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METALS RECOVERY FROM INDUSTRIAL SLUDGES - PHASE I

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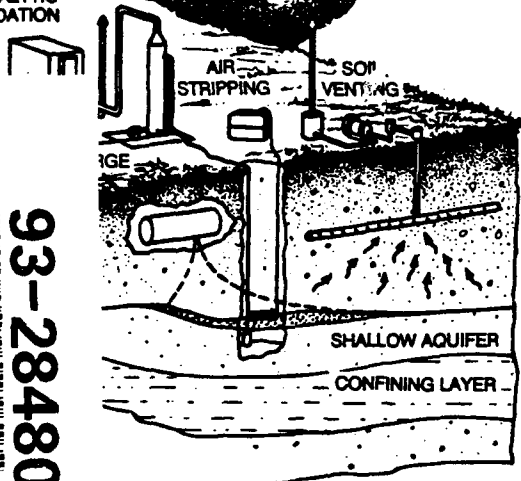
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13. ABSTRACT (Maximum 200 words)

This report describes the Metals Recovery from Industrial Sludges Program. The program objective is to identify or develop processes for reclaiming metals from a mixed, metal-bearing, waste sludge produced by the Industrial Waste Treatment Plant at Tinker Air Force Base. A Literature review, market survey, and preliminary laboratory tests have been completed. Metal separations appear promising through selective chemical precipitation processes. Other technologies including electrowinning, electrodialysis, and wet air oxidation may be combined with chemical precipitation to offer a feasible recovery process. A building was constructed around the Tinker AFB demonstration IWTP system in order to offer climatic protection to the other Air Force projects currently being performed at this site. Phase II of the program, currently under way, consists of bench-scale testing of identified unit processes and a continued search for alternative methods. At the end of Phase II an overall metals recovery scheme will have been identified and subjected to bench-scale testing and a data base generated for the design of a pilot field verification unit. Phase III of the metals recovery program will consist of design, construction, and operation and evaluation of the pilot field verification unit.

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EXECUTIVE SUMMARY

The objective of the Metals Recovery from Industrial Sludges Program is to identify or develop processes for reclaiming metals from a mixed, metal-bearing, waste sludge. The sludge is produced by the treatment of hazardous waste waters at the Industrial Waste Treatment Plant (IWTP) of Tinker Air Force Base (AFB), Oklahoma. The reclaiming process must not have an adverse impact on the operation of or the quality of effluent discharged from the IWTP.

Each day, the IWTP at Tinker AFB treats approximately 700,000 gallons of wastewater producing waste sludges which require special handling and costly disposal in approved hazardous waste landfills (\$220/ton in 1988). The relatively high concentrations of chromium and other metals, and the high cost of disposal may make recovery of these metals economically and environmentally desirable by reducing the amount of solids to be landfilled and by partial recovery due to metal recycle.

The scope of Phase I was to identify available methods for metals recovery from the metal-bearing waste sludge produced at the Tinker AFB IWTP, and to perform preliminary metals recovery laboratory studies. A literature review, survey of metal reclaimers, sludge sample characterization, and chemical leaching and precipitation separation testing have been performed. Phase II consists of bench scale testing of identified unit processes and a continued search for alternate methods. At the end of Phase II an overall metals recovery scheme will have been identified and subjected to bench scale testing and a data base generated for the design of a pilot field verification unit. In Phase III, the program will consist of design, construction, and operation, and evaluation of the pilot field verification unit.

The methodology used to conduct the research involved the identification of the existing problem. Next, the determination of potentially useful end products along with possible mechanisms for obtaining these products was made. Finally, the selected treatment options were evaluated.

Testing was conducted on the metal-bearing sulfide sludge generated at the Tinker AFB IWTP. The tests conducted involved the investigation of the sludge composition, selective leaching, and chemical precipitation for selective separations. The conditions of each test were controlled as much as possible to ensure the reproducibility of the results.

The literature review provided additional information concerning potentially applicable technologies such as wet air oxidation which could be coupled with chemical separation techniques to enhance metals recovery from sludges. Commercial ferrochromium smelters were not located, but researchers at the University of Idaho are working on a smelting process for recovering ferrochromium from electroplating waste sludges.

Results from the sequential and selective chemical leaching tests indicated that preferential leaching would be difficult due to the physical nature of the sludge. Promising results were obtained for separating trivalent chromium, iron, and aluminum from the divalent metals in the sludge. This is significant since approximately 80% of the metals within the sludge are trivalent metals.

The high concentration of oils and greases (approximately 25%) within the sludge may interfere with technologies such as ion exchange, electrowinning, and electrodialysis by coating resin, electrode, or membrane surfaces. A concerted approach involving oil removal or destruction may be the key to gaining more selectivity.

The conclusion drawn from the testing and market survey is that separation of the metals from the sludge is possible. To achieve the desired selectivity and efficiency would require a combination of technologies due to the nature of the sludge.

The market survey identified numerous reclaimers of metals which could accept a modified metal-bearing sludge if the sludge meets the reclaimer feed requirements.

The market survey could not identify a ferrochromium smelter within the U.S., but researchers at the University of Idaho are currently investigating a smelting process for recovering ferrochromium from electroplating waste sludges.

The sludge characterization study determined that oils and greases make up approximately 25% of the Tinker AFB sludge on a dry weight basis. The removal or destruction of the organic fraction in the sludge will be necessary before many of the identified technologies can be implemented. Additionally, the low metal concentration levels indicate that recovery of some metals may not be economical.

A high degree of separation of trivalent metals from divalent metals was achieved through the chemical precipitation of metal phosphate and hydroxide salts. This is significant since approximately 80% of the metal content is trivalent iron, chromium, or aluminum.

Technologies used in industry appear to be applicable to enhancing metals separation. Electrodialysis cells are available for chromic acid conversion from trivalent chromium, and other cells are capable of producing ferrous sulfate solutions from ferric solutions.

Removal of the organic fraction in the sludge is necessary for many of the identified metal separation or recovery technologies identified. It is recommended that wet air oxidation and electrochemical incineration technologies be investigated for applicability to the Tinker AFB sludge.

Commercial reclaimers should be contacted to determine what would make the Tinker AFB sludge more amenable for their processes. This may be the most economical avenue for processing the low concentration metals since some reclaimers may already be set up to process these metals.

Commercially available electrodialysis cells should be tested for chromic acid recovery from trivalent chromium. Electrodialysis may enhance the successful chemical precipitation separation of trivalent from divalent metals. Additionally, electrodialysis cells should be tested for recovering a

ferrous sulfate solution from the ferric portion of the trivalent metals. This solution could be used as a feed in the sodium sulfide-ferrous sulfate treatment process currently used at Tinker AFB. The benefit from this would be a reduction in chemical feed costs, and elimination of the ferric portion of the trivalent metals which would still have to be disposed of as waste.

The scoping report identified ion exchange as a potential separation technology if the iron could be removed. With the precipitation separation, the divalent metal portion of the waste may prove to be ideally suited for ion exchange technology. Ion exchange is a technology which should still be investigated.

Additionally, electrowinning should be further examined as a separation process.

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PREFACE

This report was prepared by the U.S. Department of Energy, Idaho Operations Office, EG&G Idaho Inc, P.O. Box 1625, Idaho Falls, ID 83415 for the Air Force Civil Engineering Support Agency, Tyndall AFB, Florida.

This report summarizes work completed during FY90. Lieutenant Phillip P. Brown was the AFCEA/RAVS Project Officer.

This report has been reviewed by the Public Affairs Office (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

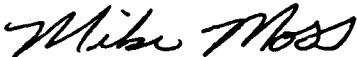
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LIST OF ABBREVIATIONS

| | |
|---------|--|
| AFB | Air Force Base |
| EDTA | Ethylenediaminetetraacetic acid |
| EPA | Environmental Protection Agency |
| ICP-AES | Inductively Coupled Plasma-Atomic Emission Spectroscopy |
| IWTP | Industrial Waste Treatment Plant |
| M | Molar |
| NPDES | National Pollutant Discharge Elimination System |
| SCC | Solids Contact Clarifier |

SECTION I INTRODUCTION

Hazardous waste waters are generated in Air Force aircraft refurbishing and maintenance operations. The contaminants are generated during metal cleaning, stripping, plating, and in paint stripping processes. The waste streams are combined producing mixed waste solutions containing metals, other inorganics, and organic contaminants. Treatment of these solutions produce sludges which must be disposed of as hazardous waste at significant cost to the Air Force. The relatively high concentrations of chromium and other metals, and the high cost of disposal may make recovery of these metals economically and environmentally desirable.

Previous efforts on the present task were reported by Suciu et al. (1). In summary, their report stated "The overall conclusion of this project is that the selectivity of the metal separation that was desired is not achievable with the technology at the conditions tested." Because of the lack of positive results or usable data from the efforts noted in Reference 1, new directions were developed for the FY-90 research and development effort. This report describes results of the FY-90 research and development program to recover metals from industrial sludges being conducted for the U.S. Air Force by the Idaho National Engineering Laboratory through the U.S. Department of Energy, Idaho Operations Office.

Subsequent to the initiation of the present study, a point source reclamation program has been put into operation at Tinker AFB. This has drastically reduced the amount of metals entering the IWTP, however, the heavy metal content of the water is still above that allowed for discharge under present and future National Pollutant Discharge Elimination System (NPDES) discharge limits.

A. OBJECTIVE

The objective of the program is to identify or develop processes for separating and recovering metals from a mixed, metal-bearing, waste sludge. The sludge is produced by the treatment of hazardous waste waters at the Industrial Waste Treatment Plant (IWTP) of Tinker Air Force Base (AFB), Oklahoma. The reclaiming process must not have an adverse impact on the operation of the IWTP or the quality of effluent discharged from this facility.

B. BACKGROUND

The Air Force aircraft refurbishing and maintenance operations include degreasing, alkaline cleaning, electrocleaning, acidizing, electrochemical deposition of protective metals, engine cleaning, and paint stripping (2). These processes produce waste streams containing oils and greases, metals, and complexing and chelating agents such as tartrates, phosphates, cyanide, ethylenediaminetetraacetic acid (EDTA), and ammonia (2). The hazardous metals include cadmium, chromium, copper, lead, nickel, and zinc. The aqueous mixed waste streams are typically treated in multiple stages. These stages include destroying cyanide, skimming off oils and greases, reducing hexavalent chromium to trivalent chromium, precipitating metals, and degrading all remaining organics by passing the remaining waste stream through an activated sludge system (2). This effluent is later filtered and readmitted to the environment.

The metal-bearing waste sludge is generated by the reduction of hexavalent chromium and subsequent precipitation of metals with the addition of sodium sulfide and ferrous sulfate at near neutral to slightly alkaline conditions. This process has maintained the effluent from the IWTP at Tinker AFB within the National Pollutant Discharge Elimination System (NPDES) Permit requirements when properly operated. Some of the NPDES metal constituent limits are listed in Table 1 (3).

TABLE 1. TINKER AFB NPDES PERMIT REQUIREMENT

| <u>Constituent</u> | <u>Effluent Limit (mg/L)</u> |
|----------------------|----------------------------------|
| Cadmium, total | 0.03 |
| Chromium, total | 1.0 |
| Chromium, hexavalent | 0.1 |
| Copper, total | 0.1 |
| Lead, total | 0.1 |
| Nickel, total | 1.0 |
| Zinc, total | 1.0 |

The IWTP at Tinker AFB treats approximately 700,000 gallons of wastewater per day producing between eight to nine tons of waste sludges per day. This waste sludge is approximately six to eight percent solids and is comprised of waste biological sludges from the activated sludge system and metal-bearing sludge from the IWTP Solids Contact Clarifier (SCC). The waste biological sludge contributes six to seven tons per day to the mixture, with the remaining one to two tons coming from the metal-bearing sludge. The waste biological and metal-bearing sludges are combined in a thickener tank. The sludges produced from these wastes are classified as EPA F006 wastes and require special handling and costly disposal (\$220/ton in 1988) in approved hazardous waste repositories. Additionally, the composition of the metal-bearing sludge is not constant and is dependent upon the daily aircraft refurbishing operations at Tinker AFB.

