

*Department of Energy
Federal Energy Technology Center
Morgantown, West Virginia*

FINAL

Bench-Scale Topical Report for:

**Mercury Removal from DOE Solid Mixed
Waste Using the GEMEPSM Technology**

Contract No. DE-AC21-97MC33139 --01

March 1999

Prepared By:



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DISCLAIMER

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**Mercury Removal from DOE Solid Mixed Waste
Using the GEMEP Technology
Final Bench-Scale Topical Report**

TABLE OF CONTENTS

	<u>Page</u>
EXECUTIVE SUMMARY	ES-1
1.0 INTRODUCTION	1-1
1.1 HISTORICAL BACKGROUND AND PROBLEM DEFINITION	1-2
1.2 SCIENTIFIC BACKGROUND OF THE GEMEP TECHNOLOGY	1-3
1.2.1 Process Description	1-3
1.3 PROJECT OBJECTIVES	1-7
1.3.1 Laboratory-Scale Tests	1-7
1.3.2 Bench-Scale Tests	1-9
1.3.3 Technical and Economic Evaluation	1-11
1.4 DESCRIPTIONS OF WASTES TESTED	1-11
1.4.1 Wastes Tested at GE-CRD	1-12
1.4.2 Wastes Tested at CMRI	1-13
2.0 TEST PROCEDURES	2-1
2.1 LABORATORY-SCALE TESTS	2-1
2.1.1 Extractions	2-1
2.1.2 Mercury Dissolution	2-2
2.2 BENCH-SCALE TESTS	2-2
2.2.1 Initial Waste Characterization	2-2
2.2.2 Pre-Segregation Tests	2-6
2.2.3 Bench-Scale Extraction Tests	2-9
2.2.4 Locked-Cycle Tests	2-16
3.0 RESULTS OF LABORATORY-SCALE TESTS	3-1
3.1 EXTRACTION TESTS ON FLUORESCENT LAMP WASTE	3-1
3.1.1 Minimizing Residual Mercury	3-2
3.1.2 Minimizing Iodine Loss	3-15
3.1.3 Cycle Testing of the GEMEP Process on Fluorescent Lamp Waste ..	3-22
3.1.4 Effect of Sizing (Pre-Segregation) of Lamp	3-24
3.1.5 Cycle Test for the Minus 30 Mesh Fractions	3-25

TABLE OF CONTENTS (Continued)

	<u>Page</u>
3.1.6 Economic Analysis	3-27
3.2 DISSOLUTION OF MERCURY DURING PRE-SEGREGATION	3-30
3.3 CONCLUSIONS FROM LABORATORY-SCALE TESTS	3-31
4.0 RESULTS OF BENCH-SCALE TESTS	4-1
4.1 EAST FORK POPLAR CREEK SEDIMENTS	4-1
4.1.1 Initial Waste Characterization	4-1
4.1.2 Pre-Segregation Tests	4-4
4.1.3 Bench-Scale Extraction Tests	4-5
4.1.4 Locked-Cycle Tests	4-6
4.2 FLUORESCENT LAMPS	4-8
4.2.1 Crushing Methods Investigation and Initial Waste Characterization ...	4-8
4.2.2 Pre-Segregation Tests	4-11
4.2.3 Bench-Scale Extraction Tests	4-12
4.2.4 Locked-Cycle Tests	4-15
4.3 INEEL SOIL/SLUDGE	4-20
4.3.1 Initial Waste Characterization	4-20
4.3.2 Pre-Segregation Tests	4-24
4.3.3 Bench-Scale Extraction Tests	4-25
4.3.4 Locked-Cycle Tests	4-29
5.0 CONCLUSIONS AND COMPARISON AGAINST SUCCESS CRITERIA	5-1
5.1 SUCCESS CRITERIA	5-1
5.2 GEMEP EFFECTIVENESS FOR MERCURY REMOVAL	5-2
5.2.1 Fluorescent Lamps	5-2
5.2.2 East Fork Poplar Creek Sediment	5-5
5.2.3 INEEL Soil/Sludge	5-5
5.3 PILOT-SCALE DESIGN AND OPERATION	5-7
5.3.1 Pilot System Equipment and Process Description	5-7
5.3.2 Mass Balances	5-12
5.4 COST ESTIMATE FOR PILOT-SCALE TREATMENT OF INEEL WASTE .	5-15
5.4.1 Cost Basis	5-15
5.4.2 Pilot Testing Costs	5-25
5.5 COMPARISON WITH COMPETING TECHNOLOGIES	
& RECOMMENDATIONS	5-26
5.5.1 INEEL Soil/Sludge Waste	5-26
5.5.2 Fluorescent Lamps	5-30

Appendices

Appendix A Data Sheets for Bench-Scale Testing

- A-1 Data Sheets for Bench-Scale Extractions of East Fork Poplar Creek Sediment
- A-2 Data Sheets for Bench-Scale Extractions of Fluorescent Lamps
- A-3 Data Sheets for Locked-Cycle Testing of Fluorescent Lamps
- A-4 Data Sheets for Bench-Scale Extractions of INEEL Soil/Sludge
- A-5 Data Sheets for Locked-Cycle Testing of INEEL Soil/Sludge

Appendix B Equipment List and Mass Balance

- B-1 Equipment List for Pilot-Scale GEMEP System
- B-2 Mass Balance for GEMEP Treatment of INEEL Waste
- B-3 Thermal Treatment Cost Information

LIST OF FIGURES

	<u>Page</u>
1-1 Flowchart for the GEMEP Process	1-4
2-1 Waste De-Agglomeration and Sizing Flowsheet	2-4
2-2 Settling Test Data Sheet	2-5
2-3 Fluorescent Lamp Glass Crushing, Sizing and Magnetic Separation	2-8
2-4 Chemical Leaching Flowsheet	2-11
2-5 Chemical Leaching Datasheet	2-12
2-6 Flowsheet for Locked-Cycle Tests	2-17
3-1 Main Effects Plot for Screening Design of Experiments	3-4
3-2 3D Plot for the Surface Generated by the Residual Hg Prediction Equation	3-7
3-3 Contour Plot Generated by the Prediction Equation for Residual Mercury	3-8
3-4 3D Graph of the Standard Deviation (s)	3-10
3-5 Contour Plot of $Z_{95\%}$ for Residual Mercury	3-12
3-6 Capability Analysis for Residual Mercury (mg) at 0.02 M I_2 and 3/1 L/S	3-14
3-7 3D Plot of mg of I_2 Lost/g of Glass Treated as a Function of Time and Temperature	3-17
3-8 Capability Analysis for mg of I_2 Loss/g Glass Treated, Using All Data from the Design of Experiments	3-19
3-9 Dissolution of Mercury as a Function of Time	3-21
3-10 Total Moles of Iodine/Iodide in Solution as a Function of Time	3-22
3-11 Capability Analysis for I_2 Loss for Extraction of the Phosphors (-30 Fraction) Only .	3-28
4-1 Size, Mercury, and Uranium Distribution in East Fork Poplar Creek Sediment	4-3

LIST OF FIGURES (Continued)

	<u>Page</u>
4-2 Effect of Initial Iodine/KI Molarity on Residual Mercury	4-7
4-3 Impact Mill - Overall View	4-9
4-4 Impact Mill - Cutaway View	4-10
4-5 Effect of Final Iodine Concentration on Residual Mercury	4-14
4-6 Flowsheet for Locked-Cycle Leaching of Crushed Lamp Glass	4-16
4-7 Size and Mercury Distribution in INEEL Sludge	4-23
4-8 Settling of INEEL Minus 4-Mesh Soil	4-26
4-9 Effect of Iodine Concentration, Temperature, and pH on Residual Mercury	4-28
4-10 Locked-Cycle Flowsheet for Processing INEEL Soil	4-30
4-11 Effect of Second Stage Iodine Concentration on Residual Mercury	4-32
5-1 GE Mercury Extraction Process Flowsheet	5-8

LIST OF TABLES

<u>Number</u>	<u>Page</u>
1-1 Treatment of Mercury-Contaminated Samples	1-5
1-2 Breakdown of the Components of a 4-Foot F40T12 Lamp	1-13
1-3 X-Ray Fluorescence Analysis: East Fork Poplar Creek Sediment	1-15
1-4 X-Ray Fluorescence Analysis: INEEL Soil/Sludge	1-16
1-5 Radioactive Composition of INEEL Soil/Sludge: Drum 708	1-17
2-1 Chemical Extraction Test Matrix for East Fork Poplar Creek Sediment (Waste Type EFPC)	2-13

LIST OF TABLES (Continued)

	<u>Page</u>
2-2 Chemical Extraction Test Matrix for Fluorescent Lamp Glass (Waste Type LAMP)	2-14
2-3 Chemical Extraction Test Matrix for INEEL Soil/Sludge (Waste Type INEEL)	2-15
3-1 High, Mid, and Low Values for Screening Design of Experiments	3-2
3-2 Screening Design of Experiments	3-3
3-3 High, Low, and Mid Values for Full Design of Experiments	3-5
3-4 Full Factorial Central Composite Design of Experiments	3-5
3-5 The Standard Deviation (s) and 95% UCL for s for Six Design Points	3-9
3-6 $Z_{95\%}$ Values for the 9 Design Points from the Full Factorial Design of Experiments ..	3-11
3-7 High, Low, and Mid Values for Full Design of Experiments for I_2 Loss	3-15
3-8 Full Factorial Central Composite Design of Experiments	3-16
3-9 Standard Deviation (s) and 95% UCL for s for Iodine Loss	3-18
3-10 $Z_{95\%}$ and Cost of Iodine per Ton of Glass (Lamp Waste)	3-18
3-11 Initial and Final Concentrations for I_2 , I^- and Hg	3-23
3-12 Iodine and Water Added During the GEMEP Cycles and the Total Mass Balance ..	3-23
3-13 Extraction of -30 Fractions at 45°C, 1 Hour and Liquid/Solid Ratio of 5	3-24
3-14 TCLP Results for Treated and Untreated Phosphors (-30 Fraction)	3-25
3-15 Cycle Extraction of -30 Fraction (0.1 M I_2 /0.2 M KI, 1 Hour, 45°C, L/S ~4)	3-26
3-16 Cycle Extraction of -30 Fraction (0.2 M I_2 /0.4 M KI, 1 Hour, 45°C, L/S ~4)	3-26
3-17 Loss of I_2 to the -30 Fraction at Two Different I_2 Concentrations, Other Conditions (1 Hour, 45°C, L/S ~4)	3-27

LIST OF TABLES (Continued)

	<u>Page</u>
3-18 Cost Comparison	3-29
3-19 Dissolution of Mercury During Pre-Segregation Step	3-30
3-20 Dissolution of Mercury in the Presence of TMT-15	3-31
4-1 Weight, Mercury, and Uranium Distribution in East Fork Poplar Creek Sediment	4-2
4-2 X-Ray Fluorescence Analysis of East Fork Poplar Creek Sediment	4-4
4-3 Bench-Scale Leaching Results of East Fork Poplar Creek Sediment	4-6
4-4 Weight and Mercury Distribution in Crushed Fluorescent Lamps	4-11
4-5 Bench-Scale Leaching of Minus 65-Mesh Fluorescent Lamp Glass	4-13
4-6 Locked-Cycle Results from Leaching Crushed Lamp Glass	4-17
4-7 TCLP Values for Locked-Cycle Leached Crushed Fluorescent Lamp Glass	4-18
4-8 Efficiency of Mercury Precipitation by Iron Turnings	4-18
4-9 Data From Iron Precipitation	4-19
4-10 X-Ray Fluorescence Analysis of INEEL Soil/Sludge	4-21
4-11 Size and Mercury Distribution in INEEL Soil/Sludge	4-22
4-12 Viscosity of INEEL Sludge at Pulp Densities	4-24
4-13 Bench-Scale Leaching of INEEL Soil/Sludge	4-27
4-14 Locked Cycle Leaching of INEEL Soil	4-31
4-15 TCLP Results of INEEL Locked-Cycle Residues	4-33
4-16 Cementation of Mercury from INEEL Leach Solution Using Iron Columns	4-33
4-17 Iron Precipitation Data, INEEL Locked-Cycle Tests	4-34
5-1 Simplified Mass Balance for Fluorescent Lamp Glass	5-13

LIST OF TABLES (Continued)

	<u>Page</u>
5-2 Simplified Mass Balance for INEEL Soil/Sludge Waste	5-14
5-3 Cost of Feed Preparation Module	5-17
5-4 Cost of Leach Module	5-18
5-5 Cost of Wash Module	5-19
5-6 Cost of Solution Processing Module	5-20
5-7 Summary of Fabricated Module Costs	5-21
5-8 Operating Costs	5-23
5-9 Total Costs for Pilot Plant Operation	5-24
5-10 Operating Cost Estimate: Thermal Treatment Pilot Test	5-28
5-11 Total Costs for Pilot Plant Operation: Thermal Treatment	5-29

EXECUTIVE SUMMARY

Under the sponsorship of the Federal Energy Technology Center (FETC), Metcalf & Eddy (M&E), in association with General Electric Corporate Research and Development Center (GE-CRD), Colorado Minerals Research Institute (CMRI), and Oak Ridge National Laboratory (ORNL), conducted laboratory-scale and bench-scale tests of the General Electric Mercury Extraction Process technology on two mercury-contaminated mixed solid wastes from U. S. Department of Energy sites: sediment from the East Fork of Poplar Creek, Oak Ridge (samples supplied by Oak Ridge National Laboratory), and drummed soils from Idaho National Environmental and Engineering Laboratory (INEEL). Fluorescent lamps provided by GE-CRD were also studied.

The GEMEP technology, invented and patented by the General Electric Company, uses an extraction solution composed of aqueous potassium iodide plus iodine to remove mercury from soils and other wastes. The extraction solution is regenerated by chemical oxidation and reused, after the solubilized mercury is removed from solution by reducing it to the metallic state.

Results

The laboratory- and bench-scale testing conducted for this project included: (1) GEMEP extraction tests to optimize extraction conditions and determine the extent of co-extraction of radionuclides; (2) pre-screening (pre-segregation) tests to determine if initial separation steps could be used effectively to reduce the volume of material needing GEMEP extraction; and (3) demonstration of the complete extraction, mercury recovery, and iodine recovery and regeneration process (known as locked-cycle testing).

Fluorescent Lamps. Tests on fluorescent lamps included evaluation of crushing and pre-segregation methods, laboratory- and bench-scale GEMEP extraction tests, and locked-cycle tests of the entire extraction, mercury recovery, reagent recycle, and iodine recovery processes.

It was possible to concentrate the mercury content in fluorescent lamps, after crushing, into the non-metal fraction that was finer than 65 mesh. This fraction contained about 425 mg Hg/kg. Bench-scale testing of minus 65-mesh glass reduced the residual mercury to concentrations ranging from 7 to 15 mg/kg, with corresponding mercury extraction efficiencies of 96.4 to 98.3%. The treated glass readily passed the 0.2 mg Hg/L TCLP limit in all samples tested. TCLP extraction results ranged from 0.01 to 0.10 mg Hg/L, with all but one of the results also below the Universal Treatment Standard (UTS) of 0.025 mg/L.

The mercury-laden extraction solution was passed through iron columns to reduce the mercury, and the solution was then made basic by the addition of lime to remove soluble iron. The efficiency of mercury removal from the combined iron column treatment and lime precipitation steps exceeded 99.9% in six of the ten test cycles.

East Fork Poplar Creek Sediment. Bench-scale GEMEP extraction testing of East Fork Poplar Creek sediment reduced the residual mercury from about 800 mg/kg in the raw waste to about 3 mg/kg. The solubilization of uranium present in the sediments was low, ranging from 0.01% to 0.13%. This result indicates that the GEMEP extraction solution would not become a radioactive waste, and that nearly all the the uranium initially present in the sediments (99 to 99.99%) would remain with the treated sediments, while mercury would be effectively extracted.

INEEL Soil/Sludge. The INEEL waste was subjected to bench-scale testing of pre-segregation steps, GEMEP extraction, and locked-cycle testing of the entire GEMEP process. Pre-segregation by wet screening on sieves was able to effectively separate the INEEL waste into a plus 4 mesh fraction that passed TCLP and consisted of rock, asphalt, and a light colored tar/polymer. The minus 4 mesh material was a fine silt with a mercury concentration in the range of 700 to 800 mg/kg; this material underwent GEMEP extraction and locked-cycle testing.

Seventeen bench-scale extraction tests were run at varying conditions of iodine/iodide concentration, temperature, and pH. Mercury concentrations were reduced from an initial level of

700-800 mg/kg to 17 to 64 mg/kg. For nine of the 17 tests, the treated solids were also analyzed by TCLP. All of the extracts of treated solids contained mercury at concentrations less than the TCLP limit of 0.2 mg/L, while for four of the nine samples, the mercury extract concentration was also less than the UTS of 0.025 mg/L. Hence, it was possible to conduct bench-scale extractions of the INEEL waste using conditions that would produce treated solids that could be directly land-disposed.

Results of the locked-cycle tests were less successful. The mercury concentrations in TCLP extracts of treated solids were all above the UTS of 0.025 mg/L, although they were below the TCLP limit of 0.2 mg/L. Because attaining the UTS was not an objective of this test program (since testing preceded its promulgation), no locked-cycle tests were performed to see if the UTS could be attained by adjustments in test conditions.

It was possible to efficiently remove solubilized mercury from the extraction solution during locked-cycle testing of the INEEL waste. The combined efficiency of mercury removal by the mercury precipitation and iron precipitation steps was 99.8% or greater in six of the ten cycles. Regeneration of iodine was less efficient, with a range of 60 to 100% and an average of 93%.

Cost Estimates

A cost estimate for construction and operation of a pilot-scale system for GEMEP treatment of INEEL waste was developed. The estimate assumes that the system will be constructed and operated at CMRI in Golden, Colorado, and does not consider costs for shipping wastes for pilot testing to CMRI, or disposal of treated solids and secondary wastes. A treatment rate of 24 kg/hr (53 lb/hr) and a thirty day operational period were assumed.

The estimated cost for the pilot-scale equipment modules plus trailers is \$223,060. Operating costs for processing 24 kg/hr of soil (not including disposal of treated solids or secondary wastes) are estimated as follows:

	<u>Daily Cost</u>	<u>Cost per Metric Ton (1,000 kg)</u>
Reagents	\$928	\$1,637
Utilities	58	100
Rentals	90	156
Labor	<u>5,500</u>	<u>9,550</u>
Total	\$ 6,576	\$ 11,443

The major operating costs are for labor and for make-up iodine. The make-up iodine requirement is based on locked-cycle test results. In these tests, no effort was made to scrub iodine from vapors exiting vessels and because of the batch nature of the testing, there were other losses. The pilot plant would include a scrubbing system to recover iodine vapors and reduce the iodine make-up requirement. Full-scale costs for treatment of the INEEL waste are anticipated to be roughly equivalent to the pilot-scale estimated costs, due to the limited volume of INEEL waste requiring treatment.

Comparison with Competing Technologies

INEEL Waste. Thermal desorption is a competitive process for treating the INEEL soil/sludge. An attempt was made to locate costs for thermal treatment of mercury-contaminated wastes. An Internet search identified two full-scale soil treatment projects of approximately 1,000 tons, with costs on the order of \$400 to \$460 per U.S. ton. The cost detail provided was not sufficient to determine whether costs for disposal of secondary wastes and treated solids were included.

The pilot-scale GEMEP system costed here was sized to process only 17,000 kg of waste (approximately 20 U.S. tons). While treating larger quantities offers an economy of scale with respect to capital and labor costs, the cost per ton of waste treated for reagents such as iodine would not be significantly reduced by going to a larger scale. Costs for iodine alone are projected to be on the order of \$1,000 per ton for GEMEP treatment of the INEEL waste, based on the iodine make-up requirements observed during the locked-cycle tests. As stated previously,

it should be possible to significantly reduce iodine consumption by adding a scrubber system to capture and reclaim iodine vapor. However, in the absence of testing to determine the effectiveness of a scrubber system or other iodine recovery system, it is not possible to predict to what degree iodine consumption costs could be reduced. Based on the currently available data and cost projections, thermal treatment is likely to be the more cost effective option for treatment of INEEL waste. When one also considers the small quantity of INEEL waste in storage, further effort to develop the GEMEP technology for this particular waste is not recommended.

Fluorescent Lamps. The competing technology for management of spent or off-spec, non-rad fluorescent lamps is to ship them to a lamp recycling facility, where thermal treatment is used to recover the mercury. According to estimates obtained by GE-CRD, recycling facilities charge \$0.25 per linear foot to accept fluorescent lamps, which equates to \$3,300 per U.S. ton for standard four-foot lamps. If reagent and utility costs for pre-segregation and GEMEP treatment are on the order of \$1,000 per ton (a conservatively high estimate), it would be necessary to build a plant large enough to reduce labor costs to the \$2,000 per ton range, for the GEMEP process to be competitive with recycling. However, in the face of a competing technology it is probable that the recyclers would reduce their prices to some degree. Hence, it is difficult to predict whether the GEMEP process would be competitive with recycling (including thermal treatment) of fluorescent lamps.

With respect to radioactive fluorescent lamp waste stored at DOE sites, it appears that there is no longer a need for development of an alternative treatment technology to handle these wastes. The DOE sites have been able to dispose of this type of waste recently, and hence no sites were interested in providing lamp waste for this project. It is not anticipated that there will be future interest in using the GEMEP technology for this particular waste, since the DOE sites are currently disposing of it without undue difficulty. While further pilot testing of the GEMEP technology may be of interest to manufacturers of lamps such as General Electric, it is not recommended that DOE pursue further testing, due to limited interest at the DOE sites.

SECTION 1.0

INTRODUCTION

The GEMEP (General Electric Mercury Extraction Process) technology was invented by the General Electric Company [1] and is being commercialized by Metcalf & Eddy. The process involves the chemical extraction and isolation of mercury from a variety of matrices including soils, sediments and plastics. This project was sponsored by the Federal Energy Technology Center (FETC) on behalf of the U.S. Department of Energy's Office of Science and Technology, and was performed by Metcalf & Eddy, Inc. (M&E) and team subcontractors General Electric Corporate Research and Development Center (GE-CRD), Colorado Minerals Research Institute (CMRI), and Oak Ridge National Laboratory (ORNL). The objective of the project was to demonstrate, at the bench scale, the ability of the GEMEP extraction process to remove mercury from several types of mixed waste for which DOE is responsible. The project also investigated the use of conventional separation technology to remove metallic mercury or waste fractions containing *de minimis* concentrations of mercury from the wastes prior to extraction.

The overall project is intended to include up to three stages:

- I. Base Phase (the subject of this report): Evaluation of process parameters through laboratory - and bench-scale testing of selected waste types
- II. Option 1: Design and demonstration of a pilot-scale mercury removal system at a DOE site
- III. Option 2: Design, fabrication, and demonstration of a full-scale mercury removal system at a DOE site

This Topical Report describes the efforts conducted under Stage I of the project, which consisted of laboratory studies on fluorescent lamps and surrogates for DOE mixed wastes, and bench-scale testing of fluorescent lamps and two actual DOE mixed wastes contaminated with mercury.

Stages II and III, the pilot- and full-scale demonstrations, are options to this contract which may or may not be exercised, depending on the results of the preceding stages.

M&E managed the base phase of this project, helped prepare a design and cost estimate for a pilot-scale system, and prepared all submissions to DOE. GE-CRD was charged with optimizing the GEMEP process for crushed fluorescent lamps, and determining ways of suppressing mercury dissolution during the initial wet sizing of the waste streams (pre-segregation). GE-CRD also provided technical support as needed throughout the project. CMRI conducted bench-scale tests of the GEMEP process on DOE mixed wastes at their facility, developed methods for pre-segregation of wastes at the bench scale, and prepared a pilot-scale system design, equipment list, and cost estimate. ORNL provided characterization data for candidate wastes to be tested, collected and shipped a representative DOE waste to the testing facilities at CMRI, assisted with disposal of excess samples of waste, and assisted with development of surrogates for DOE mixed wastes.

