≧ 2 ml) through a 0.2 μ Teflon filter.

3. A portion of the methylene chloride extract is then adjusted to an appropriate volume prior to capillary GC/MS analysis by determining the total solvent extractable constant (TSEC) and screening by capillary GC/FID.

a. Transfer 0.1 ml of the supernatant from above to a tared aluminum weighing dish.

b. Place sample 8 cm from heat lamp and allow solvent to evaporate, and weigh on microbalance.

c. From the residue weight, adjust an appropriate aliquot of sample extract, using Kuderna-Danish concentration, to a final volume such that the TSEC is 1 to 2 mg/ml.

d. Analyze the appropriately concentrated extract by gas chromatography using the following conditions:

Detection: Flame ionization
Column: 30 m x 0.25 mm ID fused silica capillary column
  coated with SE-52 methyl silicone
Column temperature: 30-40°C (4-min hold), then 8°C/min
  to 300°C (10-min hold)
Column flow: 30 cm/sec linear velocity (He)
Injection temperature: 280°C
Detection temperature: 300°C
Injection: Grob-type, splitless
Sample volume: 1 μl

e. Further adjust volume of extract if necessary, prior to GC/MS analysis, so that the average peak height of five largest peaks corresponds to 500 to 100 ng as determined from analysis of an external anthracene calibration standard.

4. The sample is now ready for spiking with internal standards and analysis by GC/MS.

a. To 1.0 ml of the sample extract with the optimum concentration, add a volume of internal standard solution containing 50 µg of the internal standard, D_{10}-anthracene.

b. Determine the concentration relative to the original sample that is represented by the 50 µg of each internal standard in the 1.0 ml aiquot of volume-adjusted extract.

c. Tune and calibrate Finnigan 4000 GC/MS instrument so that EPA-specified tuning criteria are met for DFTPP and so that 50 ng of D_{10} phenanthrene yields about 200,000 counts.

d. Analyze the sample by GC/MS using the same conditions described above for the GC/FID analysis. The initial column temperature should be 30°C. The mass spectrometer should be scanned from 40 to 450 amu with a 0.75 - 1.0 search per scan time.

5. The GC/MS data from the sample is then ready for inspection, interpretation, evaluation, and compilation as described below.

a. The raw data are searched for the target compounds of interest using a computer automated reverse search routine.

b. The quantitation report from the reverse search is inspected to ensure that internal standards were found by the search routine.

c. The amounts of the target compounds found in the sample are corrected to original sample concentrations.

d. The mass spectra of major peaks in the sample, which are not target compounds, are searched against the NBS library.

e. The results of the NBS library search results are examined manually to determine if they provide a reasonable identification for the compound. Additional manual interpretation of the mass spectra is applied when needed.

f. Quantification of non-target compounds is estimated based on the area counts from the total ionization of the mass spectrum compared to the total ionization area counts of the internal standard.

6. The GC/MS data are compiled into a table listing the compounds found and their concentration in the soil samples. If any compounds cannot be identified, the characteristic mass spectral fragmentation ions and the estimated concentration of the compound will be reported.

III. Quality Assurance/Quality Control

The objective of the QA/QC activities associated with this project will be to provide data of known quality. In case the results of the analyses are contested, the information used to generate the data will have been thoroughly documented.

A. QA Objectives

The objectives of the QA/QC activities for this program will be to make certain that the chemical analyses are performed under controlled conditions and that all experimental work is recorded for archival storage.

B. Documentation and Records

The documents for this program will include data reports, letters of transmittal, records of relevant telephone conversations, and all data and all hard-copies data and records associated with effort on the program.

The specific data for which records will be kept will include the following:

- Sample Handling

  Date received
  Condition and appearance of the samples
  Location and temperature of storage
  Date extracted
  Location and temperature of storage of extracts

- Analytical Data

  Date of GC/MS analysis
  All volumes and weights used
  Dilution and concentration factors
  Amounts of internal standards used
  Internal standard area response
  Injection volumes
  Relative response factors used for quantification
  Total solvent extractable content (TSEC)
  Scan number and retention time of GC/MS peaks
  Most intense mass spectral fragmentations
  Compound identification
  Total ion chromatograms
  Library search results
  9-Track tape storage files of all GC/MS data
  Results of analysis of calibration standards
  Mass spectrometer tuning results
  Instrument maintenance records

C. Blanks and Spikes

One method blank and one fortified blank will be generated during analysis of the six contaminated soil samples. The method blank sample will consist of all reagents used in sample preparation and carried through the entire sample preparation process and analyzed by GC/MS. This activity will assess purity of reagents and cleanliness of apparatus and environment. The fortified blank will consist of all reagents used in sample preparation plus the 10 target compounds at a level equivalent to 10 μg/g. This will be carried through the entire sample preparation process and analyzed by GC/MS. This activity will monitor the method recovery for the targeted compounds.

METHODS FOR BULK ANALYSIS OF ROCKY MOUNTAIN ARSENAL (RMA) SURFACE SOIL SAMPLES FOR INORGANICS

I. Introduction

MRI personnel have recently investigated analysis procedures for soil samples, including digestion studies and instrument optimization. The methods described below are expected to be applicable to the soil samples from RMA.

II. Analytical Method

A. Scope and Summary

MRI will apply accepted analytical methods for the analysis of soils and sediments for inorganic constituents. A vigorous nitric acid wet digestion of each sediment sample will be performed to quantitatively remove all Hg, As, F, and other constituents of interest. The acid leachates will first be quantitatively analyzed by inductively coupled plasma (ICP) emission spectrometry for the 28 elements listed on Table 1. Mercury and arsenic will also be determined in the digests by cold vapor and hydride generation atomic absorption spectrophotometry, respectively. Fluoride will be determined by a U.S. Environmental Protection Agency ion selective electrode method.

B. Safety

All samples and extracts will be considered hazardous and will be handled with utmost care. Rigid sample and extract control will be exercised to ensure sample integrity and minimize human exposure. All actual samples and digests will be stored in hoods when not being manipulated in the laboratory.

All pertinent regulations of the MRI Safety and Health Manual and the MRI General Safety Regulations for the Use of Carcinogenic Materials will be followed. In particular, all equipment and containers will be decontaminated as prescribed.

C. Sample Storage, Preservation, Handling

Subsamples for inorganic analysis will be placed in clean plastic bottles fully labelled with the sample name, description, date, and other necessary information. Extract prepared for elemental analysis shall be stored at room temperature in the Atomic Spectroscopy Preparation Lab. Samples for F^- analysis will be stored at 4°C in darkness and will be analyzed as soon as possible after preparation.

TABLE 1

AVAILABLE ANALYTICAL CHANNELS

Element	Wavelength (Å)	Estimated Detection Limit (μg/g sediment
Sn	1899	1.2
Tl	1908	1.6
As	1936	2.0
Hg	1942	1.2
Se	1960	3.0
Mo	2020	0.32
Sb	2068	1.2
Zn	2138	0.16
P	2149	2.4
Pb	2203	1.6
Co	2286	0.28
Cd	2288	0.16
Ni	2316	0.60
Be	2348	0.05
Al	2373	1.0
B	2496	0.20
Mn	2576	0.08
Fe	2599	0.28
Cr	2677	0.28
Fe	2714	2.0
Mg	2795	1.2
Al	3082	1.8
Cu	3247	0.24
Ag	3280	0.28
Ti	3349	0.16
Y	3710	0.08
Ca	3968	0.40
Ba	4934	0.08
Na	5890	1.2
K	7665	12

D. Method Descriptions

1. Sample preparation: The sediments will be wet digested for dissolution of the total amount of metals and fluoride present:

a. Five grams of homogenized sediment and 20 g of concentrated Baker Ultrex® HNO_3 will be placed in 250-ml, acid-cleaned Pyrex® graduated Erlenmeyer flasks.

b. The flasks will be capped with a cleaned glass cap and placed in an oven at 80°C for 2 hr.

c. The tube will mildly agitate for approximately 5 sec every half hour.

d. The flasks will be removed and cooled to room temperature.

e. The samples will be diluted to volume with deionized water.

2. Instrumental analysis

a. ICP emission spectrometry

(1) Instrument description: A 30-channel Jarrell-Ash Model 1155A direct-reading ICP emission spectrometry will be used. This instrument has the following features to enhance sample analysis quality and to be cost-effective:

* Triple point background correction

* Automatic interelement spectral interference correction

* Spectrum scanning for sample matrix diagnostics

* 200 sample autosampler

* Digital Equipment Corp. PDP 11/23 computer with advanced data management capability

Table 1 lists the analyte emission channels and the instrument detection limits for sediments. The detection limits are defined as three times the standard deviation of replicate midrange analyses.