C. APPROACH/SCOPE

The approach used in this investigation is identifying of technologies available for treatment of this type of sludge, and determining physical characteristics of the sludge. This information provides a basis for the testing. This report presents the information obtained from literature, a survey of commercial metal reclaimers, and preliminary testing data. Included in the report are appendices describing the procedures used in testing, and specifications for the building enclosure for the Tinker AFB IWTP Field Demonstration Unit. The results, conclusions and recommendations are also presented.

SECTION II

PROGRAM DESCRIPTION

The program consists of three phases. In the scoping study performed in FY-1989, a literature search and market survey, physical characteristic studies, biological leaching studies, chemical leaching studies, and various metal recovery techniques were examined. The following is a description of the three program phases.

A. PHASE I: LITERATURE SEARCH/MARKET SURVEY AND PRELIMINARY LABORATORY STUDIES

1. Task 1: Focused Literature Search/Market Survey

In the scoping study recommendations section, chemical precipitation, smelting to obtain a ferrochromium product, ion exchange, and combinations of technologies were suggested to be the most promising metals recovery options. An additional literature review was performed to determine the extent, availability, and applicability of these processes. The option of identifying commercial reclaimers capable of accepting and processing the metal-bearing sludge was explored in a market survey.

2. Task 2: Sludge Characterization Studies

The initial characterization of the sludge in the scoping studies were inadequate for metals separation and recovery process identification. Analytical methods have been developed for determining the metals content of the sludge, extractable organics in the sludge, and dry solids content for the thickened metal-bearing sludge and vacuum filtered wet sludge cake. These methods were used for sludge characterization.

3. Task 3: Chemical Leaching and Precipitation Studies

The sequential dissolution method proposed in the scoping studies for selectively extracting individual metals from the metal-bearing sludge was

evaluated. This method incorporated the ability of selected leaching solutions to preferentially remove specific metals from the metal-bearing sludge.

Additionally, studies were performed to determine the most effective leaching solution for complete dissolution of the sludges in preparation for chemical precipitation separations.

4. Task 4: Other Metal Recovery Studies

Other technologies identified in Task 1 were explored further. These technologies included electrodialysis, wet air oxidation, and electrowinning.

5. Task 5: Construction of Building Around Tinker AFB Field Demonstration IWTP System

A portable building has been erected around the Tinker AFB field demonstration IWTP system. The building is equipped with all of the environmental controls necessary to allow research and operation of the field demonstration unit throughout the year without interruption due to weather conditions.

B. PHASE II: PROCESS OPTIMIZATION/FIELD DEMONSTRATION DESIGN

Phase II will consist of bench scale testing of identified unit processes and a continued search for alternative methods. At the end of Phase II an overall metals recovery scheme will have been identified and subjected to bench scale testing and a data base generation for the design of a field demonstration unit.

1. Task 1: Completion of Phase I Report

This report is the draft Phase I Final Report and describes work accomplished in FY-90.

A draft of this report will be distributed for review and comment. Comments will be resolved and the final report submitted to the Air Force Engineering and Services Center.

2. Task 2: Revise Cost Account Plan

A revised Cost Account Plan for this program is to be prepared and issued. This revised plan will identify a detailed schedule of the work to be accomplished in FY-91 and is to include additional testing of alternate metal recovery schemes. The revised plan will include any changes made necessary by the previous year's research.

3. Task 3: Laboratory and Bench Scale Dynamic Testing

Laboratory testing is required to further define a process that will operate effectively on the material designated for effective metals recovery. Bench scale tests will continue to be conducted to determine the effect of continuous operation on the efficiency of metal recovery from each unit process. Each unit process and operation will be tested for compatibility with the "total process." A total process will be selected, based on feasibility, compatibility, and economics. Included in this task are efforts on sludge characterization, test plan preparation, and safety analysis for both the Idaho Falls and Tinker AFB laboratories and test facilities. The process will be evaluated for compliance with state and federal regulations.

4. Task 4: Process Economic Evaluation and Regulatory Analysis

The process(es) selected for metals recovery will be subjected to economic analysis to determine viability as a method for sludge treatment.

5. Task 5: Pilot Field Verification Unit Engineering Design

The Pilot Field Verification Unit will be designed to accomplish the metals recovery required by this program. The design is to be based on the results of Task 3. Sufficient detail will be provided for modification of the

existing pilot plant to permit integration of the metals recovery system into that facility at Tinker AFB, Oklahoma. A final report on the results of laboratory testing, process selection, and economics will be prepared.

6. Task 6: Procurement of Materials and Components

Procurement shall be initiated for long lead materials and materials and components that will be needed to construct the pilot field verification unit. Initiation of this task shall be conducted in a timely manner to eliminate delays in construction and operation of this unit.

C. PHASE III: PILOT FIELD VERIFICATION UNIT TESTING

Phase III of the metals recovery program will consist of design, construction, and operation and evaluation of the pilot field verification unit. This phase will begin in FY-92.

1. Task 1: Construction of Pilot Field Verification Unit

A site will be prepared for construction of the pilot field verification unit at Tinker AFB. The pilot field verification unit is to be constructed and integrated into the existing pilot plant at Tinker AFB. The unit is to consist of all unit processes and operations selected for optimal metal recovery from the sludge produced by the sodium sulfide/ferrous sulfate process.

2. Task 2: Operation of Pilot Field Verification Unit

The pilot field verification unit will to be used to optimize each of the processes with respect to concentration, flow rate, temperature, etc. As each step is optimized, it is anticipated that some previous steps may require modifications. Following optimization, the pilot plant process is to be operated continuously for a period of time to determine the impact of continuous operation and changes in the sludge on the overall process. The

resulting data generation will provide sufficient information to completely design, construct, and operate a system at the IWTP for recovery of heavy metals from the Tinker AFB industrial effluent.

3. Task 3: Preliminary Process Economics Determination

The economic analysis will include disposition costs or salvage value of the final product from the metal recovery system in the system's recommended configuration. The analysis will evaluate the unit operations and unit processes and apply appropriate costs for construction and continuous operation. The analysis will consider all of the necessary regulatory requirements for discharge, transportation, etc. of the materials involved.

4. Task 4: Final Report Preparation

A final report on the operation of the metals recovery field demonstration unit will be prepared and will include the operating ranges found for the system and the interaction of the metals recovery system on overall field demonstration operation. Further, the report will include all environmental and economical information necessary to determine whether or not to implement full-scale. The report will contain all the necessary data and information from all of the listed tasks which is necessary to support the conclusions. The report will contain the necessary data to design, construct, and maintain a full-scale system.

SECTION III

RESULTS

A. PHASE I: LITERATURE SEARCH/MARKET SURVEY AND PRELIMINARY LABORATORY STUDIES

1. Task 1: Literature Search/Market Survey

a. Literature Search

A literature review focusing on metal separations was performed to determine the extent, availability, and applicability of chemical precipitation, smelting to obtain ferrochromium, and ion exchange technologies. During this literature review, other technologies became apparent including selective electrowinning, electrodialysis, and wet air oxidation. Some technologies which appear promising are described below.

Chemical Precipitation. Chemical precipitation processes involve the addition of chemicals to form insoluble metal salts. Metals may be precipitated as hydroxides, sulfides, carbonates, phosphates, or other insoluble salts (4,5,6,7). Since metals have various optimum pH conditions at which they precipitate, pH can be adjusted to obtain metal separations. A 1988 report by Twidwell recommends the use of phosphates as a means of precipitating iron and chromium in the presence of divalent heavy metal cations (5). It was shown that the addition of stoichiometric quantities of phosphate ions could be used to precipitate primarily one metal at a time (5).

Ion Exchange. Ion exchange has been used as a preliminary step with many technologies for the concentration of metal species (8,9). Ion exchange resins have "exchangeable" ions bound within the resin. Ion exchange depends on the electrochemical potential of the ion to be recovered versus that of the exchange ion, and on the concentration of ions in solution. Once the resin is spent, it is recharged by exposing it to a concentrated solution of the original exchange ion so a reverse reaction takes place, and a concentrated solution of the removed ion is formed. In the scoping studies, it was

suggested that ion exchange resins may offer a cost-effective way of recovering metals if a low-cost means of removing iron can be developed. Ion exchange is becoming important for waste reduction and recovery of metals for reincorporation in electroplating rinse baths (10,11,12).

Electrowinning. Electrowinning is frequently evaluated as a technique for metal recovery (13,14,15). Its increasing popularity is most likely due to the relatively low amounts of chemicals required for its use and the small quantities of wastes that are generated. Research has been performed to test the possibilities of direct electrowinning from acidified sludges and sludge incinerator ash with limited success (14,15). It has been found that some selectivity can be achieved in electrowinning depending upon metal ion types and solution pH (14).

Electrodialysis. Electrodialysis is becoming important as a waste reduction and recovery method in the electroplating industry (12,16). This technique concentrates ionic species contained in a water solution by applying an electrical potential across an ion-selective membrane to provide the force for ion migration. The ion-selective membranes are normally thin sheets of ion-exchange resin reinforced with synthetic fiber backing. Electrodialysis plays an important role in the conversion and separation of the metal species for electroplating bath reincorporation (12,16).

Wet Air Oxidation. Approximately 25% of the metal-bearing sludge at the Tinker AFB IWTP, based upon dry weight, was found to consist of extractable organic compounds which were mainly oils and greases. The presence of organics in such large amounts may cause problems with previously identified technologies such as ion exchange, electrowinning, and electrodialysis. The problem is that the organics can coat the resins and/or electrode surfaces and blind or reduce the process efficiency of each respective technology. This prompted the search for a method of destruction of organics. The technologies found include roasting or incineration, solvent extraction, electrochemical incineration and wet-air oxidation. Wet-air oxidation destroys organics in an aqueous solution. This oxidation reaction is exothermic and can be self-sustaining if sufficient organics are present

(17). The gaseous effluent is predominantly nitrogen and carbon dioxide so there is no requirement for offgas processing.

Electrochemical Incineration. An electrochemical incineration process for the safe, low-temperature destruction of organic waste material is currently being developed by the United Kingdom Atomic Energy Authority's Chemical Processes Section in the Reprocessing Group at Dounreay, Scotland (18). The process utilizes the electrochemical production of the Ag(II) cation which reacts with water to form oxidizing and reactive hydroxide radicals. A number of organics have been shown to be amenable to treatment. The list to date includes rubber gloves, polyurethane, epoxy resins, hydraulic fluid, lubricating oil, ion exchange resins, tributyl phosphate and kerosene (18). Dounreay currently has a pilot facility which is being used to determine chemical engineering parameters required for the commercialization of the process.

b. Market Survey

A market survey was performed to identify commercial reclaimers with the ability to accept and process the metal-bearing sludge produced at the Tinker AFB IWTP. The commercial reclaimers contacted appear in Table 2. In order for a reclaimer to accept the waste sludges, the waste generator generally pays a fee depending on the sludge make-up. Most of the reclaimers listed in Table 2 cannot accept the Tinker AFB sludge in its present form due to feed requirements for their processes. If the sludge were tailored to meet the feed requirements of a reclaimer, the degree to which the sludge metals would have to be separated would be substantially reduced. A sludge sample should be sent to each firm for analysis in order for the reclaimers to suggest modifications which would allow the sludge to become acceptable for their processes. In general, unwanted components of the sludge include all organic matter, cyanide, iron, and aluminum.

