



HAZARDOUS  
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DIVISION

**Remedial  
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(REM/FIT)**

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Ecology &  
Environment

FINAL  
WASTE SOIL TREATMENT STUDY  
CELTOR CHEMICAL WORKS SITE  
HOOPA, CALIFORNIA  
WA 121.9L280  
W69628.00  
SEPTEMBER 1986

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RD/R16/031

## EXECUTIVE SUMMARY

The Celtor Chemical Works site is located within the Hoopa Valley Indian Reservation in northwestern California. The plantsite was leased from the U.S. Bureau of Indian Affairs (BIA), acting as a representative for the Hoopa Valley Indian Tribe. The site covers approximately 2.5 acres and is located at the base of a steep hill and just above a relatively flat pasture. The Trinity River is located about 800 feet west of the site.

The ore processing plant that operated at this location between 1957 and 1962 primarily handled sulfide ore from the nearby Copper Bluff Mine. Ore brought to the plant was processed for copper, zinc, and precious metals. Ore was hauled into the plant, unloaded at the upper (eastern) side, and moved through several processes down to the lower side. The complete process used at the plant is not known, but based on the equipment and structures left on the site, it is thought to have included crushing and vat leaching of the ore.

Tailings were stored in piles situated at the lower south corner of the plantsite. Wastewater containing some tailings was reportedly sluiced down the gully northward to a settling pond on the gravel bar adjacent to the Trinity River.

The plant operations were suspended in 1962, following California Fish and Game citations for fishkills apparently resulting from an uncontrolled discharge from a waste holding pond located next to the Trinity River. It is not known if other factors also prompted this closure.

Following closure, some processing wastes (tailings) remained in the plant area, and apparently contaminated soil migrated into the adjacent pasture. Ponded water with high pH was also present in the plant and pasture areas. Processing vats and ore bins in the plant contained ore and tailings. A drainage ditch leading north from the plant to the Trinity River may have been used as a sluice trench and had apparently collected deposits of tailings.

The present owner of the land on which the site is located is the Hoopa Valley Indian Tribe.

## PREVIOUS INVESTIGATIONS

In July 1981, the site was identified in a California state-wide abandoned industrial waste facility survey. In August

1981, the Indian Health Service submitted to EPA a Notification of Hazardous Waste Site under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA). Soil sampling on the site and laboratory analyses were performed in July 1981 by the California Department of Health Services (DOHS). In February 1982, soil samples were taken and analyzed by the EPA Field Investigation Team.

In April 1982, the site was placed on the California State Priority List, and on December 20, 1982, the site was proposed for inclusion on the National Priorities List. In late 1982, DOHS again took surface soil samples. Analyses of these samples indicated that portions of the site contained soils with concentrations of heavy metals exceeding the state criteria that defined materials as hazardous. Metals identified on the site in concentrations exceeding these limits included arsenic, cadmium, copper, mercury, and zinc. Concentrations of lead in some areas approached, but did not exceed, the limits.

To reduce the threat of human exposure of hazardous substances, EPA decided to expedite removal of all known sources of contamination. A focused feasibility study to evaluate and select initial remedial measures (IRM's) for the Celtor Chemical Works site was completed on August 15, 1983. Results of the focused feasibility study indicated that removing approximately 1,400 cubic yards of contaminated material from the site and adjacent pasture to an acceptable Class I disposal site was the most cost-effective solution. Therefore, EPA and the state selected this removal alternative, and the removal was completed by December 18, 1983.

During the IRM implementation, material similar in appearance to the tailings was identified in other areas of the site that had not been identified in the previous samplings. There also appeared to be contaminated material beneath the tailings pile. Therefore, it was likely that more work would be required to provide a permanent remedy. In October 1984, a remedial investigation (RI) was begun to further define the extent of remaining hazardous materials.

The RI involved an extensive sampling program to characterize the soil, surface water, and groundwater at the site. A total of 181 surface and subsurface soil samples were taken, 3 monitoring wells were drilled and sampled, and 19 surface water samples were taken from throughout the site and the adjacent river.

California Assessment Manual (CAM) TTLC criteria were used to determine if hazardous materials were present. Metals most frequently found to be present in excess of CAM limits

were cadmium, copper, iron, lead, and zinc. Many of the soil samples were found to have pH values in the range of 2 to 4.

U.S. EPA 24-Hour Ambient Water Quality Criteria for Aquatic Life (WQCAL) and Federal Drinking Water Regulations (DWR's) were used to evaluate water quality for the project. Surface water flowing within and leaving the site exceeds the DWR's for cadmium, copper, iron, and zinc, and the WQCAL for cadmium, copper, mercury, silver, and zinc. Water in the lower drainage gully that discharges into the Trinity River (combined upgradient and site runoff) exceeds both DWR's and WQCAL for cadmium, copper, iron, and zinc.

A public health assessment was performed to evaluate the release mechanisms and the potential for human health and environmental exposure risks. Based on this assessment, implementation of the No-Action Alternative (doing nothing to remedy the site conditions) may result in an excessive risk of cancer from exposure to arsenic and cadmium and long-term toxic effects related to copper and lead exposure.

Subsequent to the remedial investigation report, a draft feasibility study report was issued on June 28, 1985. This report presented five alternatives which were to be used by EPA for recommendation for a cost-effective remedial action. In addition to a summary of previously gathered information and a recap of site history, the feasibility study provided a conceptual overview of the five selected alternatives and a summary of the methodology used to develop these alternatives. The five alternatives selected were:

1. No action
2. Capping
3. Removal
4. Encapsulation
5. Treatment

Although treatment (Alternative 5) appeared to be the most costly, it had the advantages of permanently removing the contaminants from access to human contact and possibly preventing the wastes from being taken a great distance to acceptable landfills. Because of these advantages, a program was initiated to study the feasibility of treating the Celtor wastes.

#### TREATMENT STUDY

The draft feasibility study indicated that additional laboratory testing was necessary to define the extraction process to be used for treatment of hazardous soils. Once the treatment process was better defined, a more refined

cost estimate could be developed. The Treatment Alternative provides benefits to the public health and welfare and to the environment that are similar to those provided by the Removal Alternative, and better than those provided by Capping or Encapsulation. It would comply with all state and federal environmental laws. Long-term use of the land for other beneficial uses would be allowed, and no ground-water or surface water monitoring program would be necessary. Maintenance on the site would only be necessary while new vegetation was becoming established.

The purpose of the treatment study was to further define the extraction process needed to remove the hazardous substances to established cleanup levels and develop a cost estimate to implement the treatment alternative.

The treatment study was initiated by collecting bulk soil samples from six locations at the Celter site. Samples from two of these locations were combined and tested because of their similarities, and the remaining four samples were tested individually. Each of the soil samples was characterized to determine its mass fraction and content of arsenic, cadmium, copper, lead, and zinc for the following gradations: No. 10 passing, No. 10 to No. 6, No. 6 to No. 3, No. 3 to 1/2-inch, 1/2-inch to 1-inch, and 1-inch plus.

Extraction testing was performed on the No. 10-passing portion and on the No. 10 to 1-inch portion of each soil sample (five samples total including the one combined sample). No extractions were attempted for the 1-inch-plus portions of the soil sample.

Five solutions were used to perform the extraction tests:

1. Sodium gluconate--10 percent by weight aqueous solution adjusted to a pH of 12 to 13
2. Ethylenediaminetetraacetic acid (EDTA)--13 percent by weight aqueous solution adjusted to a pH of 6 to 7
3. Acetic acid--10 percent by volume aqueous solution
4. Hydrochloric acid (HCl)--Aqueous solution pH of 1.0 (approximately 1/10 molar)--some of the extractions were accomplished with a pH 0.8 solution (see text)
5. Hydrochloric acid/hydrogen peroxide (HCl/H<sub>2</sub>O<sub>2</sub>)--HCl aqueous solution pH of 1.0, H<sub>2</sub>O<sub>2</sub> concentration of approximately 0.05 molar (H<sub>2</sub>O<sub>2</sub> added to the pH 1.0 solution)



Except in a few cases, none of the extractants were able to reduce the concentration of metals in the soil to below cleanup levels if the soil was initially above the cleanup level. In no case did an extractant reduce the concentration of more than one metal to below cleanup levels. The most likely reason that extraction efficiencies were poor is that a large portion of the metals in the soil are bound up as insoluble sulfides which require severe conditions (high temperature oxidation) for mobilization and subsequent removal of metals.

One goal of this study was to perform a cost estimate of the full-scale extraction process. Because of the lack of extraction success, it was decided that a cost estimate would not be meaningful, and it was therefore not performed.

RD/R34/030

## Chapter 1 INTRODUCTION

The draft feasibility study report dated June 28, 1985, for the Celtor Chemical Works site presented five remedial action alternatives for the site. These were:

- o Alternative 1--No Action
- o Alternative 2--Capping
- o Alternative 3--Removal
- o Alternative 4--Encapsulation
- o Alternative 5--Treatment

The draft feasibility study report stated "The Treatment Alternative is the most expensive to implement, costing an estimated \$5.8 million. Additional laboratory testing is necessary to define the acid extraction process to be used for treatment of hazardous soils. Once the treatment process is better defined, a more refined cost estimate can be developed. The Treatment Alternative provides benefits to the public health and welfare and to the environment that are similar to those provided by the Removal Alternative, and better than those provided by Capping or Encapsulation. It would comply with all state and federal environmental laws. Long-term use of the land for other beneficial uses would be allowed, and no groundwater or surface water monitoring program would be necessary. Maintenance on the site would only be necessary while new vegetation was becoming established."

Although the above quote refers specifically to an acid extraction process, the scope of the bench-scale extraction study was not limited to low pH extractants, as will be described in the following sections.

### CONCEPTUAL TREATMENT PROCESS AND OBJECTIVES OF STUDY

The conceptual treatment process that was developed during the preparation of the feasibility study is shown in Figure 1-1. This process is consistent with the technology that is in the process of development and field verification by the EPA Office of Research and Development (ORD). The basic concept is to remove the metals of concern (arsenic, cadmium, copper, lead, and zinc) from the contaminated soil by dissolving the metals into a leaching (extractant) solution. The metals would then be removed from the leaching solution by precipitating them as insoluble salts such as sulfides, hydroxides, etc., thus producing a metal sludge. The metal sludge would be dewatered and hauled to a landfill. The soil remaining from the extraction process would be washed and then used as backfill at the site.

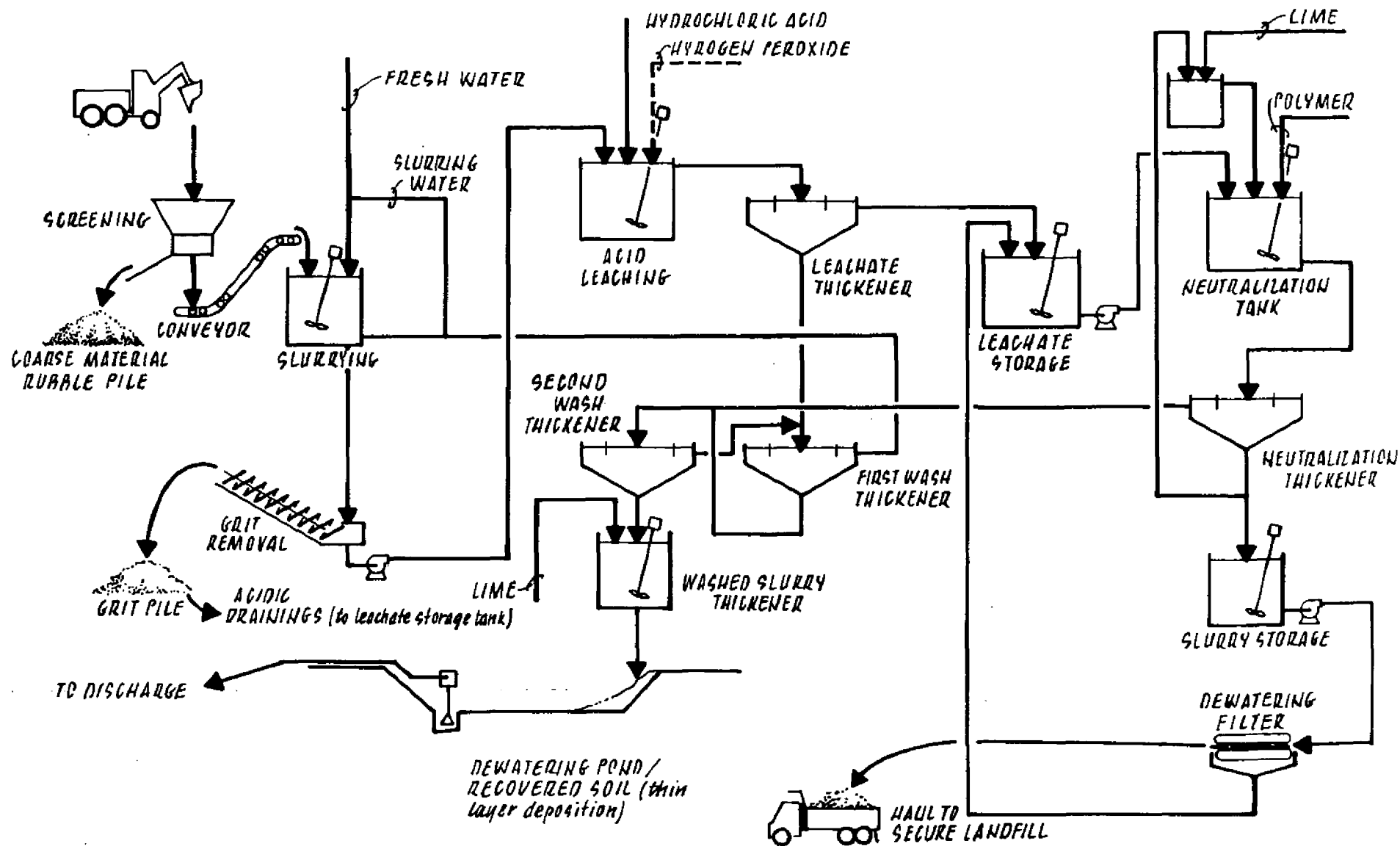


Figure 1-1

# PROCESS SCHEMATIC FOR ON-SITE TREATMENT OF CONTAMINATED CELTOR CHEMICAL WORKS SOIL

The objective of the treatment process is to reduce the concentration of metals in the remaining soils to below the following site-specific cleanup levels for the Celtor site, as described in the final feasibility study (issued January 1986):

Arsenic	100 mg/kg
Cadmium	25 mg/kg
Copper	2,500 mg/kg
Lead	500 mg/kg
Zinc	5,000 mg/kg

The objective of the laboratory study was to obtain data for the cost estimate of a leaching (extraction) facility for the Celtor Chemical Works wastes (raw ore, tailings, and soils).

Questions to be answered by the laboratory study included:

1. Which of the Celtor wastes are suited for leaching?
2. What particle sizes of the above wastes are best suited for leaching, and which are best suited for direct disposal as solids?
3. Which chemicals are best suited to the leaching processes?
4. What are the optimum processing methods? What are the best processing techniques that would be applicable to the wastes, such as type of reactor (batch, flow, leaching heap), residence time, pH, mixing energy, chemical dosage, etc.?
5. What are the capital and maintenance costs of the process?

#### SCOPE OF WORK

The scope of work to accomplish the above objectives consisted of six major items, as follows:

1. A literature review and consultation with EPA and EPA's consultants Science Applications International Corporation (SAIC), who are working on a similar project, to determine which chemicals seemed to be best suited for the proposed leaching studies.

2. Soil sample collection at the Celtor site and soil characterization at the CH2M HILL laboratory.
3. Performance of bench-scale studies to identify which of the chemicals selected in Step 1 are effective extractants, and which soils are best suited to extraction by these chemicals.
4. Further bench-scale studies to optimize chemical dosages and contact times with the most effective chemicals as determined in Step 3.
5. In consultation with EPA and SAIC, development of a process train for full-scale operation and bench-scale simulation of this train to obtain data for a cost estimate of the process.
6. Determine if the EPA mobile soil flushing unit could potentially be used for onsite soil extraction.

RD/R16/026

Chapter 2  
PERFORMANCE OF EXTRACTION STUDY

MECHANICAL SOIL CHARACTERIZATION

Soil samples were obtained at the Celtor site and transported to the Redding laboratory of CH2M HILL for characterization and extraction. The samples were taken from the locations shown in Figure 2-1 and are described in Table 2-1. ("Soil" will be used to mean rock, tailings, and soil hereinafter.)

