

**BENCH SCALE STUDY OF A  
SEQUENTIAL METAL LEACHING  
SYSTEM TO REMOVE METALS FROM  
WELLAND RIVER SEDIMENTS**

**Final Report**

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## 1.0 INTRODUCTION

Contaminated sediments can serve as long-term sources of pollution affecting regional water quality even after regulated discharges are controlled or eliminated. Various studies have shown that contaminants are being released from sediments at a number of locations in the Great Lakes basin. These releases are exceeding environmental criteria to an extent that use impairment likely exists. In 1987, the Great Lakes Water Quality Board reported to the International Joint Commission that 41 of the previously identified 42 Areas of Concern contained contaminated sediments that were releasing chemicals into the Great Lakes Basin.

The Contaminated Sediment Treatment Technology Program (CoSTTeP) is one of three separate but coordinated sediment programs sponsored by the Great Lakes Cleanup Fund. The objective of CoSTTeP is to assess sediment treatment technologies by sponsoring a series of technology demonstration projects to treat sediments from five areas of concern. The areas of concern are:

- Hamilton Harbour;
- Toronto Harbour;
- Welland River;
- St. Mary River at Sault Ste. Marie; and
- Thunder Bay Harbour.

The remediation of sediments contaminated by elevated concentrations of priority metals faces several key challenges. Firstly, priority metals are generally not biotransformed and elevated concentrations of certain metals may be toxic to microorganisms. The success of bioremedial strategies to clean up sediments containing elevated concentrations of priority metals may be limited. Elevated metal concentrations may also limit the success of

physical treatment technologies such as incineration as ash may contain metal concentrations which still exceed disposal guidelines.

Beak Consultants Limited (BEAK) was contracted by the Wastewater Technology Centre, with funds provided by the Environment Canada Great Lakes Cleanup Fund, to demonstrate the efficacy of a sequential metal leaching system to remove priority metals from sediments. Removal of metals from sediments involves:

- leaching metals from sediment surfaces into an aqueous solution using inorganic and organic acids,
- separation of the solids and extraction solution(s) by physical methods such as gravity separation, filtration or centrifugation,
- capture and removal of metals from the extraction solution(s) by means of recoverable chelators or resins, and
- recovery of the metals and regeneration of the extraction solutions, if appropriate.

The sequential metal leaching system can be used as a stand-alone treatment for metal-contaminated sediments or in conjunction with other treatment technologies (*i.e.*, to remove organic contaminants) as part of an overall treatment chain.

Brief reviews of the theories and technical considerations for each step of the metal extraction procedure are provided in Sections 1.1 to 1.4. The methods employed during the bench-scale study of the sequential metal leaching system are provided in Section 2.0. Results and interpretation of the bench-scale study are presented in Section 3.0. Optimization of the sequential metal leaching technique, through use of a validated

chemical speciation model, is discussed in Section 4.0. Section 5.0 provides a design and cost analysis for the sequential metal leaching system at full-scale. Parameters which control extraction efficiency and process cost at full-scale are also addressed in Section 5.0.

## 1.1 Metal Leaching from Sediment Surfaces

Organic and mineral colloids (clays) in sediments possess charged surfaces that bind metals and metalloids. Surface charges can be attributed to the permanent negative charge associated with layered silicates or can be attributed to pH-dependant charges associated with surface functional groups (*i.e.*, carboxyl (COOH) or hydroxyl (OH) groups). In general, pH-dependant surface functional groups are positively charged at low solution pH and negatively charged at high solution pH.

Leaching metals from sediment surfaces requires breaking the bond between surface charges (mineral surfaces or surface functional groups) and metal ions either by exchange, desorption or dissolution. The leaching method depends on the type of surface charge and the speciation of the metals to be extracted. Metal speciation in solution depends on solution pH, ionic strength, competitive ions, the presence of complexing organic ligands and the properties of the solid phases in the heterogeneous mixture. The most important solution parameter is pH. At high pH, metals may precipitate as oxides and hydroxides. At low pH, the target metal exists as free ions or oxyanions.

Weakly bound, exchangeable metal species (*e.g.*, neutral salt-extractable species) and labile metal species are generally mobile and are easily leached from sediments. Metal species that are adsorbed, precipitated or occluded to a specific solid phase (*e.g.*, the association of manganese and iron oxides with organic matter) may be difficult to leach from sediments.



The leaching of metals is accomplished by sequentially passing extraction solutions (generally inorganic and organic acids) through the sediments. Extraction solutions can be combined as a "cocktail" and applied to sediments or can be passed through the sediments sequentially to leach bound metals. The sequential approach has been used to release metal ions which are tightly bound to clays (Ferrah and Pickering, 1978; Misra and Tiwari, 1966; Ramamoorthy and Kushner, 1978; and Ramamoorthy and Rust, 1975).

A primary concern in devising metal leaching strategies is the selection of extraction solutions that are effective in solubilizing a given metal species without solubilizing excessive quantities of competitive ions. Selective solubilization using pure extraction cocktails or sequences is generally used to leach metals from sediments to solution. To obtain maximum yield, extraction solutions should be selected to target specific speciation processes and selectively solubilize target analytes into the aqueous phase in forms that are easily separated or extracted.

Hydrochloric acid will leach metal ions from most sediment surfaces. The kinetics of the reaction can be controlled by the concentration of the acid and by the contact time. Initially, weakly bound species (soluble and exchangeable) will be liberated, followed by co-precipitated species, occluded and precipitated species. It is likely that the highest concentration of target metals will be surface adsorbed or precipitated. The use of hydrochloric acid as an extraction solution is beneficial as the metal species obtained from the extraction are predominantly in free ionic form and can be separated by ion exchange resins. In addition, hydrochloric acid can be regenerated from the process.

## **1.2 Separation of Solids and Extraction Solutions**

Sediment slurries can be dewatered and separated into solid and liquid phases by filtration or by gravity separation using a corrugated plate separator, a hydrocyclone, or a centrifuge. The water stream containing dissolved metals and very fine particulates (those not removed

by the separation procedure) can then be treated to recover and regenerate the extraction solutions. The selection of a separation method will depend on the desired stringency of separation and on the size distribution of the sediment targeted for recovery. Project cost and duration will be influenced by the efficiency of the separation process.

### **1.3 Removal of Metals from Extraction Solutions**

Leached metals can be captured and removed from extraction solutions by either physical (*e.g.*, centrifugation, ion exchange) or chemical (*e.g.*, chelation) methods. Chemical removal is generally more cost-effective as it requires fewer analytical steps.

Ethylenediamine tetraacetic acid (EDTA), ethylenediamine dehydroacetic acid (EDDHA), and diethyltriamine pentaacetic acid (DTPA) are multidentate ligands with varying selectivity to transition metals. These agents complex metals in extraction solutions to varying degrees in response to their concentration and the pH of the solution. These chelating agents are best used when bound to surfaces of ion exchange resins. Metals are preferentially retained on the resins where the chelators are present as surface functional groups. Upon completion of the process, the chelator can be desorbed from the metal group by acid washing and the surface-bonded chelate can be regenerated.

#### **1.3.1 Selection of Extraction Solutions**

The principle of Hard and Soft Acid and Bases (HSAB) can be used to direct the selection of extraction solutions and chelating agents. Metals are defined as Lewis acids because they can accept electrons and coordinate covalent bonding. Hard Lewis acids are defined as small cations with high oxidation number and low electronegativity. Conversely, soft Lewis acids are large cations with low oxidation number and high electronegativity. Surface sites on phyllosilicates and soluble ligands represent Lewis bases that can donate electrons to form a chemical bond. Interactions of hard Lewis acids and bases generally

result in the creation of an ionic bond. Interactions of soft Lewis acids and bases generally result in the creation of a covalent bond.

The extraction process and final ion exchange separation can be seen as a series of acid-base reactions. Aqueous metals (Lewis acids), and sediment and chelator surface sites (Lewis bases) can be classified according to their hardness and softness. By providing the appropriate chelator surface groups, target metals can be removed from aqueous solutions and the effects of competition (by other metals and metalloids) for chelator surface groups can be minimized.

#### **1.4 Recovery of Metals and Regeneration of Extraction Solutions**

The most promising option for recovery of metals and regeneration of extraction solutions involves the use of ion-specific chelation resins. These resins provide effective removal of target metals with minimum interference by competitive non-target metals. The resins also limit extraction solution fouling by various physical and chemical agents.

Ion-exchange chelation resins such as Amberlite IRC-718 (Rohm and Haas) have a high affinity for heavy metal cations relative to alkali or alkaline earth metals and may be used to remove metals from acid solutions. The removal of metals from the extraction solutions allows the reuse of the solutions in the staged sediment washing process. The resin may be backwashed in place to remove suspended particles that may not be removed during the separation of solids. The metals may be recovered from the resin using an electrowinning process or a conventional precipitation process. Information on the IRC-718 resin is presented in Appendix A.

Competitive species may interfere with ion-exchange, and physical or chemical agents may foul ion-exchange or chelating resins. As such, it may be more effective to regenerate the extraction solution by chemical precipitation. Metals that are solubilized by acidic

extraction solutions generally precipitate with base addition (*i.e.*, lime or NaOH with  $\text{CaCl}_2$ ). Several advantages favour the use of chemical precipitation to regenerate extraction solutions. The process is applicable to a wider range of operating conditions, is less sensitive to physical and chemical interferences, and is not adversely affected by the presence of fine sediment particulates. Chemical precipitation may also remove low levels of non-metallic contaminants such as polynuclear aromatic hydrocarbons (PAHs), which may be present in the extraction solution. However, the presence of oil, often associated with contaminated sediments, may generate emulsions which create operational difficulties.

Extraction solutions may also be regenerated by distillation. Using distillation, metals are concentrated in a smaller volume of extraction solution for wastewater treatment or metal recovery. Regenerated extraction solutions can be reused in the staged sediment washing process.

## 2.0 METHODS

This section presents the technical scope of work for the bench-scale sequential metal leaching study.

### 2.1 Sediment Collection and Preparation

The Wastewater Technology Centre (WTC) provided two sediment samples collected from the Welland River in St. Catharines, Ontario for sequential metal leaching. Welland River sediments were selected because they contain concentrations of copper (Cu), chromium (Cr), iron (Fe), lead (Pb), manganese (Mn), nickel (Ni), and zinc (Zn) which approach and/or exceed Ontario Ministry of Environment (MOE) Severe Effects Levels for sediment contamination.

Samples tested included an unsorted bulk sediment and a sample of sorted sediments that were nominally less than 75  $\mu\text{m}$  in size. Sediment sorting in the latter sample was not perfect as sediments greater than 75  $\mu\text{m}$  were present in the sample. Prior to processing, both sediment samples were allowed to settle and overlying water was collected and discarded. Each sediment was thoroughly homogenized and a subsample was collected for analysis of metals.

### 2.2 Metal Extraction Procedure

A metal leaching treatment and a control were prepared for each sediment sample. The experimental system consisted of 250 g aliquots of wet sediment in 500 mL polyethylene centrifuge bottles. The exact wet weights of the sorted and bulk sediment samples, before and after treatment, are provided in Table 3.1.

Four extraction solutions were used to sequentially leach metals from the sediment samples. The composition of each of the extraction solutions, in order of use is as follows:

- deionized water (Milli-Q),
- 1 M ammonium acetate adjusted to pH 5 with acetic acid,
- 0.1 M hydrochloric acid (pH 1.2), and
- 0.82 M ammonium citrate/0.15 M EDTA adjusted to pH 8.5 with sodium hydroxide.

Each treatment sample was washed four times with each of the four extraction solutions. For each wash, 200 mL of the extraction solution was added to the sediment in the centrifuge bottle and the sample was rotated end over end for 30 minutes on a box tumbler (10 rpm). The sample was then centrifuged for 15 minutes at 1500 rpm with an IEC Centra-8 centrifuge. The pH of the supernatant was measured and the supernatant was decanted into a graduated cylinder. The volume of supernatant recovered was recorded (Tables 3.2A,B and 3.4A,B) and an aliquot was pressure filtered through a 0.45  $\mu$ m cellulose-acetate membrane filter in a 1000 mL teflon lined housing. The filtered extract was preserved with nitric acid and was analyzed for metals to determine the extraction efficiency of each extraction solution.

The control samples were processed in the same manner as the treatment samples except that equal volumes of deionized water were substituted for the extraction solutions.

### 2.3 Laboratory Analysis

Samples were analyzed by BAS Laboratories in Brampton, Ontario using sequential ICP spectroscopy (JY inductively coupled plasma spectrophotometer) for aluminum (Al), beryllium (Be), cadmium (Cd), chromium (Cr), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), nickel (Ni), silver (Ag), thallium (Tl), and zinc (Zn).

Arsenic (As), antimony (Sb) and selenium (Se) were analyzed using a Perkin Elmer 3030 graphite furnace atomic absorption spectrophotometer with Zeeman background correction. Sediments and extracts that had formed a precipitate upon preservation, were digested with aqua regia before analysis.

Sediment analysis was performed on wet samples. Results are reported on an equivalent dry weight basis.

## **2.4 Equipment Cleaning Procedures**

All extraction vessels, sample bottles, and reagent bottles were soaked for 24 hours in 5% hydrochloric acid and were rinsed repeatedly with deionized water before use.

## **2.5 Chemicals**

All chemicals used to prepare the extraction solutions were American Chemical Society (ACS) grade or better. The concentrations of metals in the extraction solutions was determined to ensure that metal concentrations observed during sequential leaching were attributable to the leaching process. Table 2.1 presents the results of the extraction solution analysis. Ammonium acetate, acetic acid, hydrochloric acid, ammonium citrate, EDTA, and sodium hydroxide were obtained from Fisher Scientific Limited (Toronto, ON).

### 3.0 RESULTS AND INTERPRETATION

Several changes in the proposed metal leaching system design were made to improve the metal leaching procedure. The design of the metal leaching system initially called for sediment columns. The column concept was abandoned at the beginning of the study as it was deemed impractical to engineer a sediment column treatment system. In addition, the metal leaching efficiency would likely be reduced by the poor contact between the extraction solutions and the sediments in the column. The column system was replaced with a simplified sediment washing technique that provided better mixing of the sediments with the extraction solutions.

Sediment samples were split and were analyzed by both BAS Laboratories and WTC. Laboratory reports of analyses for both BAS Laboratories and WTC are presented in Appendix B. The initial concentration of each metal in the sorted and bulk sediment samples prior to treatment is presented in Table 3.1. Results of the bench-scale metal leaching study are presented for the sorted sediment sample and the unsorted bulk sediment sample in Sections 3.1 and 3.2, respectively. A discussion of the extraction efficiencies between the sorted and bulk samples is provided in Section 3.3.

#### 3.1 Sorted Sediments

Metal extraction results for each of the extraction solutions in the sequential extraction procedure are discussed in Sections 3.1.1 to 3.1.5. The efficiency of the extraction solutions is compared and discussed in Section 3.1.6. Silver, selenium, and thallium were not detected in the sorted sediment sample prior to treatment and thus are not discussed. Tables 3.2A and 3.2B present the cumulative mass of metals recovered in the filtered extraction solutions for the full sequential extraction and the control, respectively. The measured aqueous concentrations of metals and the calculated mass of metals in the filtered extraction solutions are presented in Appendix C.



### 3.1.1 Deionized Water

The cumulative mass of metals recovered in the filtered treatment and control extraction solutions (both deionized water) were similar. Beryllium, cadmium, and lead were not leached by deionized water. Chromium was not leached by deionized water in the treatment but was leached in the first wash of the control. Significant masses of iron, magnesium, manganese, and nickel were leached in both the treatment and control, with masses generally decreasing with each successive wash. Antimony, arsenic, copper, and zinc were leached by deionized water, however no trends were observed with the successive washing. The mass of aluminum leached by deionized water generally increased with each successive wash. After four successive washings with deionized water, the highest cumulative mass of metal leached was 5,460  $\mu\text{g}$  for magnesium. Cumulative metal extraction masses for iron, manganese, and aluminum followed at 479, 318, and 110  $\mu\text{g}$ , respectively.

The pH of the filtered extraction solution increased in each successive washing with the pH increasing from 6.92 to 7.82 in the final wash.

### 3.1.2 1 M Ammonium Acetate (pH 5.0)

Significant masses of metals were recovered in the filtered ammonium acetate treatment as compared to the control and the previous deionized water treatment. Beryllium was not leached by ammonium acetate. The leached masses of aluminum, arsenic, chromium, copper, lead, and zinc generally increased with each successive wash. The leached masses of manganese and magnesium decreased with each successive wash. Antimony, cadmium, iron, and nickel were leached by ammonium acetate, however no trends were observed with successive washings. After four successive washings with each of ammonium acetate and deionized water, the highest cumulative mass of metal leached was 198,659  $\mu\text{g}$  for iron. Cumulative metal extraction masses for aluminum and magnesium were significantly

lower at 28,740 and 25,103  $\mu\text{g}$ , respectively. Significant leaching of both magnesium (38% of treatment) and copper (123% of treatment) was measured in the control.

The pH of the filtered extraction solution decreased in each successive washing with the pH decreasing from 5.51 to 5.13 in the final wash.

### 3.1.3 0.1 M HCl

Significant masses of metals were generally recovered in the filtered dilute hydrochloric acid treatment as compared to the control. Beryllium was not leached by dilute hydrochloric acid. The leached masses of cadmium, copper, iron, manganese, magnesium, and zinc generally increased with each successive wash. The leached masses of lead, chromium, arsenic, and antimony decreased with each successive wash. Nickel, and aluminum were leached by dilute hydrochloric acid, however no trends were observed with successive washings. After four successive washings with each of dilute hydrochloric acid, ammonium acetate and deionized water, the highest cumulative mass of metal leached was 371,183  $\mu\text{g}$  for iron. Cumulative metal extraction masses for magnesium and aluminum followed at 157,853 and 56,594, respectively. Significant leaching of magnesium (70% of treatment) copper (88% of treatment), and antimony (36% of treatment) was measured in the control.

The pH of the filtered extraction solution decreased in each successive washing with the pH decreasing from 4.88 to 4.39 in the final wash.