As recommended in the scoping report, smelting to produce a ferrochromium product was explored. This technology would provide many benefits since most of the metal content of the sludge is iron and chromium.

TABLE 2. COMMERCIAL RECLAIMERS OF METALS

| | |
|--|--|
| Chem-Tech Systems (19) 3650 E. 26th St. Los Angeles, CA 90023 (213) 268-5056 | INMETCO (24) International Metals Reclamation Co. P.O. Box 720 Ellwood City, PA 16117 (412) 758-5515 |
| CP Chemicals (20) Environmental Recovery Services One Parker Plaza Fort Lee, NJ 07024 (201) 944-6020 | JC Inc., Liquid Waste Disposal (25) 3650 E. 26th St. Los Angeles, CA 90023 (213) 268-3137 |
| Cypress Miami Mining Co. (21) Resource Recovery P.O. Box 4444 Claypool, AZ 85532 (602) 473-7080 | RECONTEK (26) 28 East Yates P.O. Box 461 Newman, IL 61942-0461 (217) 83-2461 |
| Encycle (22) 5500 Up River Rd. Corpus Christi, TX 78407 (512) 289-0035 | RFE Industries (27) Smelting and Refining Division 19 Crows Mill Road Keasbey, NJ 08832 (201) 738-5200 (800) 738-7938 |
| ETICAM (23) 2095 Newlands Dr. E. Fernley, NV 89408 (800) 648-9931 (702) 575-2760 | WATH Recycling (28) Amlon Metals, Inc. 40 Rector St. New York, NY 10008 (212) 742-1043 |

Additionally, ferrochromium is a strategic material, and currently all that is used in the U.S. is imported. It was discovered that the only ferrochromium smelter in the U.S. has shut down operations. According to Cypress Miami Mining, starting up a new ferrochromium smelter with current technology is also extremely complicated and cost intensive. Currently, researchers at the University of Idaho are working on a smelting process for making ferrochromium from electroplating waste sludges. Although smelting is still an option, more readily available technologies were explored in Phase I.

2. Task 2: Sludge Characterization Studies

The initial characterization of the sludge in the scoping studies provided insufficient information on the sludge composition. A more thorough analysis on metals content, extractable organics, and dry solids content for the thickened metal-bearing sludge and vacuum-filtered sludge-cake was performed. Analytical methods for determining the sludge characteristics appear in Appendix A. The metals concentration of the sludge appear in Table 3.

These concentrations were determined using inductively coupled plasma-atomic emission spectroscopy (ICP-AES). It is apparent that the metal content of the sludge is low, and recovery of many of the metals may not be economical.

TABLE 3. METAL CONCENTRATION IN ^cDRY SLUDGE DETERMINED BY ICP-AES

| <u>Metal</u> | <u>% Concentration (Sludge Fraction)</u> |
|------------------------|--|
| ^a Aluminum | 1.02 ± 0.09 |
| ^a Barium | 0.25 ± 0.02 |
| ^a Cadmium | 0.046 ± 0.006 |
| ^a Chromium | 3.2 ± 0.4 |
| ^a Copper | 0.053 ± 0.006 |
| ^a Iron | 2.5 ± 0.3 |
| ^a Lead | 0.052 ± 0.005 |
| ^a Manganese | 0.33 ± 0.04 |
| ^a Nickel | 0.37 ± 0.03 |
| ^b Arsenic | Not Detected |
| ^b Calcium | 2.60 |
| ^b Selenium | Not Detected |
| ^b Silver | 0.039 |
| ^b Sodium | 0.048 |
| ^b Tin | 0.012 |
| ^b Zinc | 0.26 |

^aQuantitative analysis performed on these metals. (± 10%)

^bQualitative analysis performed on these metals. (± 25%)

^cOven dried at 100°C for 48 hr.

Oils and greases can interfere with the operation efficiencies of previously identified technologies. This made it is essential to know the organic content of the sludge. The results of the extractable organics analysis are shown in Table 4. To determine the nature of the hydrocarbon content, aliquots of the filtrates from the oil and grease analysis were analyzed by gas chromatography. The sludge shows essentially two different hydrocarbon products. The first component has a hydrocarbon chain of approximately C-9 through C-17, and the second component is a mixture of hydrocarbons with lengths greater than C-20. Examples of two petroleum distillates that give similar chromatographic results when mixed are diesel fuel and motor oil.

The metal-bearing sludge contains a substantial amount of water. Much of the sludge disposal costs, therefore, is associated with the disposal of water. A study was performed for the dewatering of the sludges at the Tinker AFB IWTP (29). To obtain an approximation of the solids content of the two forms of sludge that may be obtained, a thickened sludge and a vacuum filtered sludge cake were tested for dry solids content. The results of the dry solids determination on the thickened metal-bearing sludge and vacuum filtered wet sludge cake appear in Table 5.

TABLE 4. EXTRACTABLE ORGANICS FROM SLUDGE

| <u>% Extractable Organics Neutral Sludge</u> | <u>% Extractable Organics Acidified Sludge</u> |
|--|--|
| 23 \pm 2 | 27 \pm 3 |

TABLE 5. DRY SOLIDS CONTENT

| <u>Sludge Type</u> | <u>% Solids</u> |
|--------------------|-----------------|
| Thickened Sludge | 8.36 \pm 0.01 |
| Sludge Cake | 27.7 \pm 0.2 |

3. Task 3: Chemical Leaching and Precipitation Studies

a. Sequential Dissolution

A procedure proposed in the scoping studies for sequentially and selectively extracting individual metals from the metal-bearing sludge was tested. The procedure is listed in Appendix A. A sample of dry sludge was sequentially exposed to seven leaching solutions, each stronger than the previous solution. Each solution was designed to preferentially leach a specific metal within the sludge. It was anticipated that the more difficult to leach metals would remain within the sludge until becoming exposed to stronger leaching agents. As illustrated in Figure 1, the selectivity of a sequential leaching scheme will not work under the conditions tested. This may be due in part to the high oils and grease content of the sludge, and the nonhomogeneous dispersion of metals within the sludge.

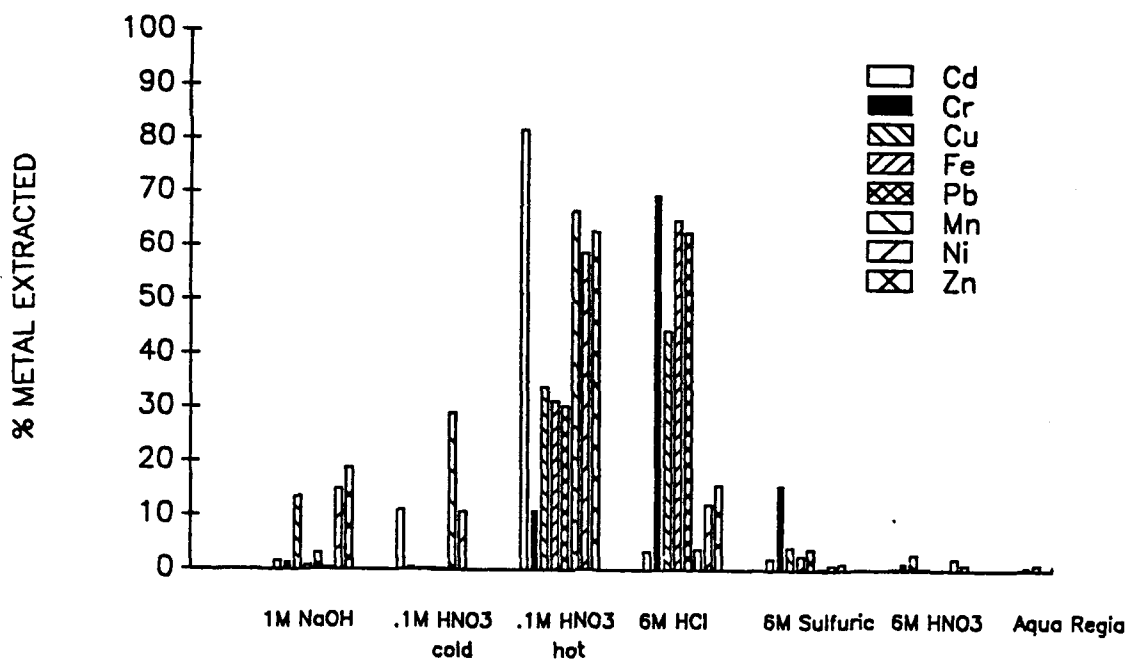


FIGURE 1. SEQUENTIAL LEACHING OF METALS

For comparison of the relative leaching strengths, each of the solutions used in the sequential dissolution were individually tested on fresh sludge samples. The results of these tests appear in Table 6. The table shows that all of the concentrated acids performed well for dissolving metals from the sludge. Aqua regia was by far the best solution for removing the metals from sludge and it was therefore used as the reference for maximum metal removed from sludge. The instrumental error in concentration determination was expected to be about 10%. An anomalous result occurred for zinc removal with sulfuric acid. Sulfuric acid removed nearly twice the amount of zinc expected, therefore that data point is suspect as an invalid quantity.

A chemical precipitation procedure for selectively precipitating iron and chromium in the presence of divalent metal cations was identified in the literature review. An initial scoping test utilizing phosphate precipitation is illustrated in Figure 2 and Table B-1 (Appendix B). It is readily apparent the iron and chromium contained in the concentrated metal solution was reduced from over ten times the concentration of other metals in the solution, to less than the concentration of many of those other metals. The precipitation occurred in a pH range in which most of the other metals in solution were not affected by the precipitation reaction.

TABLE 6. COMPARISON OF LEACHING SOLUTIONS
(percent of metal extracted based on aqua regia)

| Metal | NaOH 1M | HNO ₃ 0.1M | HNO ₃ 0.1M hot | HCl 6M | H ₂ SO ₄ 6M | HNO ₃ 6M | Aqua Regia hot |
|----------|------------|--------------------------|------------------------------|-----------|--------------------------------------|------------------------|-------------------|
| Cadmium | 0 | 4 | 8 | 97 | 93 | 97 | 100 |
| Chromium | 1 | 0 | 0 | 82 | 92 | 97 | 100 |
| Copper | 17 | 10 | 25 | 91 | 12 | 100 | 100 |
| Iron | 1 | 2 | 4 | 29 | 38 | 36 | 100 |
| Lead | 2 | 1 | 1 | 89 | 5 | 86 | 100 |
| Nickel | 18 | 61 | 20 | 89 | 96 | 88 | 100 |
| Zinc | 39 | 53 | 25 | 59 | 218 | 27 | 100 |