Table 2-1  
SAMPLE IDENTIFICATION

<u>Sample Number</u>	<u>Adjacent Borehole Number</u>	<u>Previous Sample Numbers (see RI/FS)</u>	<u>Depth (feet)</u>	<u>Soil Description</u>
1	B-6	108, 109	0-2.5	Mixed silt, sand, and gravel, dark gray-brown, moist, odor
2	B-24	001, 002	0-2.5	Sandy gravel with cobbles, brown, slightly moist, contains ore materials
3	B-22	055, 056 058, 060	0-2.5	Mixed clay, silt, sand, gravel, and cobbles, rust-brown, hard, dry
4	H-20	157, 177	0-4	Fine sand, selected tailing material, some phyllite rock pieces and soil mixed in, gray, dry
5	H-8	146	1-1.5	Sandy clay, yellow-brown-gray, nearly dry, selected layer
6	H-21	168, 171	0-2	Fine sand, selected tailing material, some phyllite rock pieces and soil mixed in, gray, moist

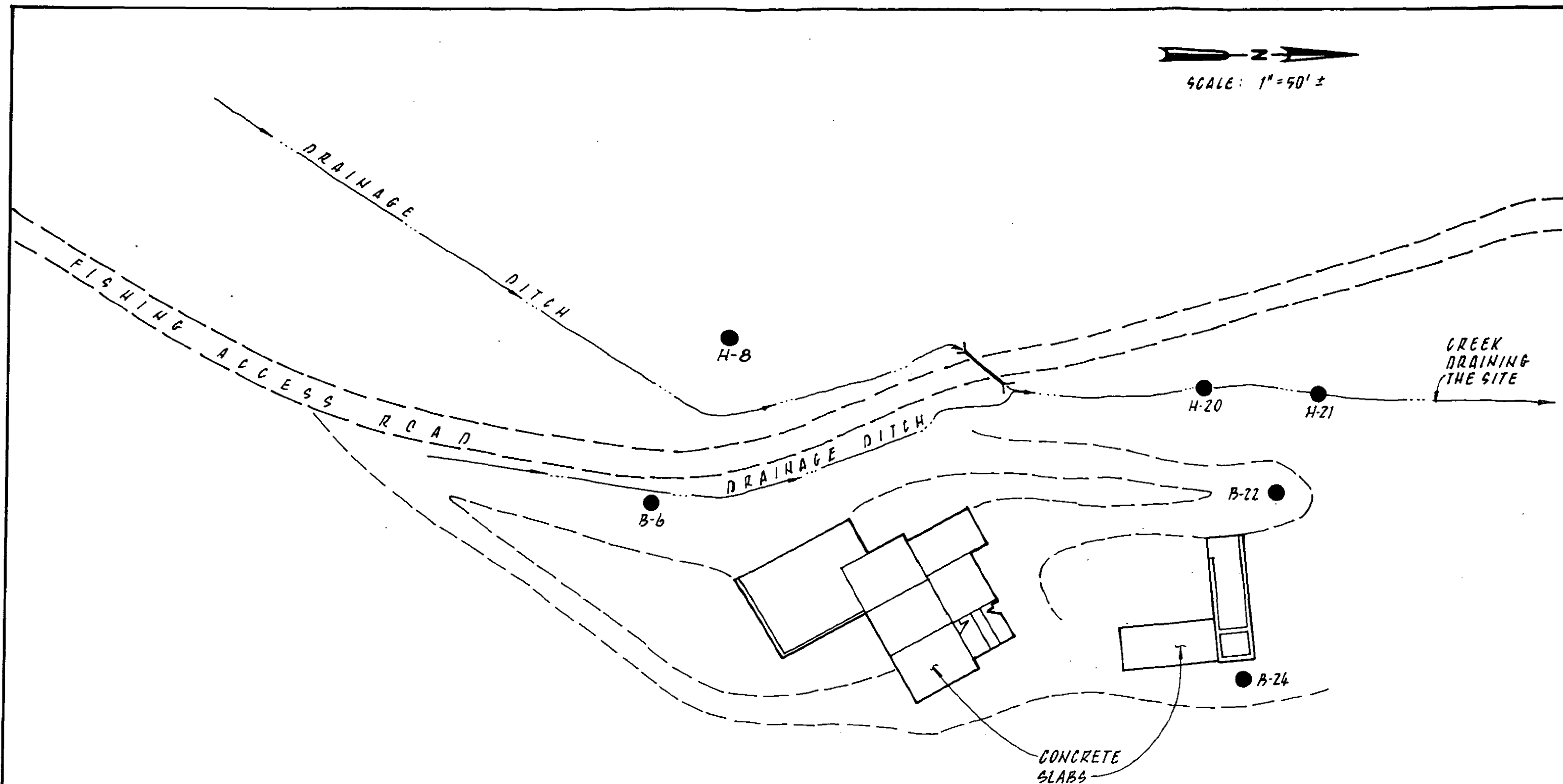


Figure 2-1  
SOIL SAMPLE LOCATIONS FOR  
CELTOR CHEMICAL WORKS  
EXTRACTION STUDIES

Each soil sample was analyzed for mass fraction of the following size gradations:

- o No. 10-passing
- o No. 10 to No. 6
- o No. 6 to No. 3
- o No. 3 to 1/2 inch
- o 1/2 inch to 1 inch
- o 1 inch retained

A complete description of soil characterization methodology is given in Appendix A. Note that Samples 4 and 6 were combined for extraction because of their similar nature. All other samples were to be extracted separately.

#### EXTRACTION METHODOLOGY

From discussions with EPA personnel (Region IX and members of ORD) and SAIC, the following procedure for handling the soil samples was agreed upon:

1. Leaching tests were to be performed on the fraction of each sample passing a No. 10 sieve (2 mm) and the fraction between the No. 10 sieve and 1 inch. These fractions correspond to the fractions treated in the mobile unit mentioned earlier, with the exception that the coarse screen on the mobile unit is 1 inch x 1-1/2 inches, and would therefore pass some solids larger than 1 inch. The total metals content of the fraction larger than 1 inch was to be analyzed, although no extraction studies were to be performed on this portion, because it could not be treated by the mobile unit.
2. Duplicate samples of each soil type, at a minimum, were to be tested for both soil size fractions (No. 10-passing and No. 10-to-inch) and each chemical chosen. Where the results of the duplicate testing were not deemed adequate to provide good confidence in the test data, a third sample was to be analyzed. (See Chapter 3 tables for information on actual number of samples taken.)
3. Soluble Threshold Limit Concentration (STLC) analyses were to be performed on the five soil types, for arsenic, cadmium, copper, lead, and zinc.
4. Analysis of a precipitate found in many locations throughout the site was to be performed. This precipitate, generally white to whitish-yellow, was suspected of having been formed from some of



the tailings onsite. It was therefore desirable to determine its composition. The method chosen for analysis was digestion and determination of aluminum and sulfate and also X-ray diffraction.

#### LEACHING AGENTS

The following chemicals were discussed as being potentially suitable for leaching of the soil samples:

1. Hydroxylamine
2. Sodium gluconate
3. Ethylenediaminetetraacetic acid (EDTA)
4. Hydroxyethylethylenediaminetriacetic acid (HEDTA)
5. Diethylenetriamine pentaacetic acid
6. Sodium pyrophosphate
7. Sodium dihydrophosphate
8. Disodium hydrogen phosphate
9. Sodium dithionite (sodium hydrosulfite)
10. Citric acid
11. Acetic acid
12. Nitric acid
13. Hydrochloric acid
14. Hydrochloric acid with hydrogen peroxide
15. Hypochlorous acid

Of the above, the following were chosen as the most appropriate solutions. (For actual starting and final pH's, see Tables 3-3 through 3-7.)

1. Sodium gluconate--10 percent by weight solution adjusted to a pH of 12 to 13. Experience with this compound indicated that it might be successful in extracting arsenic at high pH, as well as the remaining metals.
2. EDTA--13 percent by weight adjusted to a pH of 6 to 7. EDTA has been used on similar soil extraction studies. Its chelating capacity for metals made it a reasonable candidate for this study.
3. Acetic acid--10 percent by volume. Acetic acid is used in the STLC extraction procedure and was therefore assumed to be a reasonable candidate for extraction of metals.
4. HCl--Solution pH of approximately 0.8 to 1.0 (approximately 0.16 to 0.1 molar). This acid is one of the least expensive acids. It was thought that a very low pH might be ideal for leaching Cd, Cu, and Zn.

5. HCl/H<sub>2</sub>O<sub>2</sub>--HCl solution pH of 0.8 to 1.0, H<sub>2</sub>O<sub>2</sub> concentration approximately 0.16 to 0.10 molar (H<sub>2</sub>O<sub>2</sub> was added to the HCl solution).

Some of the reasons for rejecting the remaining chemicals as potential extractants were as follows:

- o Although hydroxylamine was suggested as a potential extractant, it is a selective extractant for iron and manganese oxides, and was therefore deemed to be of questionable value for the Celter soils.
- o HEDTA and diethylenetriamine pentaacetic acid (Chemicals 4 and 5) are chelating agents, as is EDTA. It was decided that EDTA would provide adequate information to determine if a chelant would be effective for this soil because EDTA has been shown to be an effective agent in some previous studies. It was therefore deemed unnecessary to use other chelants.
- o Chemicals 6, 7, and 8 are effective extractants primarily for organically bound metals. As the presence of substantial amounts of organically bound metals in the Celter soil was unlikely, this extractant was rejected.
- o Sodium dithionite (Chemical 9) is generally useful on iron oxides only and was therefore rejected for the same reasons as hydroxylamine.
- o Citric acid (Chemical 10) is generally only useful for lead extraction and was therefore considered too specific for the purposes of this study.
- o Nitric acid (Chemical 12) was rejected because of high cost (as opposed to HCl) and the concern with potential discharge of nitrogen to the Trinity River during full-scale operations.
- o Hypochlorous acid (Chemical 15) was suggested because of its oxidizing potential. As the combination of HCl/H<sub>2</sub>O<sub>2</sub> was being used for the purpose of obtaining potential oxidation, hypochlorous acid was rejected.
- o For this initial treatment study, only the concentrations of the five metals under consideration were determined. No detailed study of the chemical makeup of the soils was to be performed. The cation exchange capacity was not determined because the relatively high concentrations of extractants used should displace any cations bound

to the soil matrix. Also, most of the samples were sand and gravel size. Little fine-grained soil was present at the site.

#### LEACHING PROCEDURES

For Samples 1, 2, 3, 5, and Combined Samples 4 and 6, duplicate leaching analyses were made for both the No. 10-passing and No. 10-to-1-inch portions with each of the five leaching chemicals. The only exception was Sample 5. Because Sample 5 contained only a small mass percentage of particles larger than No. 10, leaching analyses were not run on this fraction.

For the No. 10-passing portion of each soil type (1, 2, 3, etc.), extractions were performed at ambient temperature with all five extractants. In addition, extractions were performed on pulverized (No. 200-passing) portions of each of Sample 1 and Combined Samples 4 and 6 using EDTA and HCl at ambient temperatures. Lastly, extractions on pulverized and unpulverized samples of Sample 1 and Combined Samples 4 and 6 were performed with hot hydrochloric acid.

For the No. 10-to-1-inch portion of each soil sample (1, 2, 3, etc.), extractions were performed at ambient temperatures with all extractants. No extractions were performed on pulverized samples or with hot hydrochloric acid. See Appendix B for a more detailed description of extraction procedures.

Sequential leaching by different extractants was not planned because it was desired to see if, initially, there was a single extractant that would provide a high degree of removal efficiency. Use of multiple extractants in the field would also be complicated and likely expensive; therefore, no sequential lab testing was planned.

Analyses of liquid samples (extractant) were performed in accordance with EPA "Methods for Chemical Analysis of Water and Wastewater" (EPA 600/4-79-020) and the following methods:

- Arsenic - Method 206.3
- Cadmium - Method 213.1
- Copper - Method 220.0
- Lead - Method 239.1
- Zinc - Method 289.1

Solids digestion and analyses were performed in accordance with EPA's "Test Methods for Evaluation of Solid Waste," Method SW 846.

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Chapter 3  
CHARACTERIZATION AND EXTRACTION RESULTS

SOIL CHARACTERIZATION

Table 3-1 is a detailed summary of soil characterization data for all five soils tested (Samples 1, 2, 3, 5, and Combined Samples 4 and 6). Listed below is an explanation of information contained in the table, by column and row, respectively.

- o % of Total Mass--In this column the mass percentage of the various size gradations of the sample is given. In each case, the percent refers to percent of total dry mass of raw, unsieved, unwashed sample.
- o As (mg/kg), Cd (mg/kg), Cu (mg/kg), Pb (mg/kg), Zn (mg/kg)--These columns give the concentration of metal in the particular gradation of the sample referred to by the row heading.
- o As (% of total), Cd (% of total), Cu (% of total), Pb (% of total), Zn (% of total)--These columns give the amount (as a % of the total content) of the metal contained in the particular gradation referred to in the row heading. The value calculated for the "Total" row is the weighted average of all gradations, excluding the sieved portions of the No. 10-to-1-inch fraction. (The "No. 10-to-1-inch [unsieved]" portion was used for the weighted average calculation instead of the unsieved, as it gave more consistent results with other data, specifically the "analysis of combined sample" data.)
- o >Riffle Sampler--This row presents data for the soil sample portion that did not pass through the riffle sampler (≈1-1/2 inches x 12 inches). Sample 1 was the only sample that contained pieces this large.
- o >1 inch, <Riffle--This row presents data for the sample fraction retained on a 1-inch sieve but passing the riffle sampler.
- o No. 10-to-1-Inch (Unsieved)--This row presents data for the portion passing a 1-inch sieve but retained on a No. 10 (2 mm) sieve. The sample of this size fraction being analyzed had undergone two washings to remove as many fines as possible (see Appendix A).

Table 3-1  
RAW SOIL CHARACTERIZATION\*

	% of Total Mass	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)	As(% of tot)	Cd(% of tot)	Cu(% of tot)	Pb(% of tot)	Zn(% of tot)
<b>SAMPLE 1</b>											
1" Riffle Sampler	3.9	7.1	4.5	103.0	2.4	233.0	0.2	0.4	0.2	0.0	0.1
1" (Riffle)	6.8	15.8	4.8	158.0	10.4	300.0	0.7	0.7	0.4	0.2	0.3
No. 10-to-1" (unsieved)	40.7	36.2	7.8	278.0	30.0	663.0	9.2	6.8	4.3	4.2	3.5
No. 10-to-1" (sieved)											
1"-to-1/2"	9.7	67.1	11.0	6100.0	12.6	1530.0	4.1	2.3	22.3	0.4	1.9
1/2"-to-No. 3	12.1	45.6	10.8	253.0	95.0	1280.0	3.5	2.8	1.2	4.0	2.0
No. 3-to-No. 6	11.6	57.3	11.5	570.0	82.5	1030.0	4.2	2.9	2.5	3.3	1.5
No. 6-to-No. 10	7.1	81.1	14.8	663.0	84.0	1180.0	3.6	2.3	1.8	2.1	1.1
Total	40.7	64.6	12.6	1945.8	74.7	1345.2	16.5	11.1	29.8	10.6	7.1
No. 10-Passing	48.6	295.0	87.5	5200.0	566.0	15300.0	89.9	92.1	95.2	95.5	96.1
Total (excludes sieved 1"-to-No. 10)	100.0	159.5	46.2	2655.1	288.1	7735.1	100.0	100.0	100.0	100.0	100.0
Analysis of combined sample		236.0	46.5	2750.0	215.0	7630.0					
<b>SAMPLE 2</b>											
1" (Riffle)	7.7	175.0	38.0	1860.0	921.0	7600.0	6.8	20.2	4.7	18.0	21.4
No. 10-to-1" (unsieved)	39.9	56.2	13.3	3370.0	113.0	2200.0	11.3	36.5	44.5	11.4	32.1
No. 10-to-1" (sieved)											
1"-to-1/2"	12.6	148.0	34.3	4830.0	276.0	6750.0	9.4	29.8	20.1	8.8	31.1
1/2"-to-No. 3	11.9	86.9	13.8	4780.0	240.0	2280.0	5.2	11.3	18.8	7.2	9.9
No. 3-to-No. 6	9.9	80.1	10.0	3640.0	272.0	1580.0	4.0	6.8	11.9	6.8	5.7
No. 6-to-No. 10	5.5	117.0	7.8	2010.0	207.0	1230.0	3.2	2.9	3.7	2.9	2.5
Total	39.9	108.7	18.5	4131.1	254.8	3373.2	21.8	50.8	54.5	25.7	49.2
No. 10-Passing	52.4	311.0	12.0	2930.0	532.0	2430.0	81.9	43.3	50.8	70.6	46.5
Total (excludes sieved 1"-to-No. 10)	100.0	198.9	14.5	3023.2	394.8	2736.3	100.0	38.8	100.0	100.0	100.0
Analysis of combined sample		124.0	13.3	3290.0	508.0	2340.0					
<b>SAMPLE 3</b>											
1" (Riffle)	9.4	81.9	32.7	7480.0	180.0	7130.0	27.5	42.4	33.4	8.5	43.4
No. 10-to-1" (unsieved)	32.7	9.3	2.1	963.0	147.0	405.0	10.9	9.6	14.9	24.1	8.6
No. 10-to-1" (sieved)											
1"-to-1/2"	8.3	9.4	1.8	788.0	7.9	265.0	2.8	2.0	3.1	0.3	1.4
1/2"-to-No. 3	9.0	7.7	6.0	1820.0	37.3	1050.0	2.5	7.5	7.8	1.7	6.1
No. 3-to-No. 6	9.5	11.1	2.2	1180.0	25.3	505.0	3.8	2.8	5.3	1.2	3.1
No. 6-to-No. 10	5.9	11.7	1.6	715.0	24.5	383.0	2.5	1.3	2.0	0.7	1.5
Total	32.7	9.8	3.0	1172.7	24.0	572.1	11.5	13.6	18.2	3.9	12.1
No. 10-Passing	57.9	29.7	6.0	1880.0	232.0	1280.0	61.5	48.0	51.7	67.4	48.0
Total (excludes sieved 1"-to-No. 10)	100.0	27.9	7.2	2106.5	199.3	1543.8	100.0	100.0	100.0	100.0	100.0
Analysis of combined sample		23.7	3.0	968.0	89.0	668.0					
<b>SAMPLE 4&amp;5</b>											
1" (Riffle)	3.2	14.3	1.4	143.0	55.3	208.0	0.2	0.1	0.1	0.6	0.1
No. 10-to-1" (unsieved)	18.1	63.6	2.1	328.0	38.0	408.0	4.7	0.9	1.4	2.2	0.8
No. 10-to-1" (sieved)											
1"-to-1/2"	5.4	17.4	1.4	158.0	50.8	270.0	0.4	0.2	0.2	0.9	0.2
1/2"-to-No. 3	4.7	4.7	1.8	273.0	34.0	420.0	0.1	0.2	0.3	0.5	0.2
No. 3-to-No. 6	5.0	74.8	2.0	293.0	48.3	435.0	1.5	0.2	0.3	0.8	0.2
No. 6-to-No. 10	3.0	122.0	2.2	410.0	68.8	553.0	1.5	0.2	0.3	0.6	0.2
Total	18.1	47.3	1.8	266.9	48.7	401.4	3.5	0.8	1.2	2.8	0.8
No. 10-Passing	78.7	297.0	52.0	5240.0	394.0	12200.0	95.1	99.0	98.5	97.3	99.2
Total (excludes sieved 1"-to-No. 10)	100.0	245.7	41.3	4187.8	318.7	9681.9	100.0	100.0	100.0	100.0	100.0
Analysis of combined sample		241.0	41.8	4380.0	400.0	9030.0					
<b>SAMPLE 5</b>											
No. 10-to-1" (unsieved)	1.5										
No. 10-Passing	98.5	345.0	12.5	658.0	1350.0	2350.0					
Analysis of combined sample	--	289.0	11.3	585.0	1150.0	1980.0					

\* Cleanup Criteria: Arsenic--100 mg/kg, Cadmium--25 mg/kg, Copper--2,500 mg/kg, Lead--500 mg/kg, Zinc--5,000 mg/kg

- o No. 10-to-1-Inch (Sieved)--The subheadings under this heading present data on various sized gradations within the No. 10-to-1-inch portion. The sample that was sieved for these analyses had been washed only once (see Appendix A). The fines (<No. 10) from this sieving were not analyzed, but their weight was determined and this information used for final calculation of mass fraction of all size gradations.
- o No. 10 and Smaller--This row presents data from analyses of the portion of sample passing the No. 10 (2 mm openings) sieve, including the fines from washings of the No. 10-to-1-inch and 1-inch and larger portions (see Appendix A).
- o Total (Excludes Sieved 1-inch to No. 10)--This row gives the weighted average concentration of each sample as determined from all of the size gradations excluding the sieved No. 10-to-1-inch portion.
- o Analysis of Combined Sample--This row presents the results of analysis of the unsegregated sample which was originally separated for analyses using the riffle sampler.