(2) ICP analysis procedure: ICP analysis will follow the U.S. EPA Interim Method 200.7, "Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," November 1980. The analysis quality control features of this procedure are discussed in the next section. The exceptions are that an interference check sample shall be analyzed on an as-needed basis and a 10% nitric acid matrix will be used for calibration standards. Appendix A contains a copy of Interim Method 200.7.

b. Fluoride potentiometry

(1) Instrument description: An Orion Model 601A digital potentiometer will be used for F analysis. An Orion F^- selective solid electrode will be the working electrode and a Ag/AgCl electrode will be the reference.

(2) F potentiometry procedure: Fluoride analysis will follow U.S. EPA Method 340.2. If necessary, the samples will be distilled according to the Rocky Mountain Arsenal procedure.

c. Atomic absorption analysis for As and Hg

(1) Instrument description: A Varian AA6 and Varian AA5 spectrophotometer will be used for As and Hg analysis, respectively. Both instruments have hydrogen-lamp background correction.

(2) As and Hg analysis procedures: Arsenic analysis will follow both Rocky Mountain Arsenal procedures and U.S. EPA Method 206.3. A 1 hr hot HCl incubation at 90°C will be used to reduce any As(V) to As(III) instead of KI and $SnCl_2$.

Mercury analysis will follow both Rocky Mountain Arsenal procedures and U.S. EPA Method 245.1.

III. Quality Assurance Plan

This plan conforms with the overall project QA plan and details actions specific to inorganic analysis of trace metals and fluoride in digestions of contaminated pond sediments.

A. Personnel Responsibilities

Dr. L. Petrie will act as the inorganic analysis task leader for this program. He will:

- Maintain document control of laboratory data, field data, notes, records, etc.
- Verify that each entry is valid by initialing at the bottom of each workbook page.
- Be responsible for chain of custody.
- Immediately report in memo form any problems which arise during the course of the task.
- Enforce instrument calibration and maintenance procedures and schedule.

B. Sample Custody

1. All field samples shall be stored in a locked refrigerator at 4°C. Analysts shall record in a bound logbook the name, date, time, and amount of each sample taken for preparation and analysis.

2. The analyst checking out samples shall complete the appropriate entries on one of the three project data recording sheets (Figures 1-3):

- Field samples - for samples and duplicate subsamples
- Spikes - for samples fortified for analyte recovery study
- Blanks - for method blanks

C. Calibration Procedures and Frequency

1. Each instrument shall be calibrated at the frequency stated in the analytical methods described in Section D. Generally, calibration shall be at least once every time a batch of samples is analyzed.

2. For ICP emission spectrometry, an Instrument Check Standard (ICS) is analyzed after calibration according to Jarrell-Ash instrument operating procedures. If the measured concentration values for the analytes of the ICS are within ± 5% of the correct values, samples can then be analyzed. If not, the instrument must be recalibrated.

3. For atomic absorption analysis, four calibration standards and a reagent blank are analyzed in triplicate. A linear regression of the absorbance values versus standard concentration is performed. If the coefficient of determination (r^2) is ≥ 0.996, the calibration curve is sufficiently linear and sample analysis may begin. Otherwise, instrument calibration must be repeated.

4. For fluoride potentiometric analysis, a six standard calibration curve will be generated before analysis of each sample batch. An Instrument Check Standard (ICS) will be analyzed every hour to assure the instrument is still calibrated.

5. A bound Instrument Log Book (ILB) will be kept for each major laboratory instrument requiring calibration and routine maintenance:

- Mettler Gram-Atic analytical balance
- Varian AA6 spectrophotometer
- Varian AA5 spectrophotometer
- General Electric X-ray diffractometer
- Jarrell-Ash Model 1155A emission spectrometer
- Perkin-Elmer 306 spectrophotometer

6. The ILB shall be kept beside the appropriate instrument.

7. Each ILB will be divided into two sections:

- Calibration
- Maintenance

Field Samples

RMA Sample No. ____________ MRI Sample No. 7278-A ___

Matrix ____________________

Composite Date (Soils Only) __________________

Amount Received __________ Wt/Vol Used for Analysis __________

Analyses (Specify GC/MS, GC/OP, Etc.) ______________________________

Date of Sample Preparation __________________

Date(s) of Analyte Detection __________________

Extraction Solvent (Specify) __________________

Solvent Volume __________

Solvent Aliquot Taken for Cleanup or Final Concentration __________________

Cleanup Fraction or Final Concentrate Volume ______________________________

Aliquot of Final Concentrate Taken for:

Dilution _______________ ml Diluted to _______________ ml

Concentration __________ ml Concentrated to __________ ml

Additional Dilution/Concentration?

Describe _______________ ml to __________ ml

QA Samples Associated with these Analyses: 7278 QB __________ # __________

7278 QS __________ # __________

	Compounds Detected	Calculation Location (Book #. p. #)	Concentration in Original Matrix
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Figure 1

Spikes

QA Sample No. 7278-QS ________________ # ______________________

Specify Analysis — Cons. Nos. for Specified Analyses

Matrix Spiked __________

Matrix Volume __________

	Analytes Spiked	Amount
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Date of Sample Preparation ________________

Date of Analyte Detection ________________

Extraction Solvent (Specify) ________________ **Solvent Volume** __________

Solvent Aliquot Taken for Cleanup or Final Concentration ________________

Cleanup Fraction or Final Concentrate Volume ________________________

Aliquot of Final Concentrate Taken for:

Dilution ____________ **ml Diluted to** ____________ **ml**

Concentration __________ **ml Concentrated to** __________ **ml**

Additional Dilution/Concentration?

Describe ____________ **ml to** __________ **ml**

	Compounds Detected	Calculation Location (Book #. page #)	Concentration Analyte (µg)/Matrix (g or ml)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Figure 2

Blanks

QA Sample No. 7278-QB ____________ # ____________________
Specify Analysis   Cons. Nos. for Specified Analyses

Date of Sample Preparation ____________
Date of Analyte Detection ____________
Extraction Solvent (Specify) ____________
Solvent Volume ________
Solvent Aliquot Taken for Cleanup or Final Concentration ____________
Cleanup Fraction or Final Concentrate Volume ____________________

Aliquot of Final Concentrate Taken for:
Dilution __________ ml Diluted to __________ ml
Concentration ________ ml Concentrated to ________ ml

Additional Dilution/Concentration?
Describe __________ ml to ________ ml

	Compounds Detected	Calculation Location (Book #, page #)	Concentration Analyte (μg)/Matrix (g or ml)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

Figure 3

8. The "Calibration" section will contain a tabular listing of the following entries made in chronological order:

- Date
- Time
- Analyst
- Sample Lab ID
- Analyte
- Calibration curve data (atomic absorption)
- Instrument profiling data (ICP emission spectrometry)

9. The "Maintenance" section will contain a chronological narrative entry of any operation difficulties, repairs, or routine maintenance:

- Date
- Time
- Analyte
- Description of Event
- Corrective Action

D. Data Analysis, Validation and Reporting

1. General policy

a. A record shall be kept of all samples entering the laboratory according to project, sample type, and arrival date.

b. The task leader will be responsible for assuring adherence to this procedure.

2. General data entry

a. All entries of original data or information shall be made with waterproof ink directly into the appropriate permanent record medium.

b. Entries shall be both complete and timely.

c. Calculations and entries of all measured numbers shall be according to the usual significant figure convention.

d. All original data entry shall be placed in an MRI Technical Record Book.

3. Sample preparation

a. A "Sample Preparation Sheet" (Figure 4) will be prepared by the task leader.

b. All samples to be prepared will be batched to include all necessary QC samples.

c. The project data recording sheets shall be updated by the analyst performing the sample preparation.

SAMPLE PREPARATION SHEET

Project No. ____________________ Digestion Code: ____________________

Elements: ____

Analyst: ______________________________________

Date Begun: ___________________ Date Completed: ____________________

Prep Description:

Sample Volume (ml) or Mass/g): ____________________________________
Fortification Levels (Total g): __________________________________
Digest Final Volume (ml): ____

	Sample Code	Comments		Sample Code	Comments
1.			26.		
2.			27.		
3.			28.		
4.			29.		
5.			30.		
6.			31.		
7.			32.		
8.			33.		
9.			34.		
10.			35.		
11.			36.		
12.			37.		
13.			38.		
14.			39.		
15.			40.		
16.			41.		
17.			42.		
18.			43.		
19.			44.		
20.			45.		
21.			46.		
22.			47.		
23.			48.		
24.			49.		
25.			50.		