1.1 HISTORICAL BACKGROUND AND PROBLEM DEFINITION

DOE used large quantities of mercury in lithium isotope enrichment processes and other defense-related activities. These activities have resulted in the generation of a significant volume of mercury-contaminated waste. Much of this waste is characterized as “mixed waste,” that is, it also contains radioactive materials. This mercury-contaminated mixed waste was often stockpiled at DOE sites due to difficulties in disposing of it. Such material often cannot be disposed of as low-level radioactive waste, because it contains mercury. It cannot be disposed of as a hazardous waste, because it contains radioactive material. An efficient and cost-effective means of removing the mercury from the waste would be beneficial to DOE in managing those stockpiled wastes for which practical treatment and disposal options are yet to be identified.

The baseline technology for removal of mercury from solid wastes is thermal treatment, in which the waste is heated to volatilize the mercury into the gas phase. The gas is cooled to condense the mercury vapor. The off gas, containing dust and uncondensed mercury, is passed through air pollution control equipment consisting of air filters, scrubbers and/or adsorbers to remove these materials.

Thermal treatment has significant limitations related primarily to the generation and treatment of off gas. There are safety concerns with respect to air emissions containing fugitive mercury compounds. The air pollution control equipment generates wastewater containing mercury that requires additional treatment, as well as adsorbent materials that require treatment or disposal. Limitations such as these have led DOE to become interested in alternative processes that can remove mercury from mixed wastes.

1.2 SCIENTIFIC BACKGROUND OF THE GEMEP TECHNOLOGY

The GEMEP process is a chemical extraction process that uses iodine (I_2) as an oxidant and iodide (I^-) as a solubilizing ion. General Electric Company holds a patent [1] on this process and has published a number of internal reports detailing the development of the process [2-5]. When implemented the process is closed, with extractant, oxidant and water being recycled. A flow sheet for the process is shown in Figure 1-1. Optimization of the process involves the minimization of iodine use and loss as well as producing treated solids that may be disposed of on land without further treatment. The technology is able to extract a variety of mercury-containing species from soils, sediments, glass wastes, and other materials, as demonstrated by numerous laboratory-scale tests conducted by General Electric. Representative results from tests performed by General Electric are presented in Table 1-1.

1.2.1 Process Description

The mercury removal process, shown in Figure 1-1, is a closed-loop process that contains and recycles the extraction components. The process treats mercury-contaminated media and produces mercury-free media, elemental mercury, and a metal precipitate consisting primarily of iron hydroxides.

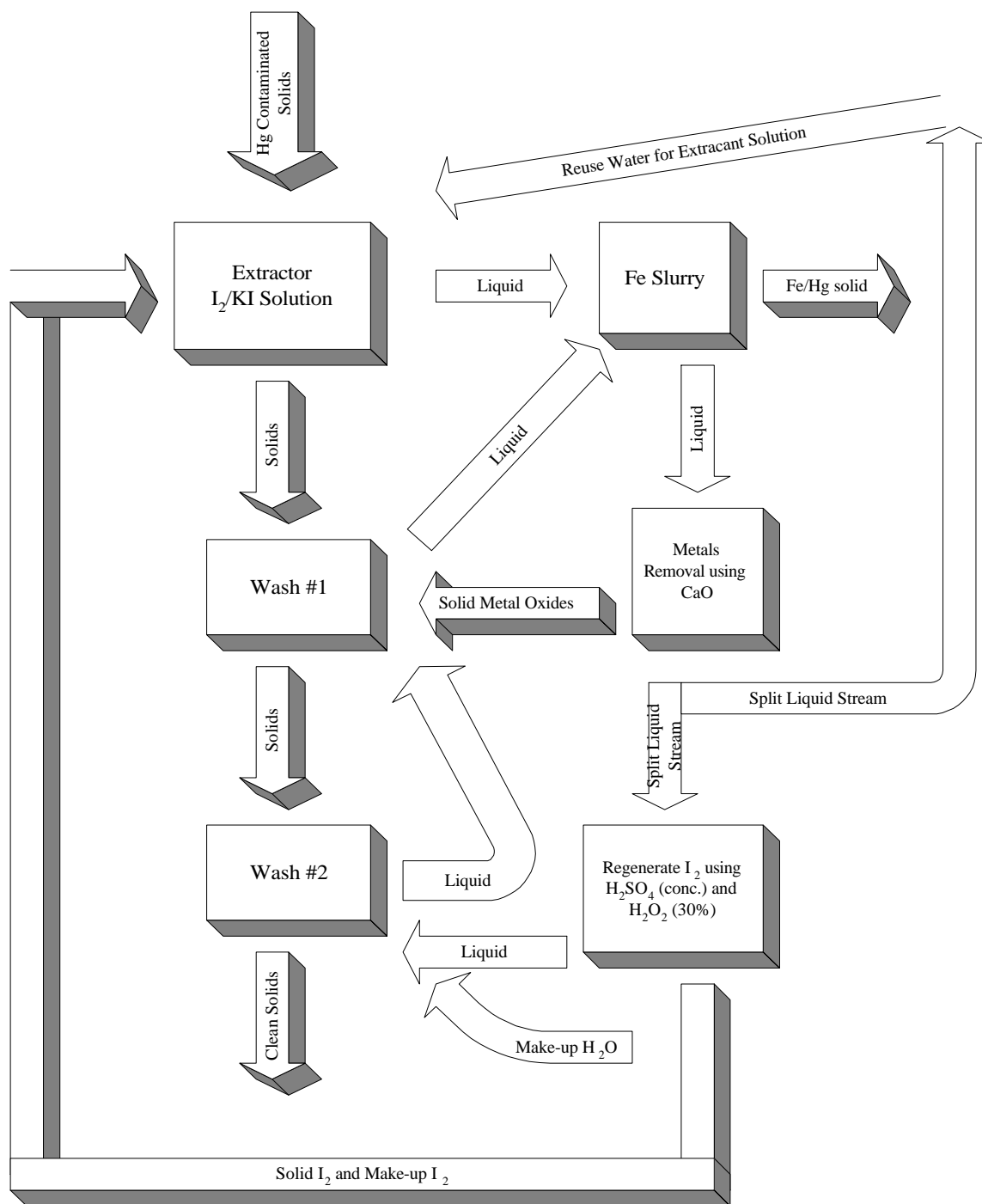


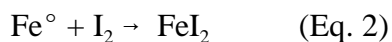
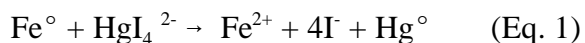
FIGURE 1-1. FLOWCHART FOR THE GEMEP PROCESS

TABLE 1-1. TREATMENT OF MERCURY-CONTAMINATED SAMPLES

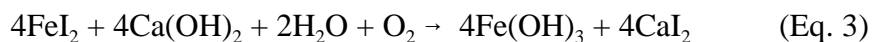
Site	Material	Initial Mercury Level (ppm)	Final Mercury Level (ppm)	Comments
Superfund, PR	Plastic	350-400	<15	
Superfund, PR	Soil	144	<4	
Industrial, MA	Sump Sludge	623-1,003	36	28% oil/2,000 ppm PCBs
Not Applicable	Fluorescent Lamp Phosphor	3,666-6,309	<10	Used Lamp
Not Applicable	Fluorescent Lamp Phosphor	1,815-2,890	6.8-7.0	Used Lamp
Not Applicable	Fluorescent Lamp Glass	3.3-5.8	0.46-0.49	Used Lamp
Oak Ridge, TN	Sediment	3,347	46	HgS
Oak Ridge, TN	Sediment	650	11	HgS
Oak Ridge, TN	Sediment	113	2	HgS
Oak Ridge, TN	Sediment	70	0.9	HgS
Port Refinery, NY	Soil	6,217-32,000	10	Hg°
Port Refinery, NY	Concrete	221-486	1	Hg°
Port Refinery, NY	Brick	36-112	1	Hg°
Natural Gas Pipeline	Soil/Gravel	7,070-34,700	27	Hg°
Natural Gas Pipeline	Soil/Gravel	328-544	11	Hg°
Mercury Reclaim Facility	Soil	22,800-23,600	<10	Hg°
Chlor-Alkali Facility	Soil	243-514	14-15	Hg°

Mercury Extraction. Mercury-contaminated media are subjected to an aqueous extraction with potassium iodide and iodine (KI/I₂). The pH and the temperature may be controlled to improve extraction efficiency. Mercury in its various forms is oxidized by the iodine to the 2⁺ oxidation state, which is then believed to form soluble complexes of the form HgI₄²⁻ by complexation with the iodide present in the extraction medium. After sufficient reaction time to solubilize the mercury, the treated media are dewatered, rinsed to remove residual extraction reagent and dissolved mercury, and backfilled or disposed. The extracted mercury remains in the aqueous phase and serves as the feed stream for the mercury reduction and removal step, along with the water generated from the rinsing of the treated media.

Mercury Reduction and Removal. The aqueous stream from the extraction step is reacted with finely divided elemental iron to reduce the mercury to its elemental form. The iron also reduces iodine to iodide:

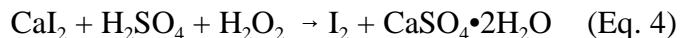


The metallic mercury produced in this step is recovered and isolated for off-site recycle. The remaining aqueous phase, containing dissolved ferrous iron and extraction reagent, is treated by pH adjustment to precipitate the iron as ferric hydroxide:



After dewatering, the precipitate can be combined with the treated solids from the extraction step for disposal, or it can be managed separately. A portion of the aqueous phase remaining after precipitation and dewatering becomes the feed stream for the iodine regeneration and recycle step.

Reagent Regeneration and Recycle. Iodine is regenerated through a combination of pH adjustment and chemical oxidation using hydrogen peroxide to oxidize iodide to iodine:



1.3 PROJECT OBJECTIVES

The objectives of Stage I of this project are to determine, by laboratory- and bench-scale testing, the capability of the GEMEP mercury extraction technology to treat representative DOE mixed wastes, and to determine if there are technical and economic advantages of the technology over alternative technologies. Secondary objectives are to determine the fate of radionuclides in the treatment process, evaluate the potential for generation of secondary waste, and generate data that will assist in the design of a pilot-scale system. GE-CRD conducted laboratory-scale tests on fluorescent lamps and on surrogates for DOE mixed wastes, while all bench-scale tests and tests of actual DOE wastes were conducted at CMRI. The different types of tests and the objectives of each are described further below. The wastes that were tested at each facility are described in Section 1.4. Details regarding the test procedures (materials and methods) are presented in Section 2.0. Results and conclusions are presented in Sections 3.0, 4.0, and 5.0.

1.3.1 Laboratory-Scale Tests

The primary objective of the laboratory scale tests performed by GE-CRD on fluorescent lamp wastes was to develop extraction conditions that would produce treated solids for which the mercury concentration in a TCLP extract would be less than the toxicity characteristic (TC) limit of 0.2 mg/L (200 ppb). This objective was established, and the studies were carried out, before EPA promulgated new Land Disposal Restriction (LDR) treatment standards for TC metal wastes. The new LDR universal treatment standard (UTS) for mercury in TC metal wastes is 0.025 mg/L (25 ppb) of mercury in the TCLP extract [7]. The UTS must be attained before TC

metal wastes can be land disposed. The UTS will go into effect for mixed TC metal/radioactive wastes on May 26, 2000.

Because the studies described herein were conducted before the new LDR treatment standards were established, the studies are based on attaining a TCLP extract concentration of no more than 0.2 mg/L mercury, rather than no more than 0.025 mg/L mercury. The latter objective would now need to be met to allow land disposal of treated lamp waste without further treatment.

To determine whether treated solids would pass TCLP, a TCLP test can be run on each batch of treated solids and the results evaluated. However, based on historical data of how fluorescent lamps behave in the TCLP test, it is known that 4 foot long, F40T12 model lamps will pass the test if the total mercury content is below 3 mg [8]. To allow for a margin of safety, GE-CRD established a clean-up goal of 2 mg (or 7.4 ppm for a 270 g lamp). Treated solids with less than 2 mg total mercury were considered to have “passed” TCLP. Lamps with less than 1 mg total mercury were judged unable to fail the TCLP test as there is not enough mercury to fail even if all the mercury dissolves (270 g lamp, 5.4 kg extracting solution, 1 mg mercury/5.4 kg = 185 ppb) [9].

Another objective of GE-CRD’s tests was to establish process conditions for treatment of fluorescent lamps that would minimize the loss of iodine (as either I_2 or I^-), because this reagent is the most costly component of the GEMEP process. When the GEMEP process was run on a soil/plastic matrix the loss of iodine was as high as 20%. Under these conditions it was necessary to add an iodine recovery step to the process flow chart to make the process economical. It was hoped that this would not be necessary for lamps. The goal was to keep iodine loss to under 1% or 10 g I_2 /kg solid. The current market price for bulk technical grade iodine is \$30-40/kg, so the iodine cost would be \$0.30-0.40/kg for treating fluorescent lamp wastes if the goal were met.

Finally, it is critical that initial pre-segregation steps to separate wastes into more manageable fractions do not result in generation of a secondary waste that would be classified as hazardous

under RCRA. Pre-segregation consists of mechanical separation steps using water and various size screens to separate material that is not contaminated with mercury (e.g., external pins, end mounts, aluminum end caps) from the lamp glass and phosphor, so that only mercury-contaminated materials need to be extracted in the GEMEP process. Pre-segregation can reduce treatment costs if the uncontaminated fraction is of significant quantity compared to the mercury-contaminated fraction, and if secondary hazardous waste is not produced. To avoid creating a secondary hazardous waste during pre-segregation, the residual mercury concentration in the pre-segregation solution must be less than 200 ppb [9], based on the TCLP limit for mercury. GE-CRD conducted tests to suppress mercury dissolution during pre-segregation, as described in Section 2.0.

1.3.2 Bench-Scale Tests

Bench-scale tests were conducted at CMRI on wastes from DOE sites and on fluorescent lamp waste provided by GE-CRD. Tests were conducted using a phased approach as described further below.

Initial Waste Characterization. The waste streams were characterized both physically and chemically to determine if mercury contamination resides primarily in certain size fractions. A thorough characterization of each waste type is essential to effectively develop pre-segregation and extraction tests that will provide the most useful information. For example, if mercury is found to be concentrated primarily in the fines fraction of a soil sample, that result suggests that pre-segregation of the waste before extraction may provide substantial cost savings during full-scale treatment.

To reduce consumption of the extraction reagent and thereby reduce treatment costs, it is desirable to remove elemental mercury from the wastes by physical means if at all possible, prior to extraction. Initial examination of wastes was conducted to determine if elemental mercury was

present. For wastes containing elemental mercury, a major objective is to remove the elemental mercury without solubilizing mercury compounds or radionuclides.

Pre-Segregation Tests. These tests involved developing one or more methods for pre-segregation of elemental mercury from the three waste types, as applicable, as well as pre-segregation of metal lamp parts from crushed lamp debris. Because the iodine in the GEMEP process can react with iron, aluminum, and copper, it is desirable to remove these metal lamp components from the crushed lamp glass prior to extraction. The first round of bench-scale testing attempted to develop such a method before actual extraction tests were carried out. The methods of pre-segregation that were tested were developed based on the results of the laboratory evaluation of process parameters and the initial waste characterization tests.

Determination of the Fate of Radionuclides. Because a main objective of this project is to reduce the quantity of mixed waste that DOE must manage, it is critical to determine the fate of the radionuclides as mixed wastes are treated to remove mercury. If necessitated by the type of waste, the plan was to develop a protocol for either selectively removing mercury from the waste while leaving the radionuclides with the solid fraction; or for solubilizing the radionuclides and mercury into two separate aqueous streams that can then be managed separately. The fate of radionuclides throughout the GEMEP process was determined for each waste type to assess how recycled streams and other residuals (e.g., iron hydroxide precipitate) should be managed.

Optimization of Extraction Process Parameters. Mercury extraction tests on the wastes (previously pre-segregated, where applicable) were done to optimize extractant concentrations, extraction methods, rinsing procedures, and extraction regeneration methods. The goal was to develop operating parameters specific to each waste type that could be used in developing cost estimates for full-scale treatment, and in designing a pilot system should Option 1 be exercised.

1.3.3 Technical and Economic Evaluation

The results of the laboratory- and bench-scale studies were used to perform an initial evaluation of the pre-segregation and GEMEP extraction technologies relative to competing technologies, in terms of: 1) ability to attain cleanup goals for mercury, 2) ability to reduce or eliminate mixed wastes, 3) quantities for secondary wastes requiring treatment/disposal, 4) potential for fugitive emissions, and 5) cost. The goal of this evaluation was to determine the viability of the GEMEP process for treatment of each waste type tested, and determine whether the technology is sufficiently promising to warrant pilot-scale studies on one or more of the waste types.

1.4 DESCRIPTIONS OF WASTES TESTED

DOE mixed wastes originally considered for testing were all located at Oak Ridge and included: storm sewer sediments stored on the Oak Ridge reservation, crushed lamp glass stored at Oak Ridge Plant Y-12, and contaminated sediments from the East Fork of Poplar Creek. A fourth waste, soil from Idaho National Environmental and Engineering Laboratory (INEEL), was later added to the program. Changes to the original program are described below.

Oak Ridge Storm Sewer Sediments. ORNL collected characterization data for the drums of storm sewer sediments available for shipment to CMRI. Review of the data showed it would not be possible to ship the storm sewer sediments to CMRI for bench-scale testing, due to concentrations of PCBs in the sediments in excess of 50 parts per million (CMRI is not currently permitted for treatability testing of TSCA-regulated wastes). Drummed mercury-contaminated mixed soils from Idaho National Engineering and Environmental Laboratory (INEEL) were shipped to CMRI for testing in place of Oak Ridge storm sewer sediments. GE-CRD conducted testing on a surrogate for the Oak Ridge storm sewer sediments that was developed at Oak Ridge National Laboratory [10].

Crushed Lamp Glass. Crushed lamp glass stored at Oak Ridge was not tested because the DOE sites have found a mechanism for disposing of their crushed lamp glass waste. Hence, the DOE

sites were no longer interested in supplying crushed lamp glass for testing. Spent lamps were instead provided by GE-CRD.

East Fork Poplar Creek Sediments. A sample of this material was collected by ORNL and shipped to CMRI for bench-scale testing. Initial testing was conducted, the results of which are presented in Section 4.0. The full suite of tests were not performed, however, upon direction from DOE, because Oak Ridge was able to dispose of these soils before testing was completed. Hence, it was decided that it would better serve DOE's interests to locate another material for testing, for which a disposal option had not yet been identified.

Actual DOE mixed wastes were shipped to CMRI for bench-scale testing by ORNL or by the originating DOE site (in the case of the INEEL waste). Fluorescent lamps were supplied by GE-CRD. Because GE-CRD's laboratory does not have a license for treatability testing of radioactive wastes, no DOE wastes were shipped to GE-CRD. GE-CRD conducted their tests on their own, non-radioactive fluorescent lamps, and on surrogates for the East Fork Poplar Creek sediment and Oak Ridge storm sewer sediments. The physical and chemical characteristics of the wastes that were tested at GE-CRD and/or CMRI are presented below.

1.4.1 Wastes Tested at GE-CRD

Soil Surrogate. For the mercury dissolution tests, EPA synthetic soil [11] was used by GE-CRD as a surrogate for East Fork Poplar Creek sediment. The soil was contaminated with HgS at 1000 ppm and Hg at 1000 and 10,000 ppm. Both Hg metal (leachable mercury) and HgS (considered to be non-leachable) were used for the tests. The non-leachable HgS was included because it is considered to be a likely form for mercury to be found in soils, and it was also included for completeness.

Storm Sewer Sediment Surrogate. For the dissolution tests, a storm sewer sediment surrogate was used that was developed at Oak Ridge National Laboratory [10]. It contained 50% EPA

synthetic soil, 35% SiO₂ (silica sand) and 15% CaCO₃. This material was spiked with Hg at 490 and 4,900 ppm and HgS at 490 ppm.

Fluorescent Lamps. The lamps used for this study were used (end-of-life), 4 foot long, Model F40T12 CW and CW/MW lamps. These lamps were used at GE-CRD and removed from service when no longer functioning. They had a variety of end discolorations and were manufactured over a number of years. The mercury content was found to vary dramatically (2 mg - 80 mg per lamp). Of the lamps tested, approximately 2% of them contained less than 2 mg of total mercury. The components of a 4-foot T12 lamp are shown in Table 1-2.

TABLE 1-2. BREAKDOWN OF THE COMPONENTS OF A 4-FOOT F40T12 LAMP

Components	Mass (grams)	% of Total Mass
External pins	1.0	0.35
Aluminum endcaps	2.1	0.73
Basing Cement	2.0	0.70
End mounts	4.6	1.61
Glass	256.4	89.62
-30 mesh fraction (phosphor)	17.8	6.22
Total Lamp	286.1	100

1.4.2 Wastes Tested at CMRI

East Fork Poplar Creek Sediment. One sediment sample from the East Fork of Poplar Creek was collected by ORNL and shipped to CMRI in September 1997. Upon receipt at CMRI, a visual inspection was performed and showed that the sample contained high moisture, a high percentage of fine particles, and a small amount of humus such as wood chips and grass. The sediment was known to contain a small amount of uranium.

Physical characterization of the as-received sample was performed. The moisture content was 33% and the bulk density was 0.82 g/cm³ (51 lb/ft³). The specific gravity of the dry sediment was 2.58. A portion of the dried sediment was submitted for a 31-element X-ray fluorescence scan. The scan, presented in Table 1-3, covers most rock and soil forming elements and accounts for 85-86% of the sample mass. The average mercury concentration in the sediment was 774 mg/kg, based on analysis of individual size fractions (see Section 4, Table 4-1).

Fluorescent Lamps. The crushed lamp glass used for the bench-scale tests at CMRI was generated from the same type of lamp used by GE-CRD and described in Section 1.4.1.

INEEL Contaminated Soil/Sludge. Three, 35-gallon drums containing soil/sludge from INEEL were received at CMRI on April 15, 1998. These drums were opened and a grab sample was taken from the surface of the soil in each drum. Observation of the surface indicated that the contents of the three drums were similar. Mercury analyses of the grab samples were as follows:

Drum 558	34 mg/kg
Drum 708	735 mg/kg
Drum 710	645 mg/kg

Drum 708 was emptied, the contents blended, and split into about twelve sub-samples. Visually the soil appeared to consist of a small plus 4-mesh fraction consisting of minor debris and very fine silt. The plus 4-mesh fraction consisted of three distinctly different materials: rock, asphalt, and a light-colored tar/polymer. Physical characterization of the sample showed a moisture content of less than 3%, a bulk density of 1.23 (80 lb/ft³), and a dry soil specific gravity of 2.61.

**TABLE 1-3. X-RAY FLUORESCENCE ANALYSIS:
EAST FORK POPLAR CREEK SEDIMENT**

As Oxide	Wt, %	Element	Concentration, mg/kg
Na ₂ O	0.16	V	102
MgO	1.43	Cr	148
Al ₂ O ₃	15.1	Co	16
SiO ₂	56.3	Ni	189
P ₂ O ₅	0.24	W	< 10
K ₂ O	2.40	Cu	266
CaO	1.85	Zn	246
TiO ₂	0.92	As	161
MnO	0.12	Sn	122
Fe ₂ O ₃	5.77	Pb	122
BaO	0.08	Mo	< 10
		Sr	134
<u>Element</u>	<u>Wt, %</u>	U	74
S	0.13	Th	57
Cl	< 0.02	Nb	17
		Zr	624
		Rb	89
		Y	57

A portion of the dried soil/sludge was submitted for a 31-element X-ray fluorescence scan. The scan, presented in Table 1-4, covers most rock and soil forming elements and accounts for 92-93% of the sample mass.