#### CHARACTERIZATION OF SAMPLE 1

The bottom two rows ("Total" and "Analysis of combined sample") under Sample 1 of Table 3-1, which provide information on soil metals concentrations, compare quite closely (within 5 percent) for all metals except As and Pb, which compare within 50 percent. Due to the difficulty in obtaining representative samples, these results are considered acceptable. As can also be seen, the metals content of the soil is above the cleanup levels in all metals except Pb.

Approximately 90 percent or more of the total metal content is contained in the No. 10-passing portion although the mass fraction of this portion is 48.6 percent. This sample therefore appears to have a substantial amount of fine raw ore or tailings.

Note that the analysis of the sieved fraction of the No. 10-to-1-inch portion shows that of the total metals contained in this mass fraction, the 1/2-inch-to-1-inch portion contains a disproportionately small amount of the Pb content and a disproportionately large amount of the Cu. In all cases the figure shown in the "Total" row for the No. 10-to-1-inch portion is greater than for the analysis of the unsieved No. 10-to-1-inch portion. Ideally, these figures should be the same. Possible reasons for the discrepancy may be:

- o Fines on the sieved fraction, which would be expected to be of higher concentration than the larger size particles, may have affected the analyses.
- o Portions used for analysis may have been unrepresentative of the actual characteristics of the samples.

#### CHARACTERIZATION OF SAMPLE 2

As in Sample 1, the weighted average concentration data for the sieved sample agree fairly well with the analysis of the unsieved sample which was split off for digestion and analysis. For Sample 2, all metals compare within 25 percent except for As and Pb. Sample 2 has metals concentrations below cleanup levels for Cd and Zn, is marginal with respect to Pb, and is above cleanup levels for As and Cu. The No. 10-passing portion contains approximately 50 percent or less of the total Cd, Cu, and Zn, and approximately 82 and 71 percent of the As and Pb, respectively.

The analysis on the sieved fraction of the No. 10-to-1-inch portion showed higher metals concentrations overall than the analysis on the unsieved fraction. Also the 1/2-inch-to-1-inch portion of this fraction contains a disproportionately large amount of Cd, Cu, and Zn.

#### CHARACTERIZATION OF SAMPLE 3

The weighted average total concentrations and analysis of the unsieved sample that was split off for analysis compare rather poorly, except for As. One likely explanation for this anomaly is that one or more samples were unrepresentative of the actual soil contents. In addition, it can be seen that the No. 10-to-1-inch portion contains a small amount of all metals in comparison to its mass percentage, while the 1-inch-passing portion contains a disproportionately large amount of metals for its mass fraction. The soil sample is below cleanup levels for all five metals.

In the sieved No. 10-to-1-inch portion, as was the case for Samples 1 and 2, the weighted average content of all metals is greater than shown by the analysis of the unsieved No. 10-to-1-inch portion, except for As and Pb. The mass percent content of the metals appears to be rather evenly distributed by metal and throughout the various gradations in this fraction.

#### CHARACTERIZATION OF COMBINED SAMPLES 4 AND 6

For this sample, the weighted average concentrations show good agreement with the analyses of the combined sample,

except for Pb, which shows only approximately 25 percent disagreement. As can be seen, more than 95 percent of all metals in this soil sample are contained in the No. 10-passing portion, which comprises almost 79 percent of the total mass fraction of the material. Good agreement was obtained for the analyses of metals on the unsieved No. 10-to-1-inch portion as compared to the weighted average of the sieved material. The soil is above cleanup levels in all metals except Pb.

#### CHARACTERIZATION OF SAMPLE 5

All of Sample 5 passed a 1-inch screen, and 98.5 percent passed the No. 10 screen. After washing, it is unlikely that more than 1 percent of Sample 5 would have been retained on a No. 10 screen. Because of this, the No. 10-to-1-inch portion of Sample 5 was not analyzed. This soil sample is above cleanup levels in As and Pb.

#### CHARACTERIZATION OF WHITE PRECIPITATE

As noted, the white precipitate found at the Celtor site was analyzed by X-ray diffraction to determine its composition. This sample was found to contain approximately 33 percent water soluble material consisting of approximately equal portions of bianchite  $[(Zn, Fe^{+2}) SO_4 \cdot 6H_2O]$  and gunningite  $[(Zn, Mn) SO_4 \cdot H_2O]$ . The insoluble portion consisted of the chlorite rapidolite, a ferroan clinocllore, quartz, and oligoclase feldspars. A trace of amphibole was also present.

Analysis for aluminum and sulfate produced the following results:

<u>Sample</u>	<u>Al (%)</u>	<u>SO<sub>4</sub> (%)</u>
White precipitate	0.9	21.2
Soil containing crust of white precipitate	2.25	17.7

#### STLC ANALYSIS

Results of the soluble threshold limit concentration (STLC) analysis on the five soil samples are presented in Table 3-2. This extraction was performed on the No. 10-passing portion of the soils.

These values show that the following soils had metal concentrations above STLC criteria:



Sample 1--Cd, Pb  
 Sample 2--Cu  
 Sample 3--Cu, Pb  
 Combined Samples 4 and 6--As, Pb

Table 3-2  
 STLC VALUES OF SOIL SAMPLES

<u>Sample</u>	<u>STLC Values</u>				
	<u>As</u> <u>mg/kg</u>	<u>Cd</u> <u>mg/kg</u>	<u>Cu</u> <u>mg/kg</u>	<u>Pb</u> <u>mg/kg</u>	<u>Zn</u> <u>mg/kg</u>
1	3.38	1.27	0.19	8.30	106
2	0.482	0.32	35.6	3.84	74.2
3	2.29	0.12	36.4	8.44	24.2
4 & 6	8.64	0.30	0.37	26.5	11.3
5	3.94	0.51	12.9	1.40	76.8

#### LEACHING DATA

##### EXTRACTION RESULTS

Tables 3-3 through 3-7 present the results of the leaching studies performed on the five soil samples. Each table contains information for one soil sample only. All tables are organized similarly. Following is an explanation of the data contained in these tables.

- o The first section of the table presents a portion of the soil characterization data contained in Table 3-1, for comparison of beginning and final metals concentrations.
- o Column headings:
  - Extractant Solution (After Extraction)--Under this heading are presented the analyses for the five metals of concern in the extractant solution. Note that in some cases hot HCl was used for extraction and that some samples were pulverized before extraction. See the appendixes for more details.

Table 3-3  
 EXTRACTION OF SOIL SAMPLE 1

Extractant	SOIL ANALYSIS SUMMARY																
	Size	% of total	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)										
	No. 10-passing No. 10-to-1"	48.6 40.7	295.0 35.2	87.5 7.8	5200.0 278.0	566.0 30.0	15300.0 663.0										
Extractant	Extractant Solution(after extraction)						EXTRACTION SUMMARY					Mass Balance (Fraction of Starting Amount Recovered)					
	Soil(after extraction)																
	As(mg/l)	Cd(mg/l)	Cu(mg/l)	Pb(mg/l)	Zn(mg/l)	pH(note 1)	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)	As	Cd	Cu	Pb	Zn	
Sodium Gluconate																	
No. 10-passing																	
Sample 1	19.4	3.20	56.0	119	277	12.7	199.0	84.5	5550	259	15800	0.81	1.04	1.09	0.88	1.07	
Sample 2	20.0	3.19	61.0	121	271	12.7	185.0	83.0	5200	260	14900	0.76	1.02	1.02	0.89	1.01	
No. 10-to-1" (note 2)																	
Sample 1	1.87	0.06	9.2	0.38	8.70	12.0	30.2	5.25	250	31.4	445	0.94	0.69	0.97	1.07	0.70	
Sample 2	2.20	0.04	9.0	0.77	10.1	12.1	39.5	6.25	475	25.8	523	1.21	0.82	1.77	0.91	0.82	
EDTA																	
No. 10-passing																	
Sample 1	0.512	12.7	168.0	10.0	792	5.59	197.0	61.0	4980	514	13200	0.67	0.99	1.02	0.94	0.97	
Sample 2	0.756	12.9	183.0	10.4	856	NA	203.0	61.8	5080	498	13400	0.69	1.00	1.05	0.92	0.99	
No. 10-to-1"																	
Sample 1	0.188	1.11	23.5	1.66	85.0	6.14	40.7	3.13	373	25.4	475	1.13	0.69	1.51	0.96	0.97	
Sample 2	0.196	0.86	17.2	1.62	55.0	NA	34.0	2.78	248	29.8	400	0.95	0.58	1.02	1.10	0.77	
No. 10-passing (note 3)																	
Sample 1 (note 4)	NA	2.5	26.7	5.6	140	7.04	NA	54	4980	403	13600	NA	0.90	1.01	0.81	0.98	
Sample 2 (note 4)	NA	2.6	27.6	6.3	144	7.04	NA	62	4980	450	13900	NA	1.01	1.01	0.91	1.00	
Acetic Acid																	
No. 10-passing																	
Sample 1	0.832	12.8	164	0.12	731	2.47	212.0	57.4	4880	454	12400	0.72	0.95	1.00	0.80	0.91	
Sample 2	0.776	11.9	154	0.12	673	2.39	204.0	57.3	4560	413	11900	0.70	0.93	0.94	0.73	0.87	
No. 10-to-1"																	
Sample 1	0.316	0.98	17.7	0.05	59.0	2.47	46.3	2.63	238	22.8	318	1.30	0.59	0.98	0.76	0.66	
Sample 2	0.292	1.32	19.2	0.10	107	2.13	36.0	3.39	294	22.5	6250	1.01	0.78	1.20	0.76	9.75	
Sample 3	0.265	0.66	13.5	0.95	38.0	NA	36.3	2.38	375	57.3	323	1.02	0.48	1.45	1.97	0.60	
HCl																	
No. 10-passing																	
Sample 1	1.63	14.0	249	2.00	814	1.50	165.0	40.0	3340	363	8830	0.57	0.78	0.74	0.65	0.68	
Sample 2	1.70	14.3	248	1.83	821	1.53	225.0	61.8	4980	615	13900	0.77	1.03	1.05	1.09	1.02	
No. 10-to-1"																	
Sample 1	0.751	1.17	23.7	0.89	61.0	1.32	46.2	3.13	398	34.0	515	1.32	0.71	1.60	1.33	1.11	
Sample 2	0.765	1.13	25.1	0.41	67.0	1.22	27.4	3.20	218	22.9	420	0.80	0.70	0.96	0.79	0.84	
No. 10-passing (note 3)																	
Sample 1	NA	2.9	49.9	2.5	173.0	0.96	NA	58.3	5050	468	14100	NA	0.99	1.07	0.87	1.03	
Sample 2	NA	2.8	49.8	2.2	178.0	0.96	NA	58.5	5280	583	14000	NA	0.99	1.11	1.07	1.03	
Hot HCl																	
No. 10-passing																	
Sample 1	NA	3.3	92.8	8.1	22.8	0.93	NA	58.0	4800	335	14400	NA	1.06	1.12	0.91	1.00	
Extraction 1	NA	0.8	29.7	11.4	72.0	0.79	NA										
Extraction 2	NA																
Sample 2	NA	3.4	82.2	4.7	21.4	0.92	NA	58.0	4880	470	14100	NA	1.06	1.12	1.11	0.98	
Extraction 1	NA	0.8	30.9	12.0	75.0	0.79	NA										
Extraction 2	NA																
No. 10-passing (note 3)																	
Sample 1	NA	3.5	83.3	7.9	29.5	1.01	NA	52.0	4800	358	12800	NA	0.92	1.13	0.92	0.93	
Extraction 1	NA	0.1	39.8	9.9	113.0	0.86	NA										
Extraction 2	NA																
Sample 2	NA	3.5	82.2	7.8	254.0	1.02	NA	51.8	4800	288	12600	NA	0.92	1.11	0.82	1.02	
Extraction 1	NA	0.1	33.8	11.2	103.0	0.83	NA										
Extraction 2	NA																
HCl/H2O2																	
No. 10-passing																	
Sample 1	1.25	14.8	272	3.08	847	1.53	262.0	60.8	4900	562	14000	0.90	1.03	1.05	1.00	1.03	
Sample 2	1.10	14.2	254	2.94	781	1.51	243.0	59.5	4820	444	14000	0.83	1.00	1.02	0.79	1.02	
No. 10-to-1"																	
Sample 1	0.616	1.26	30	1.01	62.0	1.12	45.5	3.18	278	37.3	590	1.29	0.74	1.22	1.31	1.08	
Sample 2	0.822	2.18	34	0.46	621	1.13	30.6	12.3	273	27.5	9350	0.89	2.15	1.23	0.95	15.98	
Sample 3	0.439	1.22	22	0.78	88.0	NA	7.7	1.53	598	125	250	0.24	0.51	2.31	4.22	0.64	

Note 1. Initial pH of extractant solutions were as follows unless otherwise noted: Sodium Gluconate-13.40, EDTA-6.15, Acetic Acid-2.17, HCl-1.00, Hot HCl-0.80, HCl/H2O2-1.00  
 Note 2. Initial pH-12.30  
 Note 3. Sample was pulverized before extraction.  
 Note 4. Initial pH-7.00

Table 3-4  
EXTRACTION OF SOIL SAMPLE 2

Celtor Chemical Works Extraction Studies  
Job # W19628.00

Extractant	SOIL ANALYSIS SUMMARY															
	Size	% of Total	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)									
	No. 10-passing	52.5	311.00	12.0	2930	532.0	2430									
	No. 10-to-1"	39.9	56.20	13.3	3370	113.0	220									
EXTRACTION SUMMARY																
Extractant	Extractant Solution(after extraction)						Soil(after extraction)					Mass Balance(Fraction of Starting Amount Recovered)				
	As(mg/l)	Cd(mg/l)	Cu(mg/l)	Pb(mg/l)	Zn(mg/l)	pH(note 1)	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)	As	Cd	Cu	Pb	Zn
Sodium Gluconate																
No. 10-passing																
Sample 1	7.18	0.750	144	13.2	106	NA	267	13.0	3380	731	2790	0.90	1.21	1.25	1.42	1.24
Sample 2	7.06	0.700	146	13.8	109	12.01	257	11.8	3330	794	2780	0.87	1.10	1.24	1.54	1.23
No. 10-to-1"																
Sample 1	4.77	0.060	5.3	11.8	10.0	13.06	86.6	10.8	3700	92.5	1600	1.71	0.82	1.10	1.03	7.36
Sample 2	3.75	0.030	5.4	11.6	10.1	13.04	56.9	1.95	1280	86	423	1.15	0.15	0.38	0.97	2.01
EDTA																
No. 10-passing (note 2)																
Sample 1	0.137	1.43	265	55.2	339	5.65	274	11.0	3140	663	2130	0.88	1.16	1.25	1.45	1.16
Sample 2	0.175	1.43	267	54.9	343	5.73	265	11.0	3070	664	2200	0.85	1.16	1.23	1.45	1.19
No. 10-to-1"																
Sample 1	0.048	0.440	59	9.57	74.0	5.89	89	2.4	4310	118	590	1.59	0.25	1.31	1.21	3.35
Sample 2	0.034	0.420	41	3.30	70.0	5.88	41.2	9.75	3520	64	2050	0.73	0.80	1.07	0.62	9.95
Acetic Acid																
No. 10-passing																
Sample 1	0.073	1.40	251	0.690	337	2.05	265	11.0	2810	541	2080	0.85	1.15	1.13	1.02	1.13
Sample 2	0.059	1.30	232	0.770	291	2.02	280	10.5	2790	655	2030	0.90	1.09	1.11	1.23	1.07
No. 10-to-1"																
Sample 1	0.013	0.310	36.7	0.910	57.0	2.48	58.9	2.1	1250	69.3	505	1.05	0.20	0.39	0.63	2.81
Sample 2	0.013	0.200	30	0.720	39.0	2.64	69	8.75	3060	115	1980	1.23	0.69	0.93	1.03	9.35
Sample 3	0.01	0.200	25.1	0.970	37.0	NA	62.8	3.8	768	129	893	1.12	0.32	0.24	1.16	4.40
HCl																
No. 10-passing																
Sample 1	0.251	1.60	275	6.33	354	1.39	285	10.0	2740	585	2080	0.92	1.10	1.12	1.12	1.15
Sample 2	0.237	1.37	257	5.98	327	1.40	201	52.8	5330	255	12300	0.65	4.63	1.99	0.50	5.33
No. 10-to-1"																
Sample 1	0.095	0.450	83	11.2	83.0	1.20	70.7	19.5	6930	593	3600	1.26	1.53	2.11	5.45	17.12
Sample 2	0.125	0.220	30	2.49	40.0	1.28	123	3.0	750	250	913	2.19	0.26	0.24	2.26	4.51
HCl/H2O2																
No. 10-passing																
Sample 1	0.03	0.600	294	1.11	119	1.41	271	10.5	2860	500	2130	0.87	0.98	1.18	0.94	0.97
Sample 2	0.316	2.23	316	6.61	436	1.42	361	12.5	3300	760	2450	1.16	1.41	1.34	1.45	1.37
No. 10-to-1"																
Sample 1	0.165	0.200	31	5.94	36.0	1.23	69.2	3.48	1380	154	750	1.24	0.29	0.43	1.47	3.74
Sample 2	0.131	0.220	32.7	4.30	41.0	1.17	74.4	13.5	1310	108	2800	1.33	1.05	0.41	1.03	13.10

Note 1. Initial pH of solutions were as follows, unless otherwise noted: Sodium Gluconate-13.40, EDTA-6.15, Acetic Acid-2.17, HCl-1.00, HCl/H2O2-1.00.