### 3.1.4 Ammonium citrate-EDTA (pH 8.5)

Significant masses of metals were generally recovered in the filtered ammonium citrate-EDTA treatment as compared to the control. Beryllium was not leached by ammonium citrate-EDTA. The leached masses of aluminum, antimony, cadmium, iron, lead,

manganese, magnesium, and zinc generally decreased with each successive wash. Only the mass of copper showed a general increase with successive washings. Arsenic, chromium, and nickel were leached by ammonium citrate-EDTA, however no trends were observed with successive washings. After four successive washings with each of ammonium citrate-EDTA, dilute hydrochloric acid, ammonium acetate and deionized water, the highest cumulative mass of metal leached was 534,295 µg for iron. Cumulative metal extraction masses for aluminum and magnesium followed at 327,904 and 188,354 µg, respectively.

The pH of the filtered extraction solution increased in each successive washing with the pH increasing from 7.71 to 8.48 in the final wash.

### 3.1.5 Final Deionized Water Rinse

Significant masses of metals were recovered in the filtered final deionized water rinse as compared to the control and previous extraction solutions. Beryllium was not leached by the final rinse. Leached masses of antimony, arsenic, cadmium, copper, iron, nickel, and zinc were higher in the filtered final rinse than for any other extraction solution.

Aluminum, chromium, lead, magnesium, and manganese were also leached during the final rinse. Upon completion of the sequential leaching process, the highest cumulative mass of metal leached was 634,255 µg for iron. Cumulative metal extraction masses for aluminum and magnesium followed at 406,444 and 201,206 µg, respectively.

### 3.1.6 Extraction Efficiency

Table 3.3 summarizes the total percentage of each metal leached by each extraction solution in the full sequential leaching procedure. The removal of metals attributable to the loss of fines has been calculated based upon the total metal removal observed minus the total leaching observed. These results also appear in Table 3.3.

The efficiency of the overall sequential leaching system was greatest for lead (44.0%), zinc (32.5%), and cadmium (27.1%). Beryllium was not extracted as a result of the sequential leaching process. Selenium, silver, and thallium were not detected in the initial sediment analyses. As such, no comment can be made as to their potential removal from sorted sediments.

The greatest percent mass of aluminum, antimony, arsenic, cadmium, chromium, copper, lead and zinc was leached by the ammonium citrate-EDTA extraction solution. The greatest mass of iron, manganese, and nickel was leached with the ammonium acetate extraction solution. Magnesium was leached largely by the dilute hydrochloric acid solution. While the final deionized water rinse was effective at leaching metals, the initial deionized water wash leached a maximum of only 0.1 to 0.4%.

The mass of metals associated with fines was greatest for iron (43.9%), lead (26.2%), and arsenic (23.8%). Beryllium, while not removed by the sequential leaching process, was removed (10.4%) as a result of the loss of fines.

### 3.2 Bulk Sediments

Metal extraction results are discussed for each of the extraction solutions in the sequential extraction procedure and are presented in Sections 3.2.1 to 3.2.5. The efficiency of the extraction solutions is compared and discussed in Section 3.2.6. Tables 3.4A and 3.4B present the cumulative mass of metals recovered in the filtered extraction solutions for the full sequential extraction and the control, respectively. The measured aqueous concentrations of metals and the calculated mass of metals in the filtered extraction solutions are presented in Appendix C.

### 3.2.1 Deionized Water

The cumulative mass of each metal recovered in the filtered extraction solutions was higher in the control than the treatment. Beryllium, cadmium, lead, selenium, silver, and thallium were not leached by deionized water in either the control or treatment. Antimony and chromium were not leached in the treatment but were leached in the control.

Aluminum, arsenic, copper, iron, manganese, magnesium, nickel, and zinc were leached in both the treatment and control. The leached masses of metals generally decreased with each successive washing. After four successive washings with deionized water, the highest cumulative mass of metal leached was 1,548 µg for magnesium. Cumulative metal extraction masses for iron and aluminum followed at 286 and 306 µg, respectively.

The pH of the filtered extraction solution increased in each successive washing with the pH increasing from 7.10 to 7.91 in the final wash.

### 3.2.2 1 M Ammonium Acetate (pH 5.0)

Significant masses of metals were recovered in the filtered ammonium acetate treatment as compared to the control and the previous deionized water treatment. Antimony, beryllium, copper, silver, and selenium were not leached by ammonium acetate. The leached masses of aluminum, chromium, lead, and zinc generally increased with each successive wash.

The leached masses of cadmium, iron, magnesium, manganese, nickel, and thallium generally decreased with each successive extraction. Arsenic was also extracted by ammonium acetate, however no trend was observed during the successive washings. After four successive washings with each of ammonium acetate and deionized water, the highest cumulative mass of metal leached was 312,726 µg for iron. Cumulative metal extraction masses for magnesium manganese, and nickel were significantly lower at 36,741, 12,611, and 7,536 µg, respectively.

The pH of the filtered extraction solution decreased in each successive washing with the pH decreasing from 5.47 to 5.21 in the final wash.

### 3.2.3 0.1 M HCl

Significant masses of metals were generally recovered in the filtered dilute hydrochloric acid treatment as compared to the control. Antimony, beryllium, copper, selenium, silver, and thallium were not extracted by dilute hydrochloric acid. The leached masses of aluminum, arsenic, iron, manganese, magnesium, nickel, and zinc generally increased with each successive wash. Cadmium, chromium and lead were extracted by dilute hydrochloric acid, however no trends were observed with successive washings. After four successive washings with each of dilute hydrochloric acid, ammonium acetate and deionized water, the highest cumulative mass of metal leached was 525,626 µg for iron.

The pH of the filtered extraction solution decreased in each successive washing with the pH decreasing from 4.95 to 4.32 in the final wash.

### 3.2.4 Ammonium citrate-EDTA (pH 8.5)

Significant masses of metals were generally recovered in the filtered ammonium citrate-EDTA treatment as compared to the control. Beryllium, selenium, silver, and thallium were not extracted by ammonium citrate-EDTA. The leached masses of aluminum, antimony, arsenic, cadmium, chromium, iron, lead, manganese, magnesium, nickel, and zinc generally decreased with each successive wash. Only the mass of copper showed a general increase with successive washings. Antimony was extracted by ammonium citrate-EDTA, however no trend was observed with successive washings. After four successive washings with each of ammonium citrate-EDTA, dilute hydrochloric acid, ammonium acetate and deionized water, the highest cumulative mass of metal leached was 641,334 µg

for iron. Cumulative metal extraction masses for magnesium and aluminum followed at 208,619 and 47,757 µg, respectively.

The pH of the filtered extraction solution increased in each successive washing with the pH increasing from 7.91 to 8.55 in the final wash.

### 3.2.5 Final Deionized Water Rinse

Significant masses of metals were recovered in the filtered final deionized water wash as compared to the control and previous extraction solutions. Beryllium, selenium, silver, and thallium were not leached by the final deionized water wash. Leached masses of arsenic, cadmium, copper, and zinc were higher in the filtered final wash than for any other extraction solution. Aluminum, antimony, chromium, iron, lead, manganese, and nickel were also extracted during the final wash. Upon completion of the sequential leaching process, the highest cumulative mass of metal leached was 687,432 µg for iron. Cumulative metal extraction masses for magnesium and aluminum followed at 221,021 and 58,053 µg, respectively.

### 3.2.6 Extraction Efficiency

Table 3.5 summarizes the total percentage of each metal leached by each extraction solution in the full sequential leaching procedure. The removal of metals attributable to the loss of fines has been calculated based upon the total metal removal observed minus the total leaching observed.

The efficiency of the overall sequential leaching system was greatest for cadmium (61.6%), lead (38.8%), zinc (33.8%), antimony (29.7%), and manganese (25.8%). Beryllium, selenium, silver, and thallium were not extracted as a result of the sequential leaching process.

The greatest mass of aluminum, arsenic, antimony, cadmium, chromium, copper, lead, and zinc was leached by the ammonium citrate-EDTA extraction solution. The greatest mass of iron, manganese, and nickel was leached with the ammonium acetate extraction solution. Magnesium was leached largely by the dilute hydrochloric acid solution. While the final deionized water rinse was effective at leaching metals, the initial deionized water rinse leached only 0.1% of arsenic and magnesium.

In a number of cases, the loss of metals attributable to the loss of fine sediments was equal to the mass of metals extracted by the full sequential extraction. In particular, beryllium was not removed from the sediments by the extraction process, however significant beryllium removal (55.5%) was observed as a result of the loss of fines.

### **3.3 Comparison of Sediment Type on Extraction Efficiency**

The following trends in metal extraction can be drawn from the metal leaching efficiencies summarized in Tables 3.3 and 3.5:

- Initial metal concentrations were higher in the sorted sediment sample, with the exceptions of beryllium, selenium, silver, and thallium;
- The total removal of metals from sediments was higher in the sorted sediment sample, with the exceptions of beryllium, chromium, and nickel;
- The loss of fines was higher in the sorted sediment sample; and
- Leaching efficiencies were higher in the bulk sediment sample for arsenic, antimony, cadmium, chromium, iron, magnesium, manganese, nickel, and zinc.



Figures 3.1 to 3.12 illustrate the effects of changing extraction solutions on the removal of metals from the sorted and bulk sediments and provide a comparison of metal leaching between the sorted and bulk sample.

#### 4.0 THEORETICAL OPTIMIZATION OF THE SEQUENTIAL METAL LEACHING SYSTEM USING MINTEQA2

The bench-scale study of the sequential metal leaching system yielded metal removal efficiencies which were lower than anticipated. The chosen concentration of the hydrochloric acid did not sufficiently decrease the solution pH to a level optimizing the leaching of metals from sediments. In addition, it was determined that chromium and the form of copper ( $\text{Cu}^+$ ) encountered in the sediment environment were not effectively complexed by EDTA.

In order to determine the optimal efficiency of the sequential metal leaching system prior to designing a full-scale treatment system, the results of the bench-scale sequential metal leaching study were modelled using the chemical speciation model, MINTEQA2 (EPA, 1992). The kinetics of metal desorption were investigated by adjusting the concentrations of hydrochloric acid to determine the optimal pH for the release of bound metals. The efficiency of complexation was tested using different concentrations of EDTA to determine the optimal EDTA concentration for recovering released metals. Leaching efficiency was tested in the presence of a mineral iron oxide and an organic surface to determine the effect of surfaces found in the sediment material. Potential pre-treatments to increase complexation of copper and chromium were also investigated.

Optimal conditions for removing selected regulated metals, as determined through the theoretical optimization process, are presented in Table 4.1. The optimization process revealed that:

- Decreasing the pH of the extraction solution increased the extraction efficiency without decreasing the efficiency of chelation. At decreased pH, mineral surface charges became positive, thereby repulsing metal ions into

solution. The optimal pH for the metal leaching process was approximately 3.0.

- A preliminary wash with an oxidant, such as hydrogen peroxide, oxidized  $\text{Cu}^+$  in sediments to  $\text{Cu}^{2+}$  which was effectively complexed by EDTA. The oxidant wash also solubilized chromium hydroxide and permitted the complexation and removal of chromium from solution.
- The process could be streamlined by eliminating the redundant use of weak and strong organic acids.
- The ideal extraction sequence determined by modelling the results of the bench-scale study, and combined with speciation theory is: hydrogen peroxide, 1M hydrochloric acid, and 0.1M EDTA buffered at pH 3.0.

## 5.0 SEQUENTIAL METAL LEACHING AT FULL-SCALE

The implementation of the sequential metal leaching process at full-scale requires that a number of physical, chemical, cost, and design constraints be addressed. Section 5.1 outlines the general full-scale sequential metal leaching system proposed based on the results of the bench-scale study and the optimization process. Section 5.2 briefly addresses the factors which are case-specific and will require attention for each application of the general full-scale system. Section 5.3 provides a cost analysis for the sequential metal leaching process at full-scale.

### 5.1 General Full-Scale Process Design

Based on the results of the bench-scale study and on an evaluation of currently available sediment washing systems, a staged approach to sediment washing has been devised. The intensity of washing conditions progressively increases with each stage such that the most intense conditions are applied to the smallest volume of material. The staged process minimizes the use of chemicals in the process and maximizes the amount of sediment cost-effectively recovered as clean material.

Each sediment type binds metal ions to a differing degree proportional to particle size and the electrostatic properties of the sediments. It is therefore more efficient to treat the size fractions separately, often under different conditions. The full-scale sequential metal leaching process can be separated into three stages, as illustrated in Figure 5.1. In general, oversized material (e.g., gravels) is washed with water to separate out fine particle materials. Sands are washed in mild extracting chemicals to break up aggregated materials and to remove a portion of the metals associated with the finer material. Finer materials (e.g., silt) are washed under more aggressive conditions to liberate metals that are tightly bound to the sediment matrix. Brief summaries of each of the stages in the general full-scale process are provided below.

### 5.1.1 Gravel Treatment

The first step in the sequential metal leaching process will involve the separation of gravel and oversized material (>50 mm) through a coarse screen. Bulk material will be milled to break up sediment clusters. The sediment will then be passed through a magnetic separator to remove magnetic material, and will undergo a Trommel wash. The sediment will then be washed through a 1/4" screen and the clean gravel will be separated for use as fill. Sand and silt will be treated in subsequent steps.

### 5.1.2 Sand Treatment

Sand and silt will be separated and treated through a series of washings. Initially, sand and silt containing sediments will be hydro-cycloned to remove a portion of the silt and organic matter (*e.g.*, peat, coal). The remaining sand will undergo attrition washing with a weak extractant solution and will again be hydro-cycloned to remove silt. The sand portion will then be density separated and clean sand will be removed for use as fill.

### 5.1.3 Silt Treatment

Metals are generally associated with smaller silt particles and thus, washing of the silt material is more intensive. The silt sediment slurry undergoes several wash stages in a mixing tank containing acids and chelators. Oxidizing agents (such as hydrogen peroxide or pyrophosphate) can also be added to break up metal-organic ligand complexes, release metals from specific complexes, oxidize  $\text{Cu}^+$  to  $\text{Cu}^{2+}$ , and to solubilize chromium hydroxides. The oxidized slurried silt is passed from an initial wash to a second wash where the pH is adjusted to 3.0 with 1.0 M hydrochloric acid. The decreased pH results in increased leaching of metals into the extraction solution. The sediment slurry is then partially dewatered by gravity separation using a corrugated plate gravity separator, a

hydrocyclone, or a centrifuge. The water stream containing dissolved metals and very fine particulates (those not removed by gravity separation) is then treated to recover and regenerate the metal laden extraction solutions.

The extraction solution recovery process involves ion exchange, electrowinning, precipitation, or distillation to remove the high fines content. The regenerated extraction solution can be recycled back to the washing operation. The waste sludge and the metals concentrated during the recovery/regeneration process may be disposed of or alternatively, the recovered metals may be purified for re-use. The clean sediment may be used as fill or if organic contaminants are present, the sediment may be further processed by biological or physical (*e.g.*, thermal) means. The extraction of metals from the sediments will simplify or enhance the removal of organic contaminants.

The efficiency of the gravity separation and the particle size distribution of the sediments will determine the ratio of sediment cleaned to sludge produced. On this basis, a process yielding the appropriate balance of clean sediment and sludge material can be designed. The equipment and operating costs required to recover a greater percentage of fine material will increase with decreasing particle size. A sediment specific cost analysis will be required to determine what size of material should be targeted for washing and what size will be lost with the contaminated sludge.

## **5.2 Factors Affecting Full-Scale Design**

A number of physical, chemical, and/or geochemical factors will influence the efficiency of the general full-scale sediment leaching system and will control the duration and cost of the process. These factors will be case-specific (*i.e.*, sediment-specific) and should be addressed for each use of the general full-scale process.

**Sediment Texture, Structure and Mineralogy.** The sediment matrix has a profound influence on the effectiveness of an extraction solution to leach bound metals. Sediment properties should be characterized prior to metal leaching to evaluate whether the texture and structure will significantly affect the treatment process. The staged process described above allows the sequential use of appropriate chemicals to liberate metals bound to varying degrees to varying sediment material.

**Chemistry of Extraction Solution.** To control costs, extraction solutions should be tailored to the specific sediment types to be treated. Acids may be selected based on their:

- Associated anion (*i.e.*, nitrate, sulphate, and chloride all behave differently in complexation and recovery processes);
- Lewis strength (depending on whether metals to be extracted are predominantly sorbed, precipitated or occluded to sediments);
- Safety of use; and
- Unit cost.

The most efficient concentration of the acid, the appropriate sediment to solution ratio, and the contact time needed to desorb and solubilize the surface-bound metal species should all be determined prior to application of the process at full-scale.

**Solution pH.** The pH of the process system depends on the concentration and strength of acids added for extraction and on the buffering capacity (alkalinity) of the sediments. The pH of the extraction solution affects the speciation and solubility of most inorganic ions and complexes and therefore must be controlled to maintain the metal species most conducive to chelation.

**Geochemical Reactions.** The efficiency of sediment treatment can be governed by various geochemical reactions, including complexation reactions in aqueous solutions, and

sorption/desorption reactions at the solid/liquid interface. Limiting the number of components and types of interactions in each step of the separation is essential to developing a cost-effective process.

**Number of Extraction Stages.** The selection of single (*i.e.*, mixing sediment and extraction solution only once) or multiple extraction events (*i.e.*, mixing and separating sediment and extraction solution more than once in succession) will be dependant upon the sediment type, metal content and speciation, and the chemistry of the selected extraction solutions. Full-scale treatment trains commonly employ one to three replications of the treatment process. Increased replications are uncommon due to practical and logistical constraints.

**Contact Time.** Contact time is a critical parameter in developing cost estimates for full-scale applications as it dictates the duration of the process. The kinetics of the dissolution and sequestering reactions, and the process rates will require evaluation for each process.

**Temperature.** Temperature will affect the performance of sediment washing operations and the kinetics of the dissolution and sequestering reactions. Extraction processes are generally designed to operate at slightly elevated temperatures. Heat loss from process systems will influence the cost and duration of operations.

**Sediment to Solution Ratio.** The volumetric ratio of sediment to extraction solution is a critical parameter in estimating the cost of full-scale operations. It is favourable to minimize the quantity of extraction solution required to reduce the cost of wastewater treatment. However, the benefits of reduced wastewater treatment must be balanced with the reduced process rates which result from the use of smaller volumes of extraction solutions.



**Competitive Ion Effect.** Chelating agents are not perfectly ion-specific. For example, the removal of cationic forms of heavy metals in solution may be subject to interference from calcium, magnesium, manganese, and sodium that occur naturally in sediments.