**TABLE 1-4. X-RAY FLUORESCENCE ANALYSIS:
INEEL SOIL/SLUDGE**

As Oxide	Wt, %	Element	Concentration, mg/kg
Na ₂ O	0.53	V	126
MgO	3.43	Cr	145
Al ₂ O ₃	10.8	Co	18
SiO ₂	49.4	Ni	69
P ₂ O ₅	0.28	W	13
K ₂ O	2.46	Cu	63
CaO	15.2	Zn	246
TiO ₂	0.45	As	154
MnO	0.07	Sn	70
Fe ₂ O ₃	9.29	Pb	76
BaO	0.08	Mo	< 10
		Sr	263
<u>Element</u>	<u>Wt, %</u>	U	34
S	0.07	Th	34
Cl	< 0.02	Nb	12
		Zr	160
		Rb	97
		Y	48

The radionuclide characterization of the soil/sludge was provided by INEEL and is presented in Table 1-5.

**TABLE 1-5. RADIOACTIVE COMPOSITION OF INEEL SOIL/SLUDGE
DRUM 708**

Isotope	mCi	Isotope	mCi	Isotope	mCi
Pa-234m	0.0026	Pa-231	4.9E-06	U-235	0.0049
Pm-147	0.1	Ac-227	4.9E-06	U-238	0.00264
Ba-137m	0.0468	Th-228	0.00031	Ac-228	0.000334
C-14	2.1E-07	Pa-234	4.2E-06	Co-57	0.000051
Ni-63	0.00021	K-40	0.0046	Co-60	0.00021
Ni-59	2.1E-06	Th-230	0.00042	Cs-137	0.05
Fe-55	0.00021	Th-232	0.000315	Pb-212	0.000261
Th-231	0.0049	U-234	0.113	Th-234	0.00264

Uranium (234) is the only isotope of significance. Based on the net weight of Drum 708, the concentration of U(234) was only 370 pCi/g (0.5 mg/kg).

SECTION 2.0

TEST PROCEDURES

This section summarizes the procedures followed by GE-CRD and CMRI in conducting the laboratory-scale and bench-scale tests, respectively.

2.1 LABORATORY-SCALE TESTS

All chemicals were reagent grade and were used without further purification. Iodine was 99.7% from Aldrich, potassium iodide was 99.9% from Baker, sulfuric acid was 96% from Baker, hydrogen peroxide was 30% from Baker, iron was minus 6 to plus 16 mesh from Connelly, calcium oxide was 99% from Baker, and toluene was 99.9% from Baker. The water used for the extractions and washes was tap water. For washing the acid digestions the water was deionized and then further purified by passing it through a Millipore system at 18 M Ω -cm.

Iodine and iodide were determined by a method described in a separate publication [12]. Mercury was determined by cold vapor atomic absorption (low detection limit) or flame (or ICP) atomic absorption (1 ppm detection limit).

In order to gage the reproducibility and repeatability of these techniques, 4-5 solutions were measured 3-4 times by two different operators. The standard deviations for the measurements were: $I_2/I = 0.0043$ absorption units and $Hg = 0.099$ ppm.

2.1.1 Extractions. The lamps were prepared for extraction by removing the external pins and aluminum endcaps. The vacuum was relieved by using tweezers to break the thin wall of glass inside the exhaust tube. Needle nose pliers were then used to push in the wire mount on one end. This serves to reduce the integrity of the glass wall which was then tapped on the inside of a one-gallon glass jar. The glass was broken until the whole lamp fit inside the jar. The jar was capped and shaken for 2 minutes. The resulting debris was passed through a ¼ inch diameter funnel

(except the end caps, if included) into a 2-liter Erlenmeyer flask. The extraction solution (of appropriate concentration and solid/liquid ratio) was then added. The flask was capped with parafilm with a small hole stuck in the parafilm for venting. The flask was shaken in a Orbit Environ-Shaker by Lab Line at 160 rpm and at the prescribed temperature. Once the desired reaction time was obtained the slurry was filtered through a Büchner filter using #1 Whatman filter paper. The solids were washed twice with the same volume of water as was used for the extracting solution. In general the used extraction solution and washes were kept separate and analyzed for I_2 , I^- and Hg (depending on the experiment). The glass pieces, along with the filter paper, was extracted either with CaO (1 g in 250 mL) for I^- determination or concentrated HNO_3 (200 mL)/concentrated HF (100 mL) for Hg determination [13].

2.1.2 Mercury Dissolution. Samples were contacted with distilled water at a 3/1 liquid-solid ratio for 24 hours at 45 °C, shaken at 160 rpm. The solutions were filtered and the resulting filtrate analyzed for mercury using the cold vapor atomic absorption procedure.

2.2 BENCH-SCALE TESTS

Bench-scale tests were conducted at CMRI's facility in Golden, Colorado. Bench-scale testing included four components: initial waste characterization, pre-segregation tests, bench-scale extraction tests, and locked-cycle tests. The test procedures used for each step in the bench-testing process are described below.

2.2.1 Initial Waste Characterization. The three wastes selected for testing, as identified in Section 1.4, were characterized for both physical and chemical properties prior to conducting tests on each of the wastes. This information was used to determine whether mercury contamination resided primarily in certain fractions of the waste, to determine the need for pre-segregation, and to effectively develop the pre-segregation and extraction tests. The following procedure was used in characterizing each of the wastes:

1. Upon receipt, samples were logged-in and inspected, and visual observations were noted. Samples were then blended, aliquots split for moisture, dried, weighed and pulverized to nominal minus 100-mesh for analytical pulp. Samples were then submitted for some or all of the following analytical characterization, depending on the waste type:

<u>Waste Type</u>	<u>Analysis</u>	<u>Method</u>
EFPC, INEEL	metals	X-ray fluorescence scan
EFPC, INEEL	mercury	Cold vapor
EFPC	total uranium	Fluorimetric

[Note: "EFPC" refers to the East Fork Poplar Creek sediment collected by ORNL and shipped to CMRI. Similarly, "INEEL" refers to the soil/sludge shipped by INEEL to CMRI.]

The composition of the lamp glass was known; therefore nothing would have been learned from detailed analysis. Hence, analytical characterization was not performed on the lamp glass.

2. De-agglomeration tests were conducted on 5-kg aliquots of the EFPC and INEEL samples. Samples were tumbled for 30 minutes in a small cement mixer at <30% solids with small addition of surfactant (± 1 gm/ton Triton X-100). The discharge from the mixer was passed through a screen at the following sieve sizes:

<u>EFPC</u>	<u>INEEL</u>
8-mesh	4-mesh
16-mesh	8-mesh
30-mesh	14-mesh
50-mesh	30-mesh
100-mesh	50-mesh
200-mesh	100-mesh
	200-mesh

A flow sheet for the de-agglomeration and sizing is shown in Figure 2-1. The material collected on each screen was dewatered, dried and weighed.

3. Small-scale, 100 mL, settling tests were performed on aliquots of <200-mesh solids slurry using a variety of flocculants. A quantitative, two-liter settling test was run on the INEEL sample aliquots using the best performing flocculant. Data suitable for thickener sizing was collected, including dry weight of solids. The solids were then dried and weighed. The data sheet for the settling tests is included as Figure 2-2.

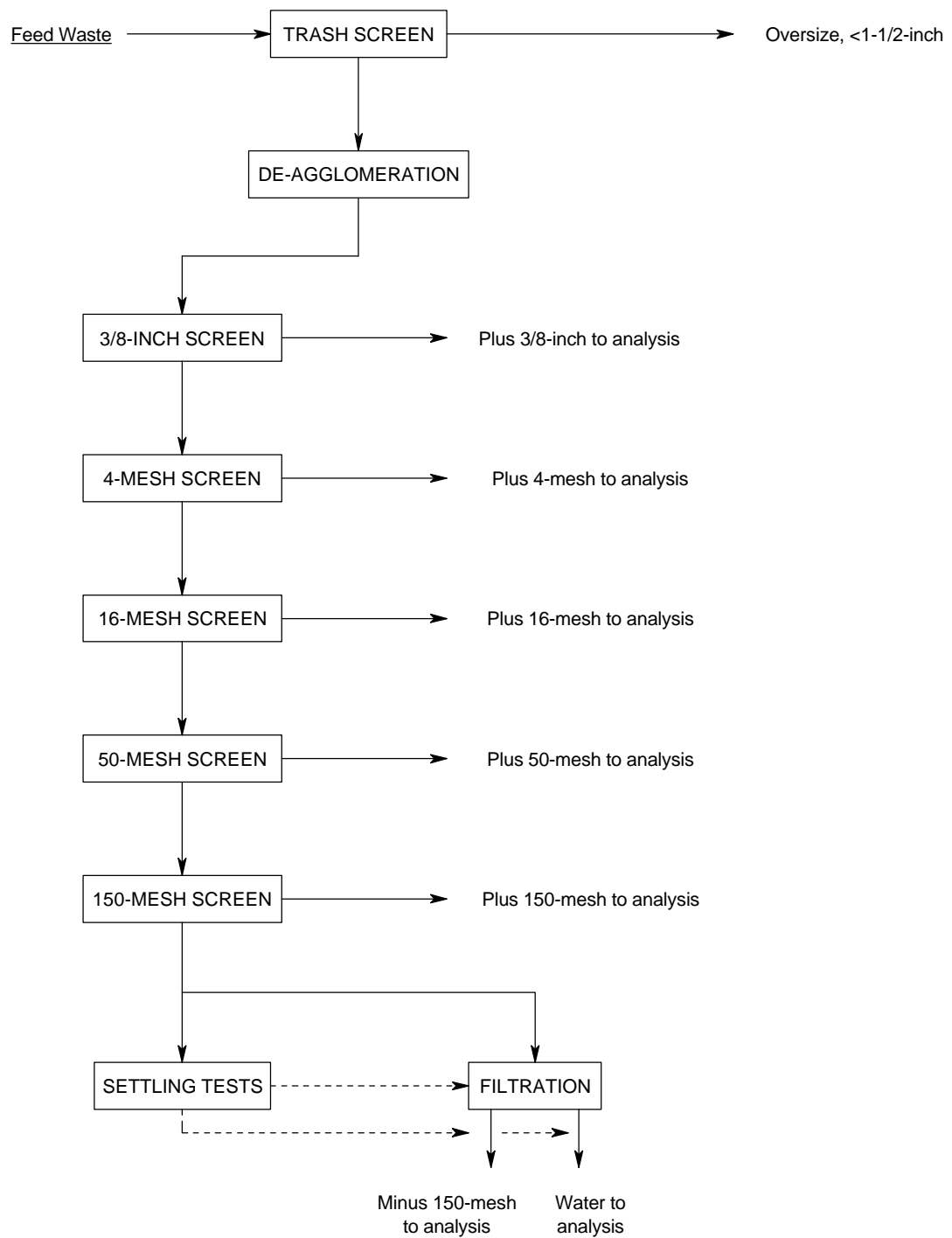


FIGURE 2-1. WASTE DE-AGGLOMERATION AND SIZING FLOWSHEET

COLORADO MINERALS RESEARCH INSTITUTE							
Metallurgical & Mineral Processing Consultants				SETTLING TEST			Project No.:
5906 McIntyre Street		(303) 279-2581		DATA AND CALCULATION SHEET			Date:
Golden, CO 80403		FAX 279-6061					
				Modified Kynch Procedure			
Test No:							
Feed Pulp				Feed Percent Solids			
				Density, g/L			
Flocculant							
Type				mL, 1 g/L		Dose, lb/st	
Type				mL, 1 g/L		Dose, lb/st	
Free Settling Rate, ft/hr				Terminal Percent Solids			0.0%
				Density, g/L			0
Settling Rate							
Time		Level				Feed Pulp	Terminal Pulp
min		mL					Clear Liquor
				Volume, mL			
				Gross pulp wt., g			
				Tare, g			
				Net pulp wt., g			
				Net dry wt., g			
				Density, g/L			
				Solids, %			
				Rake installed at		min	
				Rake rotation		min/rev	
				Thickener Unit Area Calculation			
				Initial Height, Ho		ft	
				Initial pulp density, Co (g solids/L,)			
				Co =		= st/ft ³	
				Terminal Density		solids	
				Critical time, Tx		days	
				Unit Area		ft ² /t/day	
Figure 2-2: Settling Test Data Sheet							

4. The individual size fractions were blended, aliquots were split for analysis and pulverized to nominal minus 100-mesh, and contaminant distribution was determined in the fractions and the recovered water by the analyses:

<u>Waste Type/Media</u>	<u>Analysis</u>	<u>Method</u>
EFPC/INEEL: solid fractions	Total mercury	Cold vapor
EFPC: solid fractions	Total uranium	Fluorimetric
EFPC/INEEL: water	Mercury	Cold vapor
EFPC: water	Uranium	Fluorimetric

Following completion of the initial waste characterization for each of the wastes tested, the data were compiled and evaluated. The distribution of mercury in each mesh fraction was reviewed to determine whether pre-segregation tests showed economic potential or whether proceeding directly to the extraction tests would be more economical.

2.2.2 Pre-Segregation Tests. The potential for separation of mercury-free material and metallic mercury from the balance of the sample was determined based on observations made and analytical results from the initial waste characterization. If the mercury concentration in some fractions was found to be less than what mathematically would pass the TCLP and the fraction could be separated readily, pre-segregation was conducted on the waste. As discussed previously, the purpose of the pre-segregation testing was to develop and evaluate methods of segregating mercury-free material and metallic mercury from the balance of the sample. Both the fluorescent lamps and the INEEL soil samples were subjected to pre-segregation.

The following pre-segregation test procedure was used on the fluorescent lamps to accomplish this goal. Twenty (20) kilogram batches of fluorescent tubes were crushed in a water spray using an impact crusher. The plus 3-mesh, non-mercury bearing fractions (flattened end caps) were separated by size. Gravity and magnetic separation were evaluated to separate non-mercury metallics from the balance, i.e., pins and wires from the glass. Gravity separation was done using a mineral jig but was not effective. The minus 3-mesh material then was passed over a wet magnetic separator to remove the magnetic pins and any attached wires.

For the INEEL sample, 20-kg aliquots of the sample were de-agglomerated using the small cement mixer. Non-mercury bearing fractions (the plus 4-mesh, as determined by Initial Waste Characterization) were separated by wet sieving.

A flow sheet for the separations is shown in Figure 2-3.

On completion of all separation tests, the individual fractions were blended, aliquots were split for analysis and pulverized to nominal minus 100-mesh. The following analyses were conducted:

<u>Waste Type</u>	<u>Analysis</u>	<u>Method</u>
Lamps/INEEL	Mercury	Cold vapor

Solubilization of mercury was not noted in Steps 2, 3, or 4 of the Initial Waste Characterization. Therefore, there was no need to suppress mercury solubilization during the de-agglomeration, sizing and gravity concentration steps.

Since the separation steps used for each waste were very simple, a sophisticated process mass balance was not generated. The mass balances presented in Sections 4.0 and 5.0 show the results of the separations.

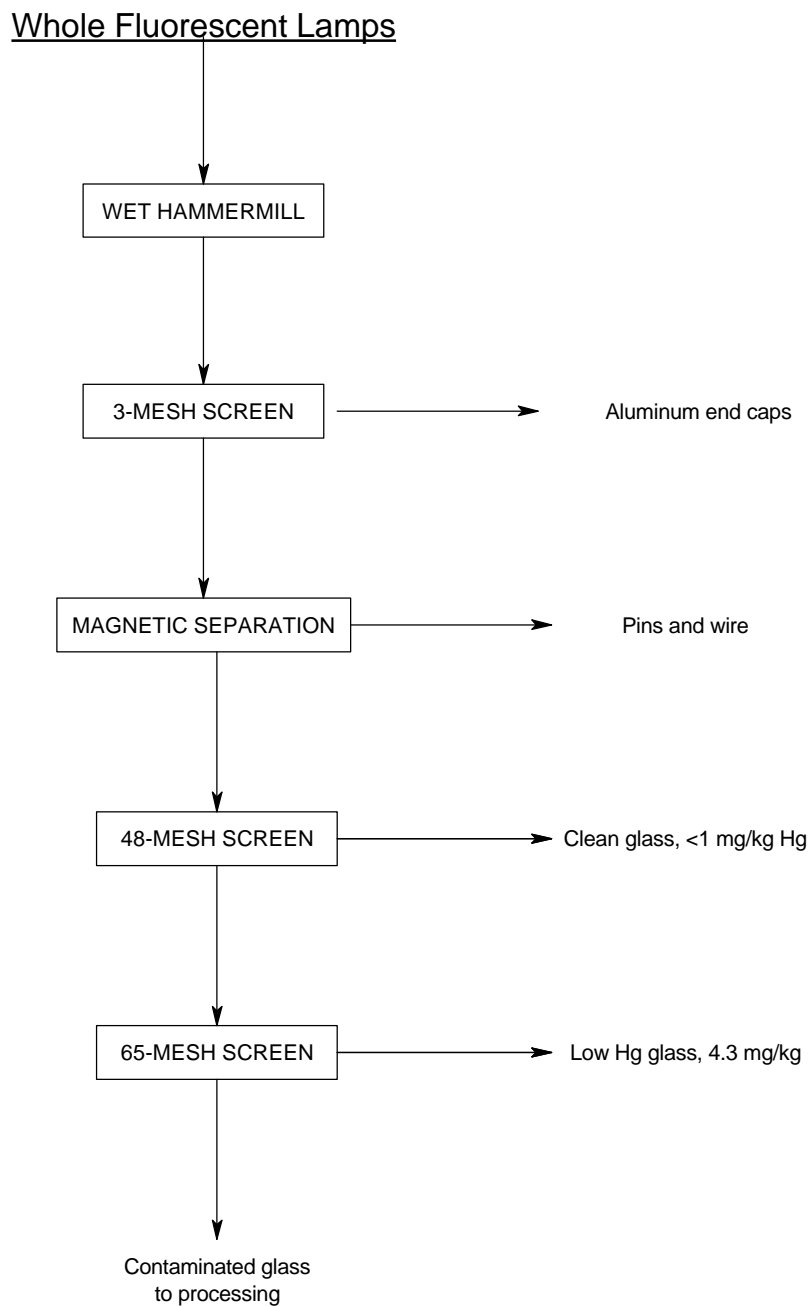


FIGURE 2-3. FLUORESCENT LAMP GLASS CRUSHING, SIZING AND MAGNETIC SEPARATION

To maximize dewatering of the solids after pre-segregation and prior to the chemical extraction of mercury, quantitative settling and/or filtering tests were performed to determine the field dewatering requirements. The crushed lamp glass settled and filtered very rapidly and was not tested. The following tests were performed on the INEEL sample:

<u>Waste Type</u>	<u>Dewatering Test</u>
INEEL, minus 4-mesh soil	Thickening

Basic data to be used for scale-up to a pilot-scale treatment system for treatment of INEEL soil/sludge were collected. These data included bulk densities of the feed material, the settling and filtration characteristics of the fine fractions, and viscosity of slurries at various solids densities (10 to 50%).

2.2.3 Bench-Scale Extraction Tests. Extraction testing, using potassium iodide/iodine (KI/I_2) lixiviant to separate mercury from the test sample, was conducted on each of the selected wastes to determine optimum conditions for mercury extraction. Reagent-grade chemicals used were: iodine (99.7%) from Aldrich, potassium iodide (99.9%) from Aldrich, sulfuric acid (96%) from Baker, and hydrogen peroxide (30%) from Baker. Commercial grade iron turnings and hydrated lime were used. The water used for the extractions and washes was tap water.

Iodine and iodide were determined volumetrically by the method described in a separate publication [12]. Low levels of mercury were determined by cold vapor atomic absorption and higher levels were determined by flame atomic absorption.

The extraction tests were used to evaluate iodine and iodide concentrations, acidity, solids density, and retention. The number of tests conducted on each waste type varied, depending on the characteristics of the waste. For the East Fork Poplar Creek sediment (Waste Type EFPC), eight (8) tests were performed; for the Crushed Lamps (Waste Type Lamps), seventeen (17) tests were performed; for the INEEL soil (Waste Type INEEL), seventeen (17) tests were performed.

The following test procedure was used.

1. The sample for extraction and the desired doses of potassium iodide and iodine were added to a volume of water. The solids sample was added at a quantity of 50 to 200 grams so as to create a 25% solids slurry (liquid to solid ratio of 3/1). The mixture was heated to the desired temperature in the range of 20° to 55°C. In some cases the pH was adjusted by the measured addition of 1000 g/L sulfuric acid solution. Once the pH was at the targeted level, the leaching period was started.
2. The slurry temperature and pH were monitored for the leaching period (2 to 24 hours).
3. At the termination of the leaching period, the slurries were vacuum filtered and washed with tap water. A flowsheet for the chemical extraction tests is presented in Figure 2-4. The residues were then blended, aliquots split for analysis, and pulverized to nominal minus 100-mesh for analysis. The following analyses were conducted on the mercury-bearing residues:

<u>Waste Type</u>	<u>Analysis</u>	<u>Method</u>
EFPC	Uranium	Fluorimetric
All	Mercury	Cold vapor
All	Soluble mercury	TCLP

The filtrates and washes were combined and analyzed as follows:

<u>Waste Type/media</u>	<u>Analysis</u>	<u>Method</u>
All	Mercury	Cold vapor or flame AA
All	Iodide	Volumetric
All	Iodine	Volumetric
EFPC	Total uranium	Fluorimetric

A data sheet for the batch chemical extraction is presented as Figure 2-5.