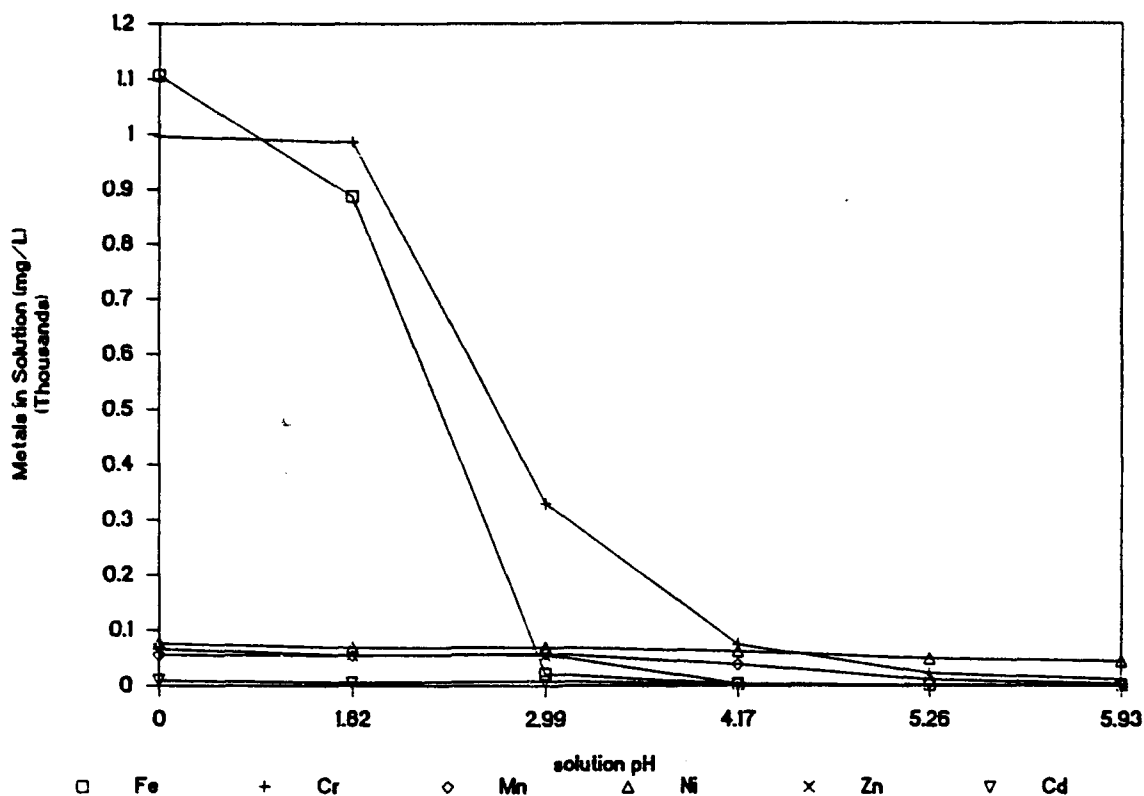


FIGURE 2. PHOSPHATE PRECIPITATION SCOPING STUDY AFTER SULFURIC ACID LEACHING

b. Phosphate Precipitation

A similar procedure, listed in Appendix A, was attempted with a metal-bearing sludge leached with sulfuric acid. The results of the chemical precipitation tests for three trivalent metals, aluminum, chromium, and iron, are shown in Figure 3 and Table B-2. It can be seen that dramatic decreases of approximately 400 mg/L to less than 4 mg/L of iron, approximately 400 mg/L to less than 3 mg/L of chromium, and approximately 27 mg/L to less than 2 mg/L of aluminum in solution has been obtained.

c. Hydroxide Precipitation

An initial scoping test utilizing a chemical precipitation with ammonium sulfate and ammonium hydroxide is illustrated in Figure 4 and Table B-3. Again it can be seen that the iron and chromium contained in the

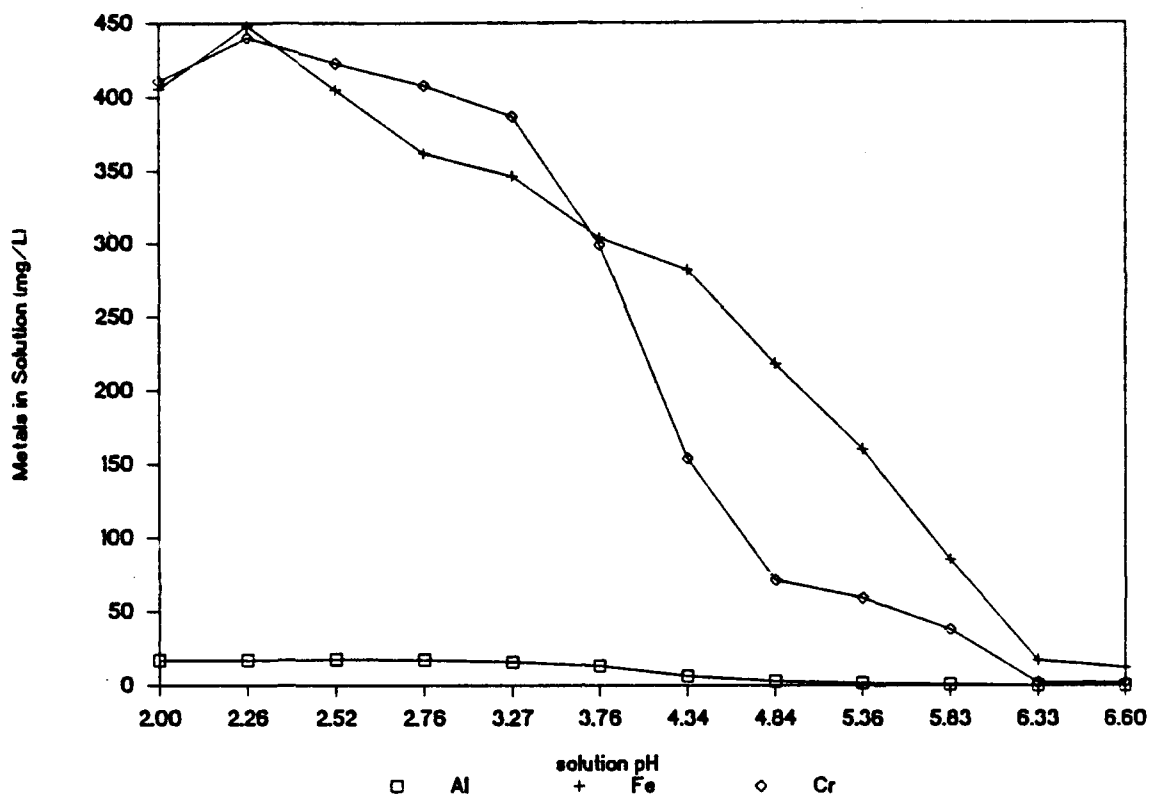


FIGURE 3. PHOSPHATE PRECIPITATION AFTER SULFURIC ACID LEACHING

concentrated metal solution was reduced to levels resembling the phosphate precipitation, while the other metals in solution remained relatively unaffected by the chemical addition. The procedure for subsequent ammonium sulfate/ammonium hydroxide, and also a sodium hydroxide precipitation reaction is listed in Appendix A. The results for aluminum, chromium, and iron appear in Figure 5 and Table B-4 for the ammonium sulfate/ammonium hydroxide precipitation, and in Figure 6 and Table B-5 for the sodium hydroxide precipitation.

The major difference noted between the different precipitations was the shifting of the precipitation pH for the phosphate precipitates versus the hydroxide precipitates. The presence of ammonium appears to have little effect on hydroxide precipitation separations.

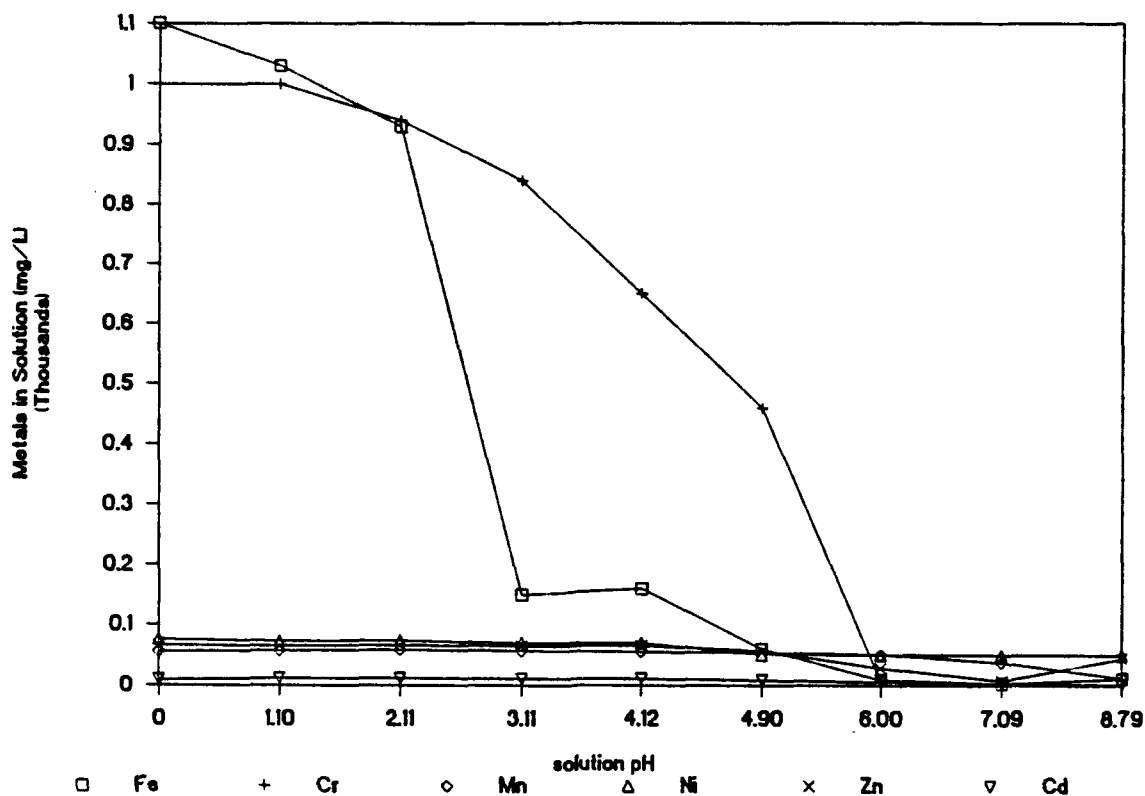


FIGURE 4. AMMONIUM SULFATE/AMMONIUM HYDROXIDE PRECIPITATION SCOPING STUDY AFTER SULFURIC ACID LEACHING

4. Task 4: Other Metal Recovery Studies

The high concentration of oils and greases in the metal-bearing sludge produced at the Tinker AFB IWTP indicate the need for pretreatment of these sludges. Wet air oxidation is a technology for organics destruction identified in the literature survey. Zimpro/Passivant in Rothschild, Wisconsin was contacted concerning this technology and its applicability to the metal-bearing sludge. Zimpro/Passivant will test sludge samples for \$3,000 to \$6,000 per sample (17).