Note 2. Initial pH-6.9

Table 3-5  
EXTRACTION OF SOIL SAMPLE 3

Geltor Chemical Works Extraction Studies  
Job # W19628.00

	SOIL ANALYSIS SUMMARY															
	Size	% of Total	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)									
	No. 10-Passing	57.9	29.7	6.0	1880	232	1280									
	No. 10-to-1"	32.7	9.34	2.1	963	147	405									
EXTRACTION SUMMARY																
Extractant	Extracted Solution (after extraction)						Soil (after extraction)					Mass Balance (Fraction of Starting Amount Recovered)				
	As(mg/l)	Cd(mg/l)	Cu(mg/l)	Pb(mg/l)	Zn(mg/l)	pH (Note 1)	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)	As	Cd	Cu	Pb	Zn
Sodium Gluconate																
No. 10-passing																
Sample 1	5.33	0.06	260	46.3	15.1	12.35	35.6	5.25	1280	121	1150	1.56	0.90	0.96	0.92	0.92
Sample 2	5.01	0.06	273	56	26	12.01	41.8	5.75	1300	144	1280	1.74	0.98	0.98	1.10	1.04
No. 10-to-1" (note 2)																
Sample 2	0.171	0.01	46	0.18	4.7	12.35	2.85	1.7	410	4.28	525	0.34	0.81	0.52	0.03	1.32
Sample 2	0.154	0.01	67	0.07	5.1	12.11	10.7	1.2	1230	8.55	278	1.18	0.57	1.42	0.06	0.71
Sample 3	0.543	0.03	83	1.76	16.3	NA	3.85	1.25	453	3.4	213	0.53	0.62	0.64	0.05	0.61
EDTA																
No. 10-passing																
Sample 1 (note 3)	0.012	0.5	248	4.65	89	7	63.9	3.88	1280	240	1110	2.15	0.81	0.94	1.07	1.01
Sample 2	0.022	0.47	232	2.37	83.4	5.87	58.4	4.75	1120	230	993	1.97	0.95	0.84	1.01	0.91
No. 10-to-1"																
Sample 1	0.033	0.22	86	3.23	26	6.17	23.9	9	3020	33	1680	2.57	4.43	3.31	0.27	4.28
Sample 2	0.024	0.13	88	0.95	25	6.17	5.37	0.9	240	4.38	135	0.58	0.54	0.43	0.04	0.46
Acetic Acid																
No. 10-passing																
Sample 1	0.016	0.45	207	0.05	92.8	2.67	60.3	5	1130	238	988	2.03	0.98	0.82	1.03	0.92
Sample 2	0.008	0.46	211	0.05	94.2	2.66	16.8	5	1180	263	1090	0.57	0.99	0.85	1.13	1.00
No. 10-to-1"																
Sample 1	0.01	0.75	53.2	0.12	124	3.27	15.2	2.25	435	20.7	535	1.63	1.76	0.56	0.14	1.93
Sample 2	0.143	0.18	178	0.05	33	2.61	7.61	3.28	1080	9.6	780	0.85	1.71	1.49	0.07	2.09
Sample 3	0.005	0.15	178	2.56	314	NA		1.13	598	386	198	NA	0.67	0.99	2.66	2.04
HCl																
No. 10-passing																
Sample 1	0.031	0.6	286	0.42	126	1.52	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sample 2	0.035	0.57	286	0.44	113	1.49	61.9	3.73	1190	274	1020	2.09	0.81	0.94	1.18	0.97
No. 10-to-1"																
Sample 1	0.026	0.31	301	0.21	65	1.32	71.7	4.75	1290	274	1030	7.68	2.52	1.96	1.87	2.86
Sample 2	0.034	0.24	212	0.16	46	1.22	6.42	1	545	6.78	215	0.69	0.69	1.01	0.05	0.76
HCl/H2O2																
No. 10-passing																
Sample 1	0.041	0.65	297	0.57	121	1.6	61.6	3.83	1080	302	1080	2.08	0.86	0.89	1.31	1.03
Sample 2	0.026	0.63	308	0.4	123	1.64	70.9	3.78	1060	228	1060	2.39	0.84	0.89	0.99	1.02
No. 10-to-1"																
Sample 1	0.033	0.22	221	0.2	52	1.13	5.38	1.73	568	5.38	370	0.58	1.02	1.05	0.04	1.17
Sample 2	0.03	0.25	187	0.61	41	1.19	4.73	0.95	438	55	328	0.51	0.68	0.84	0.38	1.01
Sample 3	0.031	0.21	137	0.2	32	NA		6.25	218	35	343	NA	3.13	0.51	0.24	1.00

Note 1. Initial pH of extractant solutions were as follows, unless otherwise noted: Sodium Gluconate-13.40, EDTA-6.15, Acetic Acid-2.17, HCl-1.00, HCl/H2O2-1.00.

Note 2. Initial pH-12.30

Note 3. Initial pH-6.9.

Table 3-6  
EXTRACTION OF COMBINED SAMPLE 486

	SOIL ANALYSIS SUMMARY																
	Size		% of Total		As(mg/kg)		Cd(mg/kg)		Cu(mg/kg)		Pb(mg/kg)		Zn(mg/kg)				
	No. 10-Passing	No. 10-to-1"	78.7	18.1	297.0	63.6	52.0	2.1	5240	328	394.0	38.0	12200	408			
EXTRACTION SUMMARY																	
	Extractant solution(after extraction)						Soil(after extraction)						Mass Balance(fraction of starting amount recovered)				
	As(mg/l)	Cd(mg/l)	Cu(mg/l)	Pb(mg/l)	Zn(mg/l)	pH(Note 1)	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)	As	Cd	Cu	Pb	Zn	
Sodium Gluconate																	
No. 10-passing																	
Sample 1	23.8	0.32	85	150	73	12.74	234	52.3	5080	286	11400	0.95	1.02	1.00	1.49	0.95	
Sample 2	24.8	0.35	98	143	77	12.66	275	49.5	5270	282	11000	1.09	0.97	1.04	1.44	0.91	
No. 10-to-1"																	
Sample 1	4.67	0.02	14.3	9.25	11	13.19	27.4	0.98	155	40	248	0.58	0.50	0.56	1.54	0.66	
Sample 2	8.88	0.02	19.1	6.61	9.2	13.26	24.7	1.1	255	41.5	280	0.67	0.56	0.89	1.44	0.73	
EDTA																	
No. 10-passing																	
Sample 1	0.39	1.58	166	35.1	359	NA	299	49	5130	208	1120	1.01	1.00	1.04	0.71	0.15	
Sample 2	0.35	1.58	158	38.4	350	5.83	301	48.5	5150	332	11300	1.02	0.99	1.04	1.04	0.98	
No. 10-to-1"																	
Sample 1	0.121	0.27	40.2	3.38	66	6.13	56.3	1	253	44	608	0.89	0.75	1.02	1.34	1.81	
Sample 2	0.113	0.16	36	1.28	46	6.16	40	0.43	150	26.7	125	0.63	0.37	0.68	0.77	0.53	
No. 10-passing (note 2)																	
Sample 1 (note 3)	NA	0.26	12.4	20.3	60	7.15	NA	48	4990	274	11300	NA	0.97	0.98	1.21	0.98	
Sample 2 (note 3)	NA	0.3	14.3	25.9	67.5	7.2	NA	59	6050	113	14200	NA	1.19	1.18	0.94	1.22	
Acetic Acid																	
No. 10-passing																	
Sample 1	0.923	1.4	135	0.13	288	1.84	276	50	4980	341	11200	0.94	1.02	1.00	0.87	0.97	
Sample 2	0.908	1.5	145	0.1	310	1.85	269	49.5	4980	284	11000	0.91	1.01	1.01	0.72	0.95	
No. 10-to-1"																	
Sample 1	0.057	0.24	54	0.84	53	NA	40.2	1.18	330	43.8	245	0.63	0.81	1.34	1.20	0.86	
Sample 2	0.051	0.14	20.3	0.05	31	2.51	31.1	0.3	168	32.5	133	0.49	0.28	0.64	0.86	0.48	
HCl																	
No. 10-passing																	
Sample 1	1.79	1.5	158	1.92	333	1.29	270	51.3	5100	445	11800	0.92	1.04	1.03	1.14	1.02	
Sample 2	1.97	1.58	164	2.04	352	1.32	243	51.8	5180	393	1900	0.83	1.06	1.05	1.01	0.21	
No. 10-to-1"																	
Sample 1	0.196	0.18	26.9	0.22	89	1.18	37	0.3	120	28.3	173	0.59	0.32	0.53	0.76	0.86	
Sample 2	0.256	0.43	49	0.34	61	1.12	32.6	0.3	135	24.3	148	0.52	0.57	0.71	0.66	0.66	
No. 10-passing (note 2)																	
Sample 1	NA	0.3	25	1.87	58	0.94	NA	47	5400	410	12000	NA	0.96	1.08	1.09	1.03	
Sample 2	NA	0.29	30.7	1.75	62	0.94	NA	52	5200	300	12000	NA	1.06	1.05	0.81	1.03	
Hot HCl																	
No. 10-passing																	
Sample 1																	
Extraction 1	NA	0.34	40.9	5.55	71	0.89	NA	54	5050	153	12200	NA	1.11	1.05	0.77	1.06	
Extraction 2	NA	0.08	11.1	10.7	17	0.78											
Sample 2																	
Extraction 1	NA	0.35	45.6	6	70.5	0.88	NA	54.5	5200	250	12600	NA	1.12	1.08	1.03	1.09	
Extraction 2	NA	0.07	10.6	10.9	15	0.77											
No. 10-passing (note 2)																	
Sample 1																	
Extraction 1	NA	0.38	41.7	9.75	71	0.95	NA	45	4850	277	11000	NA	0.95	1.02	1.26	0.97	
Extraction 2	NA	0.12	14.5	14.3	26.6	0.79											
Sample 2																	
Extraction 1	NA	0.4	47.7	8.3	79	0.97	NA	51	4950	238	1250	NA	1.06	1.05	1.12	0.18	
Extraction 2	NA	0.11	17	13.7	26.3	0.78											
HCl/H2O2																	
No. 10-passing																	
Sample 1	1.73	1.49	165	1.73	331	1.39	265	52.5	5050	503	11400	0.90	1.07	1.03	1.29	0.99	
Sample 2	1.77	0.15	178	1.91	354	1.37	257	49.3	5000	446	11000	0.88	0.95	1.02	1.14	0.96	
No. 10-to-1"																	
Sample 1	0.213	0.3	30.7	0.25	50	1.21	47.5	0.73	205	25.3	1100	0.75	0.65	0.81	0.68	2.94	
Sample 2	0.265	0.38	41	0.41	60	1.22	39.9	1.33	198	28.8	268	0.64	1.02	0.85	0.78	0.95	

Note 1. Initial pH of all extractants were as follows, unless otherwise noted: Sodium Gluconate-13.40, EDTA-6.15, Acetic Acid-2.17, HCl-1.00, Hot HCl .80, HCl/H2O2-1.00.  
 Note 2. Sample was pulverized before extraction.  
 Note 3. Initial pH-7.00

Table 3-7  
EXTRACTION OF SOIL SAMPLE 5.

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Extractant	EXTRACTION SUMMARY						SOIL ANALYSIS SUMMARY						Mass Balance(Fraction of Starting amount Recovered)				
	Extractant Solution(after extraction)						Soil(after extraction)										
	As (mg/l)	Cd(mg/l)	Cu(mg/l)	Pb(mg/l)	Zn(mg/l)	pH(Note 1)	As(mg/kg)	Cd(mg/kg)	Cu(mg/kg)	Pb(mg/kg)	Zn(mg/kg)	As	Cd	Cu	Pb	Zn	
Sodium Gluconate																	
No. 10-passing																	
Sample 1	28.3	0.02	98	50	4	11.79	219	8.5	505	1190	1950	0.80	0.68	1.07	0.96	0.83	
Sample 2	27.5	0.01	73	28	2.08	11.71	204	8.75	630	1110	2080	0.75	0.70	1.18	0.86	0.89	
Acetic Acid																	
No. 10-passing																	
Sample 1	0.041	0.27	298	0.05	48.2	2.35	282	13	755	1310	2250	0.82	1.08	2.05	0.97	1.00	
Sample 2	0.032	0.22	322	0.05	46.6	2.35	280	12.5	688	1260	2180	0.81	1.04	2.02	0.93	0.97	
HCl																	
No. 10-passing																	
Sample 1	0.08	0.36	57	0.09	62	1.37	317	12.8	533	1350	2430	0.92	1.08	0.98	1.00	1.09	
Sample 2	0.082	0.36	61	0.08	65	1.34	276	12	540	1200	2330	0.80	1.02	1.01	0.89	1.05	
EDTA																	
No. 10-passing																	
Sample 1	0.069	0.36	71	1.56	67	NA	299	10.5	518	1190	1980	0.87	0.90	1.00	0.88	0.90	
Sample 2	0.114	0.34	84	2.08	84	5.88	320	12.8	700	1340	2380	0.93	1.08	1.32	1.00	1.08	
HCl/H2O2																	
No. 10-passing																	
Sample 1	0.099	0.36	69	0.05	70	1.44	297	10.8	490	1310	2370	0.86	0.92	0.95	0.97	1.07	
Sample 2	0.147	0.37	70	0.18	73	1.55	262	10.75	650	1340	2350	0.76	0.92	1.20	0.99	1.06	

Note 1. For initial pH of extractant Solutions, see Tables 4 through 7.

- Soil (After Extraction)--Under this heading are presented the analyses of metals in the soil that had undergone extraction. Analyses were performed on the extracted soil sample after it had been pressure-filtered on a prefilter paper for removal of extractant, washed, dried, and pulverized.
- Mass Balance--Presented under this heading are results of a mass balance calculated on each metal. Values given are the fraction of the total starting mass that was recovered in the soil and extractant solutions.

For an example, refer to Table 3-1, and note that the Cd mass balance for the sodium gluconate extractant for Sample 1 of the No. 10-passing fraction equals 1.04. (This indicates greater than 100 percent recovery which, of course, is not possible. However, this value is considered to be within acceptable limits of accuracy.) This value was calculated as follows:

Beginning Mass

$$100\text{g} \times \frac{1 \text{ kg}}{1,000\text{g}} \times 87.5 \text{ mg/kg} \text{ (see No. 10-passing under Cd in the top section of the table)}$$

$$= 8.75 \text{ mg}$$

Extractant Solution

$$3.2 \text{ mg/l} \times 200 \text{ ml} \times \frac{1 \text{ liter}}{1,000 \text{ ml}}$$

$$= 0.64 \text{ mg}$$

Soil

$$84.5 \text{ mg/kg} \times 100\text{g} \times \frac{1 \text{ kg}}{1,000 \text{ g}} = 8.45 \text{ mg}$$

$$8.45 \text{ mg} + 0.64 \text{ mg} = 9.09 \text{ mg}$$

$$9.09 \text{ mg} / 8.75 \text{ mg} = 1.04$$

#### EXTRACTION EFFICIENCIES

Extraction efficiencies are summarized in Tables 3-8 through 3-12. These efficiencies were calculated by the formula

$$E = (M_e / M_s + M_e) \times 100 \quad \text{Equation 1}$$

where

E = extraction efficiency  
M<sub>e</sub> = metal in the extractant, mg  
M<sub>s</sub> = metal in the soil after extraction, mg

Referring to the values given in the previous mass balance example, the efficiency of extraction for the values given in that example would be

$$E = (0.64/8.45 + 0.64) \times 100 = 7\%$$

There are three potential methods of calculating extraction efficiencies, as follows:

1) From Equation 1

2) From the equation:

$$E = (M_i - M_s)/M_i \quad \text{Equation 2}$$

where M<sub>i</sub> is equal to the assumed initial concentration of metal in the soil, as measured by the portion originally split off for analysis

3) From the equation:

Equation 3

$$E = (M_e/M_i) \times 100$$

If our assumption that the initial concentration of metal before extraction is the same in all cases for a given sample portion is good, and all analyses are 100 percent accurate, all three equations will provide the same results. As can be seen from Tables 3-8 through 3-12, not all mass balance values equal 1.0. We therefore decided to use the first equation for calculation of efficiencies because:

- 1) If Equation 2 is used, many negative efficiencies result, which is not possible from the standpoint of conservation of mass. It is also obvious that our assumption that all samples have the same starting concentration is not necessarily true because of the heterogeneity of the sample.
- 2) If Equation 3 is used, no negative extraction efficiencies result. However, this equation still relies on the assumption that each starting sample has the same initial metals concentration.