Figure 4

4. Sample analysis

a. ICP emission spectrometry

(1) An "ICP Data Report Sheet" (Figure 5) shall be completed for each set of sample analyses. This sheet described the important instrument operating conditions and where the generated raw is stored on disk in the computer.

(2) With each sample determination, a terminal printout of the final concentration values also generated will be stored with other raw data records.

(3) Before quantitative analysis of samples is attempted, a check will be made of potential spectral interferences so that they can be compensated by the computer data calculation programs. The emission spectrum is scanned one angstom on either side of analyte emission peaks for a representative sample. The results of the spectrum scan study will be summarized (Figure 6) and placed in the appropriate MRI Technical Record Book.

b. Atomic absorption spectrophotometry: A two-page "AA Data Reporting Sheet" (Figure 7) will be completed for each sample analysis session. This sheet contains the following:

- Instrument operating parameters
- Calibration information
- Detection limit calculations
- Sample absorbance values
- Raw data calculations

c. Fluoride potentiometry: All calibration curve data, measured electrode potentials, and calculated data will be recorded directly into the appropriate MRI Technical Record Book.

d. Project data recording: The three project data recording sheets (Figures 1-3) will be completed by the analyst at the time of sample analysis.

The completed sheets will be given to the project leader and a copy will be retained in the appropriate MRI Technical Record Book.

e. Internal quality control checks: Unless specifically detailed, the following frequency of quality control samples will be carried for As, Hg, and F:

(1) Reagent blank: A minimum of one per 10 samples. For liquid samples, the same volume of deionized water will be used as used for samples. For solid samples, merely add the preparation chemicals to an empty container.

(2) Duplicate sample: A minimum of one per 10 samples.

ICP DATA REPORTING SHEET

Project No.: ____________________ Analyst: ____________________

Sample Matrix: ____________________ Date: ____________________

Elements: ____________________ Digestion Code: ____________________

Instrument Parameters

Forward Power (kw): ________ Coolant Gas Flow (l/min): ________

Reflected Power (w): ________ Plasma Gas Flow (l/min): ________

Observation Height (mm): ____ Sample Gas Flow (l/min): ________

Nebulizer Type: ____________ Solution Uptake (ml/min): ________

(FCF = Fixed crossflow) Peristaltic Pump Used?: ________

(HS = High solids)

Sample Analysis

ACT Name: ____________________

Test Performed: ___ Spectrum Scan

Integration Time (sec): ____________________

Data Files: ____________________

____

____

____

Disk Name: ____________________

___ Quantitation and Log

Command String: ____________________

Data File Name: ____________________

Disk Name: ____________________

___ Quantitation and Store

Command String: ____________________

Data File Name: ____________________

Disk Name: ____________________

Figure 5

SPECTRUM SCAN

Project No:____________________ Date:____________________

Integration Time (sec):__________ Analyst:____________________

LCN	Element	Wavelength (A)	Comments
1	LV	1001	
2	Ag	3280	
3	Al	3082	
4	Al	2373	
5	As	1937	
6	B	2496	
7	Ba	4934	
8	Be	2348	
9	Ca	3968	
10	Cd	2288	
11	Co	2286	
12	Cr	2677	
13	Cu	3247	
14	Fe	2599	
15	Fe	2714	
16	Hg	1942	
17	K	7664	
18	Mg	2795	
19	Mn	2576	
20	Mo	2020	
21	Na	5890	
22	Ni	2316	
23	P	2149	
24	Pb	2203	
25	Sb	2068	
26	Se	1960	
27	Sn	1899	
28	Ti	3349	
29	Tl	1908	
30	Y	3710	
31	Zn	2138	

Figure 6

AA DATA REPORTING SHEET

Project No.: ______________

Element: ______________

Analyst: ______________

Date: ______________

<u>Instrumental Parameters</u>

Wavelength:	______________	Photomultiplier Voltage	______________
Slit:	______________	Matrix modification	______________
Background correction	______________	Atomization mode:	______________
Lamp current:	______________	Instrument model:	______________
Background intensity	______________	Standard range:	______________

<u>Initial Calibration</u>

Standard Concentration	Absorbances	Average Absorbance	RSD (%)
blank			

Coefficient of determination (r^2): ______________

Slope of calibration curve (m): ______________

y-intercept of calibration curve (b): ______________

<u>Detection Limit</u> $= \frac{3 \times S \times C}{A} =$ ______________

Standard deviation of lowest standard (S): ______________

Concentration of lowest standard (C): ______________

Average absorbance of lowest standard (A): ______________

Figure 7

Element:____________; Date:____________; Analyst:____________; Page No.:____________

Sample Analysis

	Sample Code	Absorbances	Avg. Abs.					
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.	A.Q.C. Sample							
11.								
12.								
13.								
14.								
15.								
16.								
17.								
18.								
19.								
20.	A.Q.C. Sample							

Standard Additions

	Sample Code	(Blank + Sample)/ Absorbance	(Standard 1 + Sample)/ Absorbance	(Standard 2 + Sampl Absorbance
1.		0 /	/	/
2.		0 /	/	/

		r^2	m	b	Sample Concentration (b
1.					
2.					

Figure 7 (concluded)

(3) Spiked sample: A minimum of one per 10 samples. The sample should be spiked at a sufficiently high level to cause (1) a 50 to 150% increase in the sample analyte concentration or (2) a measurable analyte concentration three times the LOQ. Unknown samples should be spiked instead of reagent blanks.

(4) Blind QC sample: One per analysis sample set. These samples are prepared by the project QC coordinator.

f. Corrective action: Two types of corrective action formats will be used according to MRI standard operating procedure QA-10. These formats are immediate (on-the-spot) and long-term (closed loop) corrective action.

* Immediate (on-the-spot) corrective action responds quickly to indications of malfunctioning equipment or suspicious data. The QCC and principal investigator will be notified of the problem immediately. They will then take appropriate action and document any changes. The QCC is responsible for and is authorized to halt sampling or analysis if he determines that a serious problem exists.

* Long-term, closed-loop, corrective action is used to prevent the reoccurrence of unanticipated problems. Long-term corrective action steps consist of:

- Definition of the problem
- Investigation to determine the cause
- Determination of the appropriate corrective action
- Implementation of corrective action
- Verification of the effectiveness of the corrective action by followup

2. Quality assurance reports to management: The QAM will, in cooperation with the program manager, identify critical phases of the project which will be subject to inspection. The inspection will include a review of:

Data entry
Data errors, deletions, and corrections
Records and other information
Configuration control
Equipment maintenance and calibration records
Document control

The results of inspections will be reported to management according to MRI standard operating procedure QA-9.

APPENDIX A

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES

Method 200.7

INTERIM

U. S. ENVIRONMENTAL PROTECTION AGENCY
Environmental Monitoring and Support Laboratory
Cincinnati, Ohio 45268

November 1980

Foreword

This method has been prepared by the staff of the Environmental Monitoring and Support Laboratory - Cincinnati, with the cooperation of the EPA-ICP Users Group. Their cooperation and support is gratefully acknowledged.

This method represents the current state-of-the-art, but as time progresses, improvements are anticipated. Users are encouraged to identify problems and assist in updating the method by contacting the Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268.

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES

1. Scope and Application

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interference are taken into account. This is especially true when dissolved solids exceed 1500 mg/l. (See 4.)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps <u>must</u> be taken to correct for potential interference effects. (See 4.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

Figure 2

Table 1 - Recommended Wavelengths(1) and Estimated Instrumental Detection Limits

Element	Wavelength, nm	Estimated detection limit, µg/l(2)
Aluminum	308.215	45
Arsenic	193.696	53
Antimony	206.833	32
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	see(3)
Selenium	196.026	75
Silica (SiO_2)	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.354	40
Vanadium	292.402	8
Zinc	213.856	2

(1) The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 4.1.1).

(2) The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines," EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

(3) Highly dependent on operating conditions and plasma position.

2. Summary of Method

2.1 The method describes ~~a technique for~~ the ~~simultaneous or sequential multielement determination~~ of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 4.1 (and tests for their presence as described in 4.2) should also be recognized and appropriate corrections made.

3. Definitions

3.1 Dissolved -- Those elements which will pass through a 0.45 um membrane filter.

3.2 Suspended -- Those elements which are retained by a 0.45 um membrane filter.

3.3 Total -- The concentration determined on an unfiltered sample following vigorous digestion (Section 8.3), or the sum of the dissolved plus suspended concentrations. (Section 8.1 plus 8.2).