Minimization of the competitive ion effect through pH control and the choice of appropriate chelators should limit the unnecessary and inefficient use of excessive quantities of chelating agents.

**Separation of Extraction Solution from Sediments.** Effective dewatering of sediment slurries will be important to minimize the costs of wastewater treatment. A variety of solid and liquid separation processes are available at full-scale. The final selection of a separation method depends strongly on the size distribution of the sediment and on the fraction targeted for recovery.

**Recovery and Reuse of Extraction Solutions.** Efficient recovery of extraction solutions will be critical for the cost-effective application of the treatment process. Recovery of extraction solutions (acids and/or chelators) will reduce costs for raw materials in the process and will simplify the treatment of the metal bearing wastewater. The most cost-effective system will be unique for a given sediment and contaminant suite.

**Wastewater Treatment.** Sediment washing processes generate wastewater that must be treated. A number of conventional and emerging methods are available to treat this wastewater. The efficiency of each method to treat wastewater generated from a particular leaching application can be determined by bench-scale testing. The recovery of chelators and acids may or may not be feasible and cost-effective, depending on the specific sediments and the metal content and speciation.

### 5.3 Process Costs

The cost for the front end gravel and sand treatment stages of the staged soil washing process is approximately \$75-\$125 per ton (Traver, personal communication). This cost applies to a system designed to process 20 tons of material per hour. As a result of the more complex operation and smaller capacity, the cost of silt washing operations will be higher than for washing sand and gravel. The cost of silt washing will also vary greatly depending upon the fraction of the total sediment material that is silt. An estimated cost for silt treatment would be \$200-\$400 per ton. While it is recognized that the amount of silt material to be treated may be as high as 50 to 90%, a silt content of 20% (of the original sediment feed) has been used to delineate the cost of effective sediment washing. Using the 20% silt content value, the total cost of treatment per ton of bulk sediment would range from approximately \$115-\$205 per ton.

## 6.0 CONCLUSIONS

The following conclusions can be made regarding the bench-scale study of the sequential metal leaching system and the design optimization for full-scale use of the extraction process:

1. The sequential leaching system studied at bench-scale did not provide the most efficient extraction of metals from the sediment samples. Of particular note was the loss of a significant percentage of metals with fine materials (*e.g.*, silts and clays). In addition, chromium and the reduced species of copper associated with the sediments were not effectively chelated by the EDTA solution.
2. While the total removal of metals from sediments was generally higher in the sorted sediment sample, the leaching efficiencies were generally higher in the bulk sediment sample. The increased total metal removal in sorted sediments can be attributed to the loss of fines from the sample.
3. The sequential metal leaching process can be used to design a sequential extraction procedure or extraction solution cocktails which target the predominant analyte-surface bonds.
4. A simplified full-scale metal leaching system employing existing technology was developed on the basis of the sequential metal leaching process. The treatment train consists of a physical treatment (screening) stage to separate oversize materials, followed by separation of sands, addition of oxidizing agents to break up metal-organic ligand complexes, oxidize  $\text{Cu}^{1+}$  to  $\text{Cu}^{2+}$ , and to solubilize chromium hydroxides, followed by solution acidification, recovery of metals on chelating resins and regeneration of the extraction solutions and the chelation resins.

5. The estimated cost for sediment treatment using the full-scale metal leaching system is estimated to be between \$115 to \$205 (CDN) per ton, with an estimated throughput of 20 ton per hour. Metal extraction efficiencies would approach 99.9%. The potential cost effectiveness and efficiency of the designed system warrant its further development and pilot scale testing.

## 7.0 REFERENCES

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## Tables

**TABLE 2.1: METAL CONCENTRATIONS IN EXTRACTION SOLUTIONS USED IN TESTING**

Parameter	DDW	ACET	HCl	EDTA	DDW F	ACET F	HCl F	EDTA F
Ag mg/L	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Al mg/L	<0.02	0.02	<0.02	0.06	<0.02	0.02	0.02	0.06
As ug/L	<5	<2	<2	<2	<2	<2	<2	<2
Be mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cd mg/L	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Cr mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cu mg/L	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.010
Fe mg/L	<0.02	0.040	<0.02	0.040	<0.02	0.020	<0.02	0.040
Mg mg/L	<0.05	<0.05	<0.05	<0.05	0.050	<0.05	<0.05	<0.05
Mn mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Ni mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Pb mg/L	<0.02	0.040	<0.02	0.060	<0.02	0.040	<0.02	0.040
Sb ug/L	<2	<5	<5	<10	<10	10	<10	<10
Se ug/L	<5	<2	<2	<2	<2	<2	<2	<2
Tl mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Zn mg/L	<0.01	0.01	<0.01	0.03	0.01	0.01	0.03	0.11

DDW - distilled water

HCl - dilute HCl (pH 1.0)

F - Filtered

ACET - acidic ammonium acetate solution

EDTA - ammonium citrate and sodium EDTA solution

TABLE 3.1 - INITIAL CONCENTRATIONS OF METALS IN SORTED AND BULK SEDIMENTS

Parameter	BAS Laboratories		WTC	Soil Assessment
	Screened Sediment (ug/g)	Total Sediment (ug/g)	Total Sediment (ug/g)	Criteria* (ug/g)
Moisture %	52.4	57.8	--	--
FSE - Wet Wt. g	240.7/221.6	236.6/178.6	--	--
FSE - Dry Wt. g	114.6/103.5	99.8/89.1	--	--
Control - Wet Wt. g	240.2/225.1	237.2/175.2	--	--
Control - Dry Wt. g	114.3/123.6	100.1/92.7	--	--
Ag	<0.5	<0.5	2.6	2
Al	23,000	23,000	--	--
As	32	17	8.758	5
Be	1	2	--	4
Cd	2.5	1.5	5.2	0.5
Cr	2,800	530	218	20
Cu	780	290	119	30
Fe	182,000	83,000	2.55%	--
Mg	13,000	12,800	5460	--
Mn	2,200	900	382	--
Ni	2,000	770	291	20
Pb	300	170	76	25
Sb	17	<1	67	20
Se	<1	<1	0.341	1
Tl	<2	<2	--	0.5
Zn	760	730	255	60

## Notes:

FSE - Full Sequential Extraction

Wt. g. 240.7/221.6 = Weight before treatment/Weight after treatment

\* - Soil Assessment Criteria taken from CCME, 1991.



TABLE 3.2A: CUMULATIVE MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Sorted Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	ACET 1	ACET 2	ACET 3	ACET 4	HCl 1	HCl 2	HCl 3	HCl 4	EDTA 1	EDTA 2	EDTA 3	EDTA 4	DDW 5
VOL mL	192	190	216	206	184	200	198	200	202	200	196	202	200	192	198	190	238
pH	6.92	7.25	7.62	7.82	5.51	5.22	5.15	5.13	4.88	4.64	4.46	4.39	7.71	8.31	8.44	8.48	
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	19.2	42.0	76.6	110	1434	7834	17140	28740	36416	42616	47908	56594	176594	241874	297314	327904	406444
As ug	0.96	1.91	1.91	1.91	13.9	32.9	51.7	72.7	105	130	148	166	232	280	347	400	517
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cd ug	0.00	0.00	0.00	0.00	3.68	7.68	11.6	15.6	19.7	23.7	27.6	35.7	47.7	53.4	59.4	63.2	77.5
Cr ug	0.00	0.00	0.00	0.00	82.8	413	829	1269	1572	1772	1919	2090	3190	4362	6025	7165	9973
Cu ug	0.00	2.85	6.09	8.15	11.8	11.8	15.8	23.8	23.8	23.8	27.7	35.8	876	1817	3242	4306	6639
Fe ug	422	479	479	479	57519	117519	163059	198659	237443	277443	316643	371183	421183	458815	502375	534295	634255
Mg ug	2918	4780	5212	5460	13556	18756	21983	25103	33789	55189	93213	157853	173853	180573	185523	188354	201206
Mn ug	186	291	308	318	4734	8214	10769	12769	14587	16627	18939	21949	23149	23879	24710	25337	28455
Ni ug	21.1	34.4	38.7	40.8	1623	3663	5524	7284	8820	10220	11533	12967	14507	15755	17438	18673	23314
Pb ug	0.00	0.00	0.00	0.00	129	469	964	1624	1866	2006	2124	2205	7005	9328	11308	12372	15133
Sb ug	0.77	1.53	1.96	2.78	6.46	10.5	14.4	18.4	20.4	22.4	22.4	22.4	51.4	70.6	95.4	117	155
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Tl ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zn ug	5.76	9.56	18.2	20.3	324	924	1656	2676	3565	4965	7278	10288	14688	16857	19293	21117	28257

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 3.2B: CUMULATIVE MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Sorted Sediment  
TREATMENT: Control

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	DDW 5	DDW 6	DDW 7	DDW 8	DDW 9	DDW 10	DDW 11	DDW 12	DDW 13	DDW 14	DDW 15	DDW 16	DDW 17
VOL mL	197	188	216	206	190	200	200	202	200	200	200	206	200	202	200	205	222
pH	6.92	7.30	7.62	7.68	7.10	7.65	7.73	7.39	7.90	7.81	7.43	7.71	7.35	7.72	7.62	7.60	
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	19.7	42.3	98.4	123	142	158	178	198	218	242	266	295	323	360	392	428	468
As ug	1.18	1.93	1.93	1.93	2.88	3.48	4.28	4.89	5.69	6.49	7.29	8.11	9.71	10.7	11.7	12.7	16.1
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cd ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cr ug	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97	1.97
Cu ug	1.97	7.61	18.4	25.6	26.6	27.6	28.6	29.6	29.6	30.6	30.6	31.6	39.6	45.7	50.7	52.7	57.2
Fe ug	591	700	730	730	749	749	749	749	753	753	753	753	753	753	753	753	753
Mg ug	2975	4855	5276	5461	6886	7946	8786	9453	9993	10433	10793	11123	11393	11655	11865	12070	12337
Mn ug	199	304	324	334	450	520	572	612	642	666	686	705	721	737	751	765	788
Ni ug	21.7	33.0	37.3	39.3	46.9	52.9	56.9	61.0	65.0	67.0	69.0	71.0	73.0	75.1	77.1	79.1	81.3
Pb ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Sb ug	0.79	1.16	1.60	2.01	2.01	2.01	3.01	4.02	5.02	6.02	7.02	8.05	8.85	10.5	12.1	14.1	18.6
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Tl ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zn ug	11.8	13.7	18.0	20.1	22.0	22.0	22.0	22.0	22.0	22.0	22.0	22.0	22.0	24.0	24.0	28.1	43.6

DDW - distilled water

**TABLE3.3: PERCENTAGE OF METAL EXTRACTED IN DIFFERENT FRACTIONS**

SAMPLE: Sorted Sediment  
TREATMENT: Full Sequential Extraction

Metal	Mass (ug)		Percent Mass Extracted						Total Percent Mass Removed	Percent Mass Lost in Fines
	Before Treatment	After Treatment	DDW	ACET	HCl	EDTA	DDW	TOTAL		
Ag	0	0	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	nd	nd
Al	2,635,184	1,955,908	0.0%	1.1%	1.1%	10.3%	3.0%	15.4%	25.8%	10.4%
As	3,666	2,277	0.1%	1.9%	2.5%	6.4%	3.2%	14.1%	37.9%	23.8%
Be	115	103	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	10.4%	10.4%
Cd	286	155	0.0%	5.5%	7.0%	9.6%	5.0%	27.1%	45.8%	18.7%
Cr	320,805	289,764	0.0%	0.4%	0.3%	1.6%	0.9%	3.1%	9.7%	6.6%
Cu	89,367	69,336	0.0%	0.0%	0.0%	4.8%	2.6%	7.4%	22.4%	15.0%
Fe	20,852,322	11,073,130	0.0%	1.0%	0.8%	0.8%	0.5%	3.0%	46.9%	43.9%
Mg	1,489,452	1,148,708	0.4%	1.3%	8.9%	2.0%	0.9%	13.5%	22.9%	9.4%
Mn	252,061	198,695	0.1%	4.9%	3.6%	1.3%	1.2%	11.3%	21.2%	9.9%
Ni	229,146	177,998	0.0%	3.2%	2.5%	2.5%	2.0%	10.2%	22.3%	12.1%
Pb	34,372	10,245	0.0%	4.7%	1.7%	29.6%	8.0%	44.0%	70.2%	26.2%
Sb	1,948	1,449	0.1%	0.8%	0.2%	4.9%	2.0%	8.0%	25.6%	17.6%
Se	0	0	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	nd	nd
Tl	0	0	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	nd	nd
Zn	87,076	45,534	0.0%	3.1%	8.7%	12.4%	8.2%	32.5%	47.7%	15.3%

DDW - distilled water  
HCl - dilute HCl (pH 1.0)  
nd - not detected

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 3.4A: CUMULATIVE MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Bulk Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	ACET 1	ACET 2	ACET 3	ACET 4	HCl 1	HCl 2	HCl 3	HCl 4	EDTA 1	EDTA 2	EDTA 3	EDTA 4	DDW 5
VOL mL	214	200	212	210	190	204	200	204	202	202	200	200	198	188	208	198	234
pH	7.10	7.36	7.83	7.91	5.47	5.36	5.26	5.21	4.95	4.72	4.40	4.32	7.91	8.37	8.55	8.55	
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	98.4	226	277	286	438	744	1084	1512	1674	1815	2135	2615	23801	35081	43401	47757	58053
As ug	1.07	2.07	2.07	2.07	11.6	19.7	26.7	34.9	45.0	60.1	75.1	93.1	121	144	169	190	260
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cd ug	0.00	0.00	0.00	0.00	7.60	16	22	26	30	34	38	44	52	59	70	74	92
Cr ug	0.00	0.00	0.00	0.00	28.5	89.7	150	221	241	251	271	291	707	980	1219	1368	1742
Cu ug	3.21	7.21	8.27	8.27	8.27	8.27	8.27	8.27	8.27	8.27	8.27	8.27	183	483	920	1249	1904
Fe ug	120	272	306	306	85806	179646	257646	312726	357166	403626	463626	525626	575126	602762	626682	641334	687432
Mg ug	621	1021	1339	1549	15229	25021	30621	36741	49265	75525	117525	177525	194949	201717	206085	208619	221021
Mn ug	19.3	33.3	39.6	43.8	4034	7828	10408	12611	14227	16005	18005	20425	21415	21866	22220	22329	23171
Ni ug	8.56	12.6	14.7	14.7	2162	4528	6128	7536	8647	9778	10958	12518	14023	15019	15934	16508	18146
Pb ug	0.00	0.00	0.00	0.00	19.0	59.8	120	201	222	262	262	282	2242	3539	4579	4896	6581
Sb ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.97	2.97	8.17	10.15	14.83
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Tl ug	0.00	0.00	0.00	0.00	38.0	58.4	58.4	78.8	78.8	78.8	78.8	78.8	78.8	78.8	78.8	78.8	78.8
Zn ug	4.28	8.28	8.28	8.28	198	596	956	1384	1829	2435	3795	7195	10323	12711	15186	16235	24659

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 3.4B: CUMULATIVE MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Bulk Sediment  
TREATMENT: Control

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	DDW 5	DDW 6	DDW 7	DDW 8	DDW 9	DDW 10	DDW 11	DDW 12	DDW 13	DDW 14	DDW 15	DDW 16	DDW 17
VOL mL	218	204	220	198	194	200	202	200	208	208	200	204	198	208	202	204	214
pH	7.13	7.43	7.81	8.23	7.58	8.25	7.46	7.59	7.70	7.67	7.78	7.65	7.50	7.60	7.53	7.68	
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	205	360	378	409	421	433	453	469	486	507	531	551	579	599	628	685	711
As ug	1.74	2.56	3.00	3.00	3.58	3.98	3.98	4.38	4.80	4.80	5.20	5.61	6.79	7.42	8.02	8.64	9.71
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cd ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cr ug	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72	8.72
Cu ug	37.1	40.1	41.2	42.2	44.2	47.2	49.2	50.2	52.3	53.3	53.3	54.3	63.2	81.9	97.1	100	102
Fe ug	144	311	320	332	343	355	368	376	384	396	412	425	440	453	469	538.48	547.04
Mg ug	1417	2376	3168	3465	4260	4960	5546	6106	6626	7063	7453	7810	8137	8490	8813	9,139.70	9,631.90
Mn ug	54.5	83.1	98.5	104	122	136	148	158	168	177	185	191	197	203	209	217.22	225.78
Ni ug	32.7	42.9	49.5	53.5	59.3	65.3	71.3	77.3	81.5	85.7	89.7	93.7	97.7	104	110	114	121
Pb ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Sb ug	0.00	0.00	0.00	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79	0.79
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Tl ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zn ug	50.1	60.3	60.3	60.3	64.2	64.2	66.2	66.2	66.2	66.2	66.2	66.2	68.2	68.2	70.2	76.4	76.4

DDW - distilled water

**TABLE3.5: PERCENTAGE OF METAL EXTRACTED IN DIFFERENT FRACTIONS**

SAMPLE: Bulk Sediment  
TREATMENT: Full Sequential Extraction

Metal	Mass (ug)		Percent Mass Extracted						Total Percent Mass Removed	Percent Mass Lost in Fines
	Before Treatment	After Treatment	DDW	ACET	HCl	EDTA	DDW	TOTAL		
Ag	50	178	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	nd	nd
Al	2,296,440	2,317,156	0.0%	0.1%	0.0%	2.0%	0.4%	2.5%	0.0%	-2.5%
As	1,697	1,337	0.1%	1.9%	3.4%	5.7%	4.1%	15.3%	21.2%	5.9%
Be	200	89	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	55.5%	55.5%
Cd	150	134	0.0%	17.2%	12.1%	19.9%	12.5%	61.6%	10.7%	-51.0%
Cr	52,918	4,998	0.0%	0.4%	0.1%	2.0%	0.7%	3.3%	90.6%	87.3%
Cu	28,955	24,954	0.0%	0.0%	0.0%	4.3%	2.3%	6.6%	13.8%	7.2%
Fe	8,287,152	7,040,591	0.0%	3.8%	2.6%	1.4%	0.6%	8.3%	15.0%	6.7%
Mg	1,278,019	1,069,457	0.1%	2.8%	11.0%	2.4%	1.0%	17.3%	16.3%	-1.0%
Mn	89,861	61,494	0.0%	14.0%	8.7%	2.1%	0.9%	25.8%	31.6%	5.8%
Ni	76,881	54,364	0.0%	9.8%	6.5%	5.2%	2.1%	23.6%	29.3%	5.7%
Pb	16,974	7,843	0.0%	1.2%	0.5%	27.2%	9.9%	38.8%	53.8%	15.0%
Sb	50	267	0.0%	0.0%	0.0%	20.3%	9.4%	29.7%	nd	nd
Se	50	50	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Tl	100	100	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Zn	72,887	45,452	0.0%	1.9%	8.0%	12.4%	11.6%	33.8%	37.6%	3.8%