Because of the potential for lab data variations due to inaccuracies within accepted limits, it is impossible to be certain that the efficiencies calculated by Equation 1 are completely accurate. Therefore, we have used an asterisk to



highlight those samples whose mass balances show less than a 75 percent closure in Tables 3-8 through 3-12. Even though mass balance closure does not guarantee accuracy, it is another cross-check that allows greater confidence in the data.

### EXTRACTION SUMMARY

Refer to Appendix B for a detailed summary and discussion of extraction results. Presented in Figures 3-1 and 3-2 are graphical summaries of all extraction testing. Figure 3-1 presents results of testing performed on the No. 10-passing portion of the samples. Figure 3-2 presents results of testing performed on the No. 10-to-1-inch portion.

Figures 3-1 and 3-2 illustrate the patterns and differences that occur between different soils using the same extractant and between different extractants for the same soil sample. Presented below is a brief summary of the information illustrated in these figures (note that the values given for removal efficiencies in this figure are averages of the values given in Tables 3-8 through 3-12).

#### SUMMARY OF EXTRACTIONS ON NO. 10-PASSING SAMPLE

##### Sodium Gluconate Extractant

Sodium gluconate was most effective in extracting lead (up to an average value of 50.8 percent in Combined Samples 4 and 6, copper, (29.3 percent in Sample 3) and arsenic (21.2 percent in Sample 3). Observing the extraction efficiencies for the remaining extractants, note that removals of lead greater than 25 percent were obtained in only three other cases--hot HCl extraction on Sample 1 and Combined Samples 4 and 6, and EDTA extraction on Combined Samples 4 and 6. Note also that no other extractant removed a significant amount of arsenic (greatest removal was 2 percent for HCl in Soil Sample 1). In only one sample did the sodium gluconate extractant remove sufficient lead to reduce the level of this metal from above to below cleanup levels (Sample 1). No other removals to below cleanup levels were obtained.

##### EDTA Extractant

This extractant was uniformly inefficient in removing arsenic from the soils tested (greatest removal was 1 percent in Soil Sample 1). Removals of other constituents varied significantly depending on which soil was being extracted and on whether the sample was pulverized before extraction. Average removals for cadmium were as high as 31 percent, copper 46.2 percent, lead 56 percent

Table 3-8  
EXTRACTION OF SOIL SAMPLE 1  
EXTRACTION EFFICIENCIES

Extractant	Percent of Metal Removed from Soil				
	As	Cd	Cu	Pb	Zn
Sodium Gluconate					
10 mesh passing					
Sample 1	16	7	2	48	3
Sample 2	18	7	2	48	4
10 mesh to 1"					
Sample 1	11	2 *	7	2	4*
Sample 2	10	1	4*	5	4
EDTA Extractant					
10 mesh passing					
Sample 1	1 *	29	6	4	11
Sample 2	1 *	29	7	4	11
10 mesh to 1"					
Sample 1	1	41 *	11*	12	26
Sample 2	1	38 *	12	10	22
#10 passing (Pulverized)					
Sample 1	na	32	5	12	9
Sample 2	na	30	5	12	9
Acetic Acid extractant					
10 mesh passing					
Sample 1	1 *	31	6	0	11
Sample 2	1 *	29	6	0 *	10
10 mesh to 1"					
Sample 1	1 *	43 *	13	0	27*
Sample 2	2	44	12	1	3*
Sample 3	1	36 *	7*	3 *	19*
HCl extractant					
10 mesh Passing					
Sample 1	2*	41	13*	1 *	16*
Sample 2	1	32	9	1	11
10 mesh to 1"					
Sample 1	3 *	43 *	11*	4 *	17
Sample 2	5	41 *	19	3	24
10 mesh passing					
Sample 1	na	33	9	5	11
Sample 2	na	32	9	4	11
Hot HCl Extractant					
#10 passing (unpulverized)					
Sample 1					
Extraction 1	na	37	18	35	6
Extraction 2					
Sample 2					
Extraction 1	na	38	17	25	6
Extraction 2	na				
#10 passing (pulverized)					
Sample 1	na				
Extraction 1	na	36	18	31	10
Extraction 2	na				
Sample 2	na				
Extraction 1	na	36	17	38	20
Extraction 2	na				
HCl/H2O2 extractant					
10 mesh passing					
Sample 1	1	33	10	1	11
Sample 2	1	32	10	1	10
10 mesh to 1"					
Sample 1	3 *	44 *	18	5*	17
Sample 2	5	26 *	20	3	12 *
Sample 3	10 *	61 *	7 *	1 *	41 *

\* Mass balance is (0.75, or )1.25

Table 3-9  
EXTRACTION OF SOIL SAMPLE 2  
EXTRACTION EFFICIENCIES

Extractant	Percent of Metal Removed from Soil				
	As	Cd	Cu	Pb	Zn
Sodium Gluconate					
10 mesh passing					
Sample 1	2.6	5.5	4.1	1.8 *	3.7
Sample 2	2.7	5.6	4.2	1.7 *	3.8
10 mesh to 1"					
Sample 1	5.2 *	0.6	0.1	11.3	0.6 *
Sample 2	6.2	1.5 *	0.4 *	11.3	2.3 *
EDTA					
10 mesh passing					
Sample 1	0.0	11.5	7.8	7.7 *	13.7
Sample 2	0.1	11.5	8.0	7.6 *	13.5
10 mesh to 1"					
Sample 1	0.1 *	15.5 *	1.4 *	7.5	11.1 *
Sample 2	0.1 *	4.1	1.2	4.9 *	3.3 *
Acetic Acid					
10 mesh passing					
Sample 1	0.0	11.3	8.2	0.1	13.9
Sample 2	0.0	11.0	7.7	0.1	12.5
10 mesh to 1"					
Sample 1	0.0	12.9 *	2.9 *	1.3 *	10.1 *
Sample 2	0.0	2.2 *	1.0	0.6	1.9 *
Sample 3	0.0	5.0 *	3.2 *	0.7	4.0 *
HCl					
10 mesh passing					
Sample 1	0.1	13.8	9.1	1.1	14.5
Sample 2	0.1 *	2.5 *	4.6 *	2.3 *	2.6 *
10 mesh to 1"					
Sample 1	0.1 *	2.3 *	1.2 *	1.9 *	2.3 *
Sample 2	0.1 *	6.8 *	3.8 *	1.0 *	4.2 *
HCl/H2O2					
10 mesh passing					
Sample 1	0.0	5.4	9.3	0.2	5.3
Sample 2	0.1	15.1 *	8.7 *	0.9 *	15.1 *
10 mesh to 1"					
Sample 1	0.2	5.4 *	2.2 *	3.7 *	4.6 *
Sample 2	0.2 *	1.6	2.4 *	3.8	1.4 *

\* Mass balance is (0.75 or )1.25

Table 3-10  
EXTRACTION OF SOIL SAMPLE 3  
EXTRACTION EFFICIENCIES

Extractant	Percent of Metal Removed				
	As	Cd	Cu	Pb	Zn
Sodium Gluconate					
10 mesh passing					
Sample 1	23.0*	2.2	28.9	43.4	2.6
Sample 2	13.3*	2.0	29.6	43.8	3.9
10 mesh to 1"					
Sample 2	10.7*	1.2	18.3*	7.8*	1.8*
Sample 2	2.8	1.6*	9.8*	1.6*	3.5*
Sample 3	22.0*	4.6*	26.8*	50.9*	13.3*
EDTA					
10 mesh passing					
Sample 1	0.0*	20.5	27.9	3.7	13.8
Sample 2	0.1*	16.5	29.3	2.0	14.4
10 mesh to 1"					
Sample 1	0.3*	4.7*	5.4*	15.4*	3.0*
Sample 2	0.9*	22.4*	42.3*	30.3*	27.0*
Acetic Acid					
10 mesh passing					
Sample 1	0.1*	15.3	26.8	0.0	15.8
Sample 2	0.1*	15.5	26.3	0.0	14.7
10 mesh to 1"					
Sample 1	0.0*	40.0*	19.7*	1.1*	31.7*
Sample 2	3.6	9.9*	24.8*	0.0*	7.8*
Sample 3	na	21.0*	37.3	1.3*	75.0*
HCl					
10 mesh passing					
Sample 1	na	na	na	na	na
Sample 2	0.1*	23.4	32.5	0.3	16.1
10 mesh to 1"					
Sample 1	0.1*	11.5*	31.8*	0.2*	11.2*
Sample 2	1.0*	32.4*	43.8	4.5*	30.0
HCl/H2O2					
10 mesh passing					
Sample 1	0.1*	25.3	35.5	0.4*	18.3
Sample 2	0.1*	25.0	36.8	0.3	18.8
10 mesh to 1"					
Sample 1	1.2*	20.3	43.8	6.9*	21.9
Sample 2	1.3*	34.5*	46.1	2.2*	20.0
Sample 3	0.0	6.3*	55.7*	1.1*	15.7

\* Mass balance is (0.75 or )1.25

Table 3-11  
EXTRACTION OF COMBINED SOIL SAMPLES 4 AND 6

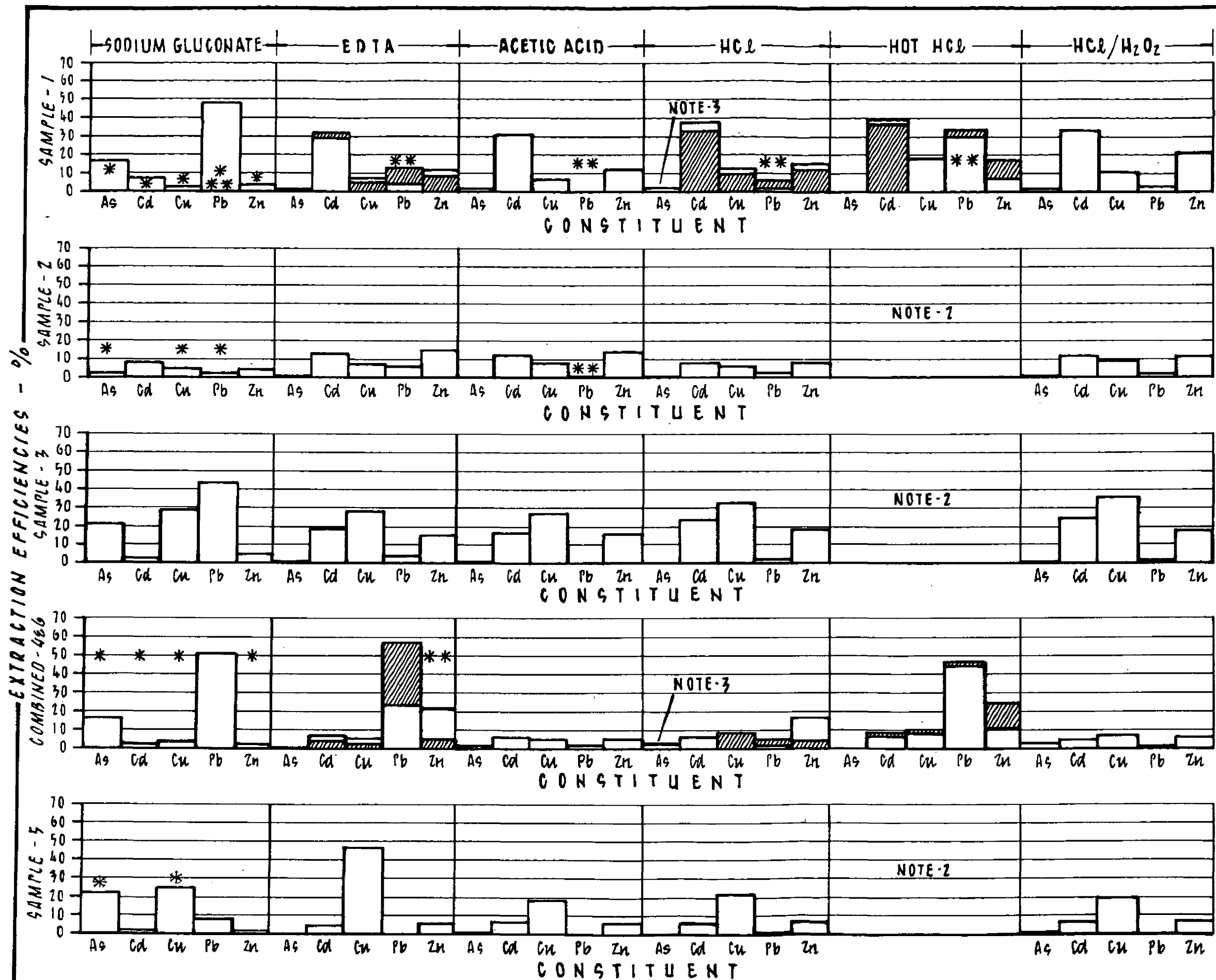
Extractant	Percent of Metal Removed from Soil				
	As	Cd	Cu	Pb	Zn
Na Gluc					
10 mesh passing					
Sample 1	15.9	1.2	3.2	51.2 *	1.3
Sample 2	15.3	1.4	3.6	50.4 *	1.4
10 mesh to 1"					
Sample 1	25.4 *	3.9*	15.6 *	31.6 *	9.1 *
Sample 2	41.8 *	3.5*	13.0	24.2 *	6.2 *
EDTA					
10 mesh passing					
Sample 1	0.3	6.1	6.1	25.2 *	39.1 *
Sample 2	0.2	6.1	5.8	18.9	5.8
10 mesh to 1"					
Sample 1	0.4	35.1	24.1	13.3 *	17.8 *
Sample 2	0.6 *	42.7*	32.4 *	8.7	42.4 *
(Pulverized)					
Sample 1	NA	5.1	2.4	42.6	5.0
Sample 2	NA	4.8	2.3	69.6	4.5
Acetic Acid					
10 mesh passing					
Sample 1	0.7	5.3	5.1	0.1	4.9
Sample 2	0.7	5.7	5.5	0.1 *	5.3
10 mesh to 1"					
Sample 1	0.3 *	28.9	24.7 *	3.7	30.2
Sample 2	0.3 *	48.3*	19.5 *	0.3	31.8 *
HCl					
10 mesh passing					
Sample 1	1.3	5.5	5.8	0.9	5.3
Sample 2	1.6	5.7	6.0	1.0	27.0 *
10 mesh to 1"					
Sample 1	1.0 *	54.5*	31.0 *	1.5	50.7
Sample 2	1.5 *	74.1*	42.1 *	2.7*	45.2 *
(Pulverized)					
Sample 1	NA	6.0	4.4	4.4	4.6
Sample 2	NA	5.3	5.6	5.5	4.3
Hot HCl					
(Unpulverized)					
Sample 1					
Extraction 1	NA	6.1	8.0	49.7	5.7
Extraction 2					
Sample 2					
Extraction 1	NA	6.0	8.3	38.6	5.4
Extraction 2					
(Pulverized)					
Sample 1					
Extraction 1	NA	8.6	9.0	44.4*	7.0
Extraction 2					
Sample 2					
Extraction 1	NA	7.8	10.0	46.1	41.7 *
Extraction 2					
HCl/H2O2					
10 mesh passing					
Sample 1	1.3	5.4	6.1	0.7*	5.5
Sample 2	1.4	0.6	6.6	0.8	6.0
10 mesh to 1"					
Sample 1	0.9	45.1*	23.0	1.9*	8.3 *
Sample 2	1.3 *	36.4	29.3	2.3	30.9

\* Mass balance is (0.75 or >1.25

Table 3-12  
EXTRACTION OF SOIL SAMPLE 5 -  
EXTRACTION EFFICIENCIES

Extractant	Percentage of Metal Extracted from Soil				
	As	Cd	Cu	Pb	Zn
Na Gluc					
10 mesh passing					
Sample 1	20.54	0.47*	27.96	7.75	0.41
Sample 2	21.24	0.23*	19.81	4.90	0.20
Acetic Acid					
10 mesh passing					
Sample 1	0.03	3.99	44.12 *	0.00	4.11
Sample 2	0.02	3.40	48.35 *	0.00	4.10
HCl					
10 mesh passing					
Sample 1	0.05	5.33	17.62	0.01	4.36
Sample 2	0.06	5.66	18.43	0.01	5.28
EDTA					
10 mesh passing					
Sample 1	0.05	6.42	21.52	0.26	6.34
Sample 2	0.07	5.04	19.35 *	0.31	6.59
HCl/H2O2					
10 mesh passing					
Sample 1	0.07	6.25	21.97	0.01	5.58
Sample 2	0.11	6.44	17.72	0.03	5.85

\* Mass balance is (0.75 or )1.25



**NOTES & LEGEND**

\* INDICATES THAT SOIL SAMPLE WAS INITIALLY ABOVE CLEAN UP CRITERIA FOR CONSTITUENT (FOR CLARITY ONLY THE SODIUM GLUCONATE EXTRACTANT GROUP IS INDICATED)

\*\* INDICATES THAT CONSTITUENT WAS REMOVED IN SUFFICIENT QUANTITIES TO BRING SOIL FROM ABOVE TO BELOW CLEAN UP CRITERIA FOR THIS CONSTITUENT. (THIS MAY HAVE ONLY OCCURED IN ONE OF TWO OR THREE EXTRACTIONS. DATA MAY INDICATE LOW PERCENTAGE REMOVALS WHICH MAY MEAN THAT THE ACTUAL SAMPLE TESTED WAS BELOW CLEAN UP CRITERIA INITIALLY - SEE TEXT ALSO).