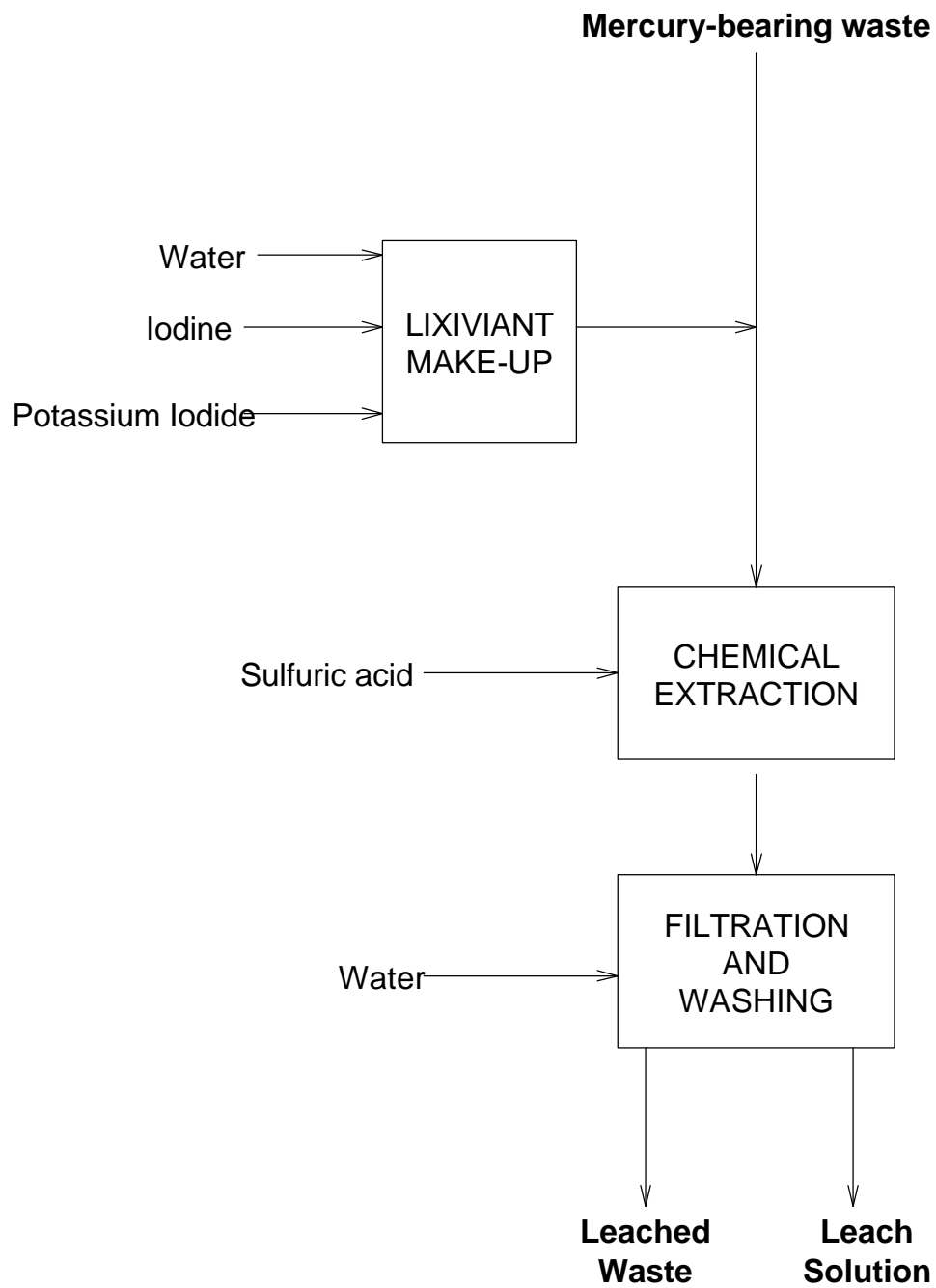


FIGURE 2-4. CHEMICAL LEACHING FLOWSHEET

COLORADO MINERALS RESEARCH INSTITUTE				CHEMICAL EXTRACTION					
Metallurgical & Mineral Processing Consultants				OF MERCURY-BEARING WASTE				Project No.: 971026-06	
5906 McIntyre Street				(303) 279-2581				Date:	
Golden, CO 80403				FAX 279-6061					
Test No:				___ °C		M KI			
				___		M I2			
						Hg,			
Feed: 0 dry grams				% moisture		Damp wt.		Hg, mg	
				0.0				0	
				0.0				0	
				0.0				0	
Leach:				% solids					
				Liquid		Potassium Iodide		Iodine	
				dry g		KI, M		I2, M	
				grams		KI, g		I2, g	
				mL					
Solids feed:				0					
Total I required						g/l		---	
Leach KI solution									
Repulp KI solution									
Heat to 50°C, adjust pH to 4.0 with 1000 g/L H2SO4									
Leach 4.0 hours									
Transfer to vacuum filter, filter, wash with 100 mL water									
Measure volume of filtrate plus wash									
Repulp in 300 ml of the same solution as used for leaching, heat to 50°C for 1 hour									
Transfer to vacuum filter, filter, wash with 100 mL water									
Measure volume of filtrate plus wash									
Dry and sample solids									
						mL			
						1000g/L			
						H2SO4			
				Time		Minutes		°C	
						pH			
				start					
				0					
				30					
				60					
				90					
				120					
				180					
				240					
Sample ID				Wet grams		Dry grams		Hg mg/kg	
						Hg mg		Hg, % Distn	
						KI g/l		KI g	
								I2 M	
								I2 g	
-1 Residue									
				mL		mg/L			
-2 Filtrate-1									
-3 Filtrate-2									
Balance, versus feed									

Figure 2-5: Chemical Leaching Data Sheet

Tests were performed in groups of two or three on each of the waste materials. This allowed time for evaluation of the analytical results of the earlier group of tests before determining the exact conditions for a subsequent group of tests.

The range of iodine and iodide concentrations depended on the waste type. For the East Fork Poplar Creek sediment, concentrations ranged from 0.025 M KI and 0.0125 M I₂ to 0.4 M KI and 0.2 M I. pH values of 4, 5, and 7 were tested. Table 2-1 presents the matrix for the chemical extraction tests on the East Fork Poplar Creek sediment (Waste Type EFPC).

TABLE 2-1. CHEMICAL EXTRACTION TEST MATRIX FOR EAST FORK POPLAR CREEK SEDIMENT (WASTE TYPE EFPC)

Test No.	KI, M	I ₂ , M	pH	Hours	Temp., °C
A-1	0.10	0.05	4	2	50
A-2	0.20	0.10	4	2	50
A-3	0.40	0.20	5	2	50
A-4	0.40	0.20	7	2	50
A-5	0.025	0.0125	4	2	50
A-6	0.05	0.025	4	2	50
A-7	0.10	0.05	5	2	50
A-8	0.10	0.05	7	2	50

For the fluorescent lamp glass, concentrations ranged from 0.04 M KI and 0.02 M I₂ to 0.12 M KI and 0.06 M I₂. Table 2-2 presents the matrix for the chemical extraction tests on the fluorescent lamp glass.

**TABLE 2-2. CHEMICAL EXTRACTION TEST MATRIX FOR
FLUORESCENT LAMP GLASS (WASTE TYPE LAMP)**

Test No.	KI, M	I ₂ , M	Temp., °C	Hours
G-1	0.04	0.02	15	2
G-2	0.04	0.02	16	6
G-3	0.04	0.02	17	24
G-4	0.04	0.02	36	2
G-5	0.04	0.02	35	6
G-6	0.04	0.02	35	6
G-7	0.04	0.02	52	2
G-8	0.04	0.02	52	6
G-9	0.08	0.04	20	2
G-10	0.08	0.04	20	4
G-11	0.08	0.04	20	6
G-12	0.08	0.04	34	2
G-13	0.08	0.04	34	4
G-14	0.12	0.06	20	2
G-15	0.12	0.06	20	4
G-16	0.08	0.06	20	2
G-17	0.08	0.06	20	4

For the INEEL soil, concentrations ranged from 0.01 M KI and 0.005 M I₂ to 0.4 M KI and 0.2 M I₂. One set of tests attempted to adjust the pH, but the high calcium carbonate content of the soil prevented significant pH adjustment without significant dissolution of the sample matrix. For tests I-10 through I-17, the residues were repulped in KI-containing water and re-filtered. Tests I-14 through I-17 were done in two stages with filtration between the stages. Table 2-3 presents the matrix for the chemical extraction tests on the INEEL soil.

**TABLE 2-3. CHEMICAL EXTRACTION TEST MATRIX FOR
INEEL SOIL/SLUDGE (WASTE TYPE INEEL)**

Test No.	KI, M	I ₂ , M	pH	Hours	Temp., °C
I-1	0.1	0.05	natural	4	25
I-2	0.2	0.1	natural	4	25
I-3	0.4	0.2	natural	4	25
I-4	0.1	0.05	4-5.6	4	45
I-5	0.2	0.1	4-5.6	4	45
I-6	0.4	0.2	4-5.6	4	45
I-7	0.1	0.05	natural	4	45
I-8	0.2	0.1	natural	4	45
I-9	0.4	0.2	natural	4	45
I-10	0.2	0.1	natural	4	45
I-11	0.3	0.15	natural	4	45
I-12	0.2	0.1	natural	4	35
I-13	0.3	0.15	natural	4	35
I-14, 2	0.2/0.2	0.1/0.1	natural	2 + 2	45
I-15, 2	0.2/0.1	0.1/0.05	natural	2 + 2	45
I-16, 2	0.2/0.2	0.1/0.1	natural	2 + 2	55
I-17, 2	0.2/0.1	0.1/0.05	natural	2 + 2	55

Only the East Fork Poplar Creek sediment sample contained significant radionuclides, i.e., 27 mg/kg total uranium. The combined filtrate plus wash solutions contained from 1 to 49 g/L (0.7 to 33 pCi/L), indicating co-extractions of 0.01 to 0.1% of the contained uranium. Because the INEEL sample contained less than 0.5 mg/kg U (234), the U(234) solubility was not followed during the bench-scale extraction studies.

2.2.4 Locked-Cycle Tests. Locked-cycle tests were conducted to demonstrate the complete extraction process, including extraction, residue rinsing, mercury recovery, and iodine regeneration. Locked-cycle tests were conducted on crushed lamp glass and on the INEEL soil. Locked-cycle tests were not conducted on the East Fork Poplar Creek sediment (Waste Type EFPC), since testing was discontinued at the direction of DOE.

The locked-cycle tests were conducted on 400-g and 1,000-g scales for the fluorescent lamp waste and the INEEL soil respectively. Ten cycles initially were performed on each material, with two later cycles added for the INEEL soil. Conditions for the locked-cycle tests were selected based on the optimum conditions determined in the bench-scale extraction tests. The locked-cycle tests can best be described by the iodine regeneration flow sheet, as shown in Figure 2-6.

A process mass balance, generated for the extraction/recovery/regeneration steps, was prepared for each test, and is presented in Section 5.0.



SECTION 3.0

RESULTS OF LABORATORY-SCALE TESTS

GE-CRD performed a series of laboratory-scale extraction tests on fluorescent lamp waste to determine the extraction conditions that would minimize the residual mercury in the waste and result in the smallest consumption of iodine. Lamps with large (> 100 mg Hg) mercury doses were used for the tests. Once the optimum extraction conditions were determined, tests that included the complete GEMEP process cycle (extraction, washing, and iodine/iodide recovery) were performed on the fluorescent lamp waste. Finally, tests to simulate possible pre-segregation steps were performed on fluorescent lamps and two surrogates for DOE mixed wastes (East Fork Poplar Creek sediments and Oak Ridge Y-12 storm sewer sediments) to assess ways of suppressing mercury dissolution during these steps. The results of these tests are described in this section.

3.1 EXTRACTION TESTS ON FLUORESCENT LAMP WASTE

The objectives of the extraction tests on fluorescent lamp waste were to establish conditions that would optimally reduce the residual mercury remaining in the treated waste to a level that will allow the waste to pass the TCLP test, while also reducing the iodine lost during the extraction process. Iodine can exit the process in several ways: 1) with the treated waste (if it adsorbs to the waste, and/or is not effectively washed from the waste), 2) by volatilization from the extraction solution during extraction, washing, or recovery steps, and 3) by less than 100% recovery during the iodine recovery step. It is important to efficiently recover the iodine because the cost of make-up iodine is generally the largest single operating cost of the GEMEP process. Conditions that produce the lowest residual mercury content in the treated waste may not necessarily be those that result in the lowest iodine cost. A series of laboratory-scale extraction tests were carried out and statistically analyzed to determine optimum conditions for extraction of

fluorescent lamp waste. The design of the experiments and the results obtained are described below.

3.1.1 Minimizing Residual Mercury. A number of factors influence the residual mercury in the treated lamp waste. These include concentration of iodine, liquid/solid ratio, time of extraction, temperature, number of washes, variations in shaker speed, mercury content, lamp history and mercury losses during handling. Concentration, liquid/solid ratio, time, temperature, mercury content and lamp history were considered the most important. The mercury content and lamp history cannot be controlled so the four other factors were examined in a screening design of experiments. High and low values for each of the four factors are listed in Table 3-1. The set up for the design of experiments is shown in Table 3-2.

TABLE 3-1. HIGH, MID, AND LOW VALUES FOR SCREENING DESIGN OF EXPERIMENTS

Factor	High Value	Mid Value	Low Value
concentration of I ₂	0.2 M	0.02 M	0.002 M
liquid/solid ratio	3	2	1
Time	24 hours	5 hours	1 hour
Temperature	65°C	45°C	25°C

TABLE 3-2. SCREENING DESIGN OF EXPERIMENTS

Exp. #	Conc. I ₂	L/S Ratio	Time	Temp.	Result (mg Hg)
1	Mid	Mid	Mid	Mid	10.8
2	Low	Low	High	High	4.5
3	Low	High	High	Low	25
4	High	Low	Low	High	0.25
5	High	Low	High	Low	0.30
6	Low	Low	Low	Low	2.2
7	Mid	Mid	Mid	Mid	5.9
8	High	High	Low	Low	0.10
9	Low	High	High	Low	27.3
10	High	High	High	High	0.10

The results of the design of experiments were analyzed using an analysis of variance approach looking at the main effects. The results are shown in Figure 3-1. The concentration of iodine and the liquid/solid ratio were found to have the largest effect on the residual mercury ($P > 0.05$), based on an analysis of the data using MINITAB statistical software. From this result a full factorial analysis was carried out using the values shown in Table 3-3 and the experiments shown in Table 3-4.

FIGURE 3-1. MAIN EFFECTS PLOT FOR SCREENING DESIGN OF EXPERIMENTS
 (-1 Represents the Low Value and +1 Represents the High Value for the Specific Factors)

♦ Centerpoint

Main Effects for Results

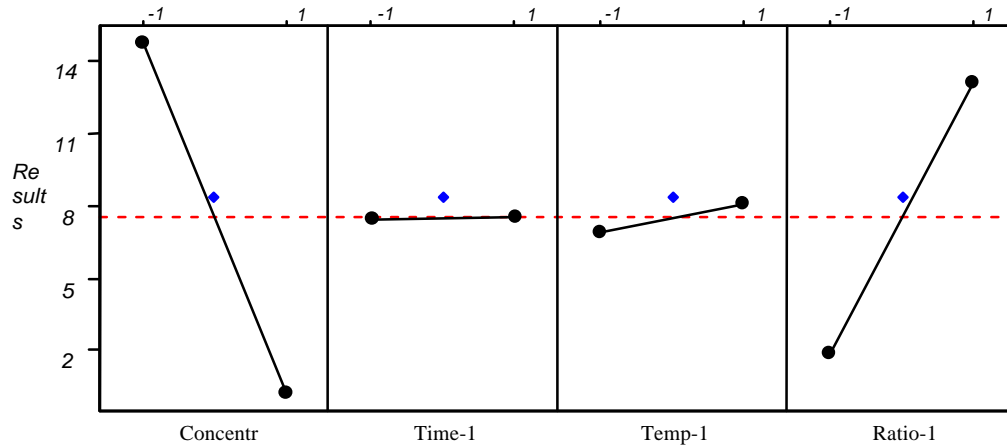


TABLE 3-3. HIGH, LOW AND MID VALUES FOR FULL DESIGN OF EXPERIMENTS

Factor	High Value	Mid Value	Low Value
Concentration I ₂	0.2	0.02	0.01
Liquid/solid Ratio	3	2	1

TABLE 3-4. FULL FACTORIAL CENTRAL COMPOSITE DESIGN OF EXPERIMENTS

Exp. #	Conc. I ₂	Ratio	# of Repeats	Avg. Result (mg Hg)
1	High	Mid	1	0.69
2	Mid	Mid	3	6.9
3	Low	Mid	1	0.6
4	Low	High	6	3.11
5	Mid	Low	2	13.6
6	Mid	High	13	0.62
7	High	High	3	0.13
8	High	Low	2	0.27
9	Low	Low	1	9

For some experiments, there are a large number of repeats because GE-CRD decided to make use of data from previously conducted experiments, and supplemented the historical data with experiments conducted solely for this project.

From the results of the full factorial experiments it was possible to generate a prediction equation for residual mercury based on concentration of iodine and the ratio of liquid to solid.

$$\text{Residual mercury (mg)} = 16.01 - 76.09(\text{conc. I}_2) - 5.00(\text{ratio}) + 24.17(\text{conc. I}_2 \times \text{ratio})$$

The concentration of iodine (conc. I₂) is in moles/liter and ratio is the liquid/solid ratio. From this equation a contour graph and 3D graph of the design space can be generated. These plots are shown in Figures 3-2 and 3-3. By using the above equation or examining the figures it was possible to determine the concentration of iodine and ratio of liquid to solid that would minimize residual mercury in the solid. The design point which gives the lowest residual mercury was the one with the highest concentration of iodine and largest liquid/solid ratio. This was not surprising. What was more useful was that it was possible to calculate the residual mercury at a number of different concentrations and ratios.

Another important consideration was the error in Figures 3-2 and 3-3. Since some points were represented by only one data point the certainty in those numbers was low. Using the design points for which more than one data point was available it was possible to calculate the standard deviation at those points. It was also possible to calculate the 95% upper confidence interval for each point using the equation below:

$$\text{Upper Confidence Limit} = s \times \sqrt{df / \chi^2_{\alpha/2, df}}$$

Where s is the standard deviation, df is the degrees of freedom = 0.05 for 95% confidence limit, and χ^2 is taken from a table of values at $\alpha/2$ and the specified degrees of freedom. Table 3-5 lists the standard deviations (s) and the UCL for the six design points with multiple values.

FIGURE 3-2. 3D PLOT FOR THE SURFACE GENERATED BY THE RESIDUAL Hg PREDICTION EQUATION

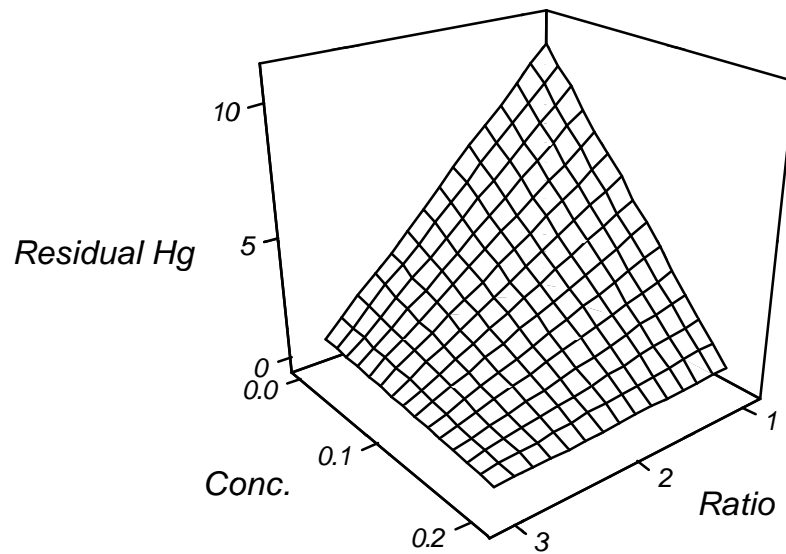
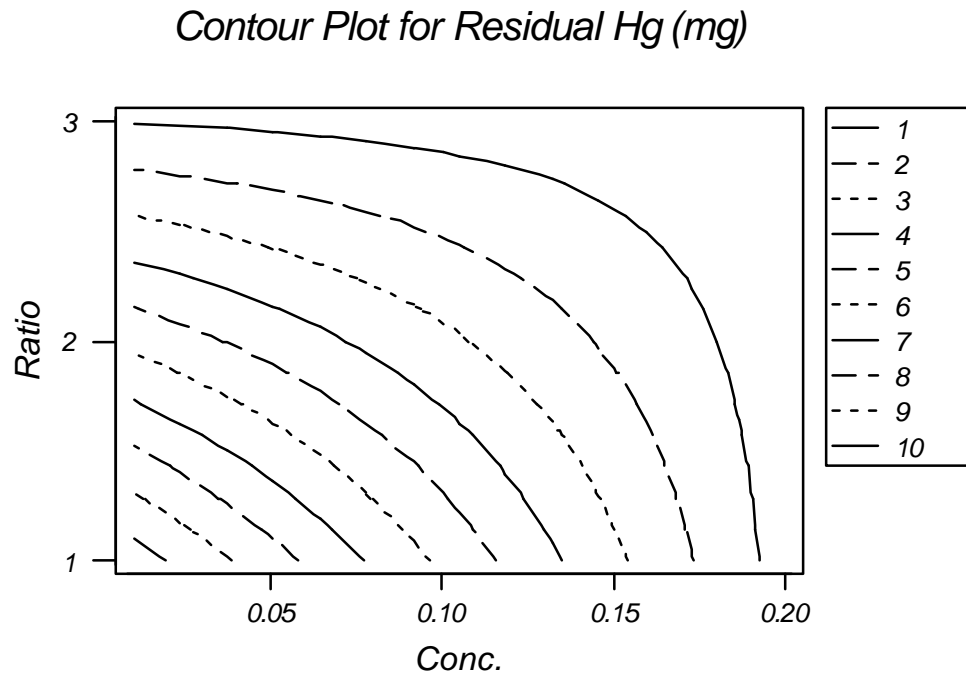


FIGURE 3-3. CONTOUR PLOT GENERATED BY THE PREDICTION EQUATION FOR RESIDUAL MERCURY



**TABLE 3-5. THE STANDARD DEVIATION (*s*) AND 95% UCL
FOR *s* FOR SIX DESIGN POINTS**

Design Point	Conc. I ₂	ratio	<i>s</i>	95% UCL
1	0.02	2	3.4	21.4
2	0.01	3	3.6	8.8
3	0.02	1	16.8	531
4	0.02	3	0.27	0.44
5	0.20	3	0.06	0.37
6	0.20	1	0.03	0.19

From the data in Table 3-5 it was possible to generate prediction equations for both *s* and the 95% UCL for *s* in terms of concentration of iodine and ratio of liquid to solid.

$$s = 23.9 - 118.9(\text{conc. I}_2) - 7.6(\text{ratio}) + 37.8(\text{conc. I}_2 \times \text{ratio})$$

$$95\% \text{UCL} = 756 - 3772(\text{conc. I}_2) - 266(\text{ratio}) + 1324(\text{conc. I}_2 \times \text{ratio})$$

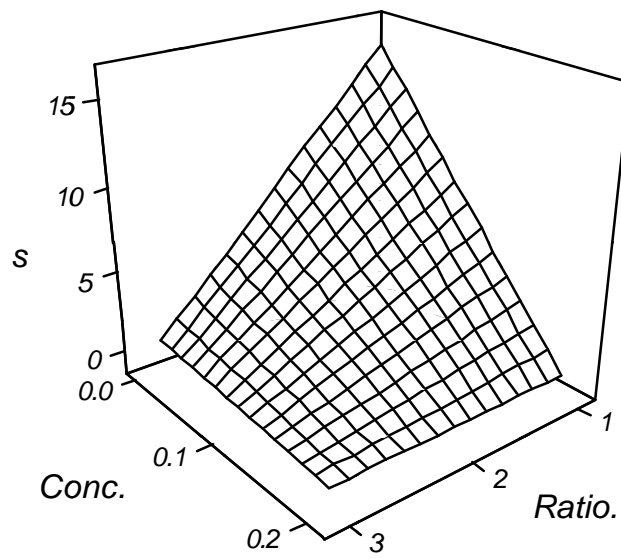
The 3D graph generated from the equation for *s* is shown in Figure 3-4. The graph for the 95%UCL was similar in appearance with larger values.