The results of the chemical precipitation studies indicated the need for additional processing technologies to obtain further metal separations. Electrodialysis methods were identified to process the fraction obtained from the chemical precipitation studies containing predominantly chromium, iron,

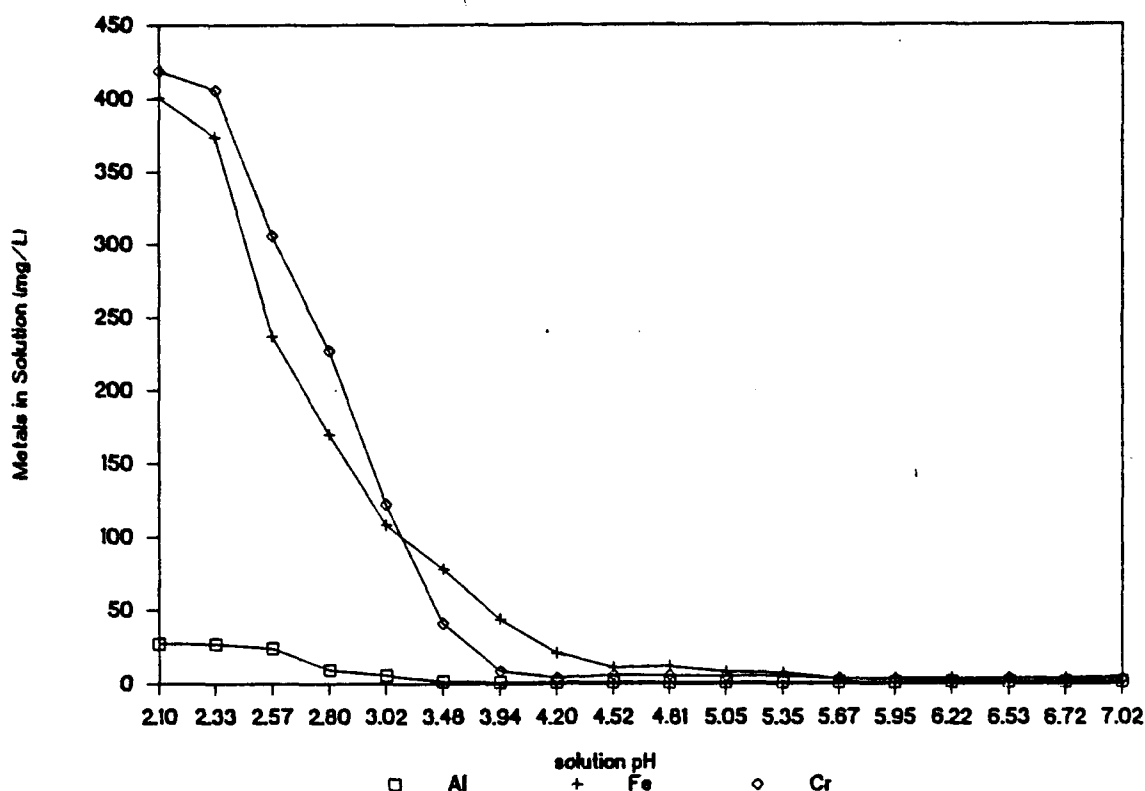


FIGURE 5. AMMONIUM SULFATE/AMMONIUM HYDROXIDE PRECIPITATION AFTER SULFURIC ACID LEACHING

and aluminum. An electrolytic cell manufactured by the IONSEP Corporation in Rockland, Delaware has been used in industry for chromic acid recovery from mixed metal solutions containing trivalent chromium. Trivalent chromium conversion to hexavalent chromium has been reported to be up to 90 to 95% efficient after one pass using a commercial IONSEP cell. Rather than paying for the disposal of the remaining iron, aluminum, and small amounts of chromium left from chromic acid conversion, a low-grade ferrous sulfate solution for use in the sodium sulfide-ferrous treatment process may be produced using another electrolytic cell. A cell for converting the trivalent ferric ion to the divalent ferrous ion has been developed by the Electrosynthesis Corporation. Conversion is expected to be between 85 to 90% efficient.

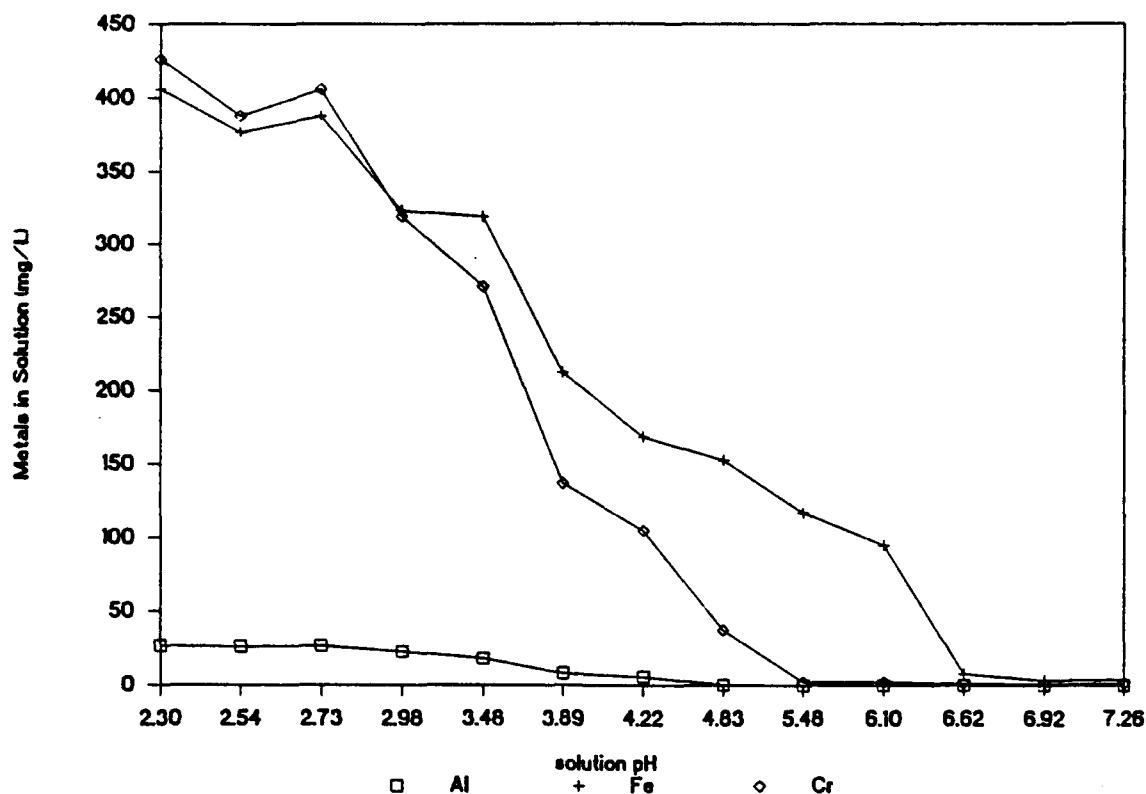


FIGURE 6. SODIUM HYDROXIDE PRECIPITATION AFTER SULFURIC ACID LEACHING

Oliver (14), reported on the selective removal of specific ions using electrochemical techniques. This was achieved by controlling solution pH, and plating potentials. The tests focused on mixed solutions of copper, zinc, nickel and cadmium. It was found that all four metals could be easily plated from neutral salt solutions, but only copper and cadmium could be plated from strongly acidic solutions.

5. Task 5: Construction of Building Around Tinker AFB Field Demonstration IWTP System

The building around the Tinker AFB field demonstration IWTP system was constructed in FY-90. This was to allow for other Air Force projects currently being performed at the Tinker AFB field demonstration IWTP to be sheltered from various climate conditions. The building is equipped with all of the environmental controls necessary to allow research and operation of the

field demonstration unit throughout the year without interruption due to weather conditions. Specifications of the building enclosure are listed in Appendix B.

SECTION IV

CONCLUSIONS

The conclusions drawn from the testing and market survey is that separation of the metals from the sludge is possible. To achieve the desired selectivity and efficiency would require a combination of technologies due to the nature of the sludge.

The market survey identified numerous reclaimers of metals which could accept a modified metal-bearing sludge if the sludge meets the reclaimer feed requirements.

The market survey could not identify a ferrochromium smelter within the U.S., but researchers at the University of Idaho are currently investigating a smelting process for recovering ferrochromium from electroplating waste sludges.

The sludge characterization study determined that oils and greases make up approximately 25% of the Tinker AFB sludge on a dry weight basis. The removal or destruction of the organic fraction in the sludge will be necessary before many of the identified technologies can be implemented. Additionally, the low metal concentration levels indicate that recovery of some metals may not be economical.

A high degree of separation of trivalent metals from divalent metals was achieved through the chemical precipitations of phosphate and hydroxide metal salts. This is significant since approximately 80% of the metal content is trivalent iron, chromium, or aluminum.

Technologies used in industry appear to be applicable to enhancing metals separation. Electrodialysis cells are available for chromic acid conversion from trivalent chromium, and other cells are capable of producing ferrous sulfate solutions from ferric solutions.

SECTION V

RECOMMENDATIONS

Removal of the organic fraction in the sludge is necessary for many of the identified metal separation or recovery technologies identified. It is recommended that wet air oxidation and electrochemical incineration technologies be investigated for applicability to the Tinker AFB sludge.

Commercial reclaimers should be contacted to determine what would make the Tinker AFB sludge more amenable for their processes. This may be the most economical avenue for processing the low concentration metals since some reclaimers may already be set up to process these metals.

Commercially available electrodialysis cells should be tested for chromic acid recovery from trivalent chromium. Electrodialysis may enhance the successful chemical precipitation separation of trivalent from divalent metals. Additionally, electrodialysis cells should be tested for recovering a ferrous sulfate solution from the ferric portion of the trivalent metals. This solution could be used as a feed in the sodium sulfide-ferrous sulfate treatment process currently used at Tinker AFB. The benefit from this would be a reduction in chemical feed costs, and elimination of the ferric portion of the trivalent metals which would still have to be disposed of as waste.

The scoping report identified ion exchange as a potential separation technology if the iron could be removed. With the precipitation separation, the divalent metal portion of the waste may prove to be ideally suited for ion exchange technology. Ion exchange is a technology which should still be investigated.

Additionally, electrowinning should be further examined as a separation process.

SECTION VI
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APPENDIX A
LABORATORY PROCEDURES

LABORATORY PROCEDURES

The following procedure was the metals analysis procedure.

Summary of Method. The samples will be weighed, digested with aqua regia in a sealed Teflon microwave digestion bomb, diluted to volume and analyzed by ICP-AES for the metals content. Calibration will be done with a 3-point calibration curve and all quantitation will include the use of yttrium and germanium as internal standards.

Sample Preparation.

1. All samples will be digested in triplicate.
2. Additional samples will include a bomb blank, a bomb blank spike (one to five ppm) and a spiked samples (levels as appropriate).
3. Accurately weigh the samples into 125 mL microwave digestion bombs between 0.1 to 0.2 grams of dry sludge.
4. Add 10 mL of aqua regia.
5. Microwave at high until boiling with the vessel open in order to drive off the hydrogen sulfide and hydrogen cyanide.
6. Add 5 more mL of aqua regia and 5 mL of deionized water to the vessel and seal.
7. Using the pressure vessel, heat the bombs in the microwave. The pressure monitor should be connected and the upper pressure set to about 25 psig. A typical power program on the microwave system might be to operate at 100% power for 5 minutes, and at 50% power for 30 minutes.
8. If necessary, repeat steps 4. and 5.
9. Dilute the samples to 100 mL with deionized water.

ICP-AES Analysis.

1. Scan the samples for the presence of yttrium and germanium as these are potential standard candidates.
2. Build a file that contains all the appropriate wavelength selections for elements to be analyzed.
3. Scan a sample and a 5 ppm standard to check for interferences, and determine background correction points and the appropriate concentration ranges for the standards.
4. Using the appropriate internal standard at a level of 5 ppm, build standards in the appropriate concentration ranges.
5. Run the quantitative analysis to determine the concentrations of the metals. ($\pm 10\%$)

The following procedure was for determining the extractable organics content of the metal-bearing sludge based upon dry weight.

Scope and Application. The methodology described below is a modification of the EPA Methods 9070 and 9071. The differences from these methods include use of different solvents and the use of jar extractions instead of a lengthy Soxhlet extraction. These changes are due to the availability of solvents and to the high levels of organics expected to be found in the sludge. This method will extract relatively nonvolatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and a variety of related matter.

Summary of Method. A sample of sludge dried for 48 hours at 100°C will be extracted. The samples will be directly extracted or first acidified to a pH < 2 with hydrochloric acid. The samples will be extracted with pentane, the organic phase separated from the remaining sample, the solvent evaporated away, and the residue weighed.