DDW - distilled water

HCl - dilute HCl (pH 1.0)

nd - not detected

ACET - acidic ammonium acetate solution

EDTA - ammonium citrate and sodium EDTA solution

**TABLE 4.1: PREDICTED PARTITIONING OF METALS WITH VARYING pH AND EDTA CONCENTRATION**

	Zn2+	Pb2+	Ni2+	Cr(OH)2+	Cu1+	Cu2+	Ca2+	Mg2+	Na2+
<b>0.1 M EDTA, pH 7.0</b>									
% bound to EDTA	100.0%	98.9%	100.0%			100.0%	100.0%	100.0%	3.3%
% bound to Soil Surface		1.1%							
% as ppt species [type]				33.8% [Cr(OH)2+]	72.8% [CuCl2]				
% as aqueous species [type]				65.0% [Cr(OH)3+]	26.3% [Cu1+]				96.7% [Na1+]
% of EDTA bound to each species	10.0%	9.9%	10.0%			10.0%	10.0%	10.0%	3.3%
<b>0.01 M EDTA, pH 7.0</b>									
% bound to EDTA	95.5%	9.6%	100.0%			98.4%	96.4%	99.7%	
% bound to Soil Surface	4.5%	90.4%				1.6%	3.6%		
% as ppt species [type]				31.8% [Cr(OH)2+]	73.6% [CuCl2]				
% as aqueous species [type]				67.3% [Cr(OH)3+]	25.5% [Cu1+]				100% [Na 1+]
% of EDTA bound to each species	19.1%	1.9%	20.0%			19.8%	19.3%	19.9%	
<b>0.001 M EDTA, pH 7.0</b>									
% bound to EDTA			98.1%					1.3%	
% bound to Soil Surface	99.9%	100.0%	1.9%			99.7%	99.6%		
% as ppt species [type]				30.7% [Cr(OH)2+]	74.2% [CuCl2]				
% as aqueous species [type]				68.5% [Cr(OH)3+]	25.1% [Cu1+]			98.7% [mg2+]	100% [Na 1+]
% of EDTA bound to each species	98.1%							1.3%	
<b>0.1 M EDTA, pH 3.0</b>									
% bound to EDTA	100.0%	100.0%	100.0%			100.0%	80.8%	25.5%	
% bound to Soil Surface									
% as ppt species [type]				1.9% [Cr(OH)2+]					
% as aqueous species [type]				98.5% [Cr3+]			19.2% [Ca2+]	74.4% [Mg2+]	100% [Na 1+]
% of EDTA bound to each species	10.0%	10.0%	10.0%			10.0%	8.1%		

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## Figures



Figure 3.1 - Cumulative Metal in Extraction Solution (Al)

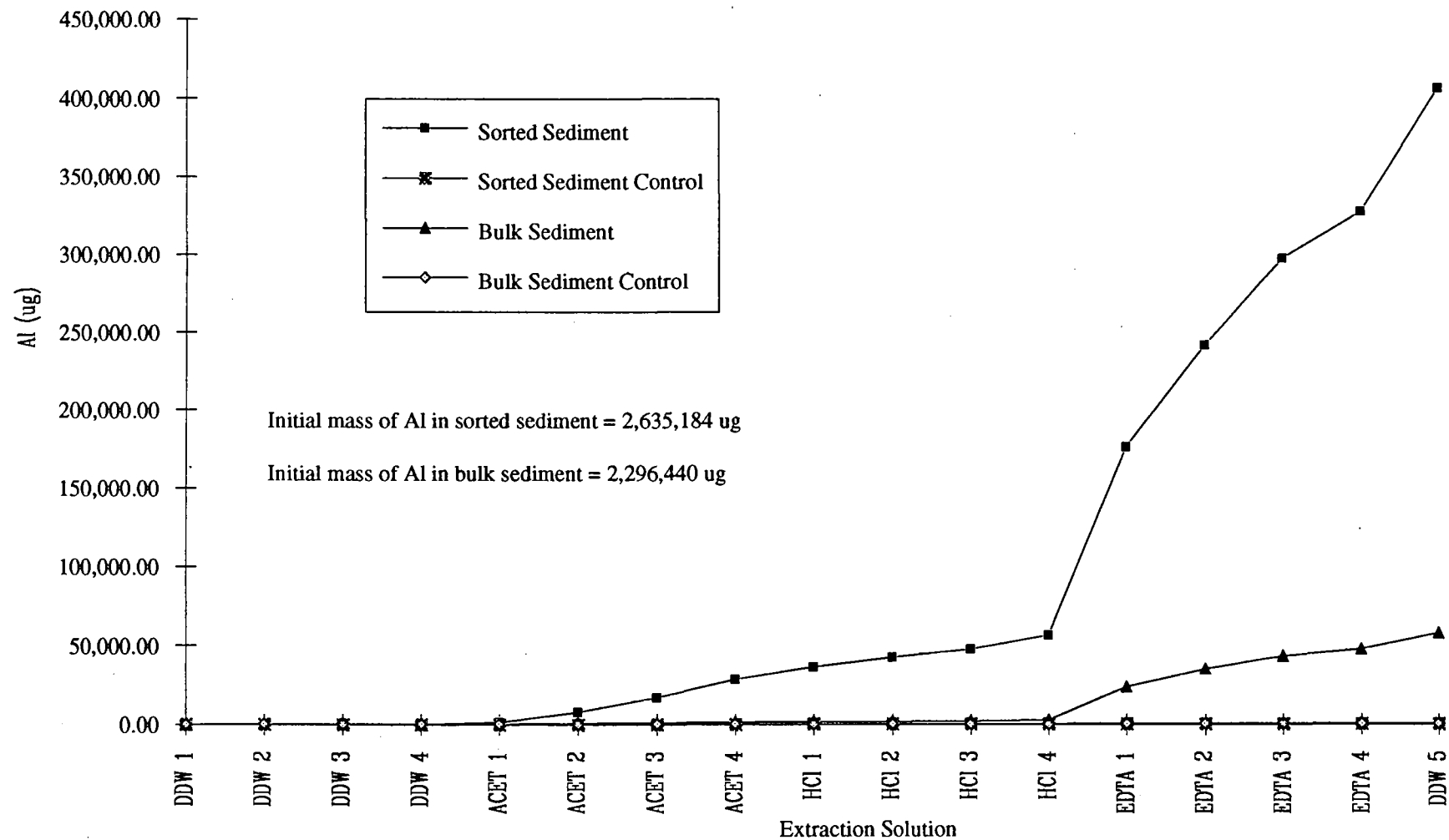
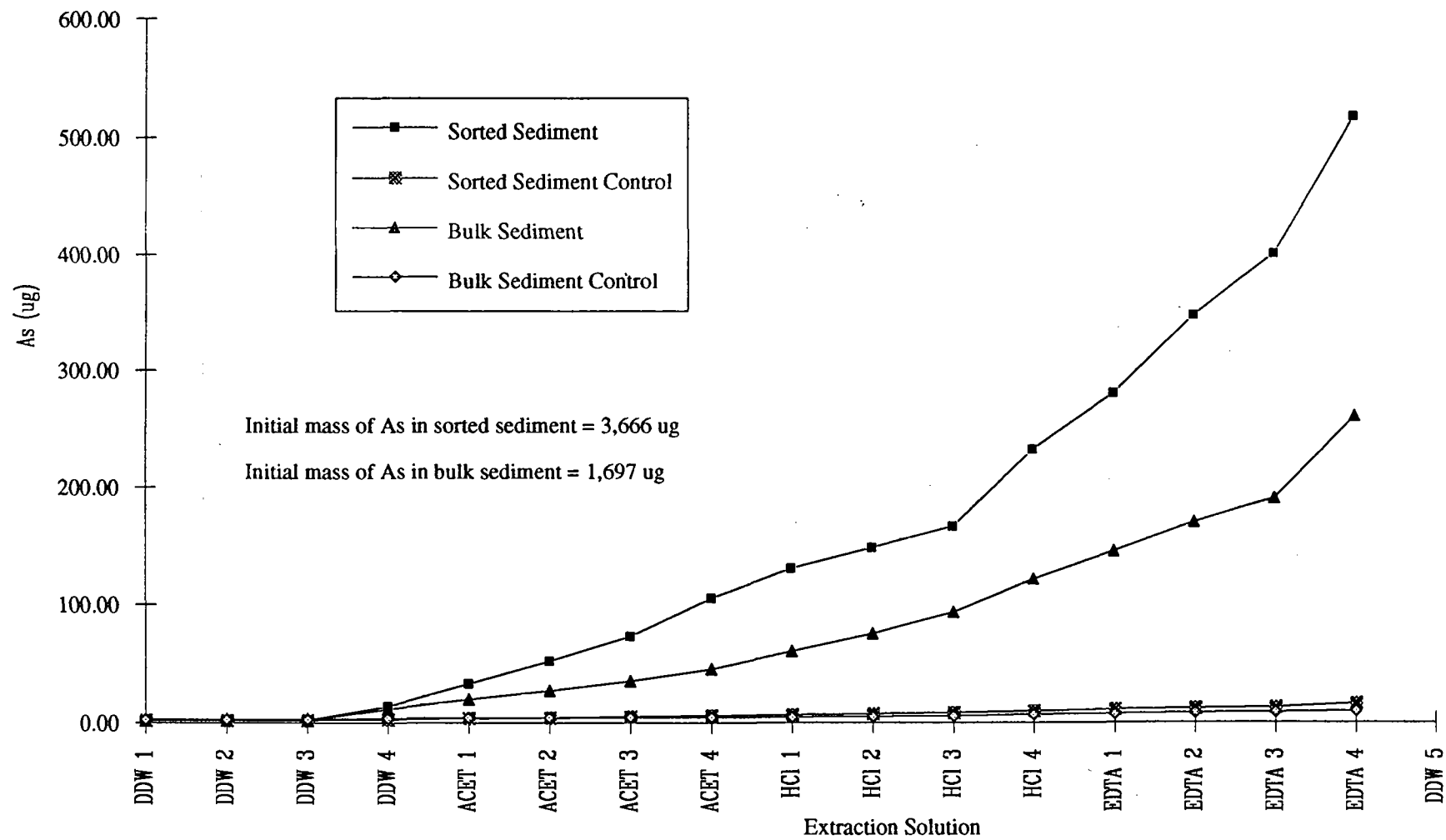


Figure 3.2 - Cumulative Metal in Extraction Solution (As)



**Figure 3.3 - Cumulative Metal in Extraction Solution (Cd)**

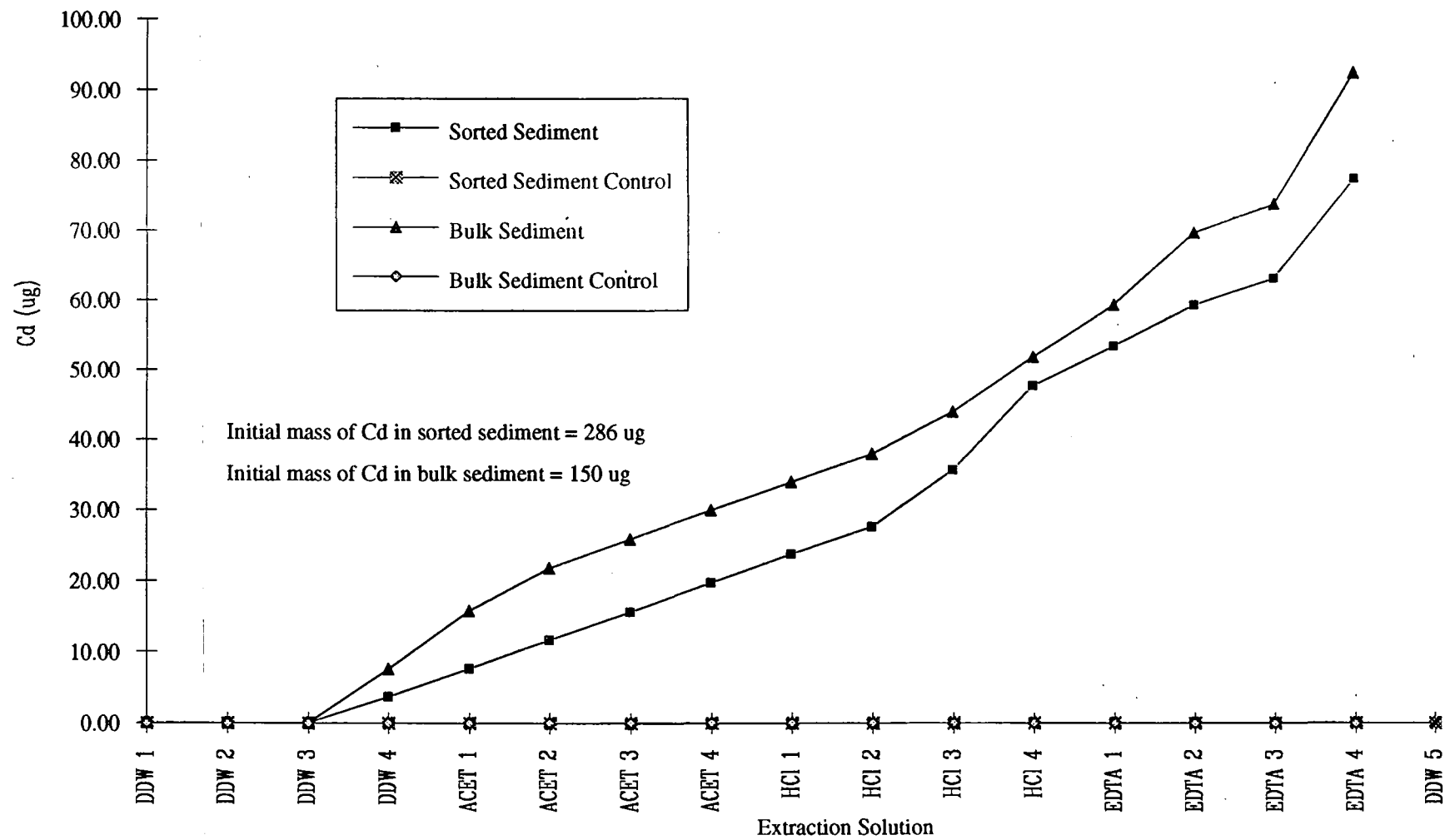


Figure 3.4 - Cumulative Metal in Extraction Solution (Cr)

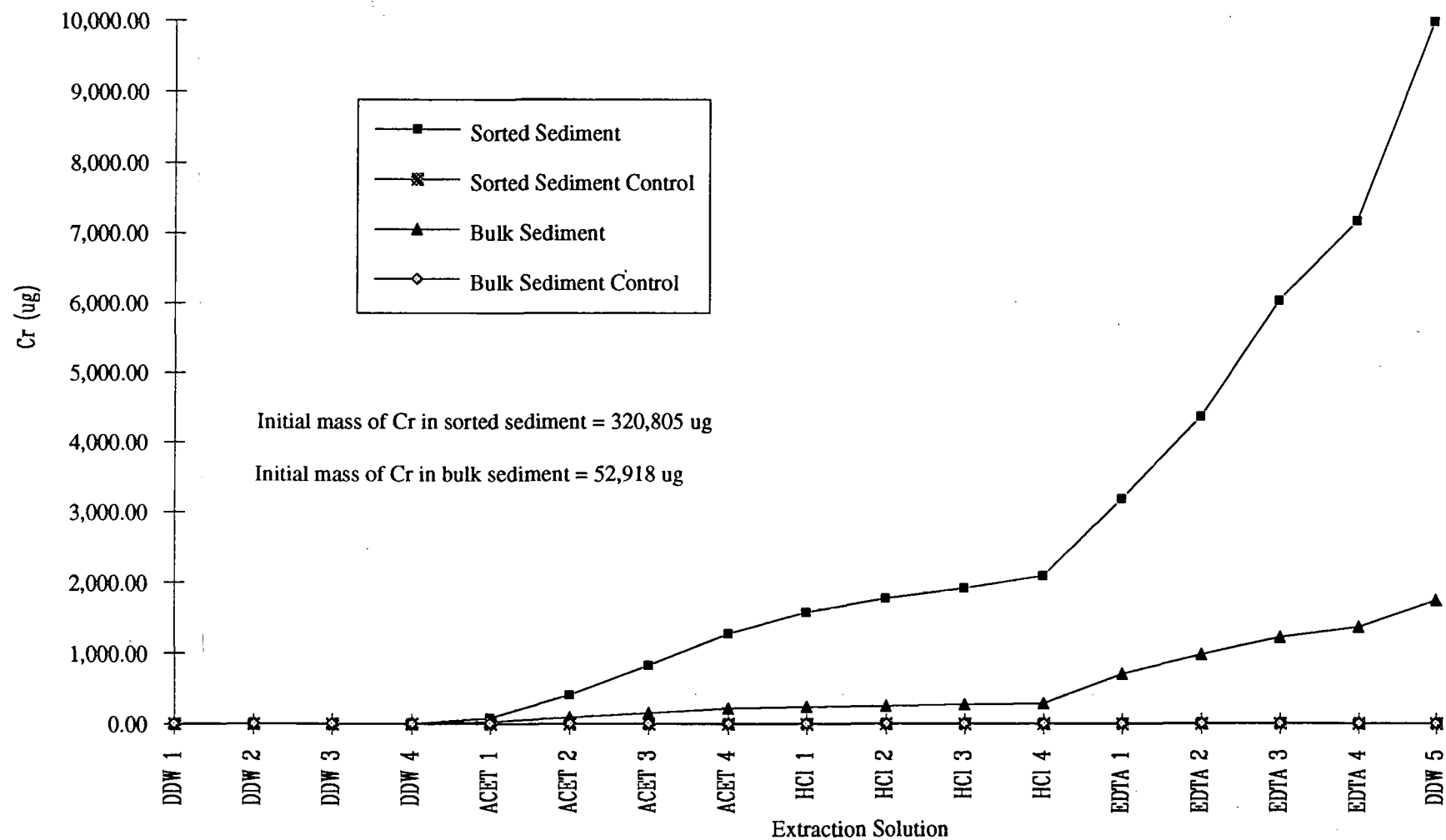


Figure 3.5 - Cumulative Metal in Extraction Solution (Cu)