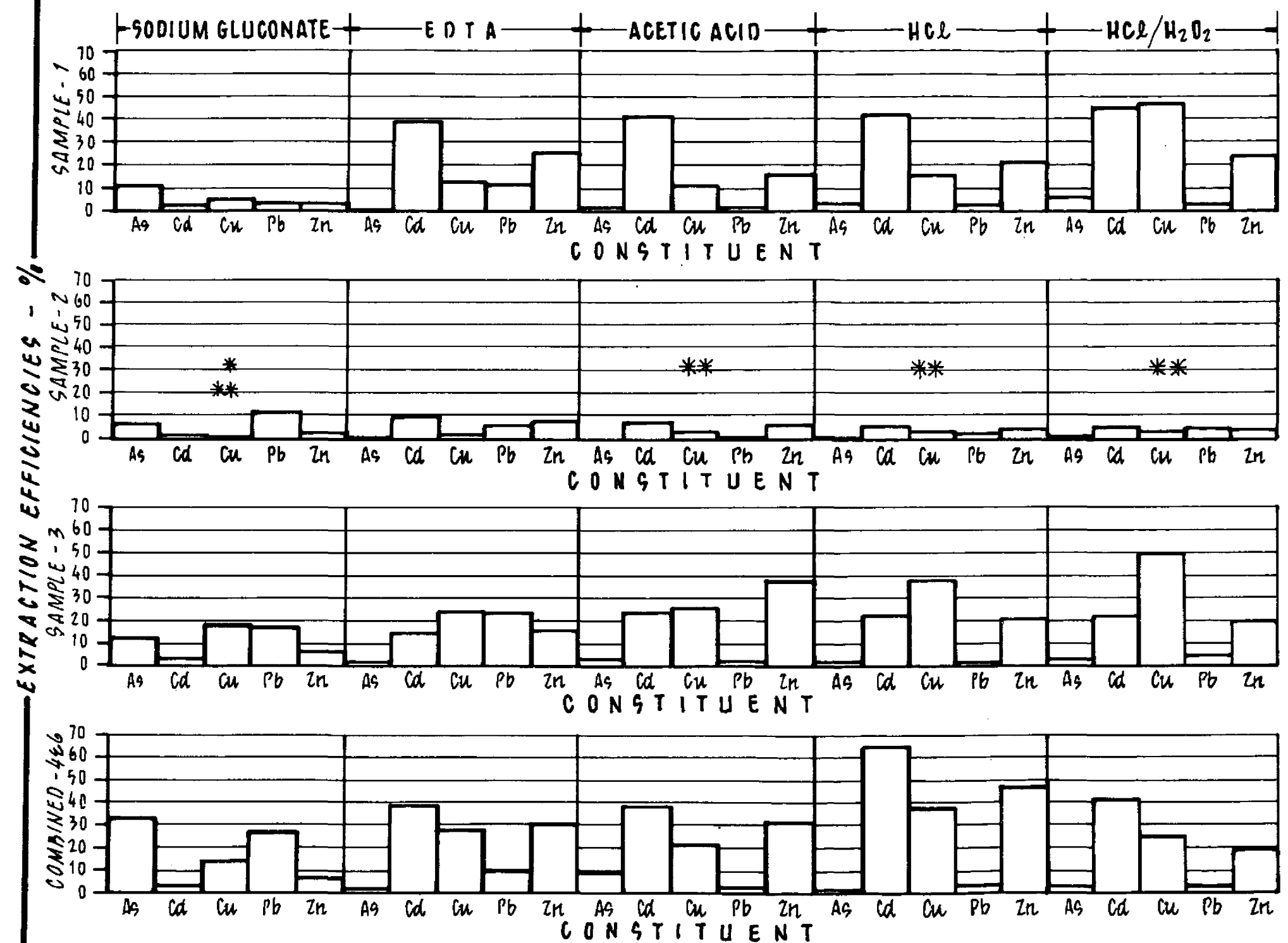
CROSS HATCHED AREAS REPRESENT ADDITIONAL OR LESSER REMOVAL OBTAINED WITH PULVERIZED SAMPLES.

1. EXTRACTION EFFICIENCIES SHOWN ARE AVERAGES OF DATA REPRESENTED IN TABLES 3-8 THROUGH 3-12.

2. EXTRACTIONS WITH HOT HCL WERE NOT PERFORMED ON THIS SOIL SAMPLE

3. ARSENIC WAS NOT ANALYZED IN THE PULVERIZED EXTRACTED SOIL

**Figure 3-1**  
**SUMMARY OF**  
**EXTRACTION EFFICIENCIES**  
**CELTOR CHEMICAL WORKS**  
**No. 10 PASSING**



**NOTES & LEGEND**

\* INDICATES THAT SOIL SAMPLE WAS INITIALLY ABOVE CLEAN UP CRITERIA FOR CONSTITUENT (FOR CLARITY ONLY THE SODIUM GLUCONATE EXTRACTANT GROUP IS INDICATED).

\*\* INDICATES THAT CONSTITUENT WAS REMOVED IN SUFFICIENT QUANTITIES TO BRING SOIL FROM ABOVE TO BELOW CLEAN UP CRITERIA FOR THIS CONSTITUENT. (THIS MAY HAVE ONLY OCCURED IN ONE OF TWO OR THREE EXTRACTIONS. DATA MAY INDICATE LOW PERCENTAGE REMOVALS WHICH MAY MEAN THAT THE ACTUAL SAMPLE TESTED WAS BELOW CLEAN UP CRITERIA INITIALLY - SEE TEXT ALSO).

1. EXTRACTION EFFICIENCIES SHOWN ARE AVERAGES OF DATA REPRESENTED IN TABLES 3-8 THROUGH 3-12.

Figure 3-2  
SUMMARY OF  
EXTRACTION EFFICIENCIES  
CELTOR CHEMICAL WORKS  
No.10 to 1 inch



(pulverized sample), and zinc 22.0 percent. Note that with some exceptions, primarily for lead, removals using EDTA showed the same overall patterns, and in many instances, percent removals similar to the remaining extractants (acetic acid, HCl, hot HCl, and HCl/H<sub>2</sub>O<sub>2</sub>).

In the two cases that showed removals to below action levels (lead in Sample 1 and zinc in Combined Samples 4 and 6), other data such as percentage removal and mass balance indicate that the actual samples tested may have initially been below cleanup levels or that the percentage recovery of the metal was not 100 percent.

#### Acetic Acid Extractant

This extractant was uniformly poor in removing arsenic and lead (greatest removals were 1 percent for arsenic in Sample 1 and 0.1 percent for lead in Sample 2 and Combined Samples 4 and 6). Removals of arsenic, cadmium, copper, and zinc were similar to those using EDTA, except for copper in Sample 5. Copper removal in Sample 5 using EDTA was approximately two and one-half times greater than the removal using acetic acid (approximately 46 percent vs 20 percent). The only removals to below cleanup levels were for lead in Sample 1. Mass balance and percentage removal data do not confirm significant removals for lead in this instance.

#### Hydrochloric Acid

As with acetic acid, removals with hydrochloric acid were uniformly poor for arsenic and lead (greatest removals were 2 percent in Sample 1 and 5.5 percent for Combined Samples 4 and 6, pulverized). The removal patterns for hydrochloric acid and acetic acid were similar, although there were some differences in actual percentages removed. Some significant removal differences between pulverized and unpulverized samples are also evident. Note that the large difference between average zinc removals in the pulverized versus unpulverized data for Combined Samples 4 and 6 is the result of the 27 percent removal shown for the second extraction on this sample (Table 3-11). Also note that the mass balance closure for this second extraction is poor (0.21).

In only one instance was a contaminant removed to below cleanup levels (lead for Sample 1), and, as was the case for previous lead removals, mass balance and percentage removal data indicate minor removals.

#### Hot Hydrochloric Acid

The major removals obtained by hot HCl were for lead and cadmium (as high as 48.7 percent for lead in Combined Samples 4 and 6 and 38 percent for cadmium in Sample 1).

However, cadmium removals were similar to ambient temperature HCl removals. Lead removals were significantly higher than for ambient HCl (as high as 44 times). Removals of copper and zinc were as high as 17.5 and 15 percent, respectively. Arsenic analyses were not performed for this extraction.

Removal to below cleanup levels was obtained for lead (Sample 1) and zinc (Combined Samples 4 and 6). Note that other data for lead and zinc in these extractions show poor mass balance closure, indicating that the sample may have started out below action levels, or that percentage recovery of the metals was not 100 percent.

#### Hydrochloric Acid/Hydrogen Peroxide

The patterns of removal with this extractant were quite similar to those of HCl; however, mass balance closure data do not corroborate high zinc removals (mass balance levels were as low as 0.1 percent). Removals to below cleanup levels were obtained for lead (Sample 1) and copper (Sample 1), although percentage removals were minor (<10 percent).

#### SUMMARY OF EXTRACTIONS ON NO. 10-TO-1-INCH PORTION

##### Sodium Gluconate Extractant

Extraction with this chemical showed uniformly poor removals for both cadmium and zinc (greatest removals were 4.6 percent for cadmium in Sample 1 and 13.3 percent for zinc in Sample 2). Removals for arsenic were as high as 33.6 percent, copper as high as 18.3 percent, and lead as high as 27.9 percent. Although final soil concentration data indicate a removal to below cleanup levels for copper in Sample 2, percentage removal data and mass balance data do not indicate large removals.

##### EDTA Extractant

This extractant showed poor removals of arsenic for all soil samples (greatest removal was 1 percent). Removals of all other metals varied considerably, depending on the particular soil sample. Maximum removals were cadmium 39.5 percent, copper 28.2 percent, lead 23.9 percent, and zinc 30.1 percent. No removals to below cleanup levels were obtained.

##### Acetic Acid Extractant

Removals with this extractant were uniformly poor for both arsenic and lead (greatest removals were 3.6 percent for arsenic in Sample 3 and 3.7 percent for lead in Combined

Samples 4 and 6.). Removals for cadmium, copper, and zinc were as high as 41, 27.3, and 38.5 percent, respectively. Removal to below cleanup levels was obtained for copper on Sample 2, although mass balance data and percent removal data do not indicate significant removals.

#### HCl Extractant

As with the acetic acid extractant, HCl was uniformly poor in removing arsenic and lead (greatest removals were 5 percent for arsenic in Sample 1 and 4.5 percent for lead in Sample 3). Removals of cadmium, copper, and zinc were as high as 64.3, 37.8, and 47.9 percent, respectively. Removal to below cleanup criteria were apparently obtained for copper (Sample 2). However, mass balance data indicate that the sample may have been below cleanup levels initially.

#### HCl/H<sub>2</sub>O<sub>2</sub> Extractant

The overall pattern of removal for this extractant was quite similar to that of HCl, although percentage removals differed substantially in some cases. Removals of arsenic and lead were uniformly poor (greatest removals were 10 percent for arsenic in Sample 1 and 6.9 percent for lead in Sample 3). Removals of cadmium, copper, and zinc were as high as 44, 48.5, and 23 percent, respectively. Copper was below cleanup levels in Sample 2, but mass balance data do not corroborate large removals.

RD/R16/029

## Chapter 4 DISCUSSION OF RESULTS

### SUMMARY

Removals of contaminants were marginal for nearly all metals, soils, and extractants. In very few cases were any soils treated successfully (reduction of a particular metal from above cleanup level to below cleanup level). There were no cases where more than one metal in a soil sample was reduced from above to below cleanup level. It therefore appears that none of the extractants used would be effective in producing a soil which would be below cleanup level in all metals of concern (if the soil were initially above cleanup level in more than one metal). The soil remaining after extraction would be nearly the same, or greater, volume than initially (because of added water). The extracted soil would remain to be dealt with as a hazardous waste, requiring one of the remaining actions outlined in the RI/FS for the site.

Of the five extractants used in this study, sodium gluconate was the only high pH extractant used. This extractant in the alkaline form has the ability to remove the four cationic metals (cadmium, copper, lead, and zinc) by binding with the chelant portion of the gluconate, and can remove arsenic by binding with its alkali portion, provided these constituents are not bound in other forms that make them chemically insoluble. The remaining four extractants are capable of extracting the cationic metals as long as they are not bound in insoluble forms (especially sulfide). The rationale for using these extractants has been covered in an earlier chapter.

While some extraction was achieved by all solutions, these removals were not adequate to meet cleanup levels. It appears from these test results that the metals are present primarily as complicated sulfides which are not soluble unless they are oxidized under severe conditions. The mining industry relies on roasting and high pressure, high temperature aqueous slurry oxidation to extract metals from sulfide ores. Although the material was not specifically analyzed for sulfides, the mining history and study results suggest strongly that the soils contained significant amounts of sulfides. The limited extraction that was observed was most likely the result of slow oxidation processes and weathering that have taken place on the exposed surfaces of the individual particles (acidic runoff from the site is evidence of this). This weathering makes metals at the particle surface susceptible to chemical extraction, while leaving the sulfide-rich interior unaltered and the sulfide-bound metal unextractable.

Examination of results on the pulverized soil versus unpulverized soil extractions tend to corroborate this conjecture, as removals of the two are quite similar. In other words, exposing the interior, unoxidized portions of the soil does not significantly increase extraction. A notable exception to this occurred for lead (which was extracted in approximately 3 to 5 times the quantity in pulverized versus unpulverized tests for all but the hot hydrochloric acid extractants). Lead removals were still minor in all cases that showed a significant difference between pulverized and unpulverized extractions (less than 15 percent). Also, note that for the hot hydrochloric acid extraction on Soil Sample 1, more zinc was extracted on the pulverized sample, and for the EDTA extraction on combined Soil Samples 4 and 6, less copper was extracted on the pulverized sample. There is no apparent explanation for this last phenomenon. (Samples whose mass balance did not close within 75 percent of unity are not included in the above discussion.)

In general, extractions with hot hydrochloric acid showed better or equal removals compared to extractions at ambient temperatures. The most dramatic example of this was for lead. The best efficiency for lead extraction at ambient temperatures was 5.5 percent, whereas with hot HCl, the best extraction efficiency was almost 50 percent and averaged 38.5 percent. This phenomenon is to be expected as most sulfides are more soluble at higher temperatures. (As in the discussion in the preceding paragraph, samples whose mass balance did not close within 75 percent of unity were not considered here.)

### CONCLUSIONS

Results of attempted extraction of five metals (arsenic, cadmium, copper, lead, and zinc) from five different soil types from the Celtor Chemical Works waste were generally unsuccessful. None of the extractants were capable of producing a soil below cleanup level for all metals if the soil was initially above cleanup level in more than one metal. The most likely reason for the failure of the extractants to perform better is that a majority of the metals in the soils are in insoluble sulfide forms. What little extraction was obtained was probably due to solubilization of weathered, oxidized surface areas of ore particles.

Extraction is not recommended as a treatment alternative for the Celtor Chemical Works wastes. Treatment of this nature would result in high capital and O&M costs, leaving the site and soils in substantially no better condition than they are at present.

Appendix A  
DESCRIPTION OF INITIAL SOIL CHARACTERIZATION

Approximately 150 to 200 pounds of each of the soil samples shown in Table 2-1 were obtained at the Celtor site. Each sample consisted of three to four 50-pound bags. Figure 2-1 is a map of the site showing sample locations.

Each of the soil samples was spread out on plywood or sampling pans and allowed to dry for 24 hours. After this drying period, the samples were broken up by hand to reduce the size of the clods, but leave all rock intact. Where necessary to obtain an adequate amount of sample, bags of soil were thoroughly mixed to form a homogeneous composite. A riffle sampler was then used to split off a representative portion for crushing, digestion, and subsequent analysis to determine the metals content of the raw, unsegregated samples. (The riffle sampler splits any sample that is put in it into two equal, representative portions.) To obtain a sample small enough to be easy to work with and pulverize, the riffle sampler was used to split the original sample in half, then the half sample into quarters, etc., until the desired sample size was produced. The openings in the riffle sampler used are approximately 1-1/2 inches wide by 12 inches long. In all soil samples but Sample 1, the riffle sampler passed the entire sample.

For Sample 1, to obtain a representative portion for crushing, digestion, and subsequent analysis, the weights of the amount passing and not passing the riffle sampler were obtained. When the portion split off for digestion was obtained by passing it through the riffle sampler several times, it was then weighed, and a proportionate amount of the "larger than riffle sampler" portion was added to it.

The sample crushing for digestion and metals analysis consisted of pulverizing the sample to No. 200 and smaller.

Each soil sample (1, 2, 3, etc.) was initially sieved into the following gradations:

- o 1 inch and larger
- o 1 inch to No. 4
- o No. 4-passing

The minus No. 4 portion was sieved into splits of minus No. 10 and plus No. 10 portions. The plus No. 10 portion was then combined with the 1-inch-to-No. 4 portion, resulting in a combined portion consisting of all the No. 10-to-1-inch particles of the sample. The entire sample was not initially sieved through a No. 10 screen because of equipment limitations.

Next, both the 1-inch-retained and the No. 10-to-1-inch portions were washed with deionized water and the fines retained for inclusion into the No. 10-passing portion. The washwater was allowed to settle for 1 to 2 hours and the supernatant decanted (a portion of the supernatant was saved for analysis of total solids and metals for later incorporation into a mass balance). Decanting of the supernatant was necessary to obtain quick drying of the washwater solids so they could be incorporated into the No. 10-passing solids, to allow extraction testing to proceed because of the necessity to complete testing quickly. After the dry washwater solids were blended into the No. 10-passing solids, a portion of this size fraction was split off from each soil for digestion and analysis.