Potential Interferences. There are a multitude of interferences that can occur that will bias the results including the co-extraction of metal complexes along with the organics and/or the inability to extract some organics.

Procedures.

1. All extractions will be run in triplicate.
2. Accurately weigh 5 grams of the dried sludge (finely ground) into a 100 mL glass bottle.
3. For the wetted acid extractions add 2 g of dried magnesium sulfate monohydrate and mix well. Let this mixture stand for 15 to 30 minutes. If mixture solidifies, break it up and grind as finely as possible.
4. Add 30 mL of pentane to the jar and agitate vigorously. Let it stand for several minutes.
5. Filter the contents of the extraction jar through either a prewashed filter paper or prewashed glass wool into a preweighed 125 mL receiving vessel.
6. Wash the jar and the solid material collected in the filter with two 30 mL aliquots of pentane.
7. Evaporate the solvent from the receiving vessel in a hood with a gentle stream of nitrogen.

8. Reweigh the vessel and calculate the % extractable organics in the sample as follows:

$$\% \text{ extractable} = (G/S) * 100\%$$

where G is the weight gain of the receiving vessel, and S is the weight of the original sample.

The following procedure was for the determination of dry solids content for the thickened metal-bearing sludge and the vacuum filtered wet sludge cake.

Procedure.

1. Accurately weigh 1 g of wet sludge cake, or 25 g of thickened sludge into a preweighed glass vessel.
2. Place samples into a 100°C oven for 48 hours.
3. Cool the samples in a desiccator for at least 30 minutes.
4. Reweigh the vessels with the now dried samples.
5. Calculate the percent of dried solids as follows:

$$\% \text{ Dry Solids} = [(DV-V)/(WV-V)] * 100\%$$

where DV is the mass of the dried sample and vessel, V is the mass of the mass of the empty vessel, and WV is the mass of the wet sample and vessel.

The following procedure was used in testing the sequential dissolution method proposed in the scoping studies for selectively extracting individual metals from the metal-bearing sludge.

Summary of Method. A 10 g sample of dry sludge will be sequentially exposed to seven leaching conditions. Each solution will be designed to preferentially leach a specific metal within the sludge, and each subsequent solution will have a greater extraction capability.

Procedure.

1. Accurately weigh 10 grams of the dried sludge (finely ground) into a 125 mL Erlenmeyer flask.

2. Add 100 mL of 1 M sodium hydroxide solution to the sludge sample and stir for 2 hours.
3. Centrifuge the mixture for 30 minutes at 8000 rpm, decant the supernatant, weigh and store for analysis.
4. Rinse precipitate with 100 mL of deionized water, centrifuge the mixture for 30 minutes at 8000 rpm, decant the liquid, weigh and store for analysis.
5. Repeat rinse operation (Step 4.).
6. Repeat Steps 2. through 5. using the following solutions sequentially in the place of the 1 M sodium hydroxide:

0.1 M nitric acid, cold
0.1 M nitric acid, hot
6 M hydrochloric acid
6 M sulfuric acid
6 M nitric acid
Aqua regia

The following procedure was used for chemical leaching of sludge to obtain a stock leach extract for the precipitation studies.

Summary of Method. A 50 gram sample of dry sludge will be dissolved with sulfuric acid and fine particulates will be filtered to prepare a stock solution of leach extract for precipitation studies.

Procedure.

1. Accurately weigh 50 grams of dried sludge in a 500 mL Erlenmeyer flask.
2. Add 250 mL of 2 M sulfuric acid and heat for 2 hours. Let solids settle and decant liquid into a 1 L Erlenmeyer flask.
3. Add an additional 250 mL of sulfuric acid and heat for 2 hours. Let solids settle and decant liquid into the 1 L Erlenmeyer flask.
4. Filter the final stock and place in another Erlenmeyer flask for the precipitation studies.
5. The scoping studies were performed with this stock at full strength. The later studies were performed with this stock diluted by adding 250 mL of stock to 250 mL of deionized water.

The following procedure was used in testing selective precipitation methods.

Summary of Method. Metals in a stock sludge leachate will be precipitated under varying pH and chemical conditions to determine the extent of metal ion removal.

Procedure for Phosphate Precipitation:

1. Add 20.0 mL of the stock leach extract from sludge to tared 50 mL centrifuge tubes and record weight.
2. Slowly add 19.01 grams of trisodium phosphate while mixing on a stir-plate and record weight of total solution.
3. Adjust the pH close to the desired level (pH - 2 to pH - 7 by 0.3 pH increments) by careful addition of dry sodium hydroxide, followed by addition of 1% sodium hydroxide as necessary to attain the final pH and record the weight of total solution. Bring the solution up to 50 mL with deionized water and record the weight. Allow the solution to precipitate overnight and check pH.
4. Centrifuge the tubes at 1000 rpm for 10 minutes, decant the solution into a tared 100 mL polypropylene bottle and record the weight of the solution.
5. Weigh the tube after decanting the solution, dissolve the remaining solid with 25 mL of 2 M sulfuric acid and decant into a 100 mL polypropylene bottle. Wash, rinse and dry the tube and record the weight.
6. Analyze metal content remaining in solution with atomic absorption spectroscopy.

Procedure for Ammonium Sulfate/Ammonium Hydroxide Precipitation:

1. Add 20.0 mL of the stock leach extract from sludge to tared 50 mL centrifuge tubes and record weight.
2. Slowly add 20 mL of 2 M ammonium sulfate/1 M ammonium hydroxide solution while mixing on a stir-plate and record weight of total solution.
3. Adjust the pH close to the desired level (pH - 2 to pH - 7 by 0.3 pH increments) by careful addition of dry sodium hydroxide, followed by addition of 1% sodium hydroxide as necessary to attain the final pH and record the weight of total solution. Bring the solution up to 50 mL with deionized water and record the weight. Allow the solution to precipitate overnight and check pH.
4. Centrifuge the tubes at 1000 rpm for 10 minutes, decant the solution into a tared 100 mL polypropylene bottle and record the weight of the solution.
5. Weigh the tube after decanting the solution, dissolve the remaining solid with 25 mL of 2 M sulfuric acid and decant into a 100 mL polypropylene bottle. Wash, rinse and dry the tube and record the weight.
6. Analyze metal content remaining in solution with atomic absorption spectroscopy.

Procedure for Sodium Hydroxide Precipitation:

1. Add 20.0 mL of the stock leach extract from sludge to tared 50 mL centrifuge tubes and record weight.
2. Adjust the pH close to the desired level (pH ~ 2 to pH ~ 7 by 0.3 pH increments) by careful addition of dry sodium hydroxide, followed by addition of 1% sodium hydroxide as necessary to attain the final pH and record the weight of total solution. Bring the solution up to 50 mL with deionized water and record the weight. Allow the solution to precipitate overnight and check pH.
3. Centrifuge the tubes at 1000 rpm for 10 minutes, decant the solution into a tared 100 mL polypropylene bottle and record the weight of the solution.
4. Weigh the tube after decanting the solution, dissolve the remaining solid with 25 mL of 2 M sulfuric acid and decant into a 100 mL polypropylene bottle. Wash, rinse and dry the tube and record the weight.
5. Analyze metal content remaining in solution with atomic absorption spectroscopy.

APPENDIX B
DATA TABLES SUPPORTING
FIGURES 2-6

TABLE B-1. PHOSPHATE PRECIPITATION SCOPING STUDY AFTER SULFURIC ACID LEACHING
(Metal in Solution in mg/L +/- 10%)

| pH | Iron | Chromium | Manganese | Nickel | Zinc | Cadmium |
|------|------|----------|-----------|--------|------|---------|
| 0 | 1107 | 997 | 55.5 | 76.3 | 66.0 | 9.41 |
| 1.82 | 887 | 987 | 56.4 | 68.7 | 54.7 | 5.70 |
| 2.99 | 21 | 330 | 57.7 | 69.5 | 55.1 | 8.20 |
| 4.17 | 3.3 | 75 | 38.7 | 62.6 | 4.0 | 2.43 |
| 5.26 | 2.2 | 23 | 11.3 | 49.2 | 1.0 | 0.78 |
| 5.93 | 2.3 | 12 | 4.5 | 45.0 | 1.6 | 0.50 |

TABLE B-2. PHOSPHATE PRECIPITATION AFTER SULFURIC ACID LEACHING
(Metal in Solution in mg/L +/- 10%)

| pH | Aluminum | Iron | Chromium |
|------|----------|------|----------|
| 2.10 | 27.3 | 401 | 419 |
| 2.33 | 27.1 | 374 | 406 |
| 2.57 | 23.9 | 237 | 306 |
| 2.80 | 9.3 | 170 | 227 |
| 3.02 | 5.6 | 108 | 122 |
| 3.48 | 1.5 | 78 | 41 |
| 3.94 | 0.0 | 44 | 8.8 |
| 4.20 | 1.2 | 21 | 4.4 |
| 4.52 | 1.2 | 11 | 6.0 |
| 4.81 | 1.1 | 12 | 5.7 |
| 5.05 | 1.2 | 8.4 | 5.3 |
| 5.35 | 1.3 | 7.5 | 5.3 |
| 5.67 | 1.2 | 3.5 | 3.9 |
| 5.95 | 1.2 | 4.1 | 3.6 |
| 6.22 | 1.4 | 4.0 | 2.6 |
| 6.53 | 1.4 | 4.0 | 3.8 |
| 6.72 | 1.6 | 4.4 | 3.3 |
| 7.02 | 1.4 | 4.9 | 3.3 |

TABLE B-3. AMMONIUM SULFATE/AMMONIUM HYDROXIDE PRECIPITATION SCOPING STUDY
AFTER SULFURIC ACID LEACHING
(Metal in Solution in mg/L +/- 10%)

| pH | Iron | Chromium | Manganese | Nickel | Zinc | Cadmium |
|------|------|----------|-----------|--------|------|---------|
| 0 | 1100 | 1000 | 55.5 | 76.3 | 66.0 | 9.4 |
| 1.10 | 1030 | 1000 | 56.9 | 73.1 | 65.2 | 11.6 |
| 2.11 | 930 | 940 | 58.4 | 74.1 | 66.0 | 11.5 |
| 3.11 | 150 | 840 | 57.0 | 70.0 | 64.6 | 11.4 |
| 4.12 | 160 | 650 | 55.3 | 70.0 | 65.3 | 11.1 |
| 4.90 | 60 | 460 | 53.6 | 50.5 | 56.0 | 8.6 |
| 6.00 | 9 | 8 | 50.9 | 50.0 | 28.5 | 5.8 |
| 7.09 | 3 | 2 | 37.5 | 50.0 | 7.2 | 4.0 |
| 8.79 | 12 | 2 | 12.4 | 50.0 | 44.4 | 0.8 |

TABLE B-4. AMMONIUM SULFATE/AMMONIUM HYDROXIDE PRECIPITATION AFTER
SULFURIC ACID LEACHING
(Metal in solution in mg/L +/- 10%)

| pH | Aluminum | Iron | Chromium |
|------|----------|------|----------|
| 2.00 | 16.9 | 406 | 411 |
| 2.26 | 16.9 | 448 | 440 |
| 2.52 | 17.5 | 405 | 423 |
| 2.76 | 17.2 | 362 | 408 |
| 3.27 | 15.6 | 346 | 387 |
| 3.76 | 13.1 | 304 | 299 |
| 4.34 | 6.3 | 282 | 154 |
| 4.84 | 2.9 | 218 | 71 |
| 5.36 | 1.6 | 160 | 59 |
| 5.83 | 0.7 | 85 | 38 |
| 6.33 | 0.1 | 17 | 1.8 |
| 6.60 | 0.0 | 12 | 1.9 |

TABLE B-5. SODIUM HYDROXIDE PRECIPITATION AFTER SULFURIC ACID LEACHING
(Metal in Solution in mg/L +/- 10%)

| pH | Aluminum | Iron | Chromium |
|------|----------|------|----------|
| 2.30 | 26.7 | 406 | 426 |
| 2.54 | 26.3 | 377 | 388 |
| 2.73 | 26.8 | 388 | 406 |
| 2.98 | 22.8 | 323 | 319 |
| 3.48 | 18.2 | 319 | 271 |
| 3.89 | 8.0 | 212 | 137 |
| 4.22 | 5.9 | 169 | 105 |
| 4.83 | 0.2 | 153 | 37.5 |
| 5.48 | 0.0 | 117 | 2.3 |
| 6.10 | 0.0 | 95 | 2.4 |
| 6.62 | 0.0 | 7.5 | 0.9 |
| 6.92 | 0.0 | 3.5 | 0.7 |
| 7.26 | 0.0 | 4.1 | 1.0 |

APPENDIX C
BUILDING ENCLOSURE SPECIFICATIONS FOR
TINKER AFB IWTP FIELD DEMONSTRATION UNIT

BUILDING ENCLOSURE SPECIFICATIONS FOR TINKER AFB IWTP FIELD DEMONSTRATION UNIT

Subcontractor shall provide all materials and equipment except as specified below. Work shall be performed per attached drawings. Work shall conform to the National Electrical Codes, meet requirements of AISC and also meet OSHA requirements.