3.4 Total recoverable -- The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (Section 8.4).

3.5 Instrumental detection limit -- The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 Sensitivity -- The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 Instrument check standard -- A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 6.6.1)

3.8 Interference check sample - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors. (See 6.6.2.)

3.9 Quality control sample -- A solution obtained from an outside source having known, concentration values to be used to verify the calibration standards. (See 6.6.3)

3.10 Calibration standards -- a series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 6.4)

3.11 Linear dynamic range -- The concentration range over which the analytical curve remains linear.

3.12 Reagent blank -- A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 6.5.2)

3.13 Calibration blank -- A volume of deionized, distilled water acidified with HNO_3 and HCl. (See 6.5.1)

3.14 Method of standard addition -- The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard. (See 9.6.1.)

4. Interferences

4.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

4.1.1 Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

The interference information, which was collected at the Ames Laboratory[1], is expressed as analyte concentration eqivalents (i.e. false analyte concentrations) arising from 100 mg/l of the interferent element. The suggested use of this information is as follows: Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/l of aluminum. According to Table 2,

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[1] Ames Laboratory, USDOE, Iowa State University, Ames Iowa 50011

Table 2. Analyte Concentration Equivalents (mg/l) Arising From Interferents at the 100 mg/l Level

Analyte	Wavelength, nm	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Boron	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Silicon	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

100 mg/l of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/l. Therefore, 10 mg/l of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/l. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system.

Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed for the interferent concentrations listed in Table 3. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2-5% of the peaks generated by the analyte concentrations also listed in Table 3.

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that second order energy from the magnesium 383.231 nm wavelength interferes with the listed potassium line at 766.491 nm.

4.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aersol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

4.1.3 Chemical Interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

4.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 4.2.1 through 4.2.4, will ensure the analyst that neither positive nor negative interference effects are operative on any of

Table 3. Interferent and Analyte Elemental Concentrations Used for Interference Measurements in Table 2.

Analytes	(mg/l)
Al	10
As	10
B	10
Ba	1
Be	1
Ca	1
Cd	10
Co	1
Cr	1
Cu	1
Fe	1
Mg	1
Mn	1
Mo	10
Na	10
Ni	10
Pb	10
Sb	10
Se	10
Si	1
Tl	10
V	1
Zn	10

Interferents	(mg/l)
Al	1000
Ca	1000
Cr	200
Cu	200
Fe	1000
Mg	1000
Mn	200
Ni	200
Ti	200
V	200

the analyte elements thereby distorting the accuracy of the reported values.

4.2.1 Serial dilution--If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 5 percent of the original determination (or within some acceptable control limit (13.3) that has been established for that matrix.). If not, a chemical or physical interference effect should be suspected.

4.2.2 Spike addition--The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect.

Caution: The standard addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended (See 4.2.3).

4.2.3 Comparison with alternate method of analysis--When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.

4.2.4 Wavelength scanning of analyte line region--If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

5. Apparatus

5.1 Inductively Coupled Plasma-Atomic Emission Spectrometer.

5.1.1 Computer controlled atomic emission spectrometer with background correction.

5.1.2 Radiofrequency generator.

5.1.3 Argon gas supply, welding grade or better.

5.2 Operating conditions -- Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

6. Reagents and standards

6.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

6.1.1 Acetic acid, conc. (sp gr 1.06).

6.1.2 Hydrochloric acid, conc. (sp gr 1.19).

6.1.3 Hydrochloric acid, (1+1): Add 500 ml conc. HCl (sp gr 1.19) to 400 ml deionized, distilled water and dilute to 1 liter.

6.1.4 Nitric acid, conc. (sp gr 1.41).

6.1.5 Nitric acid, (1+1): Add 500 ml conc. HNO_3 (sp. gr 1.41) to 400 ml deionized, distilled water and dilute to 1 liter.

6.2 Deionized, distilled water: Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (13.6).

6.3 Standard stock solutions may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 h at 105°C unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.)

Typical stock solution preparation procedures follow:

6.3.1 Aluminum solution, stock, 1 ml = 100 µg Al: Dissolve 0.100 g of aluminum metal in an acid mixture of 4 ml of (1+1) HCl and 1 ml of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask add an additional 10 ml of (1+1) HCl and dilute to 1,000 ml with deionized, distilled water.

6.3.2 Antimony solution stock, 1 ml = 100 ug Sb: Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 ml (1+1) HCl and dilute to 1000 ml with deionized, distilled water.

6.3.3 Arsenic solution, stock, 1 ml = 100 µg As: Dissolve 0.1320 g of As_2O_3 in 100 ml of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.4 Barium solution, stock, 1 ml = 100 µg Ba: Dissolve 0.1516 g $BaCl_2$ (dried at 250°C for 2 hrs) in 10 ml deionized, distilled water with 1 ml (1+1) HCl. Add 10.0 ml (1+1) HCl and dilute to 1,000 ml with deionized, distilled water.

6.3.5 Beryllium solution, stock, 1 ml = 100 µg Be: Do not dry. Dissolve 1.966 g $BeSO_4 \cdot 4H_2O$, in deionized, distilled water, add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.6 Boron solution, stock, 1 ml = 100 µg B: Do not dry. Dissolve 0.5716 g anhydrous H_3BO_3 in deionized, distilled water and dilute to 1,000 ml. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.

6.3.7 Cadmium solution, stock, 1 ml = 100 µg Cd: Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3. Heat to increase rate of dissolution. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.3 Calcium solution, stock, 1 ml = 100 µg Ca: Suspend 0.2498 g

$CaCO_3$ dried at 180°C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.9 Chromium solution, stock, 1 ml = 100 µg Cr: Dissolve 0.1923 g of CrO_3 in deionized, distilled water. When solution is complete, acidify with 10 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.10 Cobalt solution, stock, 1 ml = 100 µg Co: Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) HNO_3. Add 10.0 ml (1+1) HCl and dilute to 1,000 ml with deionized, distilled water.

6.3.11 Copper solution, stock, 1 ml = 100 µg Cu: Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.12 Iron solution, stock, 1 ml = 100 µg Fe: Dissolve 0.1430 g Fe_2O_3 in 10 ml deionized, distilled water with 1 ml (1+1) HCl. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.13 Lead solution, stock, 1 ml = 100 µg Pb: Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.14 Magnesium solution, stock, 1 ml = 100 µg Mg: Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3. Add 10.0

ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.15 Manganese solution, stock, 1 ml = 100 µg Mn: Dissolve 0.1000 g of manganese metal in the acid mixture 10 ml conc. HCl and 1 ml conc. HNO_3, and dilute to 1,000 ml with deionized, distilled water.

6.3.16 Molybdenum solution, stock, 1 ml = 100 µg Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 ml.

6.3.17 Nickel solution, stock, 1 ml = 100 µg Ni: Dissolve 0.1000 g of nickel metal in 10 ml hot conc. HNO_3, cool and dilute to 1,000 ml with deionized, distilled water.

6.3.18 Potassium solution, stock, 1 ml = 100 µg K: Dissolve 0.1907 g KCl, dried at 110°C, in deionized, distilled water dilute to 1,000 ml.

6.3.19 Selenium solution, stock, 1 ml = 100 µg Se: Do not dry. Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in deionized, distilled water and dilute to 1,000 ml.

6.3.20 Silica solution, stock, 1 ml = 100 µg SiO_2: Do not dry. Dissolve 0.4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.21 Silver solution, stock, 1 ml = 100 µg Ag: Dissolve 0.1575 g $AgNO_3$ in 100 ml of deionized, distilled water and 10 ml conc. HNO_3. Dilute to 1,000 ml with deionized, distilled water.

6.3.22 Sodium solution, stock, 1 ml = 100 µg Na: Dissolve 0.2542 g NaCl in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.23 Thallium solution, stock, 1 ml = 100 µg Tl: Dissolve 0.1303 g $TlNO_3$ in deionized, distilled water. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.24 Vanadium solution, stock, 1 ml = 100 µg V: Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc. HNO_3. Heat to increase rate of dissolution. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.3.25 Zinc solution, stock, 1 ml = 100 µg Zn: Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3. Add 10.0 ml conc. HNO_3 and dilute to 1,000 ml with deionized, distilled water.

6.4 Mixed calibration standard solutions--Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. (See 6.4.1 thru 6.4.5) Add 2 ml of (1+1) HNO_3 and 10 ml of (1+1) HCl and dilute to 100 ml with deionized, distilled water. (See Notes 1 and 6.) Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging. Calibration standards must be initially verified using a quality control sample and monitored weekly for stability (See 6.6.3). Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 1.