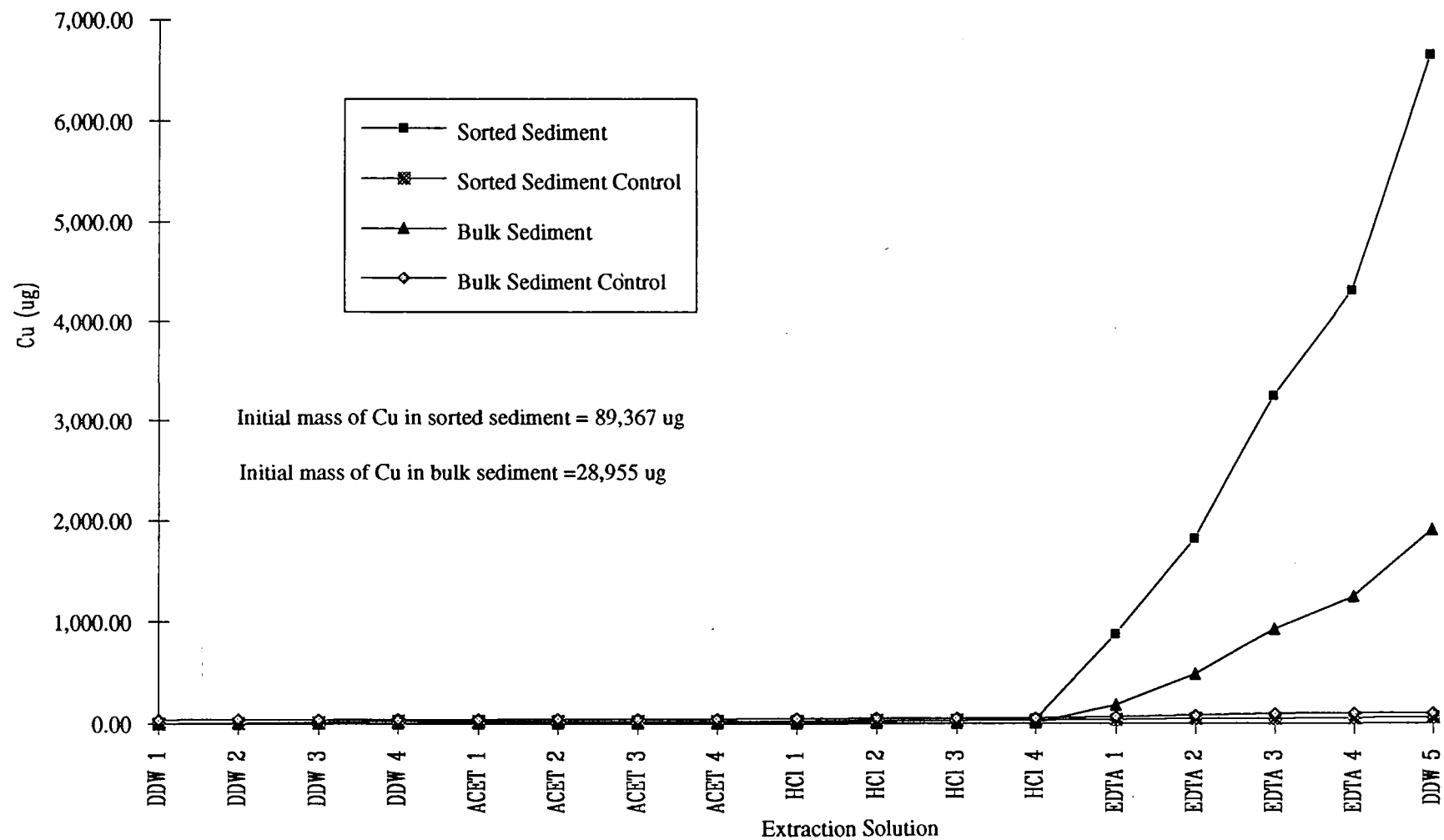
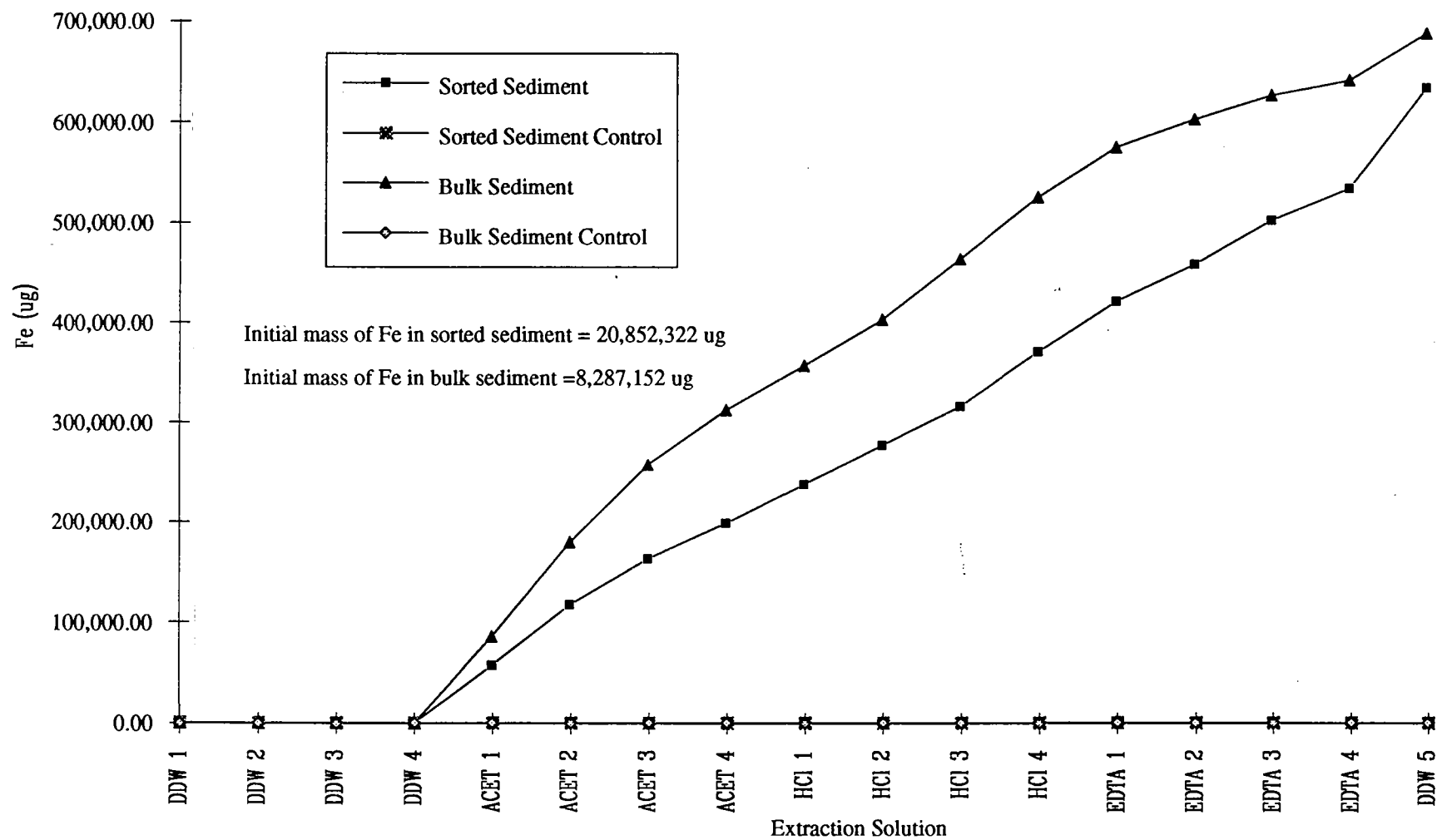


Figure 3.6 - Cumulative Metal in Extraction Solution (Fe)



**Figure 3.7 - Cumulative Metal in Extraction Solution (Mg)**

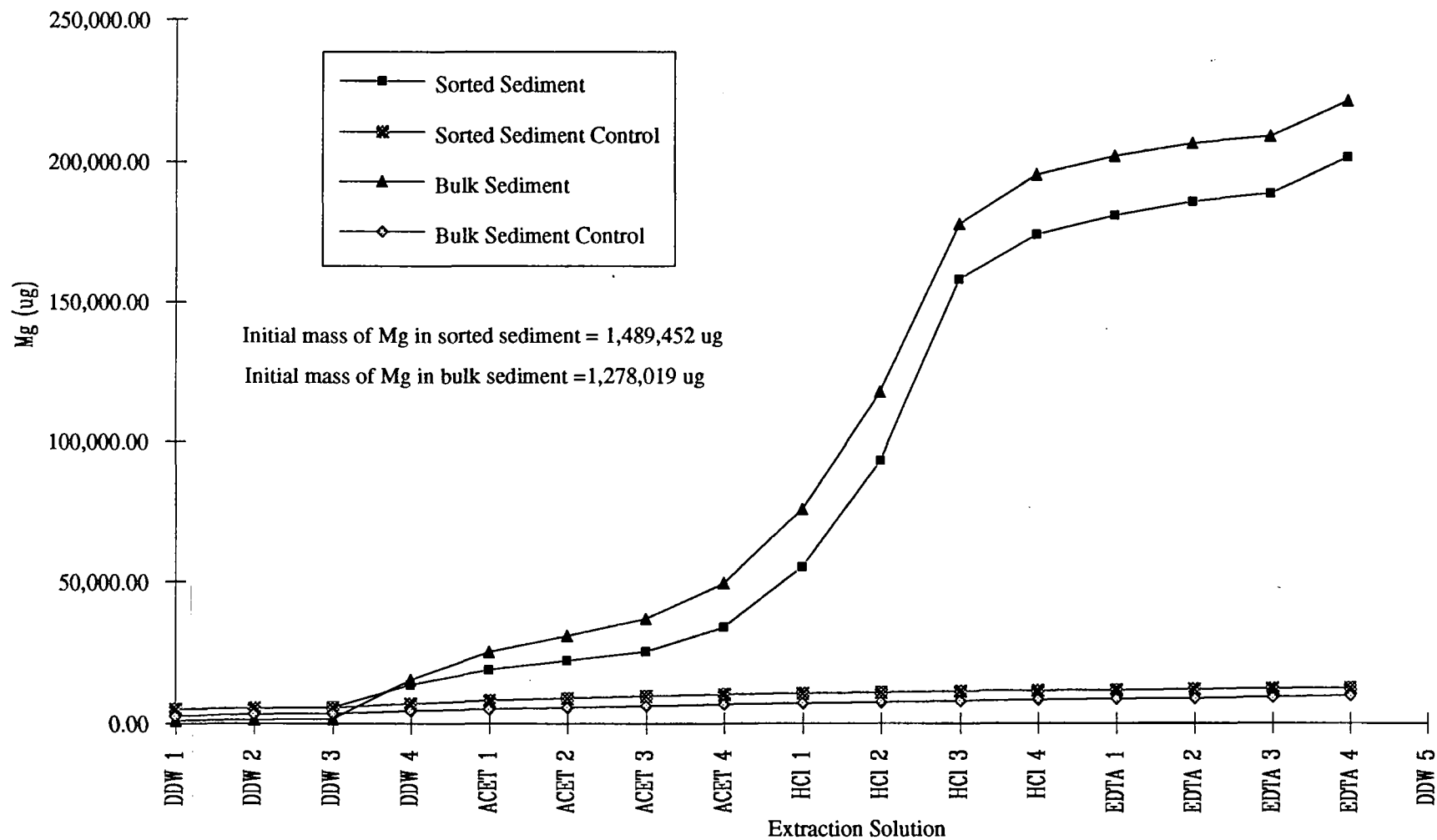
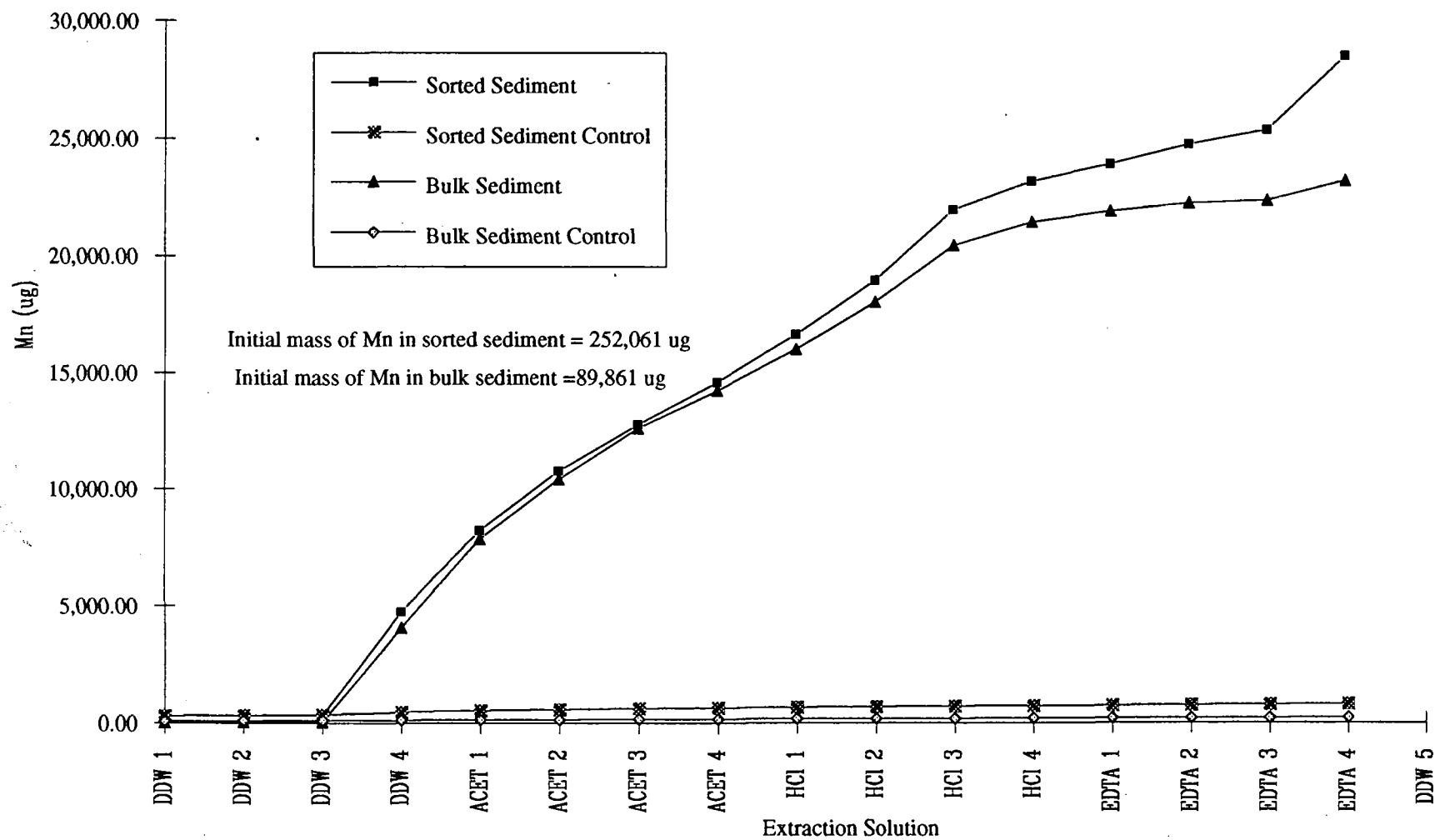


Figure 3.8 - Cumulative Metal in Extraction Solution (Mn)





**Figure 3.9 - Cumulative Metal in Extraction Solution (Ni)**

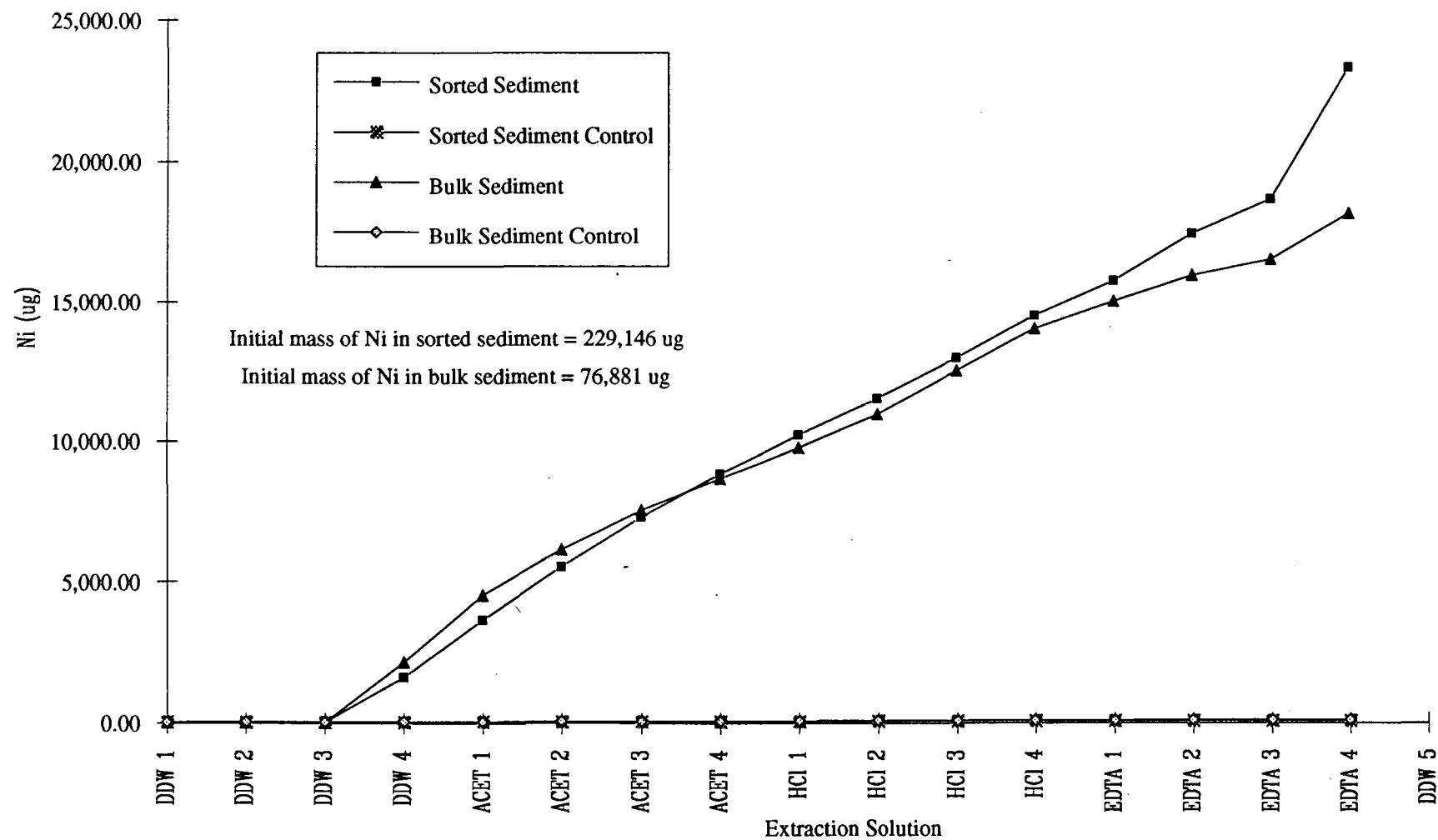


Figure 3.10 - Cumulative Metal in Extraction Solution (Pb)