Ideally, the process should be operated where both the residual mercury is low and the standard deviation is low. One way to measure this is to calculate a Z value for each design point. Z and Z_{95%} are defined below.

$$Z = (\text{Upper specification limit} - \text{residual mercury})/s$$

$$Z_{95\%} = (\text{Upper specification limit} - \text{residual mercury})/95\% \text{ UCL}$$

FIGURE 3-4. 3D GRAPH OF THE STANDARD DEVIATION (s)

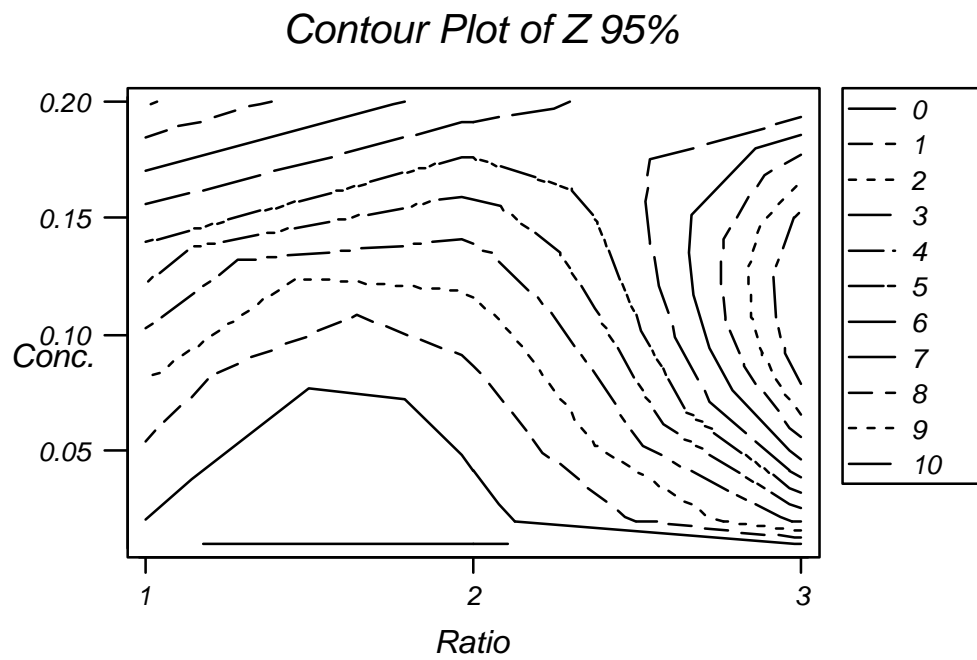


The upper specification limit in this case was 2 mg of mercury. As explained in Section 1.3.1, historical data for fluorescent lamps has shown that the lamps used in this work will pass the TCLP test if the total mercury content is below 3 mg. For a margin of safety, a residual mercury content of 2 mg (or 7.4 ppm for a 270 g lamp) was selected as the upper limit. The residual mercury values are presented in Table 3-4. The 95% UCL can be calculated from the equation above or read from Table 3-5. The values for $Z_{95\%}$ are shown in Table 3-6 for each of the design points. A contour plot of $Z_{95\%}$ is shown in Figure 3-5.

TABLE 3-6. $Z_{95\%}$ VALUES FOR THE 9 DESIGN POINTS FROM THE FULL FACTORIAL DESIGN OF EXPERIMENTS

Design Point	Conc. I_2 (M)	Ratio	$Z_{95\%}$
1	0.20	2	6.5
2	0.02	2	-0.22
3	0.01	2	0.0065
4	0.01	3	-0.12
5	0.02	1	-0.021
6	0.02	3	3.1
7	0.20	3	5.0
8	0.20	1	9.1
9	0.01	1	-0.015

FIGURE 3-5. CONTOUR PLOT OF $Z_{95\%}$ FOR RESIDUAL MERCURY



It is best to operate at conditions where $Z_{95\%}$ is above 6, because this guarantees that the number of failures (lamps with greater than 2 mg of mercury) will be less than 4 per million attempts. As shown in Figure 3-5 the area where $Z_{95\%}$ is greater than 6 is at the high concentration (0.2 M) or high ratio (3). In order to conserve I_2 the following conditions were chosen:

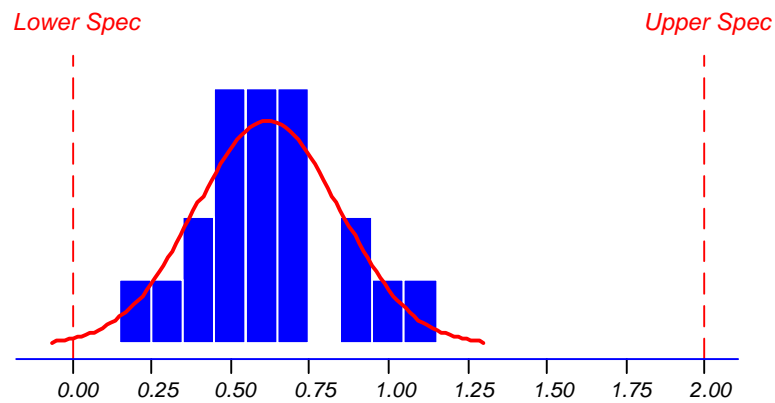
0.02 M I_2 / 0.04 M KI (the I^- concentration is always kept at 2 times the I_2 conc.)

3/1 Liquid/Solid Ratio

A number of extractions were performed at the conditions above, and the results are presented graphically in Figure 3-6. The Z value for this set of data is 6.06 (PPU*3) indicating less than 4 batches out of 1 million should have residual mercury greater than 2 mg.

FIGURE 3-6. CAPABILITY ANALYSIS FOR RESIDUAL MERCURY (mg) at 0.02 M I₂ and 3/1 L/S

Capability analysis for Residual Hg (mg)



Long-Term Capability

<i>Pp</i>	1.46	<i>Targ</i>	*	<i>Mean</i>	0.61650	<i>%>USL Exp</i>	0.00	<i>PPM>USL Exp</i>	0
<i>PPU</i>	2.02	<i>USL</i>	2.0000	<i>Mean+3s</i>	1.30040	<i>Obs</i>	0.00	<i>Obs</i>	0
<i>PPL</i>	0.90	<i>LSL</i>	0.0000	<i>Mean-3s</i>	-0.06740	<i>%<LSL Exp</i>	0.00	<i>PPM<LSL Exp</i>	0
<i>Ppk</i>	0.90	<i>k</i>	0.3835	<i>s</i>	0.22797	<i>Obs</i>	0.00	<i>Obs</i>	0
<i>Cpm</i>	*	<i>n</i>	20.0000						

3.1.2 Minimizing Iodine Loss. Iodine loss is defined here as residual iodine found on the solids. Although there are other sources of iodine loss as discussed previously, residual iodine on the treated solids is the most readily controlled source of loss.

The optimum conditions for minimizing iodine loss were determined by using a similar approach to that described above for minimizing residual mercury. Of the factors that could have an effect on iodine loss (concentration of iodine, time and temperature of extraction, ratio of liquid-solid, mercury dose, and lamp history), the concentration of iodine and the L/S ratio were the factors being used to control residual mercury, since mercury dose and lamp history were not controllable. That left time and temperature of extraction to vary to minimize iodine loss.

A full factorial design of experiments was designed around the two factors. The high, low and mid values are shown in Table 3-7. Table 3-8 lists the experiments along with the number of repeats.

TABLE 3-7. HIGH, LOW, AND MID VALUES FOR FULL DESIGN OF EXPERIMENTS FOR I₂ LOSS

Factor	High Value	Mid Value	Low Value
Time (hours)	24	5	1
Temperature (°C)	65	45	25

**TABLE 3-8. FULL FACTORIAL CENTRAL COMPOSITE
DESIGN OF EXPERIMENTS**

Exp. #	Time	Temperature	# of Repeats	mg I ₂ on solids/g solids
1	High	High	1	0.39
2	Low	Mid	1	0.15
3	Mid	Mid	5	0.75
4	Low	High	1	0.06
5	Mid	Low	1	0.54
6	Mid	High	1	1.2
7	High	Mid	1	1.0
8	Low	Low	1	0.03
9	High	Low	1	1.1

From the results presented in Table 3-8 a prediction equation could be calculated that relates mg of iodine lost/g of glass to time and temperature. The equation is shown below.

$$\text{mg of I}_2 \text{ lost/g of glass} = -0.63 + 0.27(\text{time}) + 0.011(\text{temp}) - 0.0076(\text{time}^2) - 0.0011(\text{time} \times \text{temp})$$

In the above equation, time is in hours and temperature is in °C.

From this equation a 3D surface plot could be drawn and is shown in Figure 3-7. Minimizing this function yields operating conditions of 1 hour and 25°C. It appeared from Figure 3-7 that the iodine loss goes down at longer reaction times. When the error was examined, however, it was seen that the confidence interval at longer times was very large. Since the variation due to temperature was very small it was possible to group the 1 hour, 5 hour and 24 hour data together to estimate the standard deviation at each time as well as calculate the 95% UCL for the standard deviation of iodine loss. Those results are shown in Table 3-9.

FIGURE 3-7. 3D PLOT OF mg OF I₂ LOST/g OF GLASS TREATED AS A FUNCTION OF TIME AND TEMPERATURE

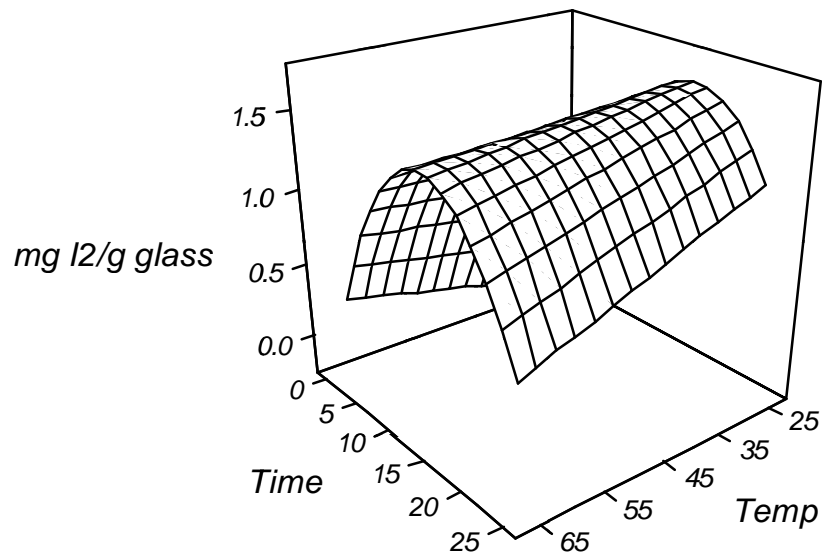


TABLE 3-9. STANDARD DEVIATION (s) AND 95% UCL FOR s FOR IODINE LOSS

Time (hours)	# Observations	s (standard dev.)	95% UCL
1	3	0.38	2.44
5	7	0.21	0.46
24	3	0.062	0.39

Using the data in Table 3-9 it was possible to generate prediction equations for both s and 95% UCL of s .

$$s = 0.094 + 0.012(\text{time})$$

$$95\% \text{ UCL} = 0.16 + 0.094 (\text{time})$$

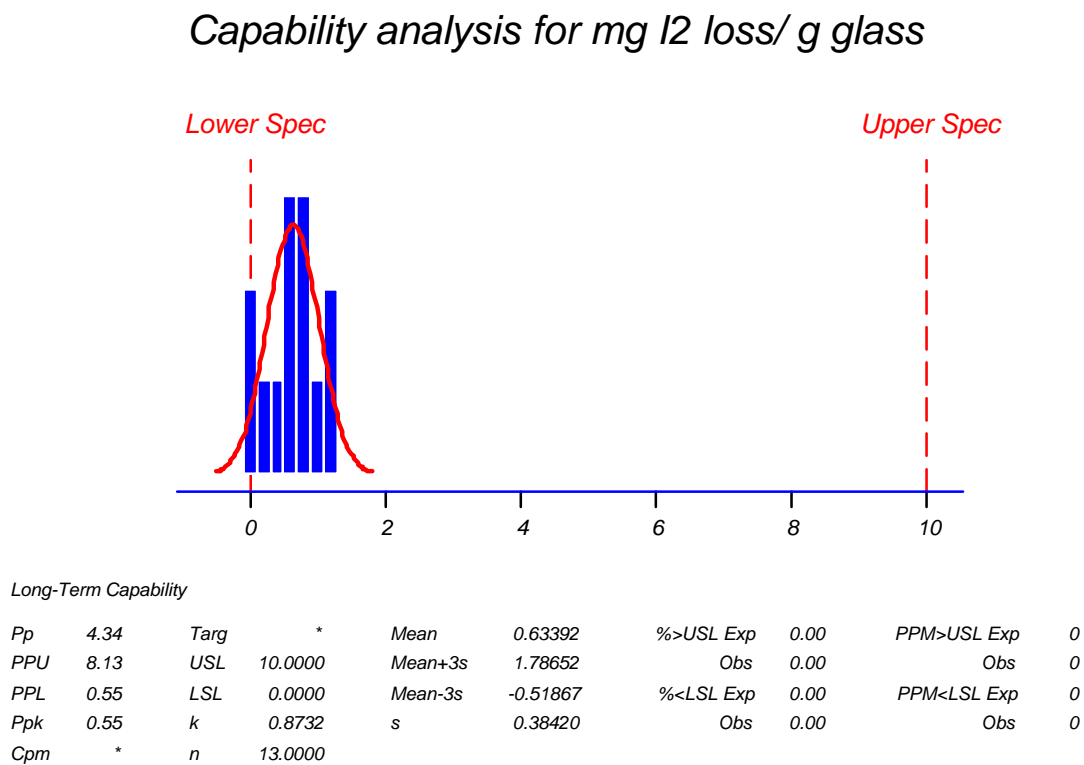
As mentioned in the previous section it is convenient to combine the average value and the error term to generate a Z value. The $Z_{95\%}$ for each time are shown in Table 3-10. Using an upper specification limit of 10 mg I_2 loss/g glass (i.e., less than 1% iodine loss), the iodine loss is very far below the upper specification, yielding very large Z values. Also, included in Table 3-10 is the cost I_2 /ton of glass treated using a unit cost of \$14/lb for iodine.

TABLE 3-10. $Z_{95\%}$ AND COST OF IODINE PER TON OF GLASS (LAMP WASTE)

Time (hours)	$Z_{95\%}$	Cost of I_2 /Ton of Glass
1	25	\$1.02
5	22	\$10.02
24	4	\$10.56

The average values for iodine loss being so far from the upper specification also means that the process capability was quite good, even when all of the data from 1, 5 and 24 hours are included (see Figure 3-8).

FIGURE 3-8. CAPABILITY ANALYSIS FOR mg OF I₂ LOSS/g GLASS TREATED, USING ALL DATA FROM THE DESIGN OF EXPERIMENTS



The Z value for this data is on the order of 24, indicating that far less than 1 batch in a million will have an iodine loss greater than 10 mg/g glass treated. Since there was an order of magnitude cost difference between running at 1 hour as opposed to 5 hours, a reaction time of 1 hour is recommended. This time should be sufficient to accomplish the extraction as presented in the above section and as confirmed by the two experiments below.

Mercury and Iodine/Iodide in Solution as a Function of Time. An extraction was run at 0.02 M I₂ at a 3/1 L/S ratio for 5 hours, with aliquots of the extraction solution removed at specific intervals and total iodine/iodide and mercury determined. The mercury results are shown in Figure 3-9. This graph indicates that the mercury was dissolved very quickly. Figure 3-10 shows that iodine loss was slow for the first couple of hours and then appeared to increase as the extraction time approached five hours.

As mentioned in the introduction to this section, these experiments were only concerned with loss of iodine with the treated solids. Throughout the whole GEMEP cycle there are other opportunities to lose iodine. Complete cycle testing was performed as described in the next section to quantify iodine losses throughout the whole GEMEP cycle.

FIGURE 3-9. DISSOLUTION OF MERCURY AS A FUNCTION OF TIME

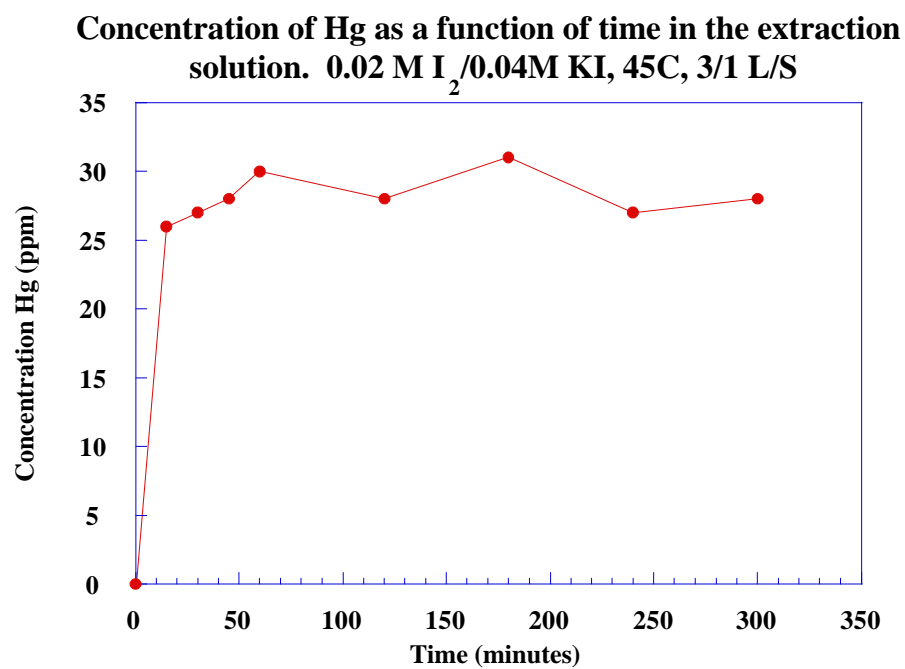
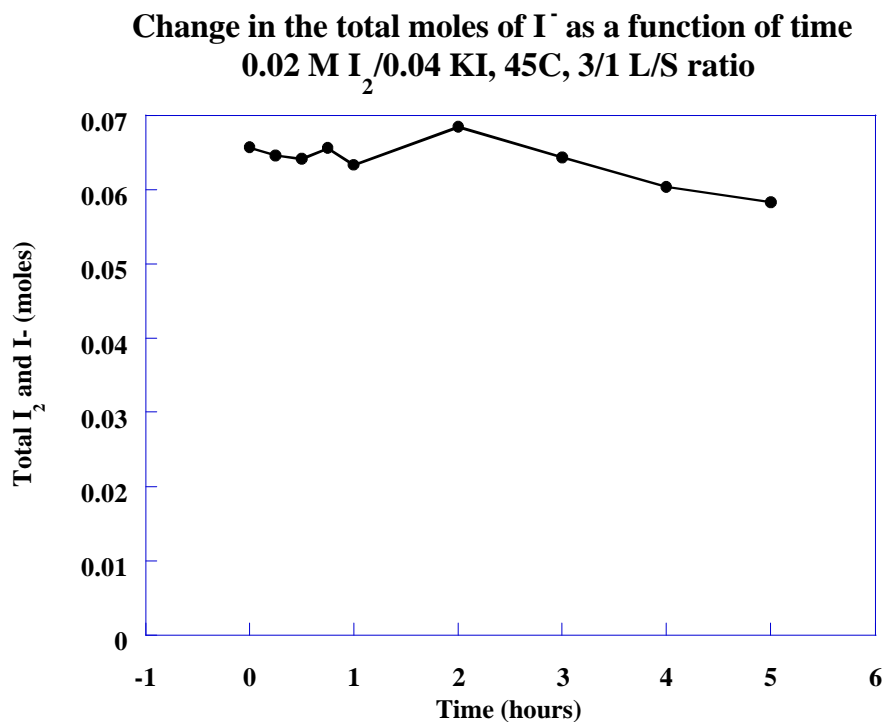


FIGURE 3-10. TOTAL MOLES OF IODINE/IODIDE IN SOLUTION AS A FUNCTION OF TIME



3.1.3 Cycle Testing of the GEMEP Process on Fluorescent Lamp Waste. Using the optimum conditions determined above, the complete GEMEP process was run through 4 complete cycles. The conditions are listed below.

0.02 M I_2 / 0.04 M KI
1 Hour extraction time
3/1 Liquid/Solid ratio
45°C Extraction temperature

The results of the cycle tests are shown in Tables 3-11 and 3-12.

TABLE 3-11. INITIAL AND FINAL CONCENTRATIONS FOR I₂, I⁻ AND Hg

Cycle	Initial [I ₂], (M)	Initial [I ⁻], (M)	Initial Hg, (mg)	Final [I ₂] ¹ , (M)	Final [I ⁻] ¹ , (M)	Final Hg, (mg)
1	0.0204	0.0397	3.1	0.014	0.0505	0.60
2	0.0207	0.0455	ND	0.0062	0.0689	0.40
3	0.0152 ²	0.0413	26.5	0.0101	0.0460	0.73
4	0.0165 ³	0.0345	52.3	0.0101	0.0455	0.75

ND = Not Determined

¹ This is the final concentration after the extraction step.

² Not enough I₂ was added due to a miscalculation (see Table 3-12 below)

³ The added I₂ had not completely dissolved

TABLE 3-12. IODINE AND WATER ADDED DURING THE GEMEP CYCLES AND THE TOTAL MASS BALANCE

Cycle	I ₂ Added	H ₂ O Added	Total Mass Balance
1	1.72 g	40 g	98.5%
2	0.30 g	20 g	98.6%
3	1.76 g	100 g	98.8%
4	--	--	98.6%

The residual mercury for each cycle was less than 1 mg, indicating that the solids would pass the TCLP test. The iodine that had to be added was higher than predicted by the work presented in Section 3.1.2. This was due to losses during the cycle that were not examined. It proved difficult to precipitate, collect and redissolve the iodine at this low concentration. This may indicate that it would be easier to work at a higher iodine concentration.

3.1.4 Effect of Sizing (Pre-Segregation) of Lamp. By dry screening the ¼" lamp fragments through a 30 mesh sieve, it was possible to separate the phosphor (and small glass pieces) from the bulk of the glass and metal pieces. Extractions were run on the minus 30 mesh fractions at two different iodine concentrations and the results are shown below in Table 3-13.

**TABLE 3-13. EXTRACTION OF -30 FRACTIONS AT 45°C, 1 HOUR
AND LIQUID/SOLID RATIO OF 5**

Concentration of I ₂ (M)	Mass of +30 Mesh Fraction (g)	Mass of -30 Mesh Fraction (g)	Residual Hg on +30 Fraction (mg)	Residual Hg on -30 Fraction After Extraction (mg)
0.02	266.1	17.8	0.34	0.85
0.20	294.8	13.5	<0.5	<0.5

While the residual mass of mercury was low for both the untreated +30 fraction and the treated -30 fraction, the concentration of mercury was quite high for the treated -30 fraction as calculated from the data in Table 3-13 (Hg concentration = 0.85 mg/0.0178 kg = 48 ppm). It is the concentration of mercury that determines what the maximum possible TCLP value could be. For these two phosphors the maximum TCLP value would be given by $48 \text{ ppm} / 20 \times 1000 = 240 \text{ ppb}$, well above the 200 ppb limit. It was therefore necessary to actually run TCLP tests on treated phosphors to determine if they would fail the test. Table 3-14 details the results of these tests on both treated and untreated phosphors.

**TABLE 3-14. TCLP RESULTS FOR TREATED AND UNTREATED PHOSPHORS
(-30 FRACTION)**

Untreated Phosphors (-30 Fraction)	Total Hg (mg)	TCLP Test Result (ppb)	Treated Phosphors (-30 Fraction)	Total Hg After Treatment (mg)	TCLP Test Result (ppb)
1	9.58	255	1	<0.02	133
2	9.50	338	2	<0.02	<23
3	25.9	357	3	<0.02	29.6
4	7.40	294	4	<0.02	31.7

The untreated phosphors all failed the TCLP test but not by a large amount, considering how much mercury was present. All the treated phosphors had very low total mercury after treatment and hence all passed the TCLP test. For one of the treated phosphors (number 2), the TCLP test result was also below the UTS limit for mercury of 0.025 mg/L (25 ppb).

3.1.5 Cycle Test for the Minus 30 Mesh Fractions. Four full GEMEP cycles were run on the minus 30 mesh fractions from lamps. In order to increase the volume of extractant that was used, phosphors from two lamps were collected and treated together. Due to the high amounts of mercury per mL of extractant and the difficulties encountered previously dealing with the recovery of iodine at low concentrations, the following cycles were run at higher iodine concentrations (0.1 M and 0.2 M I₂). The results are presented in Tables 3-15 and 3-16.