6.4.1 Mixed standard solution I--Manganese, beryllium, cadmium, lead, and zinc.

6.4.2 Mixed standard solution II--Barium, copper, iron, vanadium, and cobalt.

6.4.3 Mixed standard solution III--Molybdenum, silica, arsenic, and selenium.

6.4.4 Mixed standard solution IV--Calcium, sodium, postassium, aluminum, chromium and nickel.

6.4.5 Mixed standard solution V--Antimony, boron, magnesium, silver, and thallium.

> NOTE 1: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 ml of deionized distilled water and warm the flask until the solution clears. Cool and dilute to 100 ml with deionized, distilled water. For this acid combination the silver concentration should be limited to 2 mg/l. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl.

6.5 Two types of blanks are required for the analysis. The calibration blank (3.13) is used in establishing the analytical curve while the reagent blank (3.12) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

6.5.1 The calibration blank is prepared by diluting 2 ml of (1+1) HNO_3 and 10 ml of (1+1) HCl to 100 ml with deionized, distilled water. (See Note 6.) Prepare a sufficient quantity to be used to flush the system between standards and samples.

6.5.2 The reagent blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

6.6 In addition to the calibration standards, an instrument check standard (3.7), an interference check sample (3.8) and a quality control sample (3.9) are also required for the analyses.

6.6.1 The instrument check standard is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. (See 11.1.1.)

6.6.2 The interference check sample is prepared by the analyst in the following manner. Select a representative sample which contains minimal concentrations of the analytes of interest but known concentration of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at the approximate concentration of either 100 µg/l or 5 times the estimated detection limits given in Table 1. (For effluent samples of expected high concentrations, spike at an appropriate level.) If the type of samples analyzed are varied, a synthetically prepared sample may be used if the above criteria and intent are met. A limited supply of a synthetic interference check sample will be available from the Quality Assurance Branch of EMSL-Cincinnati. (See 11.1.2).

6.6.3 The quality control sample should be prepared in the same acid matrix as the calibration standards at a concentration near 1 mg/l and in accordance with the instructions provided by the supplier. The Quality Assurance Branch of EMSL-Cincinnati will either supply a quality control sample or information where one of equal quality can be procured. (See 11.1.3.)

7. Sample handling and preservation

7.1 For the determination of trace elements, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether polyethylene, polyproplyene or FEP-fluorocarbon) should be thoroughly washed with detergent and tap water; rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap and finally deionized, distilled water in that order (See Notes 2 and 3).

NOTE 2: Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme. A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid. Chromic acid should not be used with plastic bottles.

NOTE 3: If it can be documented through an active analytical quality control program using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

7.2 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter.

7.2.1 For the determination of dissolved elements the sample must be filtered through a 0.45-um membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Use the first 50-100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH of 2 or less. Normally, 3 ml of (1+1) acid per liter should be sufficient to preserve the sample.

7.2.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-um membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

7.2.3 For the determination of total or total recoverable elements, the sample is acidified with (1+1) HNO_3 to pH 2 or less as soon as possible, preferably at the time of collection. The sample is not filtered before processing.

8. Sample Preparation

8.1 For the determinations of dissolved elements, the filtered, preserved sample may often be analyzed as received. The acid matrix and concentration of the samples and calibration standards must be the same. (See Note 6.) If a precipitate formed upon acidification of the sample or during transit or storage, it must be redissolved before the analysis by adding additional acid and/or by heat as described in 8.3.

8.2 For the determination of suspended elements, transfer the membrane filter containing the insoluble material to a 150-ml Griffin beaker and add 4 ml conc. HNO_3. Cover the beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane. Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 ml of conc. HNO_3. Cover and continue heating until the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (2 ml), cool, add 10 ml HCl (1+1) and 15 ml deionized, distilled water per 100 ml dilution and warm the beaker gently for 15 min. to dissolve any precipitated or residue material. Allow to cool, wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined (See Note 6). The sample is now ready for analysis. Concentrations so determined shall be reported as "suspended."

NOTE 4: In place of filtering, the sample after diluting and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

8.3 For the determination of total elements, choose a measured, volume of the well mixed acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See

Note 5.) Add 3 ml of conc. HNO_3. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil and that no area of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 5 ml portion of conc. HNO_3. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.) Again, evaporate to near dryness and cool the beaker. Add 10 ml of 1+1 HCl and 15 ml of deionized, distilled water per 100 ml of final solution and warm the beaker gently for 15 min. to dissolve any precipitate or residue resulting from evaporation. Allow to cool, wash down the beaker walls and watch glass with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the sample to a predetermined volume based on the expected concentrations of elements present. The sample is now ready for analysis (See Note 6). Concentrations so determined shall be reported as "total."

NOTE 5: If low determinations of boron are critical, quartz glassware should be used.

NOTE 6: If the sample analysis solution has a different acid concentration from that given in 8.4, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

8.4 For the determination of total recoverable elements, choose a measured volume of a well mixed, acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 2 ml of (1+1) HNO_3 and 10 ml of (1+1) HCl to the sample and heat on a steam bath or hot plate until the volume has been reduced to near 25 ml making certain the sample does not boil. After this treatment, cool the sample and filter to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume to 100 ml and mix. The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

9. Procedure

9.1 Set up instrument with proper operating parameters established in Section 5.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 min. of operation prior to calibration.

9.2 Initiate appropriate operating configuration of computer.

9.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 6.4. Flush the system with the calibration blank (6.5.1) between each standard. (See Note 7.) (The use of the average intensity of multiple exposures for both standardization and sample analysis has been found to reduce random error.)

NOTE 7: For boron concentrations greater than 500 ug/l extended flush times of 1 to 2 minutes may be required.

9.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than ± 5 percent (or the established control limits whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

9.5 Begin the sample run flushing the system with the calibration blank solution (6.5.1) between each sample. (See Note 7.) Analyze the instrument check standard (6.6.1) and the calibration blank (6.5.1) each 10 samples.

9.6 If it has been found that methods of standard addition are required, the following procedure is recommended.

9.6.1 The standard addition technique (13.2) involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive interference which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a small volume V_s of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of

A and B are measured and corrected for nonanalyte signals signals. The unknown sample concentration c_x is calculated:

$$c_x = \frac{S_B V_s c_s}{(S_A - S_B) V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

10. Calculation

10.1 Reagent blanks (6.5.2) should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

10.2 If dilutions were performed, the appropriate factor must be applied to sample values.

10.3 Data should be rounded to the thousandth place and all results should be reported in mg/l up to three significant figures.

11. Quality Control (Instrumental)

11.1 Check the instrument standardization by analyzing appropriate quality control check standards as follow:

11.1.1 Analyze an appropriate instrument check standard (6.6.1) containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ± 5% of the expected values or within the established control limits, whichever is lower, the analysis is out of control. The analysis should be terminated, the problem corrected, and the instrument recalibrated.

Analyze the calibration blank (6.5.1) at a frequency of 10%. The result should be within the established control limits of 2 standard deviations of the mean value. If not, repeat the analysis two more times and average the three results. If the average is not within the control limit, terminate the analysis, correct the problem and recalibrate the instrument.

11.1.2 To verify interelement and background correction factors analyze the interference check sample (6.6.2) at the beginning, end, and at periodic intervals throughout the sample run. Results should fall within the established control limits of 1.5 times the standard deviation of the mean value. If not, terminate the analysis, correct the problem and recalibrate the instrument.

11.1.3 A quality control sample (6.6.3) obtained from an outside source must first be used for the initial verification of the calibration standards. A fresh dilution of this sample shall be analyzed every week thereafter to monitor their stability. If the results are not within ± 5% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

12. Precision and Accuracy

12.1 In an EPA round robin phase 1 study, seven laboratories applied the ICP technique to acid-distilled water matrices that had been dosed with various metal concentrates. Table 4 lists the true value, the mean reported value and the mean % relative standard deviation.

13. References

13.1 Winge, R.K., V.J. Peterson, and V.A. Fassel, "Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines, EPA-600/4-79-017.

13.2 Winefordner, J.D., "Trace Analysis: Spectroscopic Methods for Elements," Chemical Analysis, Vol. 46, pp. 41-42.

13.3 Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019.

Table 4. ICP Precision and Accuracy Data

	Sample # 1			Sample #2			Sample #3		
Element	True Value μg/l	Mean Reported Value μg/l	Mean Percent RSD	True Value μg/l	Mean Reported Value μg/l	Mean Percent RSD	True Value μg/l	Mean Reported Value ug/l	Mean Percent RSD
Be	750	733	6.2	20	20	9.8	180	176	5.2
Mn	350	345	2.7	15	15	6.7	100	99	3.3
V	750	749	1.8	70	69	2.9	170	169	1.1
As	200	208	7.5	22	19	23	60	63	17
Cr	150	149	3.8	10	10	18	50	50	3.3
Cu	250	235	5.1	11	11	40	70	67	7.9
Fe	600	594	3.0	20	19	15	180	178	6.0
Al	700	696	5.6	60	62	33	160	161	13
Cd	50	48	12	2.5	2.9	16	14	13	16
Co	500	512	10	20	20	4.1	120	108	21
Ni	250	245	5.8	30	28	11	60	55	14
Pb	250	236	16	24	30	32	80	80	14
Zn	200	201	5.6	16	19	45	80	82	9.4
Se	40	32	21.9	6	8.5	42	10	8.5	8.3

Not all elements were analyzed by all laboratories.