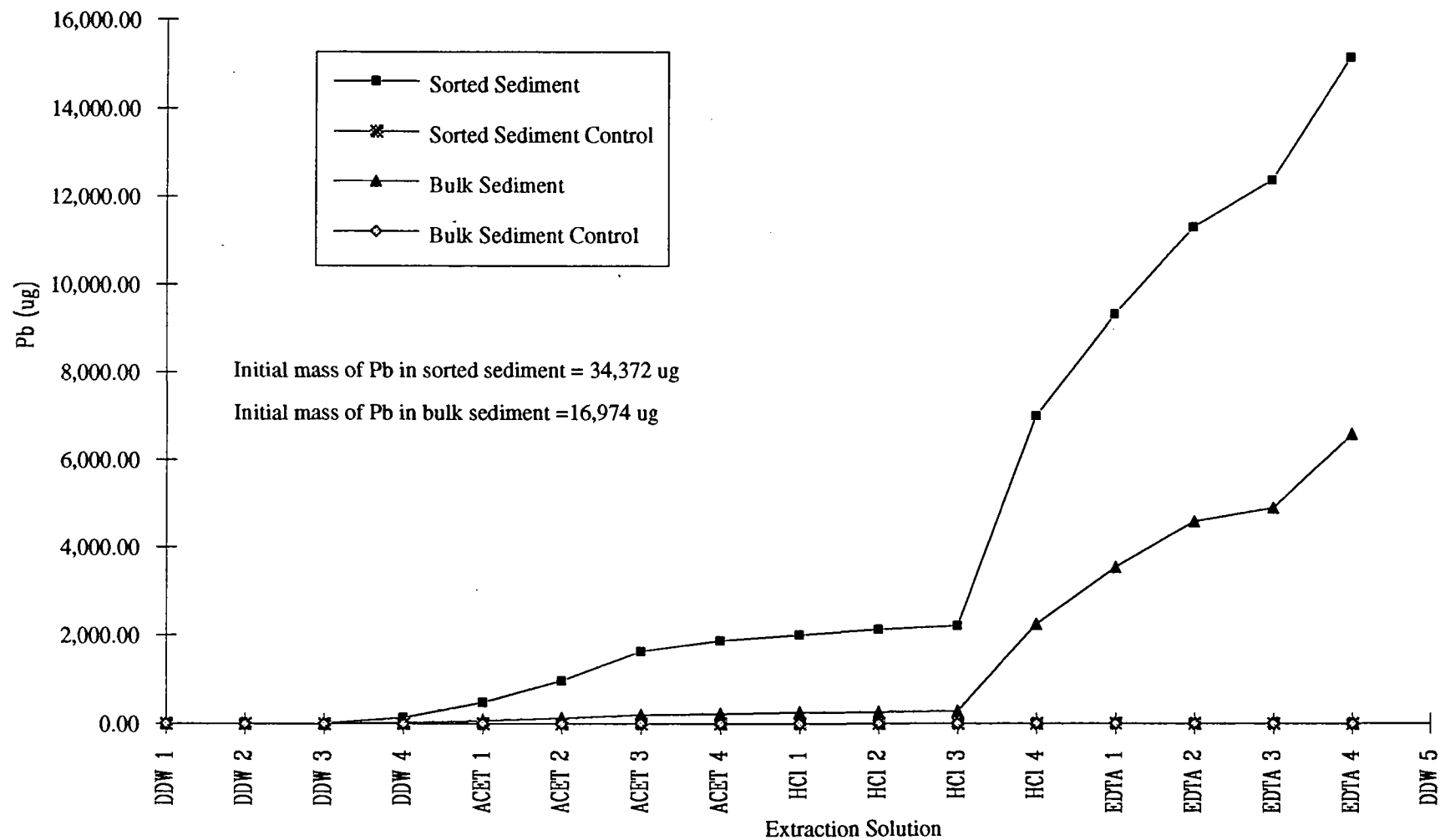


Figure 3.11 - Cumulative Metal in Extraction Solution (Sb)

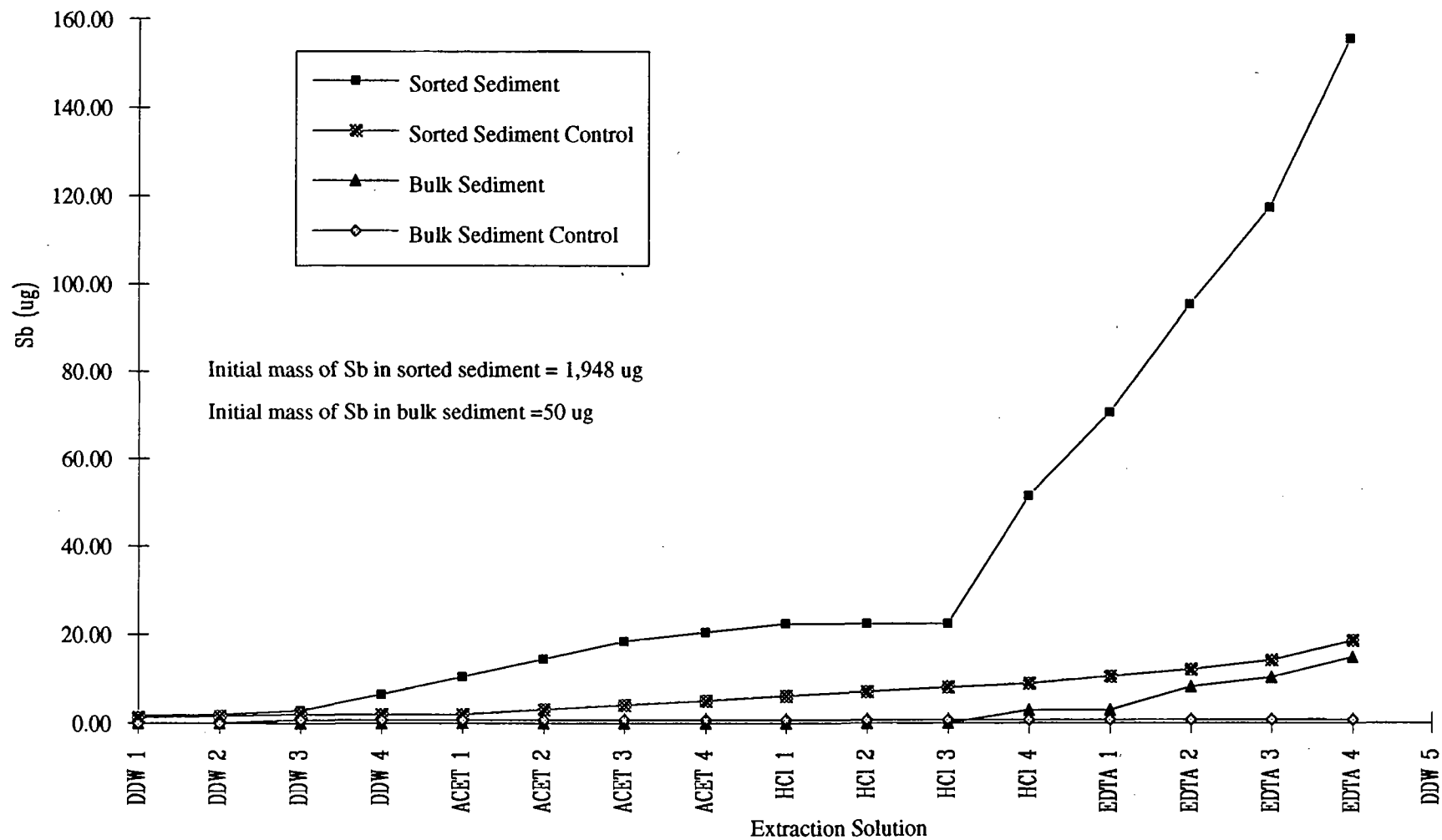
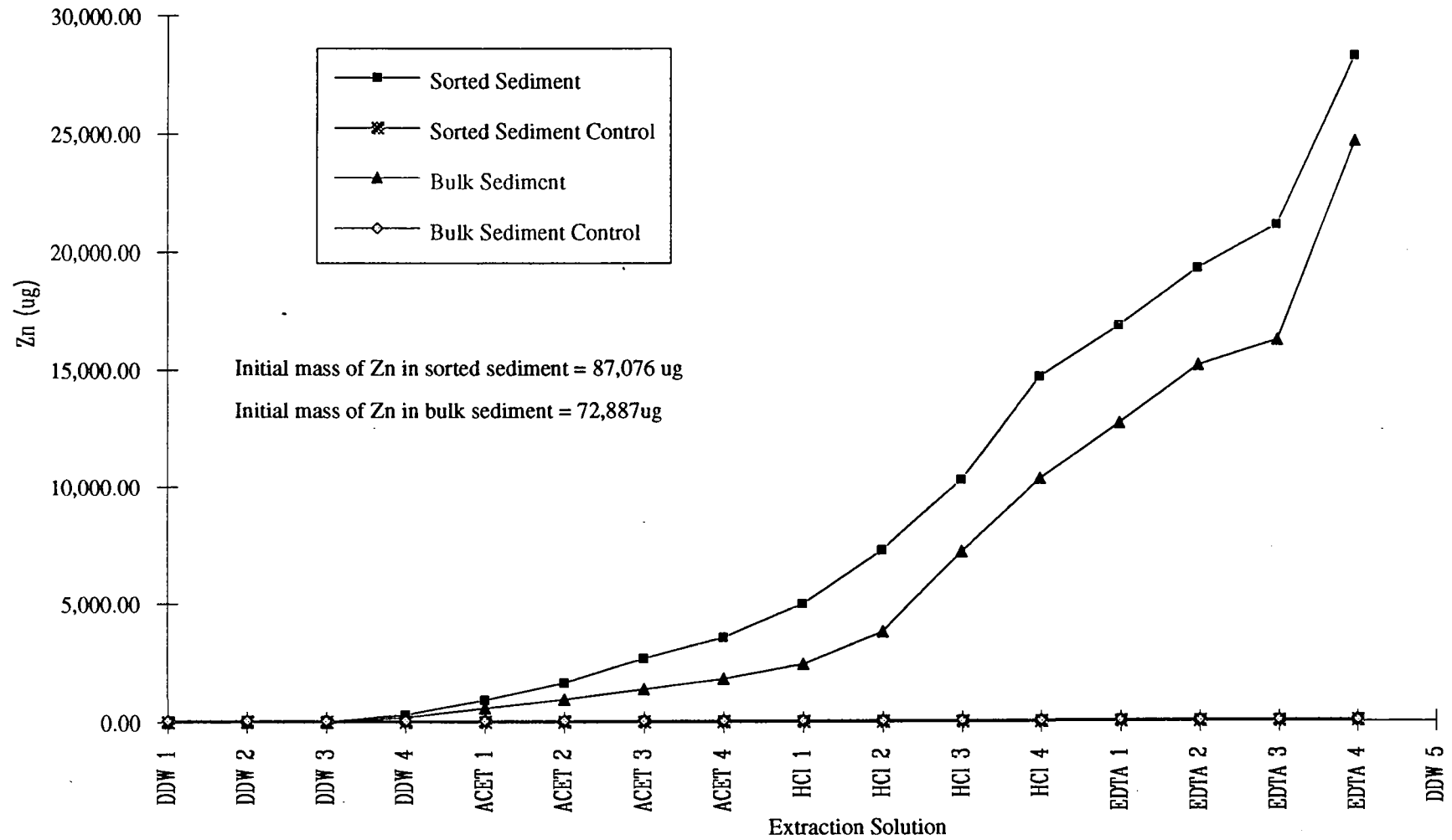


Figure 3.12 - Cumulative Metal in Extraction Solution (Zn)



**Gravel**

*Sediment*

Coarse Screen

*Oversized*

Magnetic  
Separator

*Magnetic Material*

Trommel Wash

*Water*

1/4" Screen

*Sand  
and Silt*

Clean Gravel

**Sand**

Hydro Cyclone

*Sand*

*Weak Extractant Solution*

Attrition Washing

Hydro Cyclone

*Sand*

Density Separator

Clean Sand

*Silt*

*Silt*

**Silt**

WATER  
TREATMENT

Water Reuse

ACID  
RECOVERY

*Metals/Sludge*

*Make Up Acid*

*Make Up Chemicals*

1st Stage Wash

2nd Stage Wash

Gravity Separator

SEDIMENT  
CONDITIONING

Clean Sediment

**Staged Sediment Washing Process**

**beak**

beak  
consultants  
limited

Figure  
5.1

March 1992  
Rev. 00

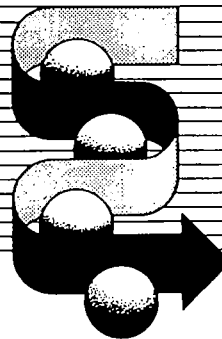
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**Appendix A**

**APPENDIX A**

**AMBERLITE IRC-718 TECHNICAL DATA**

# Ion Exchange Resins



## AMBERLITE® IRC-718

AMBERLITE IRC-718 chelating cation exchange resin has a high affinity for heavy metal cations over alkali or alkaline earth metals, such as sodium, potassium, calcium and magnesium. Selectivity is achieved by an iminodiacetic acid functionality chemically bonded to a macroreticular resin matrix. The functional group coordinates heavy metal ions with several active sites, binding the ions very tightly.

The selective nature of AMBERLITE IRC-718 resin may dramatically decrease the cost of waste treatment. When small amounts of toxic metals are in solution with larger amounts of alkali or alkaline earth cations, AMBERLITE IRC-718 resin will prefer the toxic metals over the other cations. This allows removal of the undesirable metal contaminant without the need to completely deionize the waste stream. Regenerant costs and, in some cases, capital costs can be reduced, since a smaller system can be used.

The macroreticular structure of AMBERLITE IRC-718 resin provides a number of advantages over traditional gel resins. It is highly resistant to osmotic shock, providing greater resin life than gel resins used under the same conditions. Due to the short ion diffusion path, the high porosity of AMBERLITE IRC-718 resin improves the kinetics of ion exchange.

AMBERLITE IRC-718 resin is ideal for use in non-aqueous media, such as chemical process streams, because of its macroreticular structure. The resin is based on a very stable styrene-divinyl benzene matrix. Typical physical properties are given in Table 1.

### Selectivity

The apparent selectivity of any ion exchange resin for a given metal depends upon concentration, the presence of other species, and pH. This makes absolute selectivities very difficult to determine, especially for waste treatment applications. Because of this, laboratory testing is essential when a resin is required to remove one or more types of metal ions selectively. Selectivities have been measured under various conditions

to give starting points for choosing a resin for selective metal removal.

TABLE 1

### Typical Physical Properties (These do not constitute specifications)

Appearance	Hydrated, opaque beads
Shipping Weight	42 lbs./ft. <sup>3</sup> (Na form)
Screen Grading (wet)	16 to 50 mesh (U.S. Standard Screen)
Moisture Content	62 to 68% (Na form)
Swelling	40% H → Na
Cation Exchange Capacity	1.1 meq/ml wet resin

### 1) pH = 2

Metal ion	$k^M_{Ca}$
Fe + + +	325,000
Cu + +	130,000
Hg + +	> 43,000
Au + + +	> 8,100
Ag +	4,600
Ni + +	3,200
Cd + +	620
Fe + +	190
Mn + +	120
Zn + +	120
Al + + +	50
Mg + +	20
Ca + +	1.0



2) pH = 4 Metal ion	$K_{Ca}^M$
Hg <sup>++</sup>	2,800
Cu <sup>++</sup>	2,300
Pb <sup>++</sup>	1,200
Ni <sup>++</sup>	57
Zn <sup>++</sup>	17
Cd <sup>++</sup>	15
Co <sup>++</sup>	6.7
Fe <sup>++</sup>	4.0
Mn <sup>++</sup>	1.2
Ca <sup>++</sup>	1.0

3) pH = 9, very high ammonium background (200g/L(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ). Metal ion	$K_{Ca}^M$
Ni <sup>++</sup>	30
Cd <sup>++</sup>	14
Cu <sup>++</sup>	10
Zn <sup>++</sup>	3
Ca <sup>++</sup>	1.0

These data provide a guideline of relative selectivities; no table can provide selectivities under all conditions. Using these starting points, the affinity of the resin for a given metal can be increased or decreased by adjusting pH.

### Operating Conditions

AMBERLITE IRC-718 resin is widely used in waste treatment, chemical processing, and hydrometallurgical applications where there is high variability in feed streams and competition from other species present in solution. Changes in pH, concentration, and background composition (especially complexing ions such as EDTA) may change sorption characteristics. The suggested conditions listed in Table 2 should, therefore, be considered a starting point; laboratory experiments should be done to determine the effectiveness of any ion exchange process for a desired application.