**TABLE 3-15. CYCLE EXTRACTION OF -30 FRACTION
(0.1 M I₂/0.2 M KI, 1 HOUR, 45° C, L/S ~4)**

Cycle	Mass -30 Fraction (g)	Initial [I ₂], (M)	Initial [I], (M)	Initial Hg (mg/ppm)	Final [I ₂], (M)	Final [I], (M)	Final Hg (mg/ppm)
1	21.3	0.133	0.253	63.8/3000	0.121	0.327	0.57/27
2	23.1	0.079 ¹	0.198	26.4/1140	0.067	0.153	1.4/61
3	21.6	0.121	0.244	89.6/4150	0.096	0.195	0.96/44
4	22.0	0.105	0.177	60.8/2760	ND	ND	0.69/31

¹ Not enough make-up I₂ was added.

**TABLE 3-16. CYCLE EXTRACTION OF -30 FRACTION
(0.2 M I₂/0.4 M KI, 1 HOUR, 45° C, L/S ~4)**

Cycle	Mass -30 Fraction (g)	Initial [I ₂], (M)	Initial [I], (M)	Initial Hg (mg/ppm)	Final [I ₂], (M)	Final [I], (M)	Final Hg (mg/ppm)
1	24.3	0.205	0.457	127/5230	0.183	0.450	1.7/70
2	25.1	0.174	0.309	56.4/2250	0.090	0.167	0.81/32
3	21.9	0.211	0.42	24.8/1130	0.178	0.389	0.58/26
4	23.1	0.183 ¹	0.525	25.2/1090	0.183	0.661	0.28/12

¹ Not all I₂ was dissolved when this measurement was taken.

The iodine loss was measured under similar conditions as the experiments presented in Tables 3-15 and 3-16 with the results presented in Table 3-17.

The loss of iodine was higher on a mg/g basis when treating just the -30 fraction than when treating the whole lamp, however, the loss was well below the 1% level (10 mg/g). A capability analysis was generated using the numbers in Table 3-17 and is presented in Figure 3-11.

TABLE 3-17. LOSS OF I₂ TO THE -30 FRACTION AT TWO DIFFERENT I₂ CONCENTRATIONS, OTHER CONDITIONS (1 HOUR, 45° C, L/S ~4)

Concentration of I ₂ , (M)	Mass of Phosphor (-30 Fraction), (g)	mg I ₂ Lost to Phosphor	mg I ₂ /g Phosphor
0.1	10.8	8.8	0.81
0.1	11.5	8.3	0.72
0.1	12.3	17.3	1.41
0.1	10.9	24.2	2.22
0.2	11.4	9.3	0.82
0.2	11.8	28.6	2.42
0.2	13.0	43.1	3.31
0.2	12.5	38.9	3.11

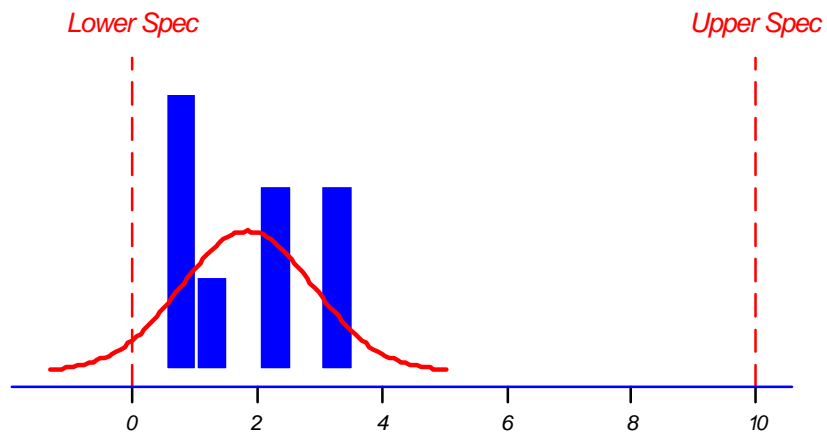
The values for iodine loss still have a Z value of greater than 6 (PPU*3 = 7.71). While the phosphors do not automatically pass the TCLP test based only on their total mercury content, the ones tested did pass.

3.1.6 Economic Analysis. Using the following assumptions an economic analysis can be done and is shown in Table 3-18.

1000 tons of lamp waste to treat
Iodine costs \$14/lb
1 ton batches

FIGURE 3-11. CAPABILITY ANALYSIS FOR I₂ LOSS FOR EXTRACTION OF THE PHOSPHORS (-30 FRACTION) ONLY

Capability analysis for I2 loss (phosphor only)



Long-Term Capability

Pp	1.58	Targ	*	Mean	1.85250	%>USL Exp	0.00	PPM>USL Exp	0
PPU	2.57	USL	10.0000	Mean+3s	5.01806	Obs	0.00	Obs	0
PPL	0.59	LSL	0.0000	Mean-3s	-1.31306	%<LSL Exp	0.00	PPM<LSL Exp	0
Ppk	0.59	k	0.6295	s	1.05519	Obs	0.00	Obs	0
Cpm	*	n	8.0000						

TABLE 3-18. COST COMPARISON

	Remove Endcaps Only [I ₂]=0.02M	Treat Only - 30 Fraction [I ₂]=0.1M	Treat Only -30 Fraction [I ₂]=0.2M
# Batches	1000	43 ¹	43
Cost for I ₂ Startup	\$0.4K	\$2.8K	\$5.7K
Cost for I ₂ Process	\$17.6K	\$1.6K	\$2.9K
Total Cost from I ₂	\$18K	\$4.4K	\$7.2K
Predicted Residual Hg (ppm)	2.2	41	35

¹ This calculation does not take into account the cost to separate the +30 and -30 fractions.

The process cost for iodine is \$1,600 per thousand tons (\$1.60/ton) for the -30 fraction at 0.1 M I₂. The current cost of disposing of lamps to a recycler is \$0.25/linear foot. One ton of lamps represents about 3,300 4-foot lamps (13,200 linear feet), or \$3,300/ton of lamps in disposal costs. This value cannot be compared to the iodine process cost directly, however, because such a comparison neglects the large capital expenditure for GEMEP process equipment, operation and maintenance costs other than for iodine, and the cost for final disposal of the treated lamps and other secondary wastes. It also does not take into account the likelihood that recycling costs would probably go down if a competing process were to become available. The purpose of the cost comparison in Table 3-18 is to compare iodine costs for various GEMEP treatment options rather than to present complete costs for GEMEP treatment. These latter costs are presented in Section 5.0.

3.2 DISSOLUTION OF MERCURY DURING PRE-SEGREGATION

The three waste streams, East Fork Poplar Creek sediment (surrogate), storm sewer sediment from Oak Ridge National Laboratory (surrogate), and fluorescent lamps were tested for mercury dissolution. Surrogate sediment samples and lamp samples were contacted with distilled water at a 3/1 liquid-solid ratio for 24 hours, to simulate pre-segregation by a wet screening method. The sample and water were then separated and the water was analyzed for mercury. The results are presented in Table 3-19.

TABLE 3-19. DISSOLUTION OF MERCURY DURING PRE-SEGREGATION STEP

Matrix	Initial Hg (ppm)	Initial HgS (ppm)	Hg in Solution (ppb)	% Hg Dissolved
EFPC Sediment	10,000		5700	0.17
EFPC Sediment	1,000		690	0.21
EFPC Sediment		1,000	<12	
ORNL Sediment	4,900		3730	0.23
ORNL Sediment	490		357	0.22
ORNL Sediment		490	<12	
Lamp			275	
Lamp			312	

Since all the solutions listed in Table 3-19 would have failed the TCLP test except those containing HgS, a metal precipitation agent (TMT-15) was added to the solutions and the experiments repeated. The results are shown in Table 3-20. Even though three of the four soil/sediment samples still failed TCLP with 1 equivalent of TMT-15, this work was stopped due to a change in the focus of the project. (The targeted waste streams changed during the course of the project.) However, it is noted that the contact time of 24 hours used for these initial

dissolution tests is extremely long compared to the likely time for pre-segregation at full scale. These results may be different if shorter contact times are used.

TABLE 3-20. DISSOLUTION OF MERCURY IN THE PRESENCE OF TMT-15

Matrix	Initial Hg (ppm)	Equivalent* TMT-15	Hg in Solution (ppb)	% Hg Dissolved
Soil	10,000	1	367	0.01
Soil	1,000	1	264	0.11
Sediment	4,900	1	261	0.07
Sediment	490	1	17	0.01
Lamp		10	62	
Lamp		100	115	

*Equivalent is based on the amount of mercury that was observed to dissolve in Table 3-18.

3.3 CONCLUSIONS FROM LABORATORY-SCALE TESTS

The GEMEP process has been optimized for the treatment of fluorescent lamps. Two sets of conditions are recommended depending on the extent of sizing of the lamps. In both cases the endcaps should be separated due to the reaction of aluminum with iodine. For whole lamps (minus endcaps and sized to $\sim \frac{1}{4}$ ") the conditions are 0.02 M I_2 /0.04 M KI, 45°C, 3/1 liquid/solid ratio and 1 hour reaction time. Under these conditions the treated solids should contain, on average, 0.6 mg of mercury and 0.63 mg of iodine. Alternatively, the crushed lamp can be divided into a +30 mesh fraction and -30 mesh fraction. The +30 mesh fraction will contain $\sim 95\%$ of the mass of the lamp and approximately 1 mg of mercury. The -30 mesh fraction should be treated using the GEMEP process at 0.1 M I_2 /0.2M KI, 45°C, 4/1 liquid/solid ratio for 1 hour. The residual solids should contain, on average, 1 mg of mercury and 1.3 mg of iodine. The residual solids will need to be tested to determine if the material passes TCLP since the concentration of mercury is high (~ 70 ppm). GE-CRD's tests indicated that the material will pass

the TCLP test. Under some conditions, Hg concentrations in TCLP extracts of the material were also below the UTS limit of 0.025 mg/L (25 ppb). Confirmation of these results at larger test scales (bench and pilot) is needed, however, because larger-scale equipment may not achieve the same efficiencies attainable at the laboratory scale. (Bench-scale work has been performed as described in Section 4.0.).

Mercury will dissolve in the pre-segregation step, except when present as HgS. TMT-15 can be used to suppress the mercury dissolution when used at 10 equivalents or 0.01% (based on the mass of solid treated), for fluorescent lamps.

SECTION 4.0

RESULTS OF BENCH-SCALE TESTS

CMRI performed bench-scale extraction, pre-segregation, and locked-cycle tests on two mixed wastes from DOE sites and on crushed fluorescent lamp glass from GE-CRD. The DOE wastes (described in Section 1.0) were East Fork Poplar Creek sediments from Oak Ridge (Waste Type ORNL) and drummed soil from INEEL (Waste Type INEEL). Objectives and methods were as described in Sections 1.0 and 2.0 respectively. The results of the tests are presented by waste type below.

4.1 EAST FORK POPLAR CREEK SEDIMENTS

4.1.1 Initial Waste Characterization

A sediment sample from East Fork Poplar Creek was received at CMRI on September 10, 1997. Visual inspection showed that the sample contained high moisture, a high percentage of fine particles, and a small amount of humus such as wood chips, grass, etc. The sediment contained 33% moisture. The bulk density of the as-received sediment was 0.82 (51 lb/ft³). The specific gravity of the dry sediment was 2.58.

A sample was wet screened using 8, 16, 30, 50, 100, and 200 mesh sieves. Screen products were filtered and dried. A split sample from each size fraction and the process water were submitted for uranium and mercury analyses. The analyses are summarized in Table 4-1 and distribution plots are shown in Figure 4-1.

TABLE 4-1. WEIGHT, MERCURY, AND URANIUM DISTRIBUTION IN EAST FORK POPLAR CREEK SEDIMENT

Fraction or Size	Fraction Weight %	Fraction Passing	Analysis U ₃ O ₈ , mg/kg	Distribution in Fraction	Cumulative Distrib.	Analysis Hg, mg/kg	Distribution in Fraction	Cumulative Distribution
					100.0			100.0
8 x 16-mesh	0.2	99.8	124	0.7	99.3	830	0.2	99.8
16 x 30-mesh	0.4	99.4	124	1.8	97.5	830	0.4	99.4
30 x 50-mesh	1.2	98.3	60	2.6	94.8	570	0.9	98.5
50 x 100-mesh	5.8	92.4	66	14.3	80.6	880	6.6	91.9
100 x 200-mesh	12.7	79.7	40	18.8	61.8	830	13.7	78.2
<200-mesh	79.7		21	61.8		760	78.2	
	Average		27			774		

The screen analysis indicated that more than 99% of the weight was finer than 30-mesh and that 80% of the weight was distributed in the minus 200 mesh fraction. Only 0.2% of the weight was humus.

The size fraction analyses indicated that 78% of the mercury (Hg) and 62% of the uranium (U₃O₈) were distributed in the minus 200 mesh fraction. Only 0.6% of the mercury and 2.5% of uranium (U₃O₈) were in the 0.6% of the weight coarser than 30 mesh.

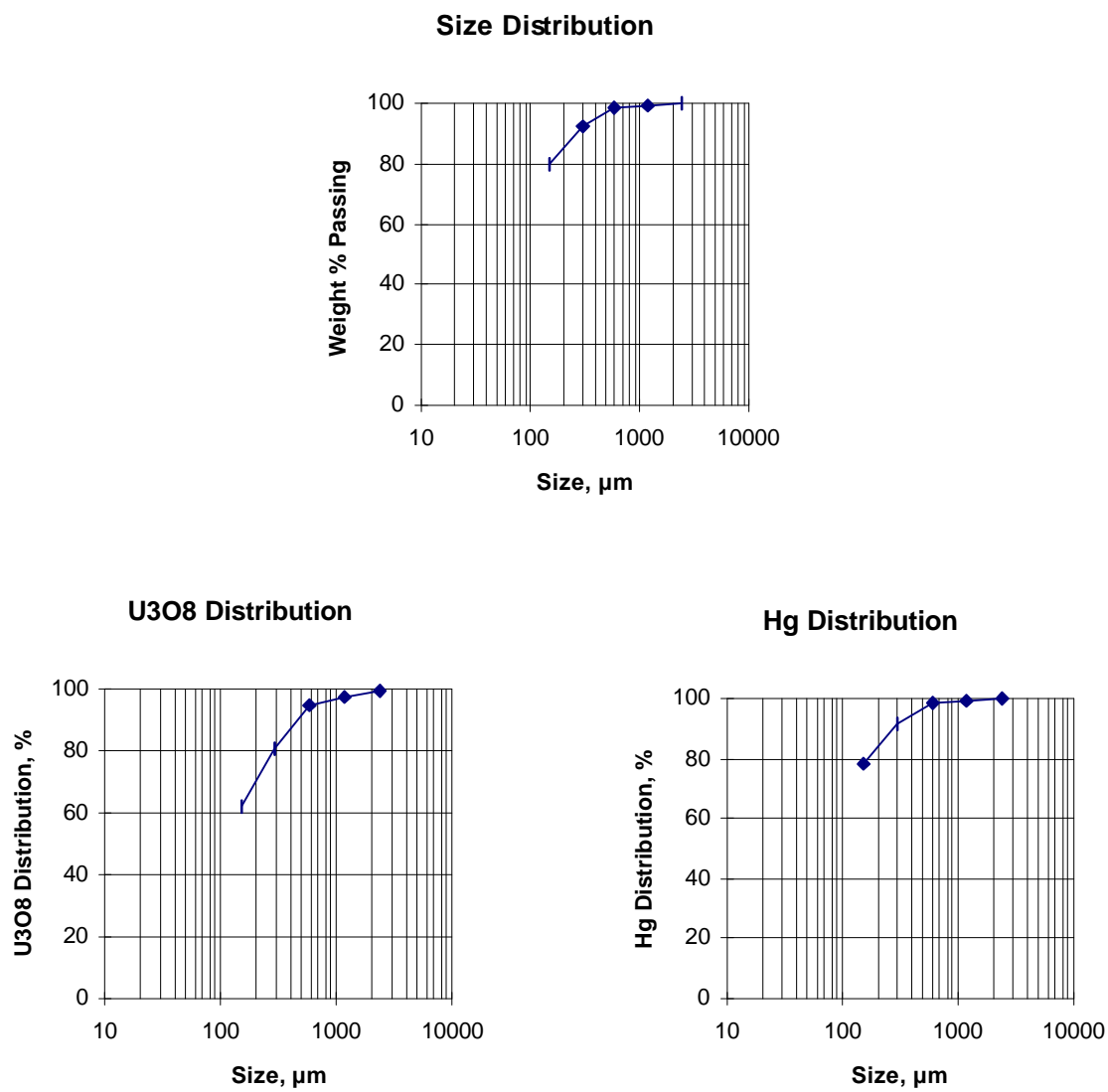


FIGURE 4-1. SIZE, MERCURY, AND URANIUM DISTRIBUTION IN EAST FORK POPLAR CREEK SEDIMENT

A 31 element X-Ray fluorescence scan was performed to determine the major, minor, and trace elements in the head sample. Table 4-2 lists the results.

TABLE 4-2. X-RAY FLUORESCENCE ANALYSIS OF EAST FORK POPLAR CREEK SEDIMENT

As Oxide	Wt, %	Element	Wt, %	Element	mg/kg	Element	mg/kg
Na ₂ O	0.16	S	0.13	V	102	Sr	134
MgO	1.43	Cl	<0.02	Cr	148	U	74
Al ₂ O ₃	15.1			Co	16	Th	57
SiO ₂	56.3			Ni	189	Nb	17
P ₂ O ₅	0.24			W	<10	Zr	624
K ₂ O	2.40			Cu	266	Rb	89
CaO	1.85			Zn	246	Y	57
TiO ₂	0.92			As	161		
MnO	0.12			Sn	122		
Fe ₂ O ₃	5.77			Pb	122		
BaO	0.08			Mo	<10		

The process water (used for wet screening) contained 0.012 ppm mercury and less than 0.001 ppm of uranium.

4.1.2 Pre-Segregation Tests

Since over 99% of the weight and mercury and 97% of the uranium are distributed in the minus 30 mesh fraction, while 63% to 80% of the weight, uranium and mercury are distributed in the minus 200 mesh fraction, gravity or other separation steps will not allow an economic separation. Therefore, the entire sediment sample was advanced for extraction tests. No pre-segregation tests were performed.

For the same reason, there was no need for information from settling tests on the minus 200 mesh fraction of the sediment, since pre-segregation is not desirable for this material. Hence, settling tests were not conducted.

4.1.3 Bench-Scale Extraction Tests

Eight potassium iodide - iodine leaching tests were performed using the East Fork Poplar Creek sediment. The eight tests evaluated the effect of the potassium iodide and iodine molarities on the solubilization of mercury and uranium from the sediment at varying pH values. The range of conditions tested was:

0.025 to 0.4M KI
0.0125 to 0.2 M I₂
pH values of 4, 5 and 7

The leaching procedure was to prepare the extraction solution containing the appropriate amounts of potassium iodide and iodine, add the sediment at a ratio of three parts liquid to one part solid (3/1 liquid-solid ratio or 25% solids), heat to 50°C with gentle mixing, measure the pH, and adjust if necessary to the target pH using 1000 g/L sulfuric acid. The leach was continued at 50°C for four hours with additional acidulation of the sediment - lixiviant slurry when necessary to maintain the target pH value. After four hours the mixture was filtered and the solids were rinsed with demineralized water. Because the observed filtration rate was quite slow, it is probable that the rinse was efficient in removing all of the soluble mercury from the solids.

Table 4-3 summarizes the results. Figure 4-2 graphically shows the effect of the potassium iodide molarity on the residual mercury.

TABLE 4-3. BENCH-SCALE LEACHING RESULTS OF EAST FORK POPLAR CREEK SEDIMENT

Test No.	KI, M	I ₂ , M	pH	U Soluble %	Residual Analyses	
					Hg, mg/kg	TCLP, mg Hg/L
A-1	0.10	0.05	4	0.13	2.7	< 0.1
A-2	0.20	0.10	4	0.13	6.6	< 0.1
A-3	0.40	0.20	5	0.06	3.1	< 0.1
A-4	0.40	0.20	7	0.06	2.8	< 0.1
A-5	0.025	0.0125	4	0.01	140	NA
A-6	0.05	0.025	4	0.01	73	NA
A-7	0.10	0.05	5	0.09	39	NA
A-8	0.10	0.05	7	0.97	42	NA

Detailed data sheets for the eight studies are included in Appendix A-1.

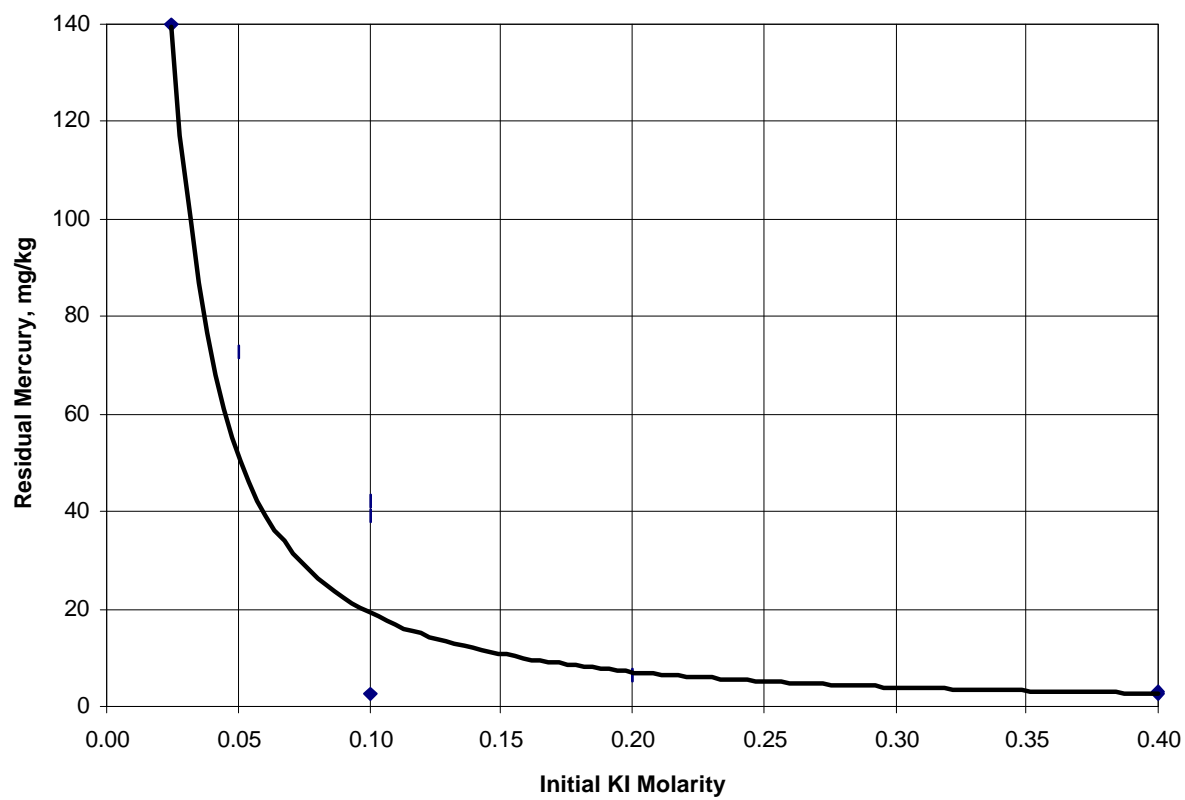
The solubilization of uranium was very low in seven of the tests. Only 0.01 to 0.13% of the initial uranium solubilized. The eighth test was a 7 pH leach at 0.1 M KI and 0.05M I₂. The solution analysis indicates that almost one percent of the contained uranium solubilized during this test. This level of uranium solubilization appears to be anomalous since another 7 pH leach solubilized only 0.06% of the uranium and other leaches at the same iodide - iodine molarities solubilized 0.09 to 0.13% of the uranium.