13.4 Garbarino, J.R. and Taylor, H.E., "An Inductively-Coupled Plasma Atomic Emission Spectrometric Method for Routine Water Quality Testing," Applied Spectroscopy 33, No. 3(1979).

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

13.6 Annual Book of ASTM Standards, Part 31.

APPENDIX F: ANALYTICAL RESULTS

List of Tables

Table F1

Analytical Results from SWLP Conducted on Samples from Boring No. 01

	Sample Identification				
	Core Subsamples				
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	3.0-4.0 ft	Overburden
pH	6.5*	8.2	8.2	8.7	7.2
Aldrin	0.30	0.61	0.71	0.40	5.07
Dieldrin	0.22	0.013	2.41	0.54	19.5
Endrin	0.40	0.20	2.22	0.91	24.4
Isodrin	0.11	0.005	0.005	0.11	0.41
DIMP	70	90	110	110	30
DMMP	-**	-	-	-	-
Dithiane	-	-	-	-	-
Sulfone	-	-	-	-	710
Sulfoxide	-	-	-	-	-
DBCP	-	-	-	-	0.010
Mercury	-	-	0.12	0.12	0.22
Arsenic	95	110	110	90	110
Fluoride (ppm)	7.0	9.5	12.3	15.2	3.3

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table 52

Analytical Results from SWLP Conducted on Samples from Boring No. 02

	Sample Identification			
	Core Subsamples			
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	3.0-4.0 ft
pH	9.1*	9.1	9.1	9.1
Aldrin	49.7	8.10	1.42	0.30
Dieldrin	59.1	19.5	4.19	0.12
Endrin	76.8	17.0	4.04	0.40
Isodrin	20.1	0.52	0.11	0.005
DIMP	20	30	30	10.8
DMMP	-**	-	-	-
Dithiane	40	-	-	-
Sulfone	1600	630	550	760
Sulfoxide	1070	440	440	760
DBCP	0.60	0.09	0.09	0.17
Mercury	0.58	1.0	1.24	0.52
Arsenic	170	230	160	120
Fluoride (ppm)	6.1	4.8	7.3	4.3

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F3

Analytical Results from SWLP Conducted on Samples from Boring No. 11

Analyte	Sample Identification				
	Core Subsamples				
	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	3.0-4.0 ft	Overburden
pH	5.3*	4.6	6.5	6.4	8.6
Aldrin	0.51	-**	0.[illegible]	-	8.51
Dieldrin	0.12	0.013	0.12	-	23.6
Endrin	0.04	-	0.71	-	39.4
Isodrin	0.005	0.005	0.005	-	0.11
DIMP	30	20	20	7	20
DMMP	-	-	-	-	0.07
Dithiane	-	-	-	-	-
Sulfone	-	-	-	-	1710
Sulfoxide	-	-	-	-	-
DBCP	-	-	0.022	-	0.07
Mercury	0.12	0.12	0.20	0.16	1.20
Arsenic	-	90	-	20	280
Fluoride (ppm)	0.75	0.95	0.71	1.2	14.8

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F4

Analytical Results from SWLP Conducted on Samples from Boring No. 12

	Sample Identification			
	Core Subsamples			
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	3.0-4.0 ft
pH	5.8*	5.4	5.5	6.0
Aldrin	-**	-	-	-
Dieldrin	-	0.12	-	-
Endrin	-	0.61	-	-
Isodrin	-	-	-	-
DIMP	30	10	10	6
DMMP	-	-	-	-
Dithiane	-	-	-	-
Sulfone	120	-	-	-
Sulfoxide	-	-	-	-
DBCP	-	0.013	-	-
Mercury	-	0.14	0.24	-
Arsenic	14	12	20	50
Fluoride (ppm)	0.48	0.54	0.95	1.95

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Analytical Results from SWLP Conducted on Samples from Boring No. 13

	Sample Identification	
	Core Subsamples	
Analyte	0.0-1.0 ft	1.0-2.0 ft
pH	8.4*	8.2
Aldrin	-**	-
Dieldrin	-	-
Endrin	-	-
Isodrin	-	-
DIMP	40	40
DMMP	0.04	0.04
Dithiane	-	-
Sulfone	-	-
Sulfoxide	-	-
DBCP	-	-
Mercury	0.14	0.14
Arsenic	64	51
Fluoride (ppm)	1.7	1.1

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F6

Analytical Results from SWLP Conducted on Samples from Boring No. 14

	Sample Identification			
	Core Subsamples			
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	Overburden
pH	5.6*	5.8	5.6	8.1
Aldrin	-**	-	0.02	1.59
Dieldrin	-	0.10	0.21	3.24
Endrin	-	-	0.07	44.5
Isodrin	-	-	-	3.1
DIMP	20	20	20	120
DMMP	-	-	-	0.39
Dithiane	-	-	-	-
Sulfone	40	-	-	9160
Sulfoxide	-	-	-	1140
DBCP	-	-	-	1.01
Mercury	0.12	-	0.12	2.14
Arsenic	38	38	71	226
Fluoride (ppm)	1.9	2.1	2.0	15.2

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F7

Analytical Results from SWLP Conducted on Samples from Boring No. 15

	Sample Identification		
	Core Subsamples		
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft
pH	5.3*	5.6	5.7
Aldrin	-**	-	-
Dieldrin	0.10	0.10	-
Endrin	0.08	0.07	-
Isodrin	-	-	-
DIMP	40	30	36
DMMP	-	-	-
Dithiane	-	-	-
Sulfone	90	-	-
Sulfoxide	-	-	-
DBCP	-	-	-
Mercury	-	0.12	-
Arsenic	160	170	20
Fluoride (ppm)	0.42	0.40	0.52

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F8

Analytical Results from SWLP Conducted on Samples from Boring No. 21

	Sample Identification		
	Core Subsamples		
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft
pH	6.9*	4.8	4.9
Aldrin	-**	0.01	-
Dieldrin	-	-	-
Endrin	-	-	-
Isodrin	-	-	-
DIMP	60	40	20
DMMP	-	-	-
Dithiane	-	-	-
Sulfone	-	-	-
Sulfoxide	-	-	-
DBCP	-	-	-
Mercury	0.58	-	-
Arsenic	-	14	11
Fluoride (ppm)	0.90	1.52	1.4

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* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F9

Analytical Results from SWLP Conducted on Samples from Boring No. 22

Analyte	Sample Identification		
	Core Subsamples		
	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft
pH	4.9*	4.8	4.4
Aldrin	0.03	-**	-
Dieldrin	0.01	-	-
Endrin	-	-	-
Isodrin	0.08	-	-
DIMP	130	140	150
DMMP	-	-	-
Dithiane	-	-	-
Sulfone	-	-	-
Sulfoxide	-	-	-
DBCP	-	-	-
Mercury	-	-	-
Arsenic	-	-	14
Fluoride (ppm)	1.4	0.855	0.24

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F10

Analytical Results from SWLP Conducted on Samples from Boring No. 23

	Sample Identification		
	Core Subsamples		
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft
pH	4.8*	5.0	5.9
Aldrin	-**	-	-
Dieldrin	-	-	0.01
Endrin	-	-	-
Isodrin	-	-	-
DIMP	60	40	30
DMMP	-	-	-
Dithiane	-	-	-
Sulfone	-	-	-
Sulfoxide	-	-	-
DBCP	-	-	-
Mercury	0.20	-	-
Arsenic	15	29	22
Fluoride (ppm)	0.48	0.64	0.59

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F11

Analytical Results from SWLP Conducted on Samples from Boring No. 31

	Sample Identification			
	Core Subsamples			
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	Overburden
pH	5.7*	5.5	5.6	8.5
Aldrin	-**	0.30	-	28.4
Dieldrin	-	1.26	-	20.2
Endrin	-	2.22	-	17.7
Isodrin	0.10	0.005	0.10	8.19
DIMP	20	20	10	310
DMMP	-	-	-	60
Dithiane	-	-	-	-
Sulfone	-	-	-	3200
Sulfoxide	-	-	-	-
DBCP	-	0.03	-	0.46
Mercury	0.16	0.22	-	0.36
Arsenic	11	25	25	360
Fluoride (ppm)	0.66	1.0	3.9	7.8