### Operating Capacity

The total exchange capacity of AMBERLITE IRC-718 resin in the sodium form is 1.1 meg/ml wet resin. In normal use, however, the resin bed is usually not completely exhausted during operation, and economic considerations may dictate less than 100 percent regeneration. Actual operating capacity will be less than the total exchange capacity and should be determined by specific applications testing.

### Backwashing

Many feed streams contain particulate matter which can clog an ion exchange bed. Backwashing is necessary at regular intervals to remove this material. When there are large amounts of suspended matter, backwashing may be necessary at each regeneration cycle. The backwash should be at a sufficient rate to give at least 50 percent bed expansion. The proper flow rate can be found in Figures 3 and 4.

### Exhaustion Flow Rate

Chelating ion exchange resins may have slightly poorer kinetic characteristics than conventional (strong electrolyte) ion exchangers. Flow rates,

TABLE 2

### Suggested Operating Conditions for AMBERLITE IRC-718

pH (minimum pH depends on application)	1.5 to 14.0
Maximum Temperature	190°F. (90°C) (Na) 160°F. (70°C) (H)
Service Flow Rate	1.0 to 4.0 gpm/ft. <sup>3</sup> (8.0 to 32.0 BV/hr.)
Regeneration	5 to 15% HCl or H <sub>2</sub> SO <sub>4</sub> 0.25 to 0.50 gpm/ft. <sup>3</sup> (2.0 to 4.0 BV/hr.) 6 to 12 lbs. acid/ft. <sup>3</sup>
Neutralization (if necessary)	1 to 3% sodium hydroxide or ammonium hydroxide 0.25 to 0.50 gpm/ft. <sup>3</sup> (2.0 to 4.0 BV/hr.) 2 to 4 lbs./ft. <sup>3</sup>
Pressure Drop	See Figures 1 and 2
Hydraulic Expansion During Backwash	See Figures 3 and 4

Figure 1. AMBERLITE IRC-718  
SODIUM FORM HMS—0.55

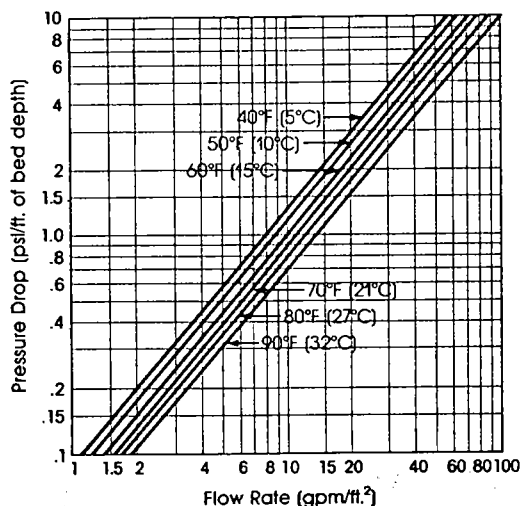


Figure 2. AMBERLITE IRC-718  
HYDROGEN FORM HMS—0.54

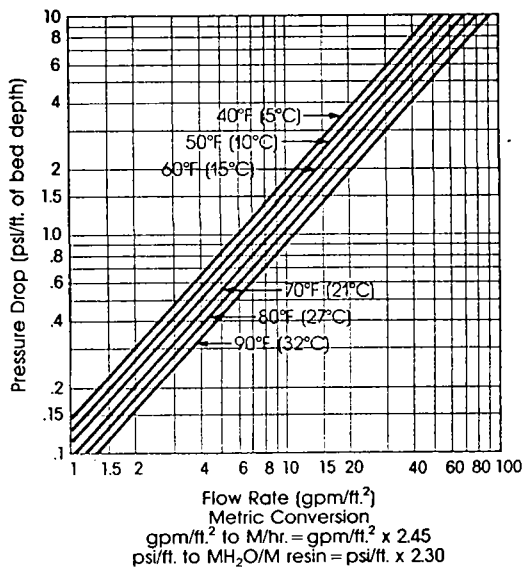


Figure 3. HYDRAULIC EXPANSION OF THE  
SODIUM FORM OF AMBERLITE IRC-718

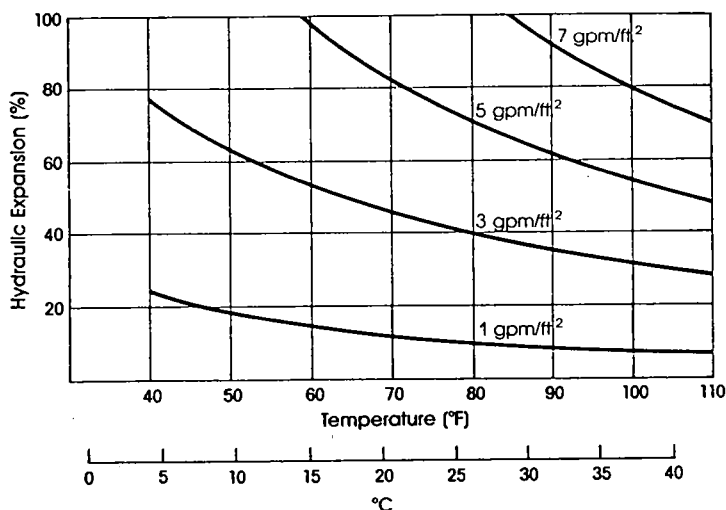
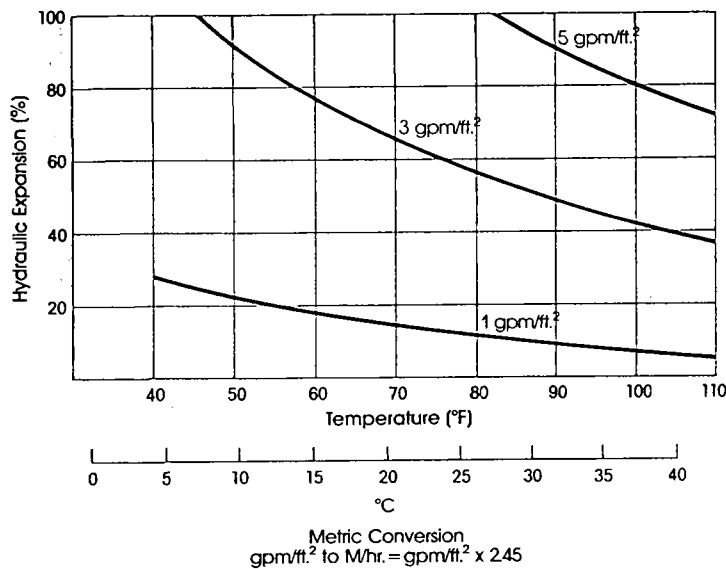


Figure 4. HYDRAULIC EXPANSION OF THE  
HYDROGEN FORM OF AMBERLITE IRC-718



therefore, may need to be somewhat slower than would be typical for water treatment resins. Flow rates of 1.0 to 4.0 gpm/ft.<sup>3</sup> (8.0 to 32.0 BV/hr.) should be acceptable for less demanding applications but should be reduced when high flow rates prevent efficient removal of the heavy metal ions.

## Regeneration

Because AMBERLITE IRC-718 resin has an extremely high affinity for metal ions, the amount of acid required is higher than that required for conventional weakly acidic ion exchange resins. A regeneration level of 6 to 10 lbs. HCl/ft.<sup>3</sup> may be

sufficient for metals with moderate selectivity, but this should be increased slightly for tightly held metals. Flow rates of 0.25 to 0.5 gpm/ft.<sup>3</sup> (2.0 to 4.0 BV/hr.) provide the best results. Acid concentration should be 5 to 15 percent, with higher concentrations needed for more tightly bound species.

## Neutralization

Because it is a weakly acidic chelating resin, AMBERLITE IRC-718 resin may require neutralization after acid regeneration. The resin should be rinsed after acid regeneration with several bed volumes of water, then treated with 2 to 4 lbs./ft.<sup>3</sup> of 1 to 3 percent sodium hydroxide or ammonium hydroxide. Neutralization may not be necessary for tightly bound ions or when the pH of the influent is high. Laboratory tests on individual influent compositions will indicate whether neutralization is needed.

## Commissioning New Resin

AMBERLITE IRC-718 resin is supplied in the sodium form and can, therefore, be used immediately after rinsing with several bed volumes of water.

## Suggested Applications

**Chlor-alkali Industry.** AMBERLITE IRC-718 resin effectively removes hardness to less than 50 ppb from brine that is fed to chlor-alkali electrolysis cells, although an aminophosphonic resin (such as DUOLITE® C-467) should be used for maximum performance.

**Purification of Process Streams Containing Trace Heavy Metals.** Objectionable levels of metals may be present in process streams due to their use as catalysts and their presence in raw mate-

rials. The high surface area and good selectivity of AMBERLITE IRC-718 resin, as well as its ability to remove metals over a wide pH range, make it a good choice for this application.

**Electroplating Industry.** The high selectivity of AMBERLITE IRC-718 resin for heavy metals is useful in the removal and recovery of metal ions from plating baths and rinse waters common to the plating industry. The macroreticular structure of this resin ensures its stability in the aggressive solutions of plating baths.

**Recovery of Heavy Metals from Hydrometallurgical Leach Streams.** Because of the ability of AMBERLITE IRC-718 resin to function over a wide pH range, it can be used to recover heavy metal cations that have been leached from ores by acids or bases. Elution of metals from this resin is extremely efficient, yielding a concentrated solution of the desired metal ion.

## Safe Handling Information

A Material Safety Data Sheet is available for AMBERLITE IRC-718. To obtain a copy contact your Rohm and Haas representative.

**CAUTION:** Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact.

Nitric acid and other strong oxidizing agents can cause explosive-type reactions when mixed with ion exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange resins, consult sources knowledgeable in the handling of these materials.

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These suggestions and data are based on information we believe to be reliable. They are offered in good faith, but without guarantee, as conditions and methods of use of our products are beyond our control. We recommend that the prospective user determine the suitability of our materials and suggestions before adopting them on a commercial scale.

Suggestions for uses of our products or the inclusion of descriptive material from patents and the citation of specific patents in this publication should not be understood as recommending the use of our products in violation of any patent or as permission or license to use any patents of the Rohm and Haas Company.



**Appendix B**

**APPENDIX B**

**LABORATORY REPORTS AND WASTEWATER TECHNOLOGY CENTRE  
AUDIT**



# MEMORANDUM

NOTE DE SERVICE

TO  
A

Dr. D. Major  
Beak Consultants Ltd.

File-N/Référence  
28896

FROM  
DE

Paul Bucens  
Wastewater Technology Centre

Date  
27 July 1992

SUBJECT  
OBJET

Wastewater Technology Centre Split Samples:  
Welland River Sediment Treatability Study

Six samples were split with Wastewater Technology Centre auditors during the treatability study of Welland River sediment. They were identified by Beak sample numbers and sample descriptions. The analyses results are presented in the accompanying table.

Results were reported by Beak for the samples identified as "Beak #02", "Beak #03", "Beak #04" and "Beak #05". With the exception of sample "Beak #04", the results reported by the WTC laboratory were very similar to those reported by Beak leaving no reason to doubt the Beak analyses. In light of this agreement, the gross differences in the results reported by the two laboratories for sample "Beak #04" are assumed to be due to mislabelling of the split sample.

No comparison could be made between the two laboratories for the analyses of "Beak #01" and "Beak #06" (untreated and treated whole sediment respectively) as the results were not reported by the Beak. However it is interesting to note that, based on the WTC analyses, the concentrations of most metals were only marginally reduced in the treated sample as compared with the untreated sample. In some cases (As, B, Fe, Pb and Ag) the concentrations increased. A word of caution, as these results represent a single sample in each case; these results should be seen as an indicator of the conditions rather than an absolute representation of the conditions.

# WELLAND RIVER SEDIMENT SAMPLE

Results from BEAK treatability testing (WTC Split Samples)

The "liquid" is filtered extract at various stages of extraction

The "solid" is the concentration on the solids in the sediment

Collected:

File:WRWTC4

Submitted: February 92

Received: April 92

WTC id. Description Beak id.	BEAK #01 Solid 11.110-02 ug/g	BEAK #02 Liquid 11.082-12 ug/mL	BEAK #03 Liquid 11.084-12 ug/mL	BEAK #04 Liquid 11.101-05 ug/mL	BEAK #05 Liquid 11.081-12 ug/mL	BEAK #06 Solid 11.110-05 ug/g
Sb	67	<0.50	<0.50	<0.50	<0.50	63
As	8758•	<1	163	699	114	10493
B	2.0	<0.05	<0.05	0.20	0.15	2.7
Cd	5.2	<0.05	<0.05	0.18	<0.05	4.7
Cr	218	<0.05	0.80	14	0.13	219
Cu	119	<0.03	1.9	12	<0.03	111
Fe	2.55%	<0.05	98	444	272	2.65%
Pb	76	<0.10	5.3	14	0.30	83
Mg	5460	2.3	30	88	208	4775
Mn	382	<0.05	2.6	18	10	380
Ni	291	<0.02	3.6	20	7.0	267
Se	341	<1	<1	<1	<1	341
Ag	2.6	<0.05	<0.05	<0.05	<0.05	2.8
Ti	746	0.02	0.30	0.13	<0.02	66
Th	5.4	<0.05	0.18	0.20	0.20	5.1
Zn	255	<0.05	10.8	25.0	13	234

As & Se - ug/kg for #01 and #06

As & Se - ug/L for #02, #03, #04 and #05

BEAK #01: Total sediment (sample), Untreated

BEAK #02: Total sediment (sample), Full sequential extraction (treatment), DDW 4

BEAK #03: Total sediment (sample), Full sequential extraction (treatment), EDTA 4

BEAK #04: Mislabeled? <75 um sediment (sample), Full sequential extraction (treatment), DDW 4

BEAK #05: Total sediment (sample), Full sequential extraction (treatment), HCL 4

BEAK #06: Total sediment (sample), Treated



## Appendix C



**APPENDIX C**

**CONCENTRATIONS AND CALCULATED MASSES OF METALS IN FILTERED  
EXTRACTION SOLUTIONS**

TABLE 1A: CONCENTRATION OF METALS IN FILTERED EXTRACTION SOLUTION

SAMPLE: Sorted Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	ACET 1	ACET 2	ACET 3	ACET 4	HCl 1	HCl 2	HCl 3	HCl 4	EDTA 1	EDTA 2	EDTA 3	EDTA 4	DDW 5
VOL mL	192	190	216	206	184	200	198	200	202	200	196	202	200	192	198	190	238
pH	6.92	7.25	7.62	7.82	5.51	5.22	5.15	5.13	4.88	4.64	4.46	4.39	7.71	8.31	8.44	8.48	
Zn mg/L	0.03	0.02	0.04	0.01	1.65	3.00	3.70	5.10	4.40	7.00	11.80	14.90	22.00	11.30	12.30	9.60	30.00
Cd mg/L	<0.002	<0.002	<0.002	<0.002	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.04	0.06	0.03	0.03	0.02	0.06
Pb mg/L	<0.02	<0.02	<0.02	<0.02	0.70	1.70	2.50	3.30	1.20	0.70	0.60	0.40	24.00	12.10	10.00	5.60	11.60
Ni mg/L	0.11	0.07	0.02	0.01	8.60	10.20	9.40	8.80	7.60	7.00	6.70	7.10	7.70	6.50	8.50	6.50	19.50
Fe mg/L	2.20	0.30	<0.02	<0.02	310.00	300.00	230.00	178.00	192.00	200.00	200.00	270.00	250.00	196.00	220.00	168.00	420.00
Mn mg/L	0.97	0.55	0.08	0.05	24.00	17.40	12.90	10.00	9.00	10.20	11.80	14.90	6.00	3.80	4.20	3.30	13.10
Cr mg/L	<0.01	<0.01	<0.01	<0.01	0.45	1.65	2.10	2.20	1.50	1.00	0.75	0.85	5.50	6.10	8.40	6.00	11.80
Mg mg/L	15.20	9.80	2.00	1.20	44.00	26.00	16.30	15.60	43.00	107.00	194.00	320.00	80.00	35.00	25.00	14.90	54.00
Be mg/L	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cu mg/L	<0.005	0.02	0.02	0.01	0.02	<0.02	0.02	0.04	<0.02	<0.02	0.02	0.04	4.20	4.90	7.20	5.60	9.80
Ag mg/L	<0.005	<0.005	<0.005	<0.005	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Al mg/L	0.10	0.12	0.16	0.16	7.20	32.00	47.00	58.00	38.00	31.00	27.00	43.00	600.00	340.00	280.00	161.00	330.00
Tl mg/L	<0.02	<0.02	<0.02	<0.02	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
As ug/L	5.00	5.00	<5	<5	65.00	95.00	95.00	105.00	160.00	125.00	90.00	90.00	330.00	250.00	340.00	280.00	490.00
Sb ug/L	4.00	4.00	2.00	4.00	20.00	20.00	20.00	20.00	10.00	10.00	<10	<10	145.00	100.00	125.00	115.00	160.00
Se ug/L	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 1B: CONCENTRATION OF METALS IN FILTERED EXTRACTION SOLUTION

SAMPLE: Sorted Sediment  
TREATMENT: Control

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	DDW 5	DDW 6	DDW 7	DDW 8	DDW 9	DDW 10	DDW 11	DDW 12	DDW 13	DDW 14	DDW 15	DDW 16	DDW 17
VOL mL	197	188	216	206	190	200	200	202	200	200	200	206	200	202	200	205	222
pH	6.92	7.30	7.62	7.68	7.10	7.65	7.73	7.39	7.90	7.81	7.43	7.71	7.35	7.72	7.62	7.60	
Zn mg/L	0.06	0.01	0.02	0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	0.02	0.07
Cd mg/L	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Pb mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Ni mg/L	0.11	0.06	0.02	0.01	0.04	0.03	0.02	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Fe mg/L	3.00	0.58	0.14	<0.02	0.10	<0.02	<0.02	<0.02	0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Mn mg/L	1.01	0.56	0.09	0.05	0.61	0.35	0.26	0.20	0.15	0.12	0.10	0.09	0.08	0.08	0.07	0.07	0.10
Cr mg/L	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mg mg/L	15.10	10.00	1.95	0.90	7.50	5.30	4.20	3.30	2.70	2.20	1.80	1.60	1.35	1.30	1.05	1.00	1.20
Be mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cu mg/L	0.01	0.03	0.05	0.04	0.01	0.01	0.01	0.01	<0.005	0.01	<0.005	0.01	0.04	0.03	0.03	0.01	0.02
Ag mg/L	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.02
Al mg/L	0.10	0.12	0.26	0.12	0.10	0.08	0.10	0.10	0.10	0.12	0.12	0.14	0.14	0.18	0.16	0.18	0.18
Tl mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.1	<0.02	<0.02	<0.02
As ug/L	6.00	4.00	<2	<2	5.00	3.00	4.00	3.00	4.00	4.00	4.00	4.00	8.00	5.00	5.00	5.00	15.00
Sb ug/L	4.00	2.00	2.00	2.00	<5	<5	5.00	5.00	5.00	5.00	5.00	5.00	4.00	8.00	8.00	10.00	20.00
Se ug/L	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<5