All coordination for site support shall be processed through the contractor.

Technical: Don Prescott 208-525-5742
Bill Martin 208-525-5738
Jonathon Specht 208-525-5747

Administration: Mike Bolender 208-525-5377

SPECIFICATIONS

Field demonstration unit enclosure/lab trailer coordination: Field demonstration unit enclosure can't be constructed totally until the 12 x 56' trailer is moved to correct location. EG&G will move the trailer to the correct location. It will take one week to coordinate move.

BUILDING

Steel 26 gauge to meet UL-90 Standard - color to be selected from manufacturer standard selection.

Frame size - Approximately 56' x 24' x 12'

Backfill and Compact - see site plan, compact 95% of maximum density at optimum moisture content as measured by ASTM-D698.

FLOOR

Concrete (4000 psi, min-564 lb. Portland Cement, adequate rock and water ratios). Interior slab on grade to be a 4-inch minimum thickness. Reinforcing to be #3 at 16 inches on center each way, supported by bolsters in the lower 1/3 of slab. Floor shall be sloped one inch in ten feet to three center drains, sealed with a silica based sealer (Stifel by Noxcrete or equivalent). Magnesium float finish. Backfill to be compacted to prevent settling. Doorways shall have a 2 inch high concrete or steel threshold to make a water tight dam across each door opening.

EXTERIOR WALLS

Wall height - 12 feet at NE corner

Insulation - 2" vinyl back

Covering - Steel 26 gauge white liner panel with trim and accessories 7'4" above floor.

Siding - Steel 26 gauge Galvolume sheeting and meet UL-90 standard.

Special item - 6 translucent panels south side approx 3' x 6' panels.

ROOF

Sloping roof to north

Insulation - 2" vinyl back

Rain gutters - 4" x 4' 26 gauge galvolum sheeting, north and south sides, continuous gutter with downspout.

WINDOWS

9 ea size 3' x 3' horz. slide windows typ.

Glazing - clear - single pane.

DOORS

Exterior - 3 doors 3070 hollow metal walk doors, one 8' x 10' overhead rollup door, pull chain drive 24 gauge.

Hardware - keyed locks

Exit signs - Install to meet OSHA requirements (3 doors).

ELECTRICAL

Electrical Service - 440 v 3 phase 150 amp.

Load Centers - 150 amps. 440 v, 110 v/208 v 3 phase.

Lighting - 16 ea. fluorescent 8' 2 tube 110 watt heavy duty industrial fixtures. Switches at each entry. Exterior light at each door on common photo switch.

Receptacles - 10 ea. 110 v duplex GFI 3 per circuit.

Conduit; below grade -3 ea. 1-1/4 inch, PVC sch 80, stub up through floor; stub into 110 v panel 3 ea. 1-1/4 inch, PVC, stub up through floor stub up under trailer. PVC conduit stubs to pass through sleeves in the concrete floor.

Emergency lights - install emergency lights (battery powered) to meet OSHA requirements.

H & V

Exhaust Fan - up blast 3865 CFM @ 1/2"SP 440 v 3 phase with shutters, to include manual on/off controls.

Air intake - 2 ea. shutters 36" x 36" motor operated, 110 v AC, interlock with exhaust fan.

Heaters - Electric radiant heater 10 ea. 6 KW 440 v 3 phase with thermostat and controls for 3 zones minimum.

PIPING

Drain - Industrial waste floor drains and stub ups PVC sch 40. Stubs ups to pass through steel sleeve.

- 3 inch PVC drain line to existing line.

Water - 34 inch PVC sch 40, 3/4 inch PVC stub ups.

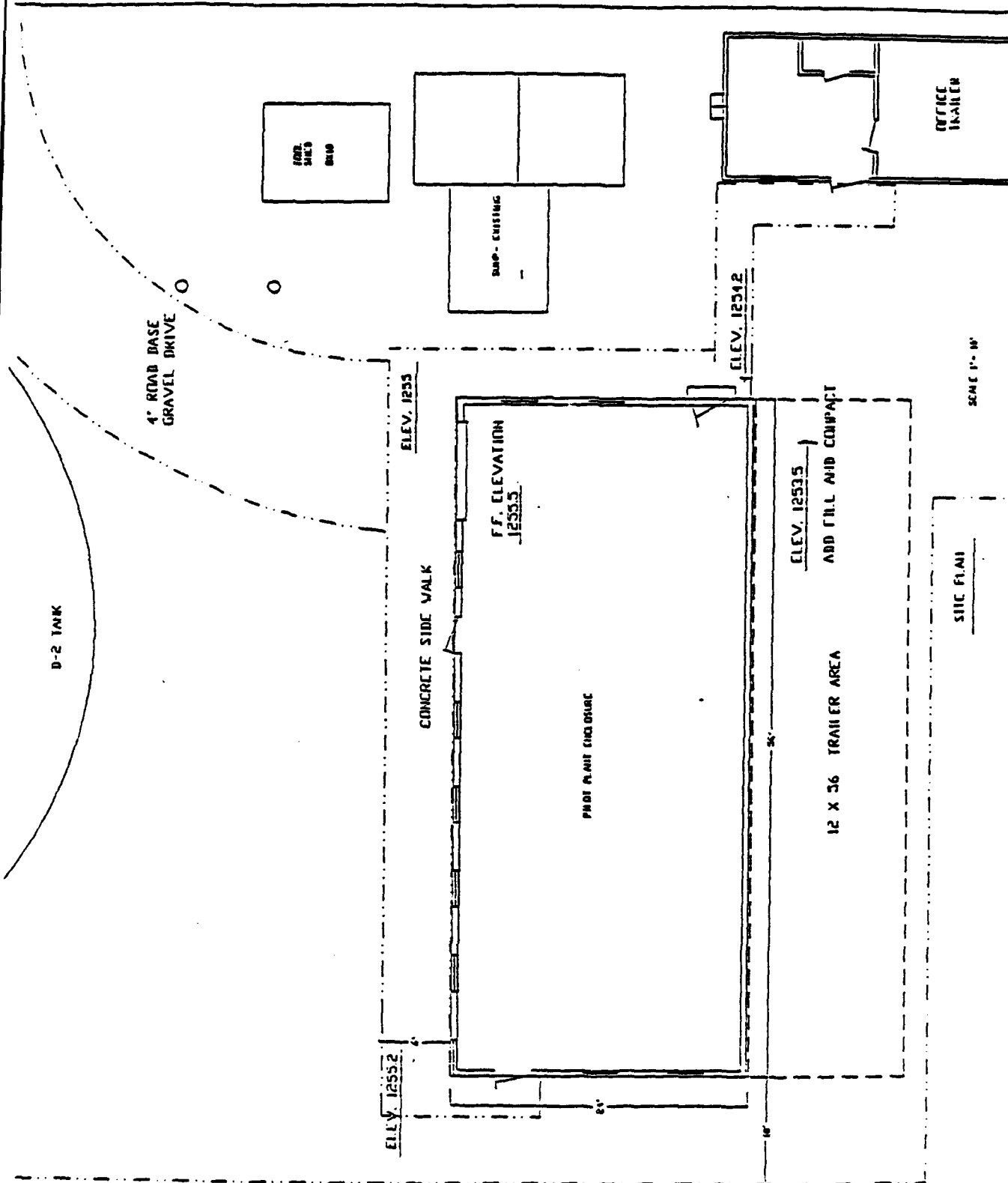
Note- Piping depth - 12 to 24" to avoid existing lines.

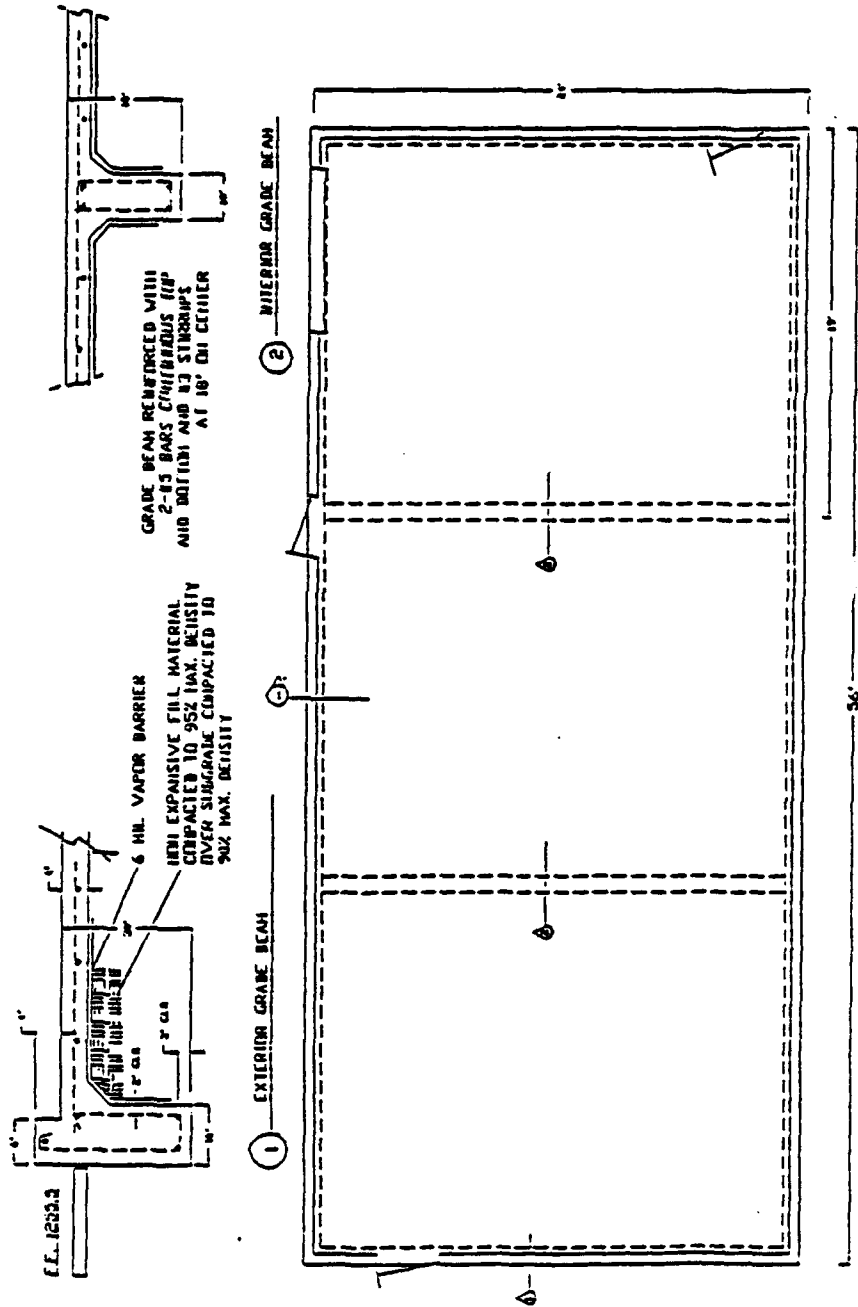
SIDEWALKS

Concrete with appropriate expansion joints, wire mesh reinforcement, broom finish, elevation - 2 inches below floor, ramp up to roll up door.