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F12

Analytical Results from SWLP Conducted on Samples from Boring No. 32

	Sample Identification		
	Core Subsamples		
Analyte	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft
pH	5.5*	5.8	5.9
Aldrin	0.20	-**	0.10
Dieldrin	0.10	-	0.10
Endrin	0.10	-	0.10
Isodrin	-	0.10	0.10
DIMP	170	150	150
DMMP	-	-	-
Dithiane	-	-	-
Sulfone	100	-	-
Sulfoxide	-	-	-
DBCP	-	0.006	-
Mercury	0.16	0.36	-
Arsenic	14	12	14
Fluoride (ppm)	0.57	0.63	0.41

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

F13

Analytical Results from SWLP Conducted on Samples from Boring No. 33

	Sample Identification	
	Core Subsamples	
Analyte	0.0-1.0 ft	1.0-2.0 ft
pH	5.5*	5.9
Aldrin	0.20	-**
Dieldrin	0.10	-
Endrin	0.20	-
Isodrin	0.10	-
DIMP	20	10
DMMP	-	-
Dithiane	-	-
Sulfone	-	-
Sulfoxide	-	-
DBCP	0.008	-
Mercury	-	0.16
Arsenic	14	28
Fluoride (ppm)	0.67	0.95

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F14

Analytical Results from SWLP Conducted on Samples from Boring No. 50

Analyte	Sample Identification			
	Core Subsamples			
	0.0-1.0 ft	1.0-2.0 ft	2.0-3.0 ft	3.0-4.0 ft
pH	6.2*	5.0	5.2	8.0
Aldrin	0.40	0.30	0.30	-**
Dieldrin	0.43	0.12	0.12	-
Endrin	0.71	0.30	0.50	-
Isodrin	0.11	0.11	0.005	-
DIMP	30	40	20	17
DMMP	-	-	-	-
Dithiane	-	-	-	-
Sulfone	40	-	-	-
Sulfoxide	-	-	-	-
DBCP	-	-	-	-
Mercury	0.12	0.12	0.40	0.38
Arsenic	-	-	-	20
Fluoride (ppm)	0.71	0.41	0.62	0.75

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

F15

Analytical Results from SWLP Conducted on Samples from Boring No. 60

	Sample Identification	
	Core Subsamples	
Analyte	0.0-1.0 ft	1.0-2.0 ft
pH	5.2*	5.6
Aldrin	-**	-
Dieldrin	-	0.10
Endrin	-	0.10
Isodrin	0.10	-
DIMP	20	20
DMMP	-	-
Dithiane	-	-
Sulfone	-	-
Sulfoxide	-	-
DBCP	0.01	-
Mercury	0.54	0.16
Arsenic	-	11
Fluoride (ppm)	0.63	0.41

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F16

Analytical Results from SWLP Conducted on Samples from Boring No. 70

Analyte	Sample Identification Core Subsamples 0.0-1.0 ft	 1.0-2.0 ft	 2.0-3.0 ft	 Overburden
pH	4.7*	5.6	5.5	8.5
Aldrin	-**	0.10	-	0.27
Dieldrin	0.10	-	-	0.10
Endrin	0.70	-	-	0.61
Isodrin	-	0.10	0.10	-
DIMP	40	20	20	40
DMMP	-	-	-	-
Dithiane	-	-	-	-
Sulfone	-	-	-	840
Sulfoxide	-	-	-	-
DBCP	-	-	-	-
Mercury	0.22	-	0.42	0.28
Arsenic	12	11	11	81
Fluoride (ppm)	0.36	0.40	0.43	6.4

---

* All values other than pH are reported as ppb unless otherwise noted.

** Less than detection limit.

Table F17

Bulk Organic Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 01

Tentative Identification	Level ($\mu g/g$)
Diisopropylmethylphosphonate	5
Toluene	0.6
1,1,2-Trichloroethane	2
Tetrachloroethylene	0.2
Unknown (*m/e* 79 base)	1
Xylene	0.1
Xylene	0.1
1,1,2,2-Tetrachloroethane	4
Pentachloroethane	0.3
Acetophenone	0.1
Unknown (*m/e* 79 base)	2
Unknown (*m/e* 79 base)	2
Unknown (*m/e* 79 base)	6
Unknown (*m/e* 79 base)	1
S_6 (molecular sulfur)	1
S_8 (molecular sulfur)	16
Unknown (*m/e* 275 base)	2
4-*tert*-Butyl-2-(*tert*-butylthio)pyridine	0.5

Table F18

Bulk Metal Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 01

Analyte	Concentration (μg/g)
Silver	1.18
Aluminum	8750
Arsenic	<1.8
Boron	7.15
Barium	170
Beryllium	<0.08
Calcium	2130
Cadmium	<0.1
Cobalt	9.29
Chromium	13.1
Copper	<100
Iron	11300
Mercury	0.023
Potassium	2630
Magnesium	3050
Manganese	384
Molybdenum	6.19
Sodium	4250
Nickel	14.3
Phosphorus	579
Lead	27.1
Antimony	45.0
Selenium	<6.2
Tin	<50
Titanium	48.0
Thallium	24.4
Yttrium	16.2
Zinc	49.4
Fluoride	152

Table F19

Bulk Organic Analysis of the Overburden from Boring No. 11

Tentative Identification	Level (μg/g)
Dimethymethylphosphonate	40
Diisopropylmethylphosphonate	-
p-chlorophenylmethylsulfone	250
Chlorophenylmethylsulfone isomer	12
Aldrin	500
Isodrin	80
Dieldrin	530
Endrin	450
Benzene	20
Cyclohexene	130
Dimethyl disulfide	2
1,1,2-trichloroethane	6
Unknown (possibly N-methylacetamide)	80
MW 98 unknown	80
Weak unknown (*m/e* 78 base)	30
2° or 3° amine unknown	20
Unknown (*m/e* 57 base)	30
Alkane	5
Alkane	4
N-nitrosodipropylamine	200
2° or 3° amine unknown	25
N-containing unknown	20
2° or 3° amine unknown	30
Methylcyclopentadiene	2
Methylcyclopentadiene isomer	2
Unknown (*m/e* 79 base)	270
Hexachlorobutadiene	70
Monochlorinated unknown (MW 158)	177
Alkane	3

(Continued)

Table F19 (Concluded)

Tentative Identification	Level (μg/g)
Hexachlorobicycloheptadiene	800
Alkane	8
Unknown	7
Aldrin-type pesticide (?), Weak!	7
Chlorinated unknown (MW 332)	300
Chlorinated unknown	10
Alkane	14
Alkane	8
Tetrachlorobenzene	70
Chlorinated unknown	250
Unknown (<u>m/e</u> 57 base)	20
S_8 (molecular sulfur)	300
Unknown	35
2° or 3° amine unknown	10
2° or 3° amine unknown	13
2° or 3° amine unknown	40
Aldrin-type chlorinated pesticide	180

Table F20

Bulk Metal Analysis of the Overburden from Boring No. 11

Analyte	Concentration (μg/g)
Silver	0.561
Aluminum	6830
Arsenic	<1.8
Boron	6.48
Barium	94.6
Beryllium	<0.08
Calcium	6110
Cadmium	0.55
Cobalt	5.66
Chromium	10.7
Copper	5220
Iron	7660
Mercury	0.057
Potassium	1810
Magnesium	2740
Manganese	189
Molybdenum	5.55
Sodium	23700
Nickel	13.8
Phosphorus	3100
Lead	35.6
Antimony	29.0
Selenium	<6.2
Tin	<50
Titanium	63.6
Thallium	15.0
Yttrium	9.56
Zinc	69.7
Fluoride	494

Table F21

Bulk Organic Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 12

Tentative Identification	Level (μg/g)
p-Chlorophenylmethylsulfone	2.1
Toluene	8
1,1,2-Trichloroethane	1.1
Tetrachloroethylene	0.2
MW 98 unknown	30
Xylene	0.2
Unknown (*m/e* 79 base)	5
1,1,2,2-Tetrachloroethane	4
Pentachloroethane	0.2
MW 98 or 134 unknown	7.9
Unknown	2
S_6 (molecular sulfur)	1

Table F22

Bulk Metal Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 12

Analyte	Concentration (µg/g)
Silver	1.26
Aluminum	7190
Arsenic	<1.8
Boron	6.31
Barium	133
Beryllium	<0.08
Calcium	18700
Cadmium	<0.1
Cobalt	6.34
Chromium	10.5
Copper	<100
Iron	10200
Mercury	0.010
Potassium	1840
Magnesium	3060
Manganese	257
Molybdenum	5.01
Sodium	811
Nickel	9.59
Phosphorus	558
Lead	19.7
Antimony	44.0
Selenium	<6.2
Tin	<50
Titanium	84.5
Thallium	22.9
Yttrium	11.8
Zinc	37.3
Fluoride	95.0