Examination of the No. 10-to-1-inch and 1-inch-retained solids after the initial washing showed a small but significant portion of attached soil and fines. Because of these fines, both of these portions were washed and dried a second time. The fines from these washings were not saved, but final weighing of both size fractions (No. 10-to-1-inch and 1-inch-retained) was obtained after drying to determine the weight of fines removed. The 1-inch-retained portion was then pulverized for analysis. Part of the No. 10-to-1-inch portion was also split off for pulverizing and analysis.

Before the second washing, a fraction of the No. 10-to-1-inch sample was split off for sieving to characterize it in terms of particle size distribution and metals content. The sieves used were No. 3, No. 6, and No. 10. Each of the portions retained was weighed and then bagged for pulverizing and metals analysis. As might be expected, a portion of this No. 10-to-1-inch fraction passed the No. 10 sieve. The portion passing the No. 10 sieve was weighed but not analyzed. Figure A-1 is a schematic of the processing used for the soils characterization.

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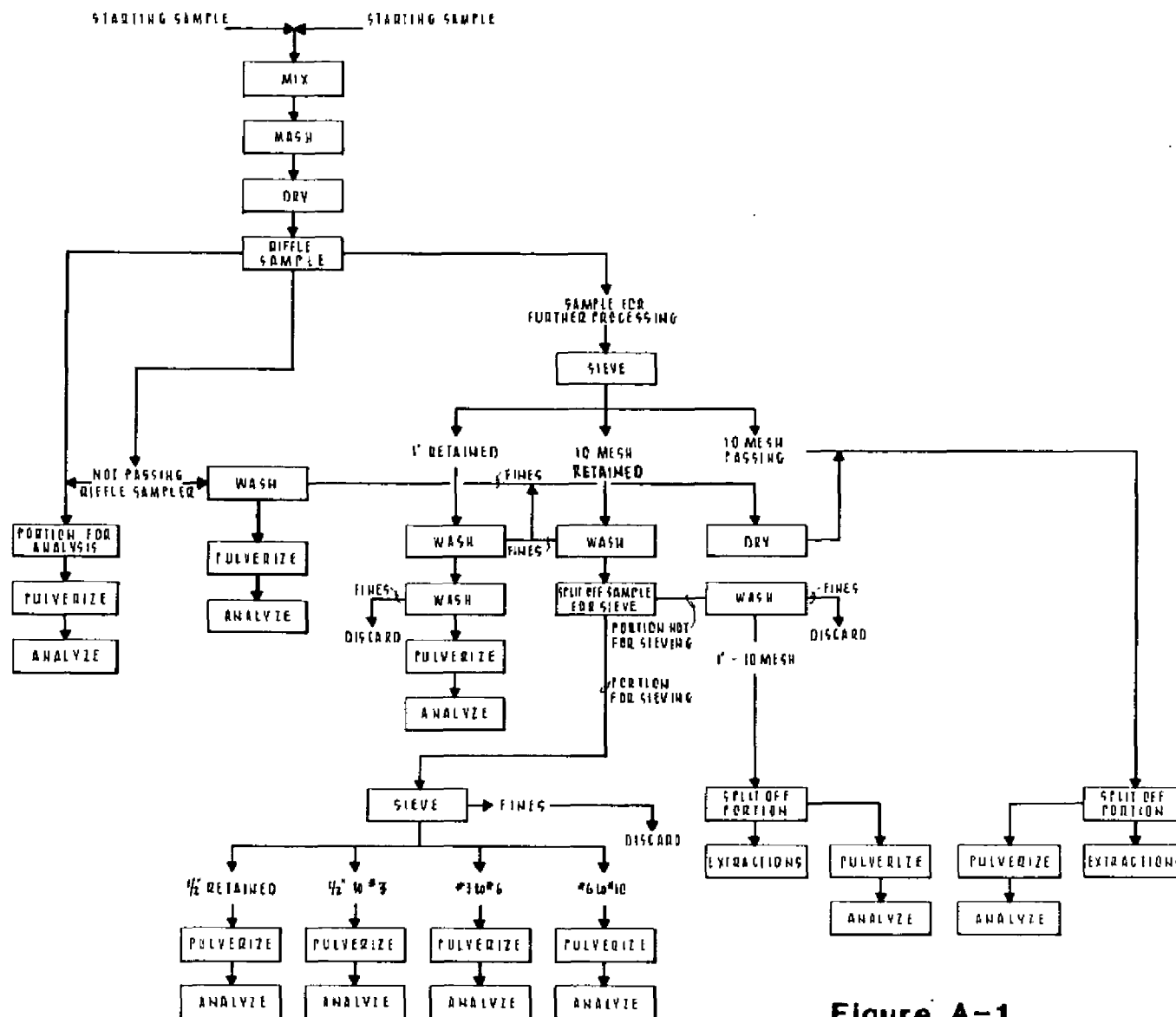


Figure A-1

# SCHEMATIC OF CELTOR CHEMICAL WORKS SOIL SAMPLE PROCESSING



Appendix B  
DETAILED DESCRIPTION OF LEACHING PROCEDURE  
AND EVALUATION OF EXTRACTION

EXTRACTION OF NO. 10-PASSING SOIL

To perform each analysis on the No. 10-passing portion, a 100-gram soil sample was first weighed and placed into a 250 ml Nalgene bottle. To assure that samples tested were representative of the bulk mass, portions to make up the 100-gram sample were taken from different areas of the bulk sample container. All weighings were done to an accuracy of  $\pm 0.1$  gram.

Two hundred milliliters of the extractant solution were added to the bottle which was then capped and placed on a shaker table for 1 hour. After the 1-hour shaking, each bottle was set aside and its contents allowed to settle until the settling rate had slowed significantly (approximately 1/2 hour to 1 hour).

The supernatant from these samples was centrifuged to remove fines and allow for easier filtration in subsequent steps. The supernatant from the centrifuge tubes was vacuum-filtered through a 0.45-micron filter and the filtrate acidified and set aside for analysis of As, Cd, Cu, Pb, and Zn.

The solids from the original shaker bottle (containing residual extractant solution) as well as the solids from the centrifuge tubes (also containing residual extractant solution) were washed with deionized water in a pressure filter apparatus using a piece of prefilter paper. After the initial filtration was finished, an additional 200 ml of deionized water was used to wash the solids. No attempt was made to quantitatively recover the solids from the extraction; rather, as much of the solid phase as was easily recoverable was saved. We estimate that the amount of solids recovered by this process was greater than 95 percent.

This procedure was followed for all samples except the samples extracted with sodium gluconate. These samples were extremely difficult to vacuum-filter, so an alternate procedure was followed with these. The entire sample was initially filtered in the pressure filter on 0.45-micron paper to collect the extractant. The solids from the pressure filtration were then transferred to a piece of prefilter paper and processed as described above.

The rationale for using a 0.45-micron filter for the extractant supernatant and the second water wash for the solids was to obtain as total a separation of the phases as possible. It was thought that this procedure would help to

ensure closure of a mass balance between the liquid- and solid-phase metals and the starting sample. Additional water washes of the solid phase were considered, but were rejected because of the excessive amount of time this would have required (some pressure filtrations took up to an hour to complete).

After filtration, the soil sample was placed in an oven to dry. After drying, the sample was crushed to No. 200 and finer and then digested and analyzed for total metals (see main text for appropriate EPA methods).

#### EXTRACTION OF NO. 10-TO 1-INCH SOIL

Processing of the No. 10-to-1-inch solids followed a procedure similar to that of the No. 10-passing solids with the following differences:

- o The extracted sample supernatant was not centrifuged after shaking; rather, it was filtered directly. Because the residue on the filter paper contained significant fines, these fines were washed back into the solids that were to be pressure-filtered.
- o In weighing out the 100-gram solids sample, it was apparent that obtaining a representative, uniform sample would be extremely difficult because of the heterogeneity of the sample. To help alleviate this difficulty, for each soil type, a sample of approximately 1,000 grams was obtained from the No. 10-to-1-inch portion (enough for 10 bottles, or duplicates for one of the five soils). The larger pieces in the 1,000-gram sample were then separated out and distributed approximately evenly to each of the 10 sample bottles. The bottles were then "topped off" to 100 grams with the smaller mesh particles of the remaining soil.

After separation of the solid and liquid phases, the liquid phase pH was measured. The color of the extractant solutions was also compared between duplicates. Any of the No. 10-to-1-inch duplicate samples that showed obvious color differences or showed pH variations of more than 0.1 unit were rerun to obtain a third analysis. Figure B-1 is a schematic process diagrams of the leaching process.

#### SECOND PHASE EXTRACTIONS

After the majority of data was obtained in the previous phase of leaching experiments, CH2M HILL, SAIC, and EPA

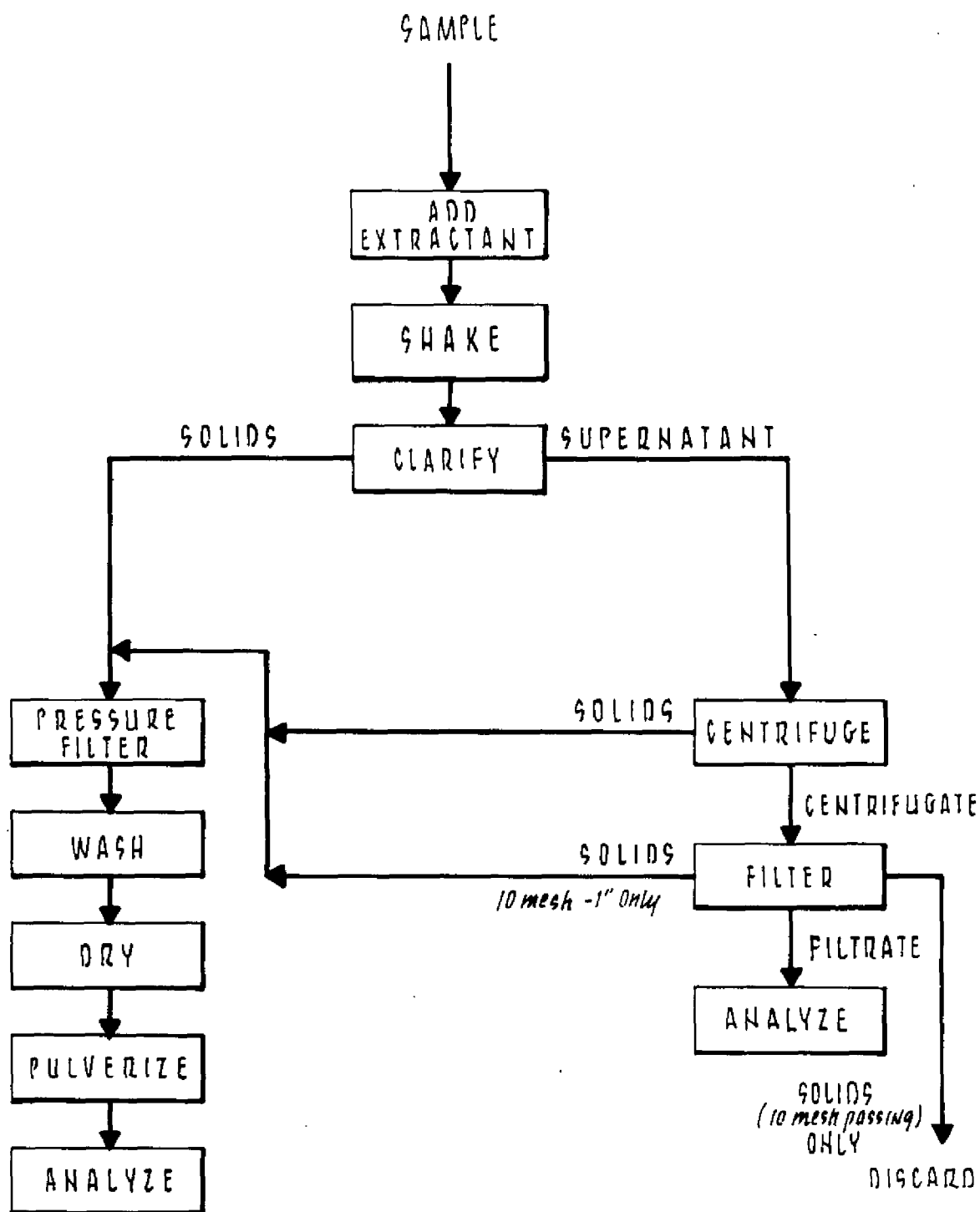


Figure B-1

**SCHEMATIC OF CHEMICAL  
LEACHING PROCESSING OF  
CELTOR CHEMICAL WORKS  
SOILS (Unheated extractants)**

participated in a conference call to determine the procedure for the next phase of studies, if any. This conference call occurred on August 2, 1985.

Because of the generally poor performance of all the chemicals on all the soils, it was decided not to proceed with any optimization studies as originally envisioned (see "Results" section for a more detailed description of results of extractions). Rather, it was decided to perform additional extractions with some of the more promising chemicals, with modifications to the process to increase the extraction efficiencies. The following additional extractions were performed:

- o Extraction of the No. 10-passing fraction of Sample 1 and Combined Samples 4 and 6 with EDTA (13 percent by weight, pH = 7.0) after the soil sample had been pulverized to finer than No. 200.
- o Extraction of the above soil samples (again after pulverizing to No. 200 and finer) with HCl (pH = 0.8, approximately 0.16 molar)
- o Hot HCl (pH = 0.8, approximately 0.16 molar) extraction of the No. 10-passing fractions of Sample 1 and Combined Samples 4 and 6 (unpulverized)
- o Hot HCl (pH = 0.8, approximately 0.16 molar) extraction of the No. 10-passing fractions of Sample 1 and Combined Samples 4 and 6 (pulverized)

The extractions using EDTA and unheated HCl were performed in essentially the same manner as the original extractions on the No. 10-passing samples, with the following differences:

- o Twenty-five grams of soil sample were used.
- o Two hundred fifty milliliters of extractant were added to each sample initially.
- o The supernatant from each sample was not centrifuged, as the solid and liquid phases separated fairly well.
- o Initial pH of HCl was 0.8.

The hot HCl extractions were performed as follows:

- o Twenty-five grams of sample were weighed into a 400 ml beaker.

- o The HCl extractant was heated to 150°F (initial pH was 0.8), and then 250 ml of the hot HCl were added to the 400 ml beaker using a graduated cylinder.
- o As soon as acid had been added to all samples, stirring of each sample was initiated to maintain all solids in suspension.
- o A hot plate stirrer was used to maintain the temperature of each solution between 135°F and 155°F. (Only one hot plate stirrer was available, so each of the samples was switched between the hot plate and standard stirrers to maintain its temperature within the limits noted.)
- o After 1 hour of mixing and heating, each sample was taken off its stirrer and allowed to clarify.
- o Two hundred milliliters of the supernatant from each sample were decanted and filtered through a 0.45-micron filter. The filtrate was saved for later analysis of As, Cd, Cu, Pb, and Zn.
- o Two hundred milliliters of fresh HCl were added to each solid sample and the above heating and stirring repeated.
- o Approximately 200 ml of the second extractant solution were filtered through a 0.45-micron filter and the filtrate saved for later analysis.
- o The solid portion was pressure-filtered and washed with two successive 200 ml/portions of deionized water.
- o The solid phase was dried for later digestion and analysis (the unpulverized No. 10-passing sample was pulverized before analysis).

See Figure B-2 for a schematic of these extractions.

#### EXTRACTION SUMMARY

##### EVALUATION OF EXTRACTION OF SAMPLE 1

This soil is above cleanup level for all metals in the No. 10-passing portion. For the No. 10-to-1-inch portion, the soil is below cleanup level for all metals.

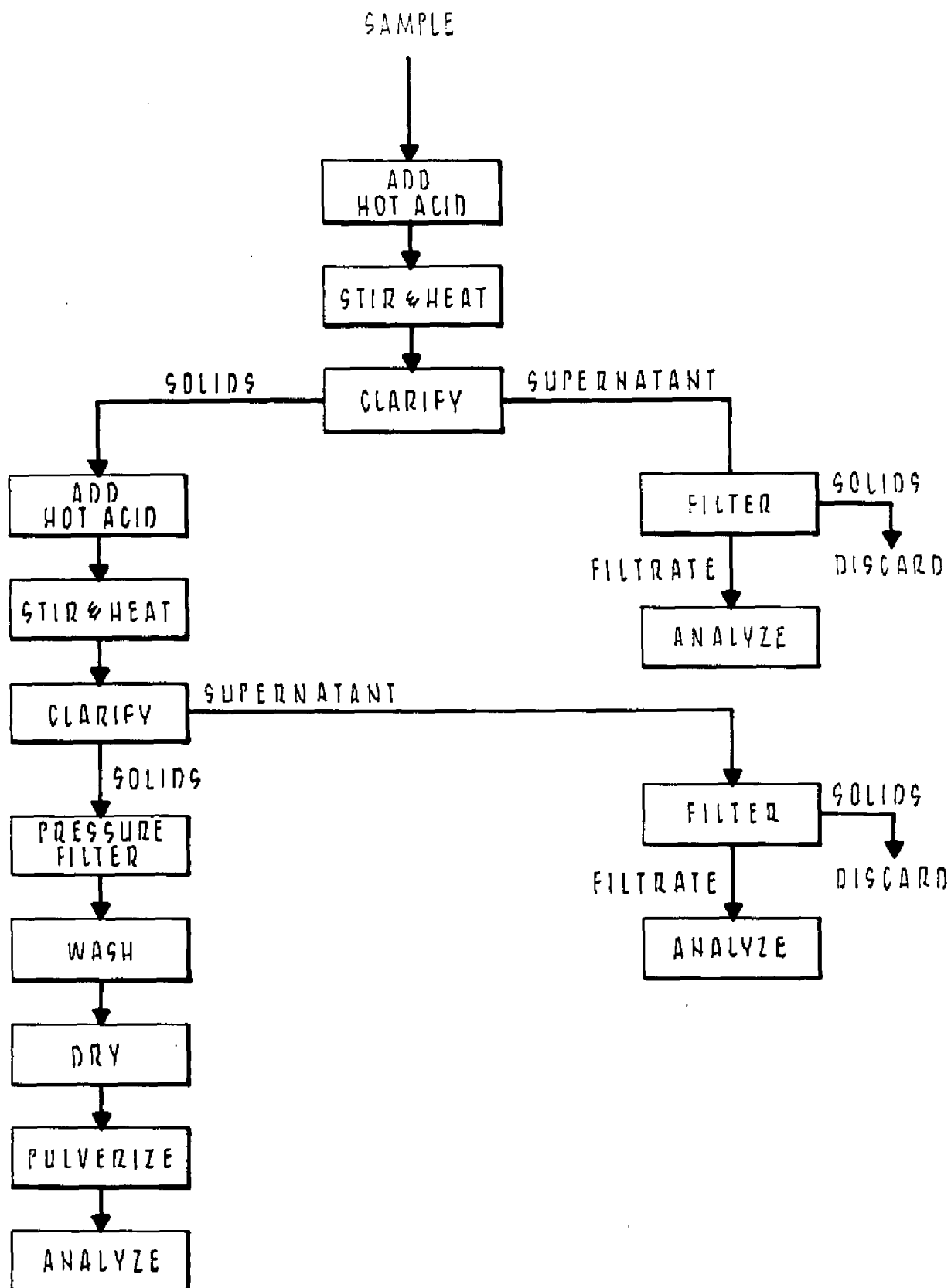


Figure B-2  
SCHEMATIC OF CHEMICAL  
LEACHING PROCESSING OF  
CELTOR CHEMICAL WORKS SOILS (Hot acid)

### Extraction with Sodium Gluconate

On the No. 10-passing portion, this extractant showed high removals of lead only (48 percent) with reduction of lead to approximately one-half its cleanup level. Removal of arsenic was as high as 18 percent, although mass balance closure for arsenic was 81 percent at best, and concentrations of arsenic in the soil were not below cleanup level after extraction. Removals of cadmium, copper, and zinc were relatively minor (all less than 10 percent), and concentrations of these constituents were above cleanup level after extraction.