DDW - distilled water

ACET - acidic ammonium acetate solution

HCl - dilute HCl (pH 1.0)

EDTA - ammonium citrate and sodium EDTA solution

TABLE 1C: CONCENTRATION OF METALS IN FILTERED EXTRACTION SOLUTION

SAMPLE: Bulk Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	ACET 1	ACET 2	ACET 3	ACET 4	HCl 1	HCl 2	HCl 3	HCl 4	EDTA 1	EDTA 2	EDTA 3	EDTA 4	DDW 5
VOL mL	214	200	212	210	190	204	200	204	202	202	200	200	198	188	208	198	234
pH	7.10	7.36	7.83	7.91	5.47	5.36	5.26	5.21	4.95	4.72	4.40	4.32	7.91	8.37	8.55	8.55	
Zn mg/L	0.02	0.02	<0.01	<0.01	1.00	1.95	1.80	2.10	2.20	3.00	6.80	17.00	15.80	12.70	11.90	5.30	36.00
Cd mg/L	<0.002	<0.002	<0.002	<0.002	0.04	0.04	0.03	0.02	0.02	0.02	0.02	0.03	0.04	0.04	0.05	0.02	0.08
Pb mg/L	<0.02	<0.02	<0.02	<0.02	0.10	0.20	0.30	0.40	0.10	0.20	<0.1	0.10	9.90	6.90	5.00	1.60	7.20
Ni mg/L	0.04	0.02	0.01	<0.01	11.30	11.60	8.00	6.90	5.50	5.60	5.90	7.80	7.60	5.30	4.40	2.90	7.00
Fe mg/L	0.56	0.76	0.16	<0.02	450.00	460.00	390.00	270.00	220.00	230.00	300.00	310.00	250.00	147.00	115.00	74.00	197.00
Mn mg/L	0.09	0.07	0.03	0.02	21.00	18.60	12.90	10.80	8.00	8.80	10.00	12.10	5.00	2.40	1.70	0.55	3.60
Cr mg/L	<0.01	<0.01	<0.01	<0.01	0.15	0.30	0.30	0.35	0.10	0.05	0.10	0.10	2.10	1.45	1.15	0.75	1.60
Mg mg/L	2.90	2.00	1.50	1.00	72.00	48.00	28.00	30.00	62.00	130.00	210.00	300.00	88.00	36.00	21.00	12.80	53.00
Be mg/L	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cu mg/L	0.02	0.02	0.01	<0.005	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.88	1.60	2.10	1.66	2.80
Ag mg/L	<0.005	<0.005	<0.005	<0.005	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Al mg/L	0.46	0.64	0.24	0.04	0.80	1.50	1.70	2.10	0.80	0.70	1.60	2.40	107.00	60.00	40.00	22.00	44.00
Tl mg/L	<0.02	<0.02	<0.02	<0.02	0.20	0.10	<0.1	0.10	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
As ug/L	5.00	5.00	<5	<5	50.00	40.00	35.00	40.00	50.00	75.00	75.00	90.00	140.00	125.00	120.00	105.00	300.00
Sb ug/L	<2	<2	<2	<2	<10	<10	<10	<10	<5	<10	<10	<10	15.00	<5	25.00	10.00	20.00
Se ug/L	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 1D: CONCENTRATION OF METALS IN FILTERED EXTRACTION SOLUTION

SAMPLE: Bulk Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	DDW 5	DDW 6	DDW 7	DDW 8	DDW 9	DDW 10	DDW 11	DDW 12	DDW 13	DDW 14	DDW 15	DDW 16	DDW 17
VOL mL	218	204	220	198	194	200	202	200	208	208	200	204	198	208	202	204	214
pH	7.13	7.43	7.81	8.23	7.58	8.25	7.46	7.59	7.70	7.67	7.78	7.65	7.50	7.60	7.53	7.68	
Zn mg/L	0.23	0.05	<0.01	<0.01	0.02	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	0.01	0.03	<0.08
Cd mg/L	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Pb mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Ni mg/L	0.15	0.05	0.03	0.02	0.03	0.03	0.03	0.03	0.02	0.02	0.02	0.02	0.02	0.03	0.03	0.02	0.03
Fe mg/L	0.66	0.82	0.04	0.06	0.06	0.06	0.06	0.04	0.04	0.06	0.08	0.06	0.08	0.06	0.08	0.34	0.04
Mn mg/L	0.25	0.14	0.07	0.03	0.09	0.07	0.06	0.05	0.05	0.04	0.04	0.03	0.03	0.03	0.03	0.04	0.04
Cr mg/L	0.04	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mg mg/L	6.50	4.70	3.60	1.50	4.10	3.50	2.90	2.80	2.50	2.10	1.95	1.75	1.65	1.70	1.60	1.60	2.30
Be mg/L	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cu mg/L	0.17	0.02	0.01	0.01	0.01	0.02	0.01	0.01	0.01	0.01	<0.005	0.01	0.05	0.09	0.08	0.02	0.01
Ag mg/L	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Al mg/L	0.94	0.76	0.08	0.16	0.06	0.06	0.10	0.08	0.08	0.10	0.12	0.10	0.14	0.10	0.14	0.28	0.12
Tl mg/L	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
As ug/L	8.00	4.00	2.00	<2	3.00	2.00	<2	2.00	2.00	<2	2.00	2.00	6.00	3.00	3.00	3.00	5.00
Sb ug/L	<2	<2	<2	4.00	<5	<5	<5	<5	<10	<5	<5	<5	<5	<5	<5	<5	<5
Se ug/L	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<5	<2	<2	<2	<2

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 2A: MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Sorted Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	ACET 1	ACET 2	ACET 3	ACET 4	HCl 1	HCl 2	HCl 3	HCl 4	EDTA 1	EDTA 2	EDTA 3	EDTA 4	DDW 5
Zn ug	5.76	3.80	8.64	2.06	303.60	600.00	732.60	1020.00	888.80	1400.00	2312.80	3009.80	4400.00	2169.60	2435.40	1824.00	7140.00
Cd ug	0.00	0.00	0.00	0.00	3.68	4.00	3.96	4.00	4.04	4.00	3.92	8.08	12.00	5.76	5.94	3.80	14.28
Pb ug	0.00	0.00	0.00	0.00	128.80	340.00	495.00	660.00	242.40	140.00	117.60	80.80	4800.00	2323.20	1980.00	1064.00	2760.80
Ni ug	21.12	13.30	4.32	2.06	1582.40	2040.00	1861.20	1760.00	1535.20	1400.00	1313.20	1434.20	1540.00	1248.00	1683.00	1235.00	4641.00
Fe ug	422.40	57.00	0.00	0.00	57040.00	60000.00	45540.00	35600.00	38784.00	40000.00	39200.00	54540.00	50000.00	37632.00	43560.00	31920.00	99960.00
Mn ug	186.24	104.50	17.28	10.30	4416.00	3480.00	2554.20	2000.00	1818.00	2040.00	2312.80	3009.80	1200.00	729.60	831.60	627.00	3117.80
Cr ug	0.00	0.00	0.00	0.00	82.80	330.00	415.80	440.00	303.00	200.00	147.00	171.70	1100.00	1171.20	1663.20	1140.00	2808.40
Mg ug	2918.40	1862.00	432.00	247.20	8096.00	5200.00	3227.40	3120.00	8686.00	21400.00	38024.00	64640.00	16000.00	6720.00	4950.00	2831.00	12852.00
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cu ug	0.00	2.85	3.24	2.06	3.68	0.00	3.96	8.00	0.00	0.00	3.92	8.08	840.00	940.80	1425.60	1064.00	2332.40
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	19.20	22.80	34.56	32.96	1324.80	6400.00	9306.00	11600.00	7676.00	6200.00	5292.00	8686.00	120000.00	65280.00	55440.00	30590.00	78540.00
Tl ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
As ug	0.96	0.95	0.00	0.00	11.96	19.00	18.81	21.00	32.32	25.00	17.64	18.18	66.00	48.00	67.32	53.20	116.62
Sb ug	0.77	0.76	0.43	0.82	3.68	4.00	3.96	4.00	2.02	2.00	0.00	0.00	29.00	19.20	24.75	21.85	38.08
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution

TABLE 2B: MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Sorted Sediment  
TREATMENT: Control

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	DDW 5	DDW 6	DDW 7	DDW 8	DDW 9	DDW 10	DDW 11	DDW 12	DDW 13	DDW 14	DDW 15	DDW 16	DDW 17
Zn ug	11.82	1.88	4.32	2.06	1.90	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.02	0.00	4.10	15.54
Cd ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Pb ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ni ug	21.67	11.28	4.32	2.06	7.60	6.00	4.00	4.04	4.00	2.00	2.00	2.06	2.00	2.02	2.00	2.05	2.22
Fe ug	591.00	109.04	30.24	0.00	19.00	0.00	0.00	0.00	4.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mn ug	198.97	105.28	19.44	10.30	115.90	70.00	52.00	40.40	30.00	24.00	20.00	18.54	16.00	16.16	14.00	14.35	22.20
Cr ug	1.97	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mg ug	2974.70	1880.00	421.20	185.40	1425.00	1060.00	840.00	666.60	540.00	440.00	360.00	329.60	270.00	262.60	210.00	205.00	266.40
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cu ug	1.97	5.64	10.80	7.21	0.95	1.00	1.00	1.01	0.00	1.00	0.00	1.03	8.00	6.06	5.00	2.05	4.44
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	19.70	22.56	56.16	24.72	19.00	16.00	20.00	20.20	20.00	24.00	24.00	28.84	28.00	36.36	32.00	36.90	39.96
Tl ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
As ug	1.18	0.75	0.00	0.00	0.95	0.60	0.80	0.61	0.80	0.80	0.80	0.82	1.60	1.01	1.00	1.03	3.33
Sb ug	0.79	0.38	0.43	0.41	0.00	0.00	1.00	1.01	1.00	1.00	1.00	1.03	0.80	1.62	1.60	2.05	4.44
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

DDW - distilled water

HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution

EDTA - ammonium citrate and sodium EDTA solution

TABLE 2C: MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Bulk Sediment  
TREATMENT: Full Sequential Extraction

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	ACET 1	ACET 2	ACET 3	ACET 4	HCl 1	HCl 2	HCl 3	HCl 4	EDTA 1	EDTA 2	EDTA 3	EDTA 4	DDW 5
Zn ug	4.28	4.00	0.00	0.00	190.00	397.80	360.00	428.40	444.40	606.00	1360.00	3400.00	3128.40	2387.60	2475.20	1049.40	8424.00
Cd ug	0.00	0.00	0.00	0.00	7.60	8.16	6.00	4.08	4.04	4.04	4.00	6.00	7.92	7.52	10.40	3.96	18.72
Pb ug	0.00	0.00	0.00	0.00	19.00	40.80	60.00	81.60	20.20	40.40	0.00	20.00	1960.20	1297.20	1040.00	316.80	1684.80
Ni ug	8.56	4.00	2.12	0.00	2147.00	2366.40	1600.00	1407.60	1111.00	1131.20	1180.00	1560.00	1504.80	996.40	915.20	574.20	1638.00
Fe ug	119.84	152.00	33.92	0.00	85500.00	93840.00	78000.00	55080.00	44440.00	46460.00	60000.00	62000.00	49500.00	27636.00	23920.00	14652.00	46098.00
Mn ug	19.26	14.00	6.36	4.20	3990.00	3794.40	2580.00	2203.20	1616.00	1777.60	2000.00	2420.00	990.00	451.20	353.60	108.90	842.40
Cr ug	0.00	0.00	0.00	0.00	28.50	61.20	60.00	71.40	20.20	10.10	20.00	20.00	415.80	272.60	239.20	148.50	374.40
Mg ug	620.60	400.00	318.00	210.00	13680.00	9792.00	5600.00	6120.00	12524.00	26260.00	42000.00	60000.00	17424.00	6768.00	4368.00	2534.40	12402.00
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cu ug	3.21	4.00	1.06	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	174.24	300.80	436.80	328.68	655.20
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	98.44	128.00	50.88	8.40	152.00	306.00	340.00	428.40	161.60	141.40	320.00	480.00	21186.00	11280.00	8320.00	4356.00	10296.00
Tl ug	0.00	0.00	0.00	0.00	38.00	20.40	0.00	20.40	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
As ug	1.07	1.00	0.00	0.00	9.50	8.16	7.00	8.16	10.10	15.15	15.00	18.00	27.72	23.50	24.96	20.79	70.20
Sb ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.97	0.00	5.20	1.98	4.68
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution



TABLE 2D: MASS OF METAL RECOVERED IN FILTERED EXTRACTION SOLUTIONS (CALCULATED)

SAMPLE: Bulk Sediment  
TREATMENT: Control

Parameter	DDW 1	DDW 2	DDW 3	DDW 4	DDW 5	DDW 6	DDW 7	DDW 8	DDW 9	DDW 10	DDW 11	DDW 12	DDW 13	DDW 14	DDW 15	DDW 16	DDW 17
Zn ug	50.14	10.20	0.00	0.00	3.88	0.00	2.02	0.00	0.00	0.00	0.00	0.00	1.98	0.00	2.02	6.12	0.00
Cd ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Pb ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ni ug	32.70	10.20	6.60	3.96	5.82	6.00	6.06	6.00	4.16	4.16	4.00	4.08	3.96	6.24	6.06	4.08	6.42
Fe ug	143.88	167.28	8.80	11.88	11.64	12.00	12.12	8.00	8.32	12.48	16.00	12.24	15.84	12.48	16.16	69.36	8.56
Mn ug	54.50	28.56	15.40	5.94	17.46	14.00	12.12	10.00	10.40	8.32	8.00	6.12	5.94	6.24	6.06	8.16	8.56
Cr ug	8.72	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mg ug	1417.00	958.80	792.00	297.00	795.40	700.00	585.80	560.00	520.00	436.80	390.00	357.00	326.70	353.60	323.20	326.40	492.20
Be ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cu ug	37.06	3.06	1.10	0.99	1.94	3.00	2.02	1.00	2.08	1.04	0.00	1.02	8.91	18.72	15.15	3.06	2.14
Ag ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Al ug	204.92	155.04	17.60	31.68	11.64	12.00	20.20	16.00	16.64	20.80	24.00	20.40	27.72	20.80	28.28	57.12	25.68
Ti ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
As ug	1.74	0.82	0.44	0.00	0.58	0.40	0.00	0.40	0.42	0.00	0.40	0.41	1.19	0.62	0.61	0.61	1.07
Sb ug	0.00	0.00	0.00	0.79	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Se ug	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

DDW - distilled water

HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution

EDTA - ammonium citrate and sodium EDTA solution

TABLE 3: METAL CONCENTRATIONS IN UNFILTERED EXTRACTION SOLUTIONS

Parameter	SS - FSE DDW1	SS - C DDW1	TS - FSE DDW1	TS - C DDW1	SS - FSE ACET 1	SS - C ACET 1	TS - FSE ACET 1	TS - C ACET 1	SS - FSE HCl 1	SS - C HCl 1	TS - FSE HCl 1	TS - C HCl 1	SS - FSE EDTA 1	SS - C EDTA 1	TS - FSE EDTA 1	TS - C EDTA 1
Ag mg/L	<0.005	<0.005	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.005	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Al mg/L	2.3	2.7	144	106	61	5.2	32	186	52	3.9	11.5	123	730	2.8	153	96
As ug/L	10	10	30	25	90	12	60	45	110	5	60	20	330	10	110	22
Be mg/L	<0.02	<0.02	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.02	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Cd mg/L	<0.002	<0.002	0.01	0.005	0.01	<0.005	<0.005	0.01	<0.005	<0.002	<0.005	0.005	0.08	<0.005	0.035	0.005
Cr mg/L	0.07	0.08	0.44	0.36	2.4	0.16	0.36	0.6	1.9	0.11	0.22	0.46	9	0.08	2.5	0.38
Cu mg/L	0.065	0.07	0.53	0.44	1.29	0.14	0.35	0.75	0.11	0.08	0.09	0.56	7.4	0.05	1.34	0.44
Fe mg/L	6.8	7.7	69	51	610	8.5	780	220	280	4.4	370	153	400	2.9	360	118
Mg mg/L	16.7	17.2	48	37	81	10.5	106	57	56	4.3	87	38	131	2.3	129	30
Mn mg/L	0.99	1.02	1.58	1.3	32	0.78	23	1.78	10.8	0.26	9.9	1.24	9.4	0.14	6.9	0.98
Ni mg/L	0.18	0.19	1.44	1.16	13.3	0.24	14	1.9	9.3	0.13	6.8	1.44	12	0.1	10.5	1.18
Pb mg/L	0.06	0.06	0.45	0.4	0.7	0.15	0.3	0.25	0.7	0.08	0.15	0.45	26	0.25	10.2	0.65
Sb ug/L	<10	<5	10	<10	30	<10	<10	10	<10	5	<10	<10	180	<10	<10	<10
Se ug/L	<5	<2	<5	<2	<5	<2	<5	<2	<5	<5	<5	<5	<5	<2	<5	<2
Tl mg/L	<0.02	<0.02	<0.05	<0.05	0.15	<0.05	0.15	<0.05	<0.05	<0.02	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Zn mg/L	0.14	0.14	3.1	2.4	3.6	0.24	2.3	3.8	4.6	0.14	2.3	2.8	24	0.08	17.8	2.4

SS - Sorted Sediment  
TS - Total (Bulk) Sediment

FSE - Full Sequential Extraction  
C - Control

DDW - distilled water  
HCl - dilute HCl (pH 1.0)

ACET - acidic ammonium acetate solution  
EDTA - ammonium citrate and sodium EDTA solution