Table F23

Bulk Organic Analysis of the Overburden from Boring No. 12

Tentative Identification	Level (μg/g)
Dieldrin	5.4
p-Chlorobenzene methyl sulfoxide	3.6
p-Chlorobenzene methyl sulfone	32
p-Chlorobenzene methyl sulfoxide isomer	1
Aldrin	1.4
Toluene	14
1,1,2-Trichloroethane	1
MW 98 unknown	3
Unknown	7
Xylene	0.3
N,N-dimethylactamide	2
Unknown	5
N-nitrosodipropylamine	20
MW 127 unknown	4
Unknown 2° or 3° amine	24
Weak unknown	1
Unknown (*m/e* 79 base)	8
Unknown	20
MW 138 chlorinated unknown	5
Methyl sulfonyl benzene (very weak)	0.3
Weak unknown	1
S_8 (molecular sulfur)	30
Unknown (275 base pk)	6
4-*tert*-Butyl-2(*tert*-butylthio)pyridine	8
Unknown MW 221 N-containing compound	4
Weak MW 131 unknown	4

Table F24

Bulk Metal Analysis of the Overburden from Boring No. 12

Analyte	Concentration (µg/g)
Silver	1.15
Aluminum	7280
Arsenic	<1.8
Boron	8.08
Barium	115
Beryllium	<0.08
Calcium	4440
Cadmium	<0.1
Cobalt	5.57
Chromium	9.76
Copper	613
Iron	9640
Mercury	0.091
Potassium	1790
Magnesium	2350
Manganese	205
Molybdenum	4.97
Sodium	14100
Nickel	10.4
Phosphorus	1550
Lead	17.4
Antimony	18.0
Selenium	<6.2
Tin	<50
Titanium	84.4
Thallium	18.1
Yttrium	9.90
Zinc	41.6
Fluoride	217

Table F25

Bulk Organic Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 14

Tentative Identification	Level (µg/g)
DMMP	2.6
DIMP	1.5
p-Chlorophenylmethylsufone	0.9
Toluene	4.7
1,1,3-Trichloroethane	3.1
Tetrachloroethylene	0.2
Possibly N-methylacetamide	0.3
MW 98 unknown	3.2
Xylene	0.2
Xylene	0.5
Weak unknown	1.3
Xylene	0.3
1,1,2,2-Tetrachloroethane	11
Pentachloroethane	0.7
Acetophenone	0.5
Unknown (m/e 79 base)	7
S_6 (molecular sulfur)	5
S_8 (molecular sulfur)	20

Table F26

Bulk Metal Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 14

Analyte	Concentration (μg/g)
Silver	1.53
Aluminum	6840
Arsenic	<1.8
Boron	9.82
Barium	120
Beryllium	<0.08
Calcium	9120
Cadmium	<0.1
Cobalt	7.90
Chromium	11.8
Copper	<100
Iron	10900
Mercury	0.015
Potassium	2200
Magnesium	4920
Manganese	294
Molybdenum	5.41
Sodium	896
Nickel	13.6
Phosphorus	606
Lead	22.5
Antimony	100
Selenium	<6.2
Tin	<50
Titanium	94.5
Thallium	28.3
Yttrium	14.5
Zinc	47.5
Fluoride	184

Table F27

Bulk Organic Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 31

Tentative Identification	Level (μg/g)
DMMP	1.9
p-Chlorophenylmethylsulfone	0.6
Toluene	1
1,1,2-Trichloroethane	2
Unknown	3
Xylene	0.2
Unknown (*m/e* 79 base)	1
Xylene	0.1
1,1,2,2-Tetrachloroethane	7
Pentachloroethane	0.3
Acetophenone	0.1
Unknown (*m/e* 79 base)	4
Weak unknown (contains *m/e* 79)	2
S_6 (molecular sulfur)	2
S_8 (molecular sulfur)	6

Table F28

Bulk Metal Analysis of the 0.1-1.0 ft Core Subsample from Boring No. 31

Analyte	Concentration (µg/g)
Silver	1.47
Aluminum	9270
Arsenic	<1.8
Boron	14.6
Barium	177
Beryllium	<0.08
Calcium	18900
Cadmium	<0.1
Cobalt	8.81
Chromium	14.2
Copper	<100
Iron	12700
Mercury	0.030
Potassium	2680
Magnesium	5050
Manganese	329
Molybdenum	6.85
Sodium	655
Nickel	15.0
Phosphorus	562
Lead	23.6
Antimony	117
Selenium	<6.2
Tin	<50
Titanium	75.1
Thallium	35.2
Yttrium	14.9
Zinc	49.9
Fluoride	224

Table F29

Bulk Organic Analysis of the Overburden from Boring No. 31

Tentative Identification	Level (μg/g)
Aldrin	3,100
Isodrin	200
p-Chlorophenylmethylsulfone	70
Toluene	30
Hexachlorobutadien	220
Monochlorinated unknown (MW 158)	100
Hexachlorobicycloheptadiene	1,700
Chlorinated unknown	500
Tetrachlorobenzene	30
S_8 (molecular sulfur)	130
Unknown (<u>m/e</u> 275 base)	30
Dieldrin	550
Chlorinated unknown	30
Aldrin-type chlorinated pesticide	200

Table F30

Bulk Metal Analysis of the Overburden from Boring No. 31

Analyte	Concentration (µg/g)
Silver	0.65
Aluminum	7460
Arsenic	<1.8
Boron	8.70
Barium	126
Beryllium	<0.08
Calcium	16100
Cadmium	0.21
Cobalt	6.03
Chromium	11.3
Copper	2110
Iron	9190
Mercury	0.031
Potassium	2050
Magnesium	3600
Manganese	219
Molybdenum	5.54
Sodium	32700
Nickel	13.1
Phosphorus	2980
Lead	25.4
Antimony	55.0
Selenium	<6.2
Tin	<50
Titanium	91.6
Thallium	19.1
Yttrium	9.72
Zinc	49.2
Fluoride	336

Table F31

Bulk Organic Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 33

Tentative Identification	Level (μg/g)
p-Chlorobenzene methyl sulfone	0.4
Toluene	6
1,1,2-Trichloroethane	2
Tetrachloroethylene	0.2
MW 98 Unknown	30
Xylene	0.2
Unknown (*m/e* 79 base)	4
Tetrachloroethane	5
Pentachloroethane	0.4
MW 98 or 134 unknown	11
Alkane	0.3

Table F32

Bulk Metal Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 33

Analyte	Concentration (μg/g)
Silver	1.35
Aluminum	11500
Arsenic	<1.8
Boron	8.58
Barium	162
Beryllium	<0.08
Calcium	2990
Cadmium	<0.1
Cobalt	8.71
Chromium	14.8
Copper	<100
Iron	13600
Mercury	0.015
Potassium	2680
Magnesium	3380
Manganese	296
Molybdenum	8.31
Sodium	1030
Nickel	15.1
Phosphorus	432
Lead	24.6
Antimony	55.0
Selenium	<6.2
Tin	<50
Titanium	70.2
Thallium	32.9
Yttrium	15.0
Zinc	49.0
Fluoride	60.8

Table F33

Bulk Organic Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 70

Tentative Identification	Level (μg/g)
Toluene	10
1,1,2-trichloroethane	3.0
Tetrachloroethylene	0.2
MW 98 unknown	30
Xylene	0.3
Unknown (m/e 79 base)	9
1,1,2,2-Tetrachloroethane	13
Pentachloroethane	0.8
MW 98 or 134 unknown	30
Unknown	0.7
Alkane	2

Table F34

Bulk Metal Analysis of the 0.0-1.0 ft Core Subsample from Boring No. 70

Analyte	Concentration (μg/g)
Silver	1.23
Aluminum	2590
Arsenic	<1.8
Boron	9.66
Barium	53.6
Beryllium	<0.08
Calcium	1300
Cadmium	<0.1
Cobalt	3.34
Chromium	4.49
Copper	<100
Iron	4040
Mercury	0.013
Potassium	885
Magnesium	1050
Manganese	123
Molybdenum	1.93
Sodium	124
Nickel	4.80
Phosphorus	230
Lead	12.4
Antimony	65.0
Selenium	<6.2
Tin	<50
Titanium	45.4
Thallium	3.77
Yttrium	5.63
Zinc	18.3
Fluoride	60.8