Figure 4-2 shows that the solubilization of the mercury was very dependent on the iodine - iodide molarities. At 0.2 M KI and 0.1 M I₂ and above (Tests A-2, A-3, and A-4), the treated sediment contained 2.8 to 6.6 mg Hg/kg. Mercury analyses of the treated solids (i.e., the residual mercury values) indicate greater than 99% solubilization of the mercury. Mercury analyses of TCLP extracts of the treated solids showed no detectable mercury, at a detection limit of 0.1 mg/L.

4.1.4 Locked-Cycle Tests

Locked-cycle tests were not performed using East Fork Poplar Creek sediment since the balance of the sediment has since been disposed by DOE. As a result, after discussion with DOE, it was decided that efforts could be more productively expended on testing of wastes that are still of concern to DOE.

FIGURE 4-2. EFFECT OF INITIAL IODINE/KI MOLARITY ON RESIDUAL MERCURY



4.2 FLUORESCENT LAMPS

4.2.1 Crushing Methods Investigation and Initial Waste Characterization

The initial evaluation of methods for crushing the fluorescent lamps included tests of both a rolls crusher and an impact mill. These tests clearly demonstrated that dry-crushing created too much hazardous dust from the phosphor. Wet-crushing using an impact mill was selected for further evaluation.

Drawings of the impact mill are shown in Figures 4-3 and 4-4. The rubber curtain, item 35, was removed and a feed chute was fitted to the mill that allowed the bulbs a straight line entry. The bulbs were inserted through the feed chute along with a spray of water. The mill's rotating blades, item 5, disintegrated the bulbs and rejected the pieces out the discharge, item 27. The collection apparatus is not shown. It includes a pan sealed tightly against the mill frame and a fabric dust collector. Note that the blades serve as an air pump and move air from the inlet to the discharge. The water spray partially atomized in the mill and wet the dust collection bag. The effect of the water spray was to prevent particulate discharges from the system. Particulate material was confined to the discharge container.

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street Golden, CO 80403	(303) 279-2581 FAX 279-0061	Project No.: 971028 Date: 2/19/98
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FIGURE 1
HAZEMAG IMPACT MILL
OVERALL VIEW

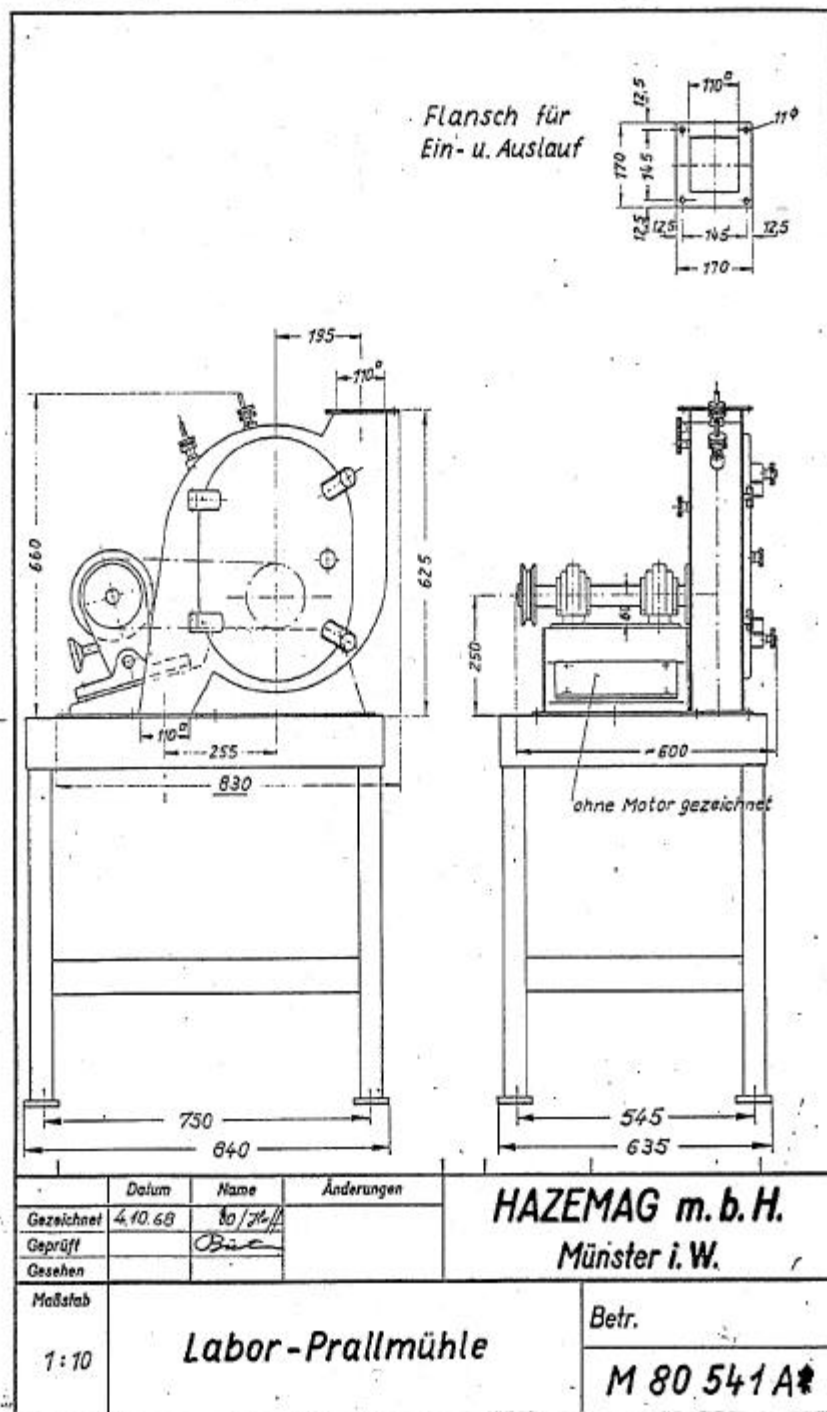


FIGURE 4-3. IMPACT MILL - OVERALL VIEW

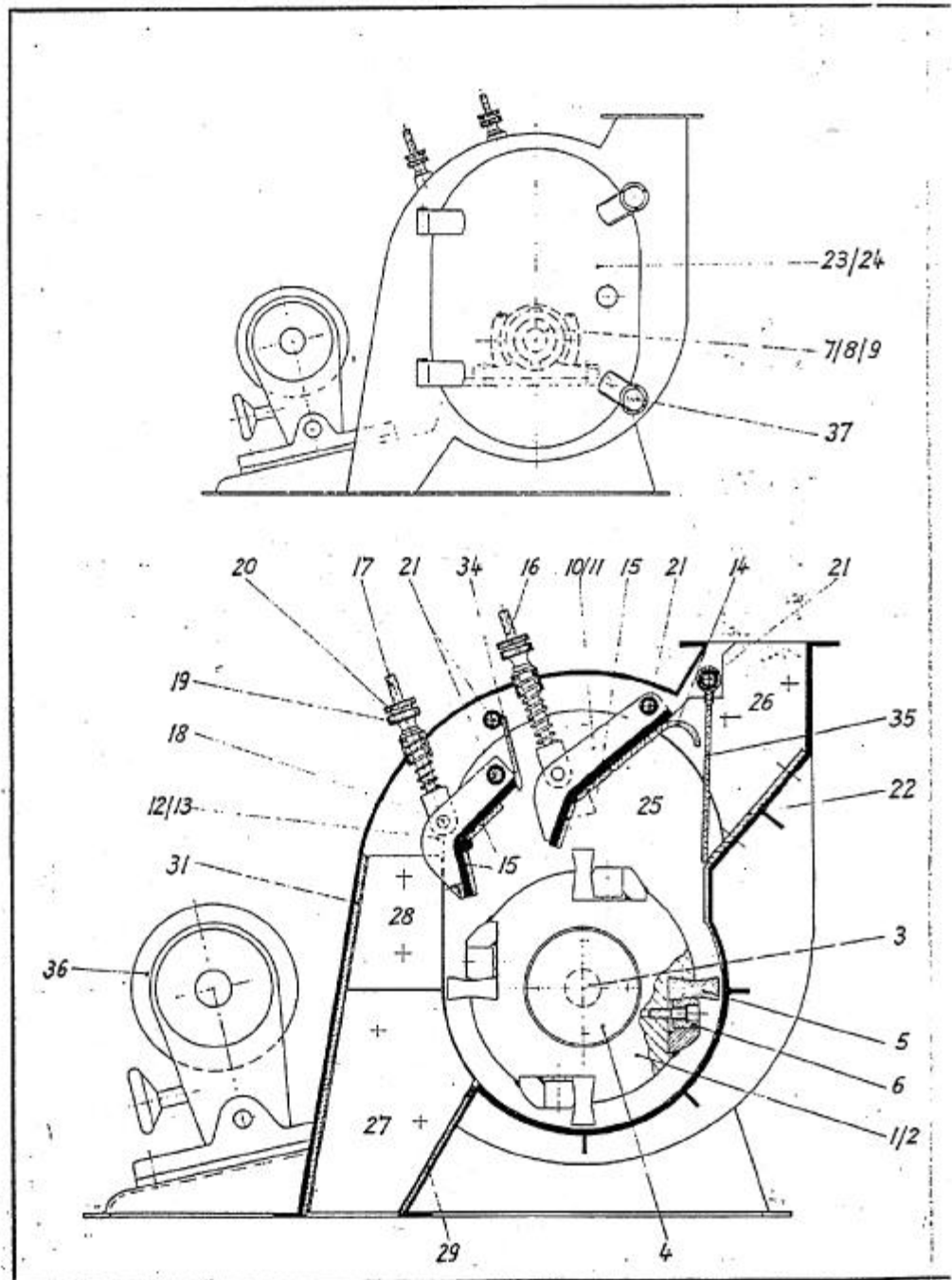


FIGURE 4-4. IMPACT MILL - CUTAWAY VIEW

4.2.2 Pre-Segregation Tests

The product from the crushing was wet screened at 3 mesh to first separate the large aluminum end caps. It then was passed over a magnetic separator to remove the pins and most of the wire (still attached to the pins). Finally, a portion of the crushed glass was again wet-screened to separate it into size fractions. Table 4-4 shows the weight and mercury distribution. The aluminum ends were 2.5% of the weight, the magnetics were 0.2% of the weight, and the glass was 97.3% of the weight.

TABLE 4-4. WEIGHT AND MERCURY DISTRIBUTION IN CRUSHED FLUORESCENT LAMPS

Size Mesh	Fraction Weight %	Weight Percent		Analysis Hg, mg/kg	Distribution in Fraction	Percent	
		Retained	Passing			Retained	Passing
Aluminum	2.5%	2.5%	97.5%	NA	0.0%	0.0%	100.0%
Magnetic	0.2%	2.7%	97.3%	NA	0.0%	0.0%	100.0%
3 x 8	13.1%	15.8%	84.2%	0.6	0.1%	0.1%	99.9%
10	13.3%	29.0%	71.0%	0.4	0.1%	0.2%	99.8%
14	15.2%	44.2%	55.8%	0.3	0.1%	0.3%	99.7%
28	23.3%	67.5%	32.5%	0.4	0.1%	0.4%	99.6%
48	13.4%	80.9%	19.1%	0.7	0.1%	0.6%	99.4%
65	4.3%	85.2%	14.8%	4.3	0.3%	0.9%	99.1%
<65	14.8%	100.0%	0.0%	425	99.1%	100.0%	0.0%

The combination of wet-milling and screening recovered 80% of the glass in fractions coarser than 48 mesh that contained less than 1 mg/kg Hg. The 48- x 65-mesh fraction was less than 5% of the total glass and contained 4.3 mg/kg Hg. The minus 65-mesh represented 15% of the glass weight and contained 425 mg/kg Hg.

The water collected from the milling and screening contained less than 100 µg Hg/L. This calculates to less than 0.6 mg of mercury per kg of initial lamp weight.

The plus 3-mesh aluminum fraction and the magnetics were not sampled and analyzed for total mercury. However, the TCLP was performed on each material. In both cases the concentration

of mercury in the TCLP extract was less than the 0.1 mg/L detection limit. The combined glass fractions coarser than 65 mesh contained an average of 0.7 mg/kg Hg, which is below the 4 mg/kg “twenty times rule” concentration below which a waste will pass TCLP for mercury, even if all the mercury present in the waste is solubilized during the TCLP extraction. Hence, subsequent leaching tests were restricted to the minus 65-mesh fraction.

4.2.3 Bench-Scale Extraction Tests

In determining appropriate conditions for bench-scale extraction of the minus 65-mesh lamp glass, the laboratory-scale results presented in Section 3.0 were reviewed to determine their applicability. Most of the laboratory-scale tests were conducted on whole lamps which were broken inside a large flask, without screening to remove large glass fragments. It was considered probable that the optimum conditions for whole lamps might not be directly applicable to the minus 65 mesh fraction. The optimum iodine concentration for whole lamps, 0.02 M iodine, was initially tried on the minus 65 mesh lamp waste, but it was quickly learned that higher iodine concentrations would be needed to effectively treat the minus 65 mesh material. Hence, a range of iodine concentrations was tested, with 0.02 M iodine as the lowest concentration. A range of times and temperatures was also investigated.

A total of seventeen leaches were performed on the minus 65-mesh material. Data sheets for the 17 studies are included in Appendix A-2. Conditions evaluated were:

0.02, 0.04 and 0.06 Molar iodine
0.04, 0.08 and 0.12 Molar potassium iodide
20, 35, and 50°C.
2, 4, 6, and 24 hours

The leaching procedure was to prepare the extraction solution containing the appropriate amounts of potassium iodide and iodine, add the crushed glass at a ratio of three parts liquid to one part

solids (3/1 liquid-solid ratio or 25% solids), and heat if desired with gentle mixing. The leach was continued at the target temperature for up to twenty-four hours. After the leach period, the mixture was filtered and the solids were rinsed with demineralized water.

The leaching results, summarized in Table 4-5, indicate that room temperature (20°C) is as efficient as elevated temperatures, the extraction time does not need to exceed four hours, and the larger of the above reagent molarities is best. The treated solids from these leaches contained as little as 7 mg/kg Hg (20°C, 4 hours, 0.04 M I₂, and 0.08 M KI).

TABLE 4-5. BENCH-SCALE LEACHING OF MINUS 65-MESH FLUORESCENT LAMP GLASS

Test No.	Time Hours	Temp	KI, M Initial	I ₂ , M		Residue Analysis	
				Initial	Final	Hg, mg/kg	TCLP, mg/L Hg
G-1	2	15	0.04	0.02	0.0058	19.8	NA
G-2	6	16	0.04	0.02	0.0030	12.6	NA
G-3	24	17	0.04	0.02	0.0005	14.3	NA
G-4	2	36	0.04	0.02	0.0014	14.4	NA
G-5	6	35	0.04	0.02	0.0002	22.4	NA
G-6	6	35	0.04	0.02	0.0002	27.4	NA
G-7	2	52	0.04	0.02	0.0001	25.6	NA
G-8	6	52	0.04	0.02	0.0000	47.6	NA
G-9	2	20	0.08	0.04	0.0125	8.5	< 0.1
G-10	4	20	0.08	0.04	0.0106	6.7	< 0.1
G-11	6	20	0.08	0.04	0.0094	7.3	< 0.1
G-12	2	34	0.08	0.04	0.0087	8.4	< 0.1
G-13	4	34	0.08	0.04	0.0061	9.2	< 0.1
G-14	2	20	0.12	0.06	NA	10.8	< 0.1
G-15	4	20	0.12	0.06	NA	8.5	< 0.1
G-16	2	20	0.08	0.06	NA	12.8	< 0.1
G-17	4	20	0.08	0.06	NA	11.4	< 0.1

Two tests were performed with 0.08 M potassium iodide and 0.06 M iodine. The amount of potassium iodide was not sufficient to allow all of the iodine to dissolve. Two tests were performed with 0.12 M potassium iodide and 0.06 M iodine. No improvement was observed in the mercury dissolution.

Nine of the treated solids samples for which the mercury concentration was less than 15 mg/kg were also tested using the TCLP test. For all eight samples, no mercury was detected in the TCLP extracts (i.e., the mercury concentrations were < 0.1 mg/L).

Figure 4-5 shows that the residual mercury concentration showed good correlation with the final iodine concentration. When the final iodine concentration was greater than 0.06 molar, the residual mercury was less than 10 mg/kg.

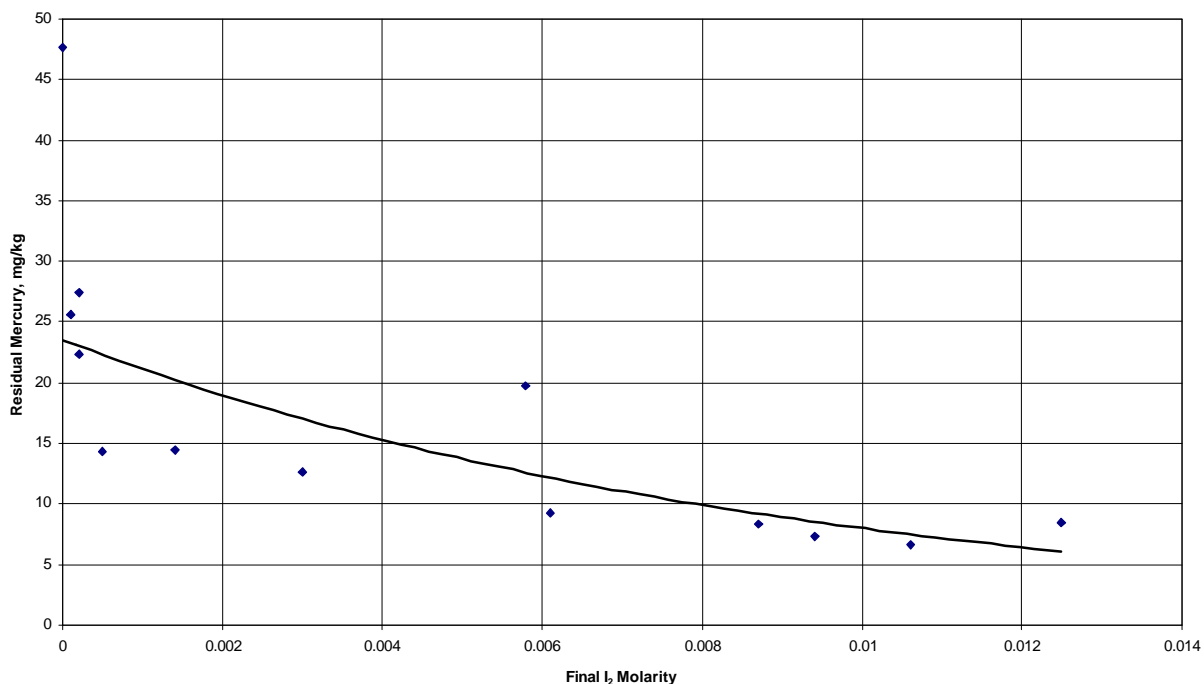


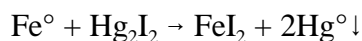
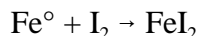
FIGURE 4-5. EFFECT OF FINAL IODINE CONCENTRATION ON RESIDUAL MERCURY

4.2.4 Locked Cycle Tests

Locked cycle leaching studies on the minus 65-mesh lamp glass were initiated. The flowsheet for the locked cycle leaching is shown in Figure 4-6.

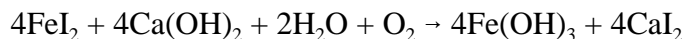
The conditions used were:

1. Four-hundred gram (dry) aliquots of the minus 65-mesh lamp glass were leached at the following conditions:
 - 0.04 Molar iodine
 - 0.08 Molar potassium iodide
 - 25% solids (3 liquid to 1 solid)
 - 6.5-7.5 pH, adjusted with sulfuric acid
 - $\pm 20^{\circ}\text{C}$
 - 4 hours
2. The leached slurry was filtered and washed with three to four liquid displacements of water for the first cycle, filtrate from iodine regeneration in the later cycles.
3. The filtrate was adjusted to approximately 4 pH and passed upflow through a column containing 500 grams of iron turnings. The volume of iron turnings in the column was approximately 680 mL. The leach flow was 20 mL per minute giving approximately 30 minutes retention. The iron turnings reduce any iodine present to iodide, ferrous iodide, and also precipitate the mercury by displacement:



A small quantity of suspended solids reported to the iron column discharge. These solids apparently were due to slight oxidation of the iron when the column was not being used, and would be minimized in continuous operation.

4. The discharge solution from the iron column then was adjusted to 10 pH using hydrated lime to precipitate the soluble iron and form soluble calcium iodide.



The iron precipitation also served to scavenge any mercury remaining after the cementation column.

LOCKED CYCLE GEMEP PROCESSING OF CRUSHED LAMP GLASS

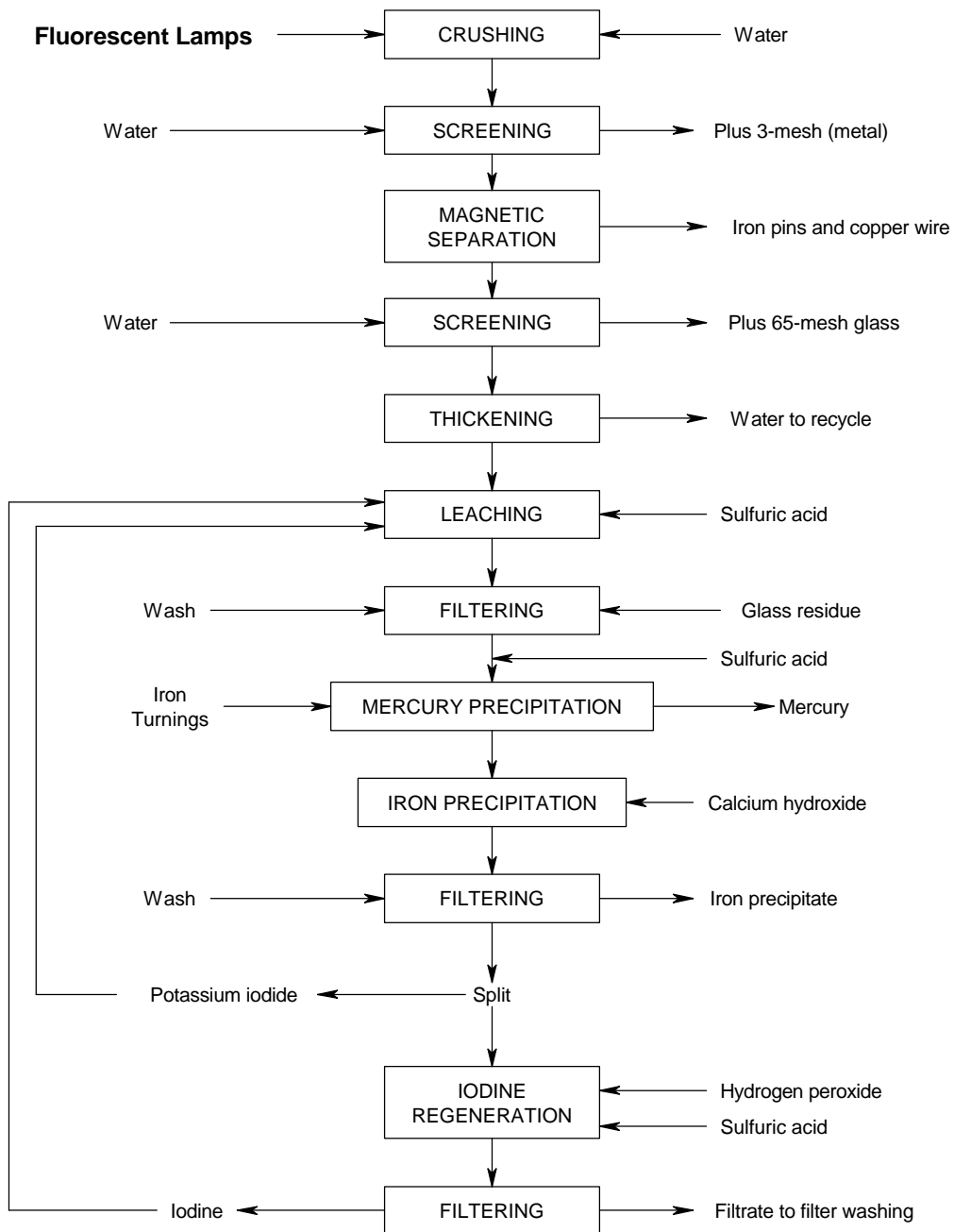
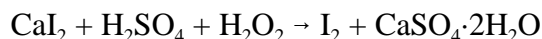


FIGURE 4-6. FLOWSHEET FOR LOCKED-CYCLE LEACHING OF CRUSHED LAMP GLASS

5. A portion of the filtrate from the iron precipitation is advanced to provide the potassium iodide for the next leaching cycle.
6. The balance of the iron precipitation filtrate was oxidized using sulfuric acid and hydrogen peroxide to generate iodine for advance to the next leaching cycle. The oxidation reaction is shown below:



The sulfuric acid and hydrogen peroxide additions were 140% and 120% of stoichiometric for the above reaction.