For the No. 10-to-1-inch portion of this soil, no significant removals of any of the five constituents were obtained. The best removal was that of arsenic which was a maximum of 11 percent.

### Extraction With EDTA

Extractions were performed on both pulverized and unpulverized samples. Comparing the pulverized and unpulverized samples, it can be seen that efficiency of extraction of all metals were similar except for lead. Arsenic was not measured on the pulverized sample. Removal of lead was approximately 4 percent in the unpulverized sample and 12 percent in the pulverized sample. Removals of cadmium, zinc, copper, and arsenic were approximately 30 percent, 10 percent, 6 percent, and 1 percent, respectively. Lead was the only metal below cleanup level after extraction, although removal efficiency data do not indicate that a major portion was extracted.

Data on the No. 10-to-1-inch portion show significant removals of cadmium and zinc (approximately 40 and 24 percent, respectively) while removal of the remaining three metals were all 12 percent or less (arsenic removal was only one percent).

### Extraction With Acetic Acid

Extraction of the No. 10-passing portion with this solution showed approximately 30 percent cadmium removal, but the soil was still above cadmium cleanup level after extraction. Removals of the remaining metals were minimal, 10 percent or less for all instances except the value of 11 percent for zinc. Although the value for lead in the extracted soil is below the cleanup level, liquid phase concentrations and extraction efficiencies indicate little removal.

Data for the No. 10-to-1-inch portion indicate that some removal of Cd, Cu, and Zn was obtained with little removal of As and Pb. Removal of Cd was as high as 43 percent.

Again, the levels of metals in this portion of the sample were all below cleanup level initially.

#### Extraction With HCl (Ambient Temperature)

For the No. 10-passing portion, extractions were performed on both pulverized and unpulverized samples. Results for these samples were similar (arsenic was not analyzed in the pulverized sample). Cadmium removal ranged from 32 to 41 percent, zinc removal from 11 to 16 percent, and copper from 9 to 13 percent. Although removal of lead was four to five times greater for the pulverized sample than the unpulverized, it was nevertheless only 4 to 5 percent.

Removal of arsenic was minimal (arsenic was not tested for in the pulverized sample). No metals were removed to below cleanup level for either the pulverized or unpulverized sample, except for two instances for lead; and removal efficiency does not indicate large removals of lead.

The data on the No. 10-to-1-inch portion indicate that 41 to 43 percent of soil Cd was removed, along with 17 to 24 percent of zinc and 11 to 19 percent of copper. Five percent or less of arsenic and lead was removed.

#### Extraction with Hot HCl

These extractions were performed on the No. 10-passing portions for both pulverized and unpulverized samples. If results are compared between the pulverized and unpulverized samples, removal of all metals appear to be similar. Lead was the only metal below cleanup level after extraction (arsenic was not measured). In general, it appears that more Zn is removed in Extraction 2 than in Extraction 1.

Although a greater percentage of cadmium was removed compared to lead, cadmium was still above cleanup level after extraction. Approximately 17.5 percent of the copper was removed from the soil, and 6 percent to an average of 15 percent of the zinc was removed in extraction of the unpulverized and pulverized samples, respectively.

#### HCl/H<sub>2</sub>O<sub>2</sub> Extractant

For the No. 10-passing portion, this extractant removed approximately 33 percent of cadmium and approximately 10 percent or less of all other metals. Note the similarities in extraction efficiencies between this extractant and the HCl (ambient temperature) extractant. In general, removal to below cleanup level was not obtained. The soil was slightly below cleanup level in copper after extraction.



Data for the No. 10-to-1-inch portion show some variation, but in general, removals were significant for cadmium only (average 44 percent removal), although the third extraction showed a 41 percent removal of zinc. Average removal of copper was 15 percent, and removals of arsenic and lead were both less than 10 percent.

#### EVALUATION OF EXTRACTION OF SAMPLE 2

In Sample 2, As, Cu, and Pb were above cleanup level in the No. 10-passing portion. For the No. 10-to-1-inch portion, Cu was the only metal found to be above cleanup level.

#### Extraction with Sodium Gluconate

Extractions on the No. 10-passing portion of Sample 2 showed little removal of any metals (less than approximately 6 percent). Note that in general the mass balances for this extraction do not close within 10 percent, with some exceptions. No metals were removed to below cleanup level.

For the No. 10-to-1-inch portion, removals were also generally minor, and although copper was removed to below its cleanup level on the average (avg concentration Cu = 2,490 mg/kg), liquid phase copper concentrations and percentage removal data do not corroborate large removals.

#### Extraction with EDTA

Removals for all metals for both the No. 10-passing portion and the No. 10-to-1-inch portions were minor (the largest removal was 15.5 percent for cadmium on the No. 10-to-1-inch portion). No removals to below cleanup level were obtained.

#### Extraction with Acetic Acid

Extraction efficiency for all metals was minor on both the No. 10-passing and the No. 10-to-1-inch portions (approximately 14 percent maximum). Again, no removals to below cleanup level were obtained, with the exception of copper in the No. 10-to-1-inch portion, and liquid phase and percentage removal data do not corroborate significant removals for copper.

#### Extraction with HCl

Removals on both the No. 10-passing and the No. 10-to-1-inch portions were minor (the maximum removal was 14.5 percent for zinc). Although lead was below cleanup level on the second extraction on the No. 10-passing portion and copper was below cleanup level on the second extraction for the No. 10-to-1-inch portion, mass balance, liquid phase and extraction efficiency data would indicate that these samples were probably below cleanup level initially.

#### Extraction with HCl/H<sub>2</sub>O<sub>2</sub>

Again, removals for all metals on both mass fractions were apparently minor (greatest removals were of cadmium and zinc in the No. 10-passing fraction, both at 15.1 percent). Although copper was below cleanup level for the extraction on the No. 10-to-1-inch portion, the mass balance, liquid phase, and extraction efficiency data again indicate that the starting sample was probably initially below the cleanup level.

#### EVALUATION OF EXTRACTION OF SAMPLE 3

Sample 3 contained no metals concentrations that were above cleanup level for the No. 10-passing and No. 10-to-1-inch portions.

#### Extraction with Sodium Gluconate

For the No. 10-passing portion, significant removals of lead, copper, and arsenic were obtained (average of 43.6, 29.3, and 21.2 percent, respectively). Removals of cadmium and zinc were less than 5 percent.

Removals varied significantly for the extractions performed on the No. 10-to-1-inch portion. Some significant removals of lead (up to 51 percent), copper (up to 26.8 percent), and arsenic (up to 22 percent) were obtained. Removals of cadmium and zinc were not significant (less than 14 percent).

#### Extraction with EDTA

Moderate removals were obtained on the No. 10-passing portion for copper, cadmium, and zinc (average removal of 28.6, 18.5, and 14.1 percent, respectively). Removals of arsenic and lead were small.

In general, there were wide variations in removals between the two extractions for the No. 10-to-1-inch portion. Average removals were copper--23.9 percent; lead--23.4 percent; cadmium--13.6 percent; zinc--15 percent; and arsenic--0.6 percent.

#### Extraction with Acetic Acid

For the No. 10-passing portion, moderate removals were obtained for copper, cadmium, and zinc (approximately 26.6, 15.4, and 15.4 percent, respectively). Little arsenic and lead were removed.

As with sodium gluconate, removals varied significantly between extractions for the No. 10-to-1-inch portion.

Removals of zinc, cadmium, and copper were as high as 76 percent, 40 percent, and 37 percent, respectively. Removals of arsenic and lead were small.

#### Extraction with HCl

Moderate removals were obtained on the No. 10-passing portion of copper, cadmium, and zinc (32.5, 23.4, and 18.1 percent, respectively). Removals of arsenic and lead were small.

Removals for the No. 10-to-1-inch portion were similar to the No. 10-to-1-inch passing portion, although significant variation was noted between extractions.

#### Extraction with HCl/H<sub>2</sub>O<sub>2</sub>

Moderate removals were obtained on the No. 10-passing portion for copper, cadmium, and zinc (average removals of 36.2, 25.2, and 18.6 percent, respectively). Removals of arsenic and lead were minor.

There were some variations in removals between extractions for the No. 10-to-1-inch portion, with moderate removals of copper, cadmium, and zinc (average removals of 48.5, 20.4, and 19.2 percent, respectively). Removals of arsenic and lead were small, less than 5 percent.

#### EVALUATION OF EXTRACTION OF COMBINED SAMPLES 4 AND 6

Of the No. 10-passing fraction, the Combined Samples 4 and 6 were above action limits in all metals except for Pb. Of the No. 10-to-1-inch portion, none of the metals were above cleanup level. The same extractions performed on Sample 1 were also performed on this sample, including the hot acid and pulverized sample extraction.

#### Extraction with Sodium Gluconate

On the No. 10-passing portion, major removals of lead (50 to 51 percent) and moderate removals of arsenic (15 to 17 percent) were obtained. Removals of the remaining metals were all less than 5 percent, and no removals to below cleanup level were obtained.

For the No. 10-to-1-inch portion, moderate removals of arsenic, lead, and copper were obtained (average removals of 33.6, 27.9, and 14.3 percent, respectively). Removals of cadmium and zinc were less than 10 percent.

#### Extraction with EDTA

As with Sample 1, extractions were performed on both pulverized and unpulverized samples of the No. 10-passing portion of the soil. Arsenic was not analyzed in the extractions on the pulverized portion. Removal of lead was about 2 to 3 times greater in the pulverized versus unpulverized portions, averaging 56.1 and 22 percent, respectively. Removals were similar for cadmium between pulverized and unpulverized samples, averaging about 5 to 6 percent. Except for the apparently anomalous first extraction on the unpulverized portion, removals of zinc were similar on both portions, averaging slightly above 5 percent. Removal of copper was approximately 2 to 3 times greater in the unpulverized than in the pulverized portion, averaging 6 and 2.4 percent, respectively. Removal of arsenic was less than 1 percent. No removals to below cleanup level were obtained, except in one case with zinc.

For the No. 10-to-1-inch portion, minor to moderate removals of all metals but arsenic were obtained (averages for cadmium, copper, zinc, and lead were 38.3, 28.2, 30.1, and 11 percent, respectively). Removal of arsenic was less than 1 percent.

#### Extraction with Acetic Acid

Removals of metals were all approximately 5 percent or less on the No. 10-passing portion, and no removals to below cleanup level were obtained.

On the No. 10-to-1-inch portion, moderate removals were obtained for cadmium, zinc, and copper (average removals of 38.6, 31.0, and 22.1 percent, respectively). Removals of lead and arsenic were less than 4 percent and 1 percent, respectively.

#### Extraction with HCl (Ambient Temperature)

As with Sample 1, for the No. 10-passing portion, extractions were performed on both pulverized and unpulverized samples. Results for the pulverized and unpulverized samples were generally similar. Generally, removals were approximately 6 percent or less, with the exception of zinc in the second extraction on the unpulverized sample, which showed a removal of 27 percent. Note that for this extraction, the amount of zinc in the soil after extraction is shown to be below action limits. Whether this sample was initially above or below cleanup level in zinc or there is a possible error in analysis is unknown. Note that lead removal for the pulverized sample was approximately 4 to 5 times greater than for the unpulverized sample, as was the case with Sample 1. Except for the case of zinc, no apparent removals to below cleanup level were obtained.

For the No. 10-to-1-inch portion, high to moderate removals of cadmium, zinc, and copper were obtained (average of 64.3, 48.0, and 36.6 percent, respectively). Removals of arsenic and lead were less than 3 percent.

#### Extraction with HCl (Hot)

As with Sample 1, extractions were performed on both pulverized and unpulverized samples of the soil. Arsenic was not analyzed in these extractions. Results for both the pulverized and unpulverized portions are quite similar, with the exception of the value for zinc on the second extraction of the pulverized sample. The value for zinc in the extracted soil for this sample indicates that the sample may have started out low in zinc or that the analysis may be incorrect. Removals were all approximately 10 percent or less, except for lead, of which approximately 44.7 percent was removed, on the average. No removals to below cleanup level, except in the one case of zinc, were obtained.

#### Extraction with HCl/H<sub>2</sub>O<sub>2</sub>

For the No. 10-passing portion, removals of all metals were minor (maximum removal of less than 7 percent). No removals to below cleanup level were obtained.

On the No. 10-to-1-inch portion, moderate removals of cadmium, copper, and zinc were obtained (average of 40.8, 26.2, and 19.6 percent, respectively). Removals of lead and arsenic were less than 3 percent.

#### EVALUATION OF EXTRACTION OF SAMPLE 5

Sample 5 was above cleanup level in lead and arsenic.

#### Extraction with Sodium Gluconate

Moderate removals of copper and arsenic were obtained (average of 23.4 and 20.9 percent, respectively). Removals of cadmium, lead, and zinc were minor, all being less than 8 percent. Removal to below cleanup level was not obtained for any metal.

#### Extraction with Acetic Acid

The only moderate removal obtained with acetic acid was with copper (average of 46.2 percent). Removal of all other metals was less than approximately 4 percent. Removal to below cleanup level was not obtained for any metal.

### Extraction with HCl, EDTA, and HCl/H<sub>2</sub>O<sub>2</sub>

As with acetic acid, the only moderate removal achieved was on copper (average removal between 17 and 22 percent). Removal of all other metals was less than 7 percent, and removal to below cleanup level was not obtained for any metal.

### SUMMARY OF EXTRACTION PERFORMANCE

In the following section, a brief summary of the effectiveness of each extractant is presented. In this discussion we have limited examination of extractant performance to those soils that had metals which were initially above cleanup level and to the unpulverized samples. This decision was made in the interest of simplifying this summary and because the metals and soils that are above cleanup level are of greatest concern.

#### Sodium Gluconate

This extractant performed best on arsenic and lead (maximum removals of approximately 21.2 and 48 percent, respectively). Removal of cadmium, copper, and zinc were poor, maximum removals being less than 10 percent.

#### EDTA

EDTA appeared to be effective for cadmium removal only, and its maximum extraction efficiency was 30 percent. Note that the analysis for zinc on the first extraction of the unpulverized sample of Combined Samples 4 and 6 was not included here because of questionable data.

Comparing extraction efficiencies for the pulverized and unpulverized samples shows little difference in efficiencies except for the case of copper and lead.

#### Acetic Acid

Acetic acid was most efficient in the extraction of cadmium, the maximum removal being 31 percent. Removals of all other metals were 10 percent or less.

#### Hydrochloric Acid (Ambient Temperatures)

This extractant was similar to acetic acid in its efficiency of extraction. Maximum removal of cadmium was 41 percent. Removals of all other metals were poor, less than 15 percent. The analysis of zinc for the second extraction on the No. 10-passing portion of Combined Samples 4 and 6 was not considered in this summary because of questionable data.

Comparison of extraction on the pulverized and unpulverized samples shows similar efficiencies for all metals except lead (arsenic was not analyzed for the pulverized sample). Although lead removal was significantly higher for the pulverized samples, it was still below 6 percent in all cases.

#### Hot Hydrochloric Acid

The use of hot HCl greatly improved efficiency of lead extraction and improved copper and zinc extraction efficiency somewhat. Pulverizing the sample seemed to improve zinc extraction efficiency slightly, but did not seem to improve extraction of any other metals greatly.

Maximum removals for lead, cadmium, zinc, and copper were 50, 37, 44, and 18 percent, respectively. The analysis for zinc on the second extraction of the pulverized sample was not included here because of questionable data.

#### HCl/H<sub>2</sub>O<sub>2</sub> Extractant

As was the case with acetic acid and hydrochloric acid, cadmium was the only metal removed to any significant extent. Its maximum removal was 33 percent.

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