DRIVEWAY

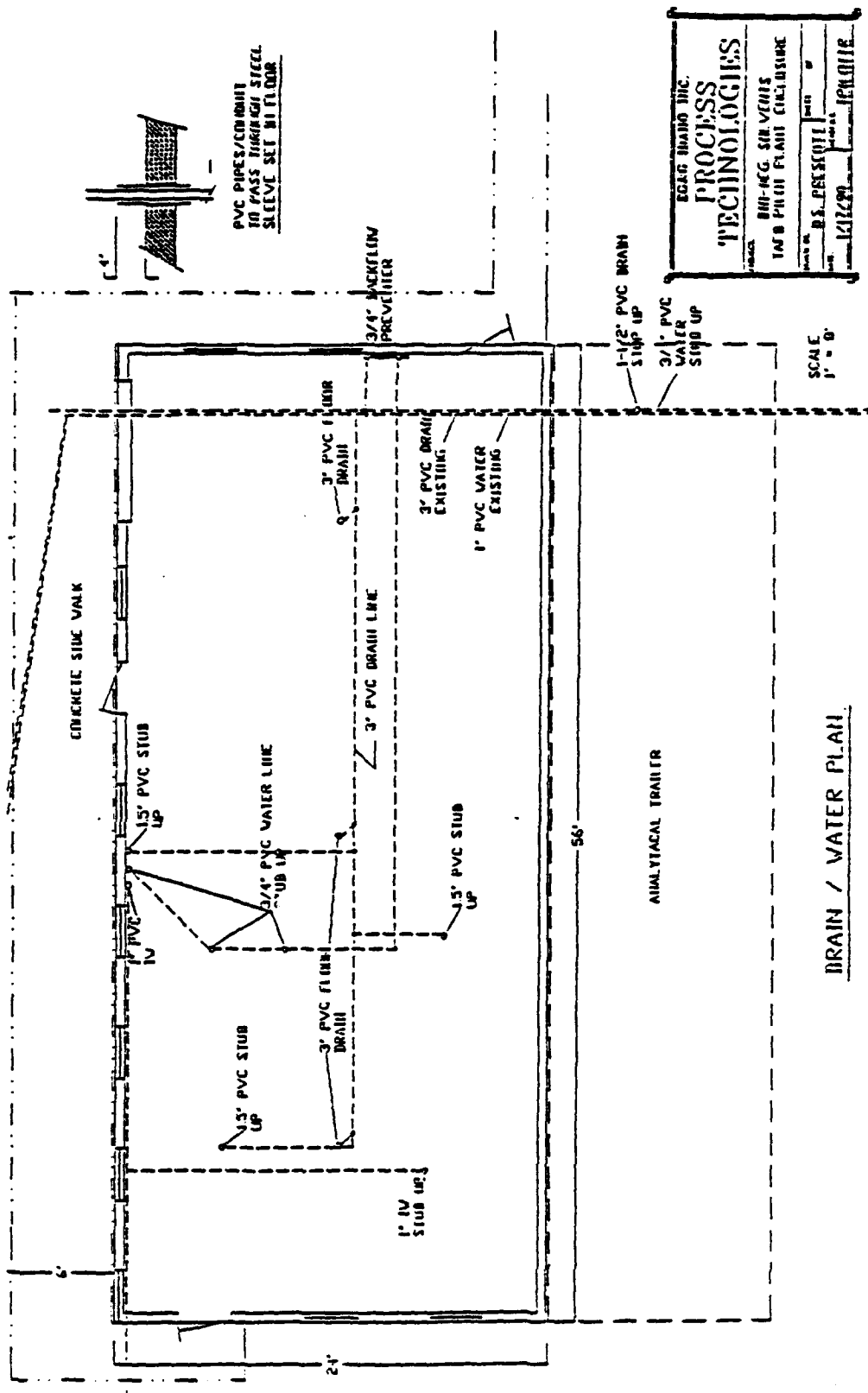
4 inches 3/4" road base gravel.

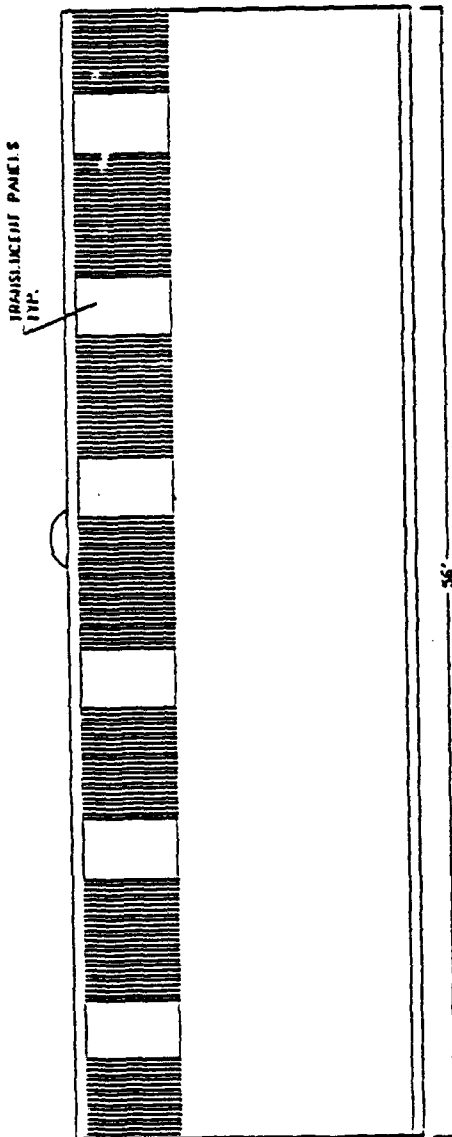




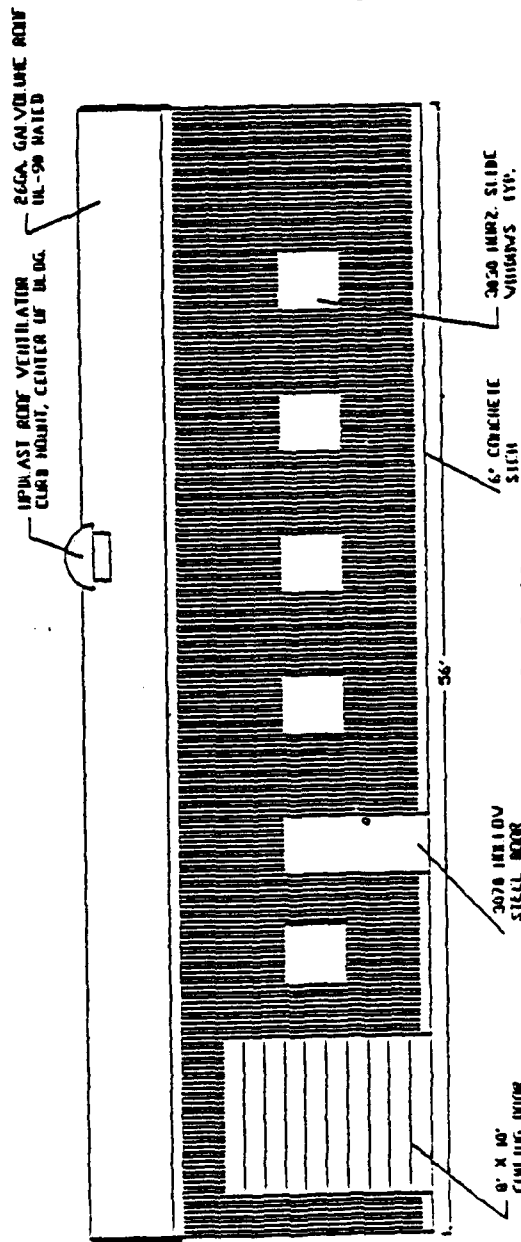
FOUNDATION PLAN

| | |
|--------------------------|--------------|
| ELEC. BOARD INC. | |
| PROCESS TECHNOLOGIES | |
| BIO-TEC. SERVICES | |
| TANK PUMP PLAN ENCLOSURE | |
| DATE: 01.08.2001 | BY: JENNIFER |
| 1/17/20 | |





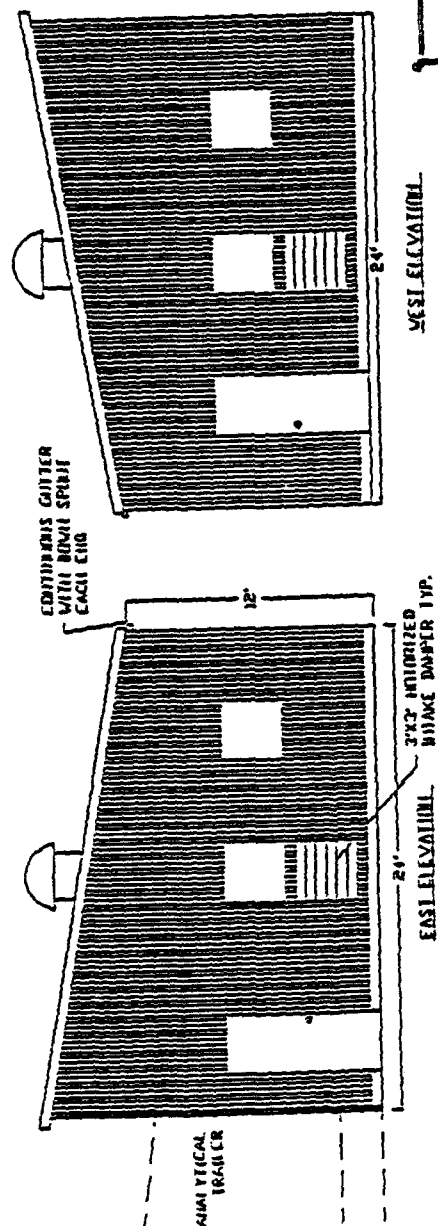
36'
SOUTH ELEVATION



36'
NORTH ELEVATION

SCALE 1" = 8'

| | |
|----------------------|-------------------|
| BRAIN TRAIL INC. | |
| PROCESS TECHNOLOGIES | |
| PROJECT | BIO-LOG. SOLVENTS |
| DATE | 1/17/79 |
| BY | AS PRESENT |
| CHECKED | 1/17/79 |



| | |
|----------------------|----------------------|
| ECHO RADIO INC. | |
| PROCESS TECHNOLOGIES | |
| PROJECT | 800-REG. SOLVENTS |
| | 1458 PLANT ENCLOSURE |
| DESIGN BY | D.S. PRESCOTT |
| DATE | 1/17/90 |
| | 1010118 |

SCALE 1" = 8'

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