Leaching results for the locked-cycle tests are summarized in Table 4-6. Data sheets are presented in Appendix A-3.

TABLE 4-6. LOCKED-CYCLE RESULTS FROM LEACHING CRUSHED LAMP GLASS

<u>Cycle</u>	<u>Residual Mercury, mg/kg</u>	<u>Percent Solubilized</u>
1	7.8	98.3
2	9.0	96.6
3	8.5	97.9
4	8.8	98.1
5	9.7	97.8
6	11.8	97.6
7	11.8	97.5
8	15.8	96.4
9	15.2	96.5
10	12.8	97.0

The slightly higher residue values in the last five cycles probably are due to leaching at a slightly lower ambient temperature. The studies were changed to a different location after the fifth cycle. The new location had a considerably lower ambient temperature (15-16°C) than the laboratory in which the initial five cycles were performed (18-21°C). This indicates that the temperature should not be less than 20°C, and that probably approximately 25°C should be the minimum.

Five of the residues were evaluated by the TCLP. The results are summarized in Table 4-7.

TABLE 4-7. TCLP VALUES FOR LOCKED-CYCLE LEACHED CRUSHED FLUORESCENT LAMP GLASS

<u>Cycle</u>	<u>Residual Mercury, mg/kg</u>	<u>TCLP Mercury, mg/L</u>
2	9.0	0.02
4	8.8	0.02
6	11.8	0.04
8	15.8	0.02
10	12.8	0.01

All TCLP mercury results were below the TCLP limit of 0.2 mg/L (200 ppb). All results but one were below the Universal Treatment Standard of 0.025 mg/L (25 ppb) that will go into effect for mixed wastes on May 26, 2000.

The mercury containing leach solution was adjusted to 4.0 ± 0.5 pH using dilute sulfuric acid prior to passing the solution through the column of iron turnings. The adjustment required an average of 0.5 grams of 100% sulfuric acid/batch (0.25 lb H₂SO₄/ton whole lamps). The mercury cementation (precipitation) results are presented in Table 4-8.

TABLE 4-8. EFFICIENCY OF MERCURY PRECIPITATION BY IRON TURNINGS

<u>Cycle</u>	<u>Leach Solution Hg, mg/kg</u>	<u>Discharge Solution Hg, mg/kg</u>
1	130	2.3
2	48	NA
3	100	<0.1
4	76	<0.1
5	100	<0.1
6	130	3.5
7	120	3.1
8	100	1.9
9	105	4.0
10	105	4.7

The lower efficiency in the precipitation during the last five tests probably is due to the lower temperature in the new location.

The effluent from the mercury precipitation was adjusted to between 10 and 11 pH by the addition of calcium hydroxide. The hydrated lime addition averaged 2.7 grams per batch (1.33 lb/ton whole lamps). This adjustment precipitated the soluble iron and any residual mercury.

Table 4-9 summarizes the precipitation data.

TABLE 4-9. DATA FROM IRON PRECIPITATION

Cycle	Precipitate		Filtrate	Removal
	Grams	Hg, mg/kg	Hg, mg/kg	Efficiency, %
1	2.3	855	<0.1	>99.9
2	5.8	405	<0.1	>99.8
3	4.2	745	<0.1	>99.9
4	6.6	1125	<0.1	>99.9
5	4.0	1430	<0.1	>99.9
6	2.6	1105	0.4	99.7
7	5.2	815	0.4	99.7
8	3.5	520	<0.1	>99.9
9	5.2	820	0.4	99.6
10	5.8	915	0.2	99.8

The efficiency of mercury removal from the combined cementation and iron precipitation steps exceeded 99.9% in six of the ten cycles.

The potassium iodide content of the iron precipitation filtrate was determined and a portion of the filtrate that contained either sufficient potassium iodide or liquid volume for the next leach cycle was set aside. The balance of the solution, if any, was advanced to iodine regeneration. A total of eight iodine regenerations were performed. The efficiency of the regeneration was proportional to the quantity of iodine. With only 3.6 grams iodine available the regeneration efficiency was only 33%; whereas when more than 10 grams iodine was available the regeneration

efficiency was greater than 70%.

After the second cycle of leaching the average make-up iodine requirement was 5.6 grams per leach (2.8 lb I₂/ton whole lamps).

4.3 INEEL SLUDGE

4.3.1 Initial Waste Characterization

Three, 35-gallon drums containing soil/sludge from INEEL were received at CMRI on April 15, 1998. These drums were opened and grab samples were taken from the surface of each drum. Observation of the surface indicated that the contents of the three drums were similar. Mercury analyses of the grab samples are shown below.

Drum 558	34 mg/kg
Drum 708	735 mg/kg
Drum 710	645 mg/kg

Drum 708 was emptied, the contents blended, and split into about twelve sub-samples. The INEEL material contained less than 3% moisture. The loose bulk density of the material was 1.23 (80 lb/ft³). The packed bulk density (as received) of the material was 1.57 g/cm³ (103 lb/ft³). The specific gravity of the dry material was 2.61.

A split of the Drum 708 material was submitted for X-ray Fluorescence analysis. This analysis is listed in Table 4-10.

TABLE 4-10. X-RAY FLUORESCENCE ANALYSIS OF INEEL SOIL/SLUDGE

As Oxide	Wt, %	Element	Wt, %	Element	mg/kg	Element	mg/kg
Na ₂ O	0.53	S	0.07	V	126	Sr	261
MgO	3.43	Cl	<0.02	Cr	145	U	34
Al ₂ O ₃	10.8			Co	18	Th	34
SiO ₂	49.4			Ni	69	Nb	12
P ₂ O ₅	0.28			W	13	Zr	160
K ₂ O	2.46			Cu	63	Rb	97
CaO	15.2			Zn	246	Y	48
TiO ₂	0.45			As	154		
MnO	0.07			Sn	70		
Fe ₂ O ₃	9.29			Pb	76		
BaO	0.08			Mo	<10		

The bench-scale studies were conducted on this drum of material. One of the sub-samples was pulped with water in a cement mixer for 30 minutes and wet screened on sieves from 4- to 100-mesh. The size distribution in the minus 100-mesh was determined using a Coulter Laser Particle size analyzer. Portions of each fraction from the wet screening were submitted for mercury analysis.

The plus 4-mesh consisted of three distinctly different materials: rock, asphalt, and a light colored tar/polymer. These three materials were separated by hand sorting and analyzed separately.

Table 4-11 lists the data from the characterization (weights and mercury analyses) and Figure 4-7 plots the weight and mercury distributions. The three plus 4-mesh materials all passed the TCLP limit for mercury of 0.2 mg/L (200 ppb):

<u>Material</u>	<u>TCLP Mercury Result</u>
Tar	0.06 mg/L
Asphalt	0.08 mg/L
Rock	0.06 mg Hg/L

TABLE 4-11. SIZE AND MERCURY DISTRIBUTION IN INEEL SOIL/SLUDGE

Size Mesh, μm	Fraction Weight %	Weight Percent	
		Retained	Passing
4	9.3%	9.3%	90.7%
8	2.9%	12.2%	87.8%
14	1.8%	14.0%	86.0%
28	1.4%	15.5%	84.5%
48	1.8%	17.2%	82.8%
100	4.4%	21.6%	78.4%
140	4.3%	26.0%	74.0%
200	3.5%	29.5%	70.5%
270	3.3%	32.8%	67.2%
400	2.9%	35.7%	64.3%
20 μm	3.9%	39.6%	60.4%
10 μm	7.1%	46.6%	53.4%
3 μm	20.4%	67.0%	33.0%
<3 μm	33.0%	100.0%	0.0%

Size Mesh	Fraction Weight %	Weight Percent		Mercury Analysis mg/kg	Distribution in Fraction	Percent	
		Retained	Passing			Retained	Passing
Tar	1.3%	1.3%	98.7%	19.2	0.05%	0.1%	99.9%
Asphalt	0.9%	2.2%	97.8%	18.0	0.03%	0.1%	99.9%
Rock	7.2%	9.3%	90.7%	59.4	0.9%	1.0%	99.0%
8	2.9%	12.2%	87.8%	170	1.1%	2.1%	97.9%
14	1.8%	14.0%	86.0%	275	1.1%	3.1%	96.9%
28	1.4%	15.5%	84.5%	490	1.5%	4.6%	95.4%
48	1.8%	17.2%	82.8%	895	3.5%	8.1%	91.9%
100	4.4%	21.6%	78.4%	870	8.2%	16.3%	83.7%
<100	78.4%	100.0%	0.0%	495	83.7%	100.0%	0.0%

The INEEL material is very fine, with 78% passing 100 mesh, 64% passing 400-mesh, and 33% finer than 3 micrometers. The mercury content increases with decreasing particle size, although the minus 100-mesh contains slightly less mercury than the 28- x 100-mesh fractions (mercury content of 495 mg/kg versus 570-895 mg/kg). A trace of humates was recovered during the set screening, about 0.03% of the weight. The humates contained 11,000 mg Hg/kg indicating that the mercury was or had been quite mobile.

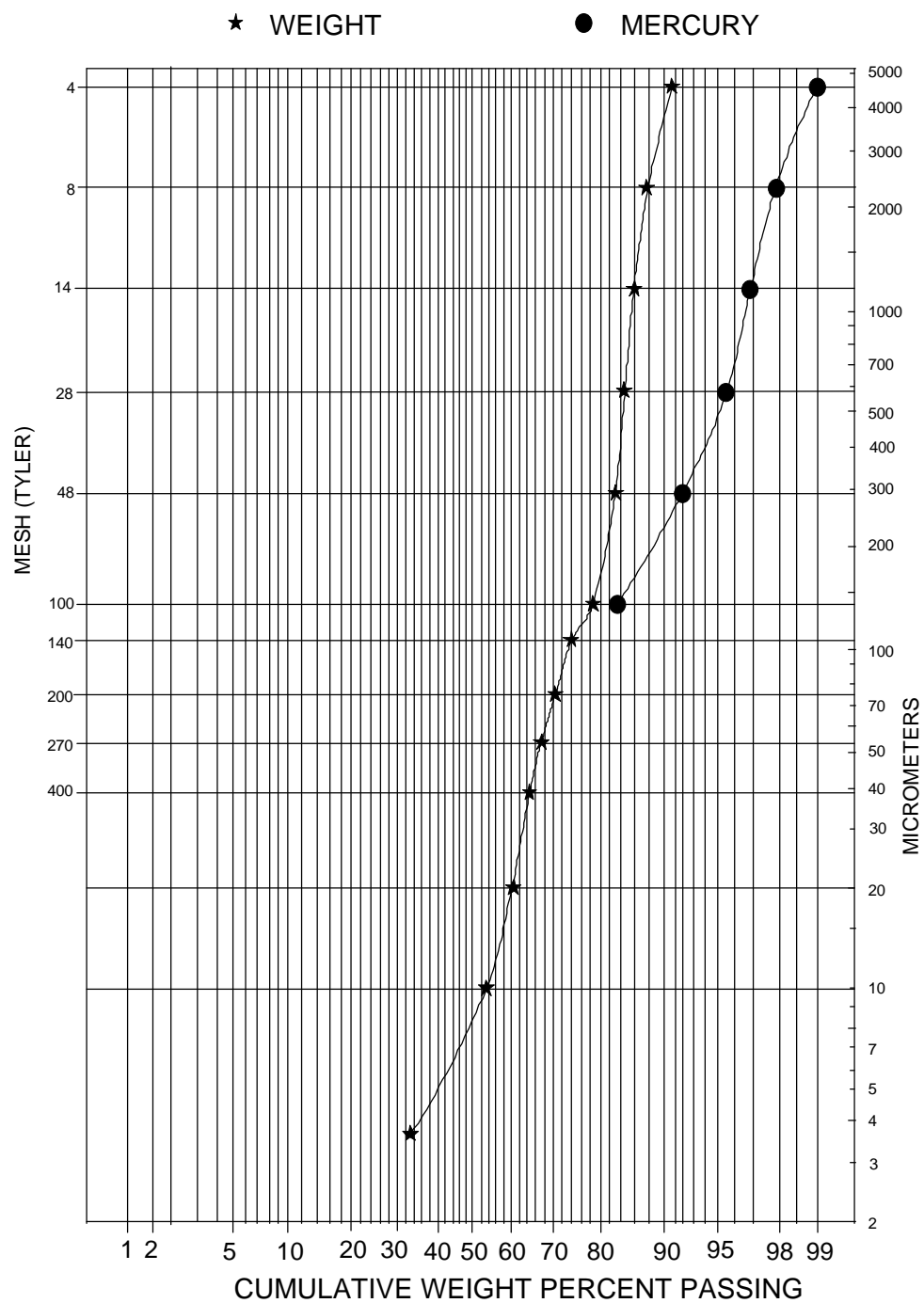


FIGURE 4-7. SIZE AND MERCURY DISTRIBUTION IN INEEL SLUDGE

The Promesh plot in Figure 4-7 indicates a near normal size distribution for the material. A perfect normal distribution plots as a straight line.

Viscosity measurements were made on slurries of the minus 4-mesh material when pulped at 10 to 50 percent solids. The viscosities measured are listed in Table 4-12.

TABLE 4-12. VISCOSITY OF INEEL SLUDGE AT PULP DENSITIES

<u>Slurry Percent Solids</u>	<u>Viscosity Centipoise</u>
10	2.4
20	3.8
30	4.5
40	14
50	55

The high viscosity at 50% solids indicates that leaching should be done at no higher than a 40% solids density to enable effective mixing and pumping of the slurry.

4.3.2 Pre-Segregation Tests

Feed for subsequent bench-scale and locked cycle leaching studies was prepared by first screening dry on 4 mesh. This step removed more than one-half the weight as minus 4-mesh material and reduced the viscosity during the de-agglomeration. The plus 4-mesh then was de-agglomerated in a cement mixer and screened on 4 mesh. The oversize, rocks-asphalt-tar, was washed and set aside.

A settling test was performed on the minus 4-mesh material from the de-agglomeration and screening. Beaker tests were used to determine that a slightly anionic polymer showed the best flocculation; however the dosage was large. A one-liter scale test then was performed. The initial slurry was 14% solids and a dose of 0.57 lb Percol 351 was added per ton of dry solids. Percol 351 is a very high molecular weight very slightly anionic polymer. Most vendors can supply a

similar polymer.

The terminal slurry density was 44.5% solids. The initial free settling rate of the slurry was six feet per hour. The calculated unit area to achieve 44.5% solids was 2.7 square feet per ton per day. The settling test data sheet is shown in Figure 4-8.

4.3.3 Bench-Scale Extraction Tests

A total of seventeen batch leaching tests were performed. The first thirteen evaluated the effect of iodine concentration, temperature, and acidulation on the dissolution of the mercury. Data sheets for the bench-scale extractions are presented in Appendix A-4.

The leaching procedure was to prepare the extraction solution containing the appropriate amounts of potassium iodide and iodine, add the minus 4-mesh material at a ratio of three parts liquid to one part solids (3/1 liquid-solid ratio or 25% solids), and heat if desired with gentle mixing. The leach was continued at the target temperature for four hours. After the leach period, the mixture was filtered and the solids were rinsed with demineralized water.

Because of the potential for some soluble mercury to be retained in the solids, four two-stage tests were performed. In the two-stage tests, the residual is repulped in iodine solution and re-filtered and washed. This allows for more efficient removal of the soluble mercury. The results from the seventeen tests are listed in Table 4-13.

The soil is quite alkaline; any adjustment of the natural pH requires a large addition of acid. The three tests with pH adjustment, I-4, I-5, and I-6, required sulfuric acid additions of 400-500 lb per ton of soil just to reduce the pH about one unit. For this material, pH adjustment would not be practical at full scale. Also, the adjustment of pH provided relatively little benefit with respect to mercury concentrations in the residual.

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street Golden, CO 80403	SETTLING TEST DATA AND CALCULATION SHEET	Project No.: 961026-06
		Date: 5/27/98

Modified Kynch Procedure

Test No: INEEL-1

Feed Pulp Large leach batch Feed Percent Solids 13.9%
Density, g/L 1074

Flocculant
Type Percol 351 45 mL, 1 g/L 0.57 Dose, lb/st

Free Settling Rate, ft/hr 6.1 Terminal Percent Solids 44.5%
Density, g/L 1284

Settling Rate	
Time min	Level mL
0	1050
1	967
2	925
3	855
4	765
5	665
6	585
7	525
8	480
9	440
10	410
14	350
20	315
24	300
30	290
38	290
55	280
1000	275

	Feed Pulp	Terminal Pulp	Clear Liquor
Volume, mL	1050	275	775
Gross pulp wt., g	1810	1052	
Tare, g	682	682	
Net pulp wt., g	1128	353	775
Net dry wt., g	157		
Density, g/L	1074	1284	1000
Solids, %	14	44	

Rake installed at 3 min

Rake rotation 10 min/rev

Thickener Unit Area Calculation

Initial Height, Ho 1.25 ft

Initial pulp density, Co (g solids/L,)

Co = 149.52 = 0.0047 st/ft³
Terminal Density 44.5% solids
Critical time, Tx 0.016 days
Unit Area 2.73 ft²/t/day

FIGURE 4-8. SETTLING OF INEEL MINUS 4-MESH SOIL

TABLE 4-13. BENCH-SCALE LEACHING OF INEEL SOIL/SLUDGE

Test No.	I ₂ , M	Temp, °C	Final pH	Residual Hg, mg/kg	TCLP, mg Hg/L
I-1	0.05	25	7.2	60	
I-2	0.1	25	7.2	64	0.02
I-3	0.2	25	7.2	53	
I-4	0.05	45	5.7	31	0.02
I-5	0.1	45	5.6	23	
I-6	0.2	45	5.6	17	0.04
I-7	0.05	45	6.8	35	
I-8	0.1	45	6.7	34	0.02
I-9	0.2	45	6.7	26	
I-10	0.1	45	6.7	49	0.01
I-11	0.15	45	6.6	48	
I-12	0.1	35	6.9	59	
I-13	0.15	35	6.6	47	
I-14, 2	0.1/0.1	45	6.6	37	0.08
I-15, 2	0.1/0.05	45	6.7	47	0.06
I-16, 2	0.1/0.1	55	6.7	34	0.06
I-17, 2	0.1/0.05	55	6.7	29	0.08

The effect of iodine molarity, pH, and leaching temperature on the residual mercury is shown in Figure 4-9. Elevated temperature improves the solubilization of the mercury.

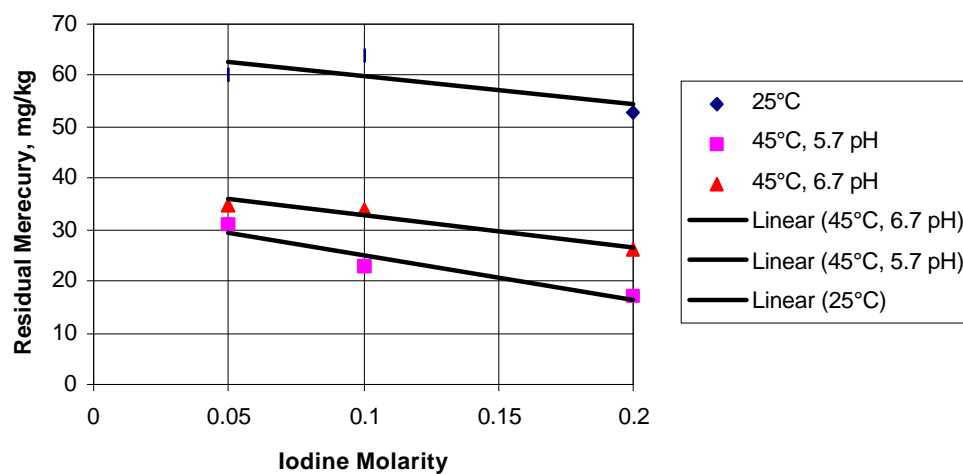
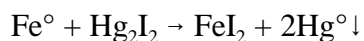
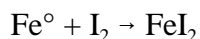


FIGURE 4-9. EFFECT OF IODINE CONCENTRATION, TEMPERATURE, AND pH ON RESIDUAL MERCURY

4.3.4 Locked Cycle Tests

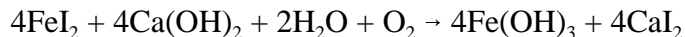
Locked cycle leaching studies on the de-agglomerated minus 4-mesh INEEL material were initiated. The flowsheet for the locked cycle leaching is shown in Figure 4-10. The tests were conducted as follows:

1. One kilogram (dry) aliquots of the minus 4-mesh material, 800 mg/kg mercury, were leached at the following conditions:
 - 0.10 Molar iodine
 - 0.20 Molar potassium iodide
 - 30% solids (2.3 liquid to 1 solid)
 - Natural pH
 - Recycle solutions adjusted 6.5-7.5 pH with sulfuric acid
 - $\pm 60^{\circ}\text{C}$
 - 2 hours in each stage
2. The leached slurry was filtered and washed with three to four liquid displacements, using water for the first two cycles and filtrate from iodine regeneration in the later cycles.
3. The filtrate was adjusted to approximately 4 pH and passed upflow through two columns in series containing a total of 1760 grams iron turnings. The void space in the columns of iron turnings was approximately 1300 mL. The leach flow was 30-35 ml per minute giving approximately 40 minutes retention. The iron turnings reduce any iodine present to iodide (ferrous iodide), and also precipitate the mercury by displacement:



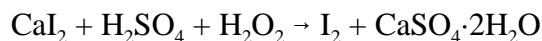
When a significant amount of suspended solids was observed in the cementation effluent, polish filtering of the leach filtrate was performed prior to feeding the leach solution to the iron columns.

4. The discharge solution from the iron columns then was adjusted to 10 pH using hydrated lime to precipitate the soluble iron and form soluble calcium iodide.



The iron precipitation also served to scavenge any mercury remaining after the iron columns.

5. A portion of the filtrate from the iron precipitation is advanced to provide the potassium iodide for the next leaching cycle.
6. The balance of the iron precipitation filtrate was oxidized using sulfuric acid and hydrogen peroxide to generate iodine for advance to the next leaching cycle. The oxidation reaction is shown below:



The sulfuric acid and hydrogen peroxide additions were 140% and 120% of stoichiometric for the above reaction.

Leaching results for the locked cycle tests are summarized in Table 4-14. Data sheets are presented in Appendix A-5.

TABLE 4-14. LOCKED CYCLE LEACHING OF INEEL SOIL

Cycle	Residual Mercury, mg/kg	Percent Solubilized
1	250	73
2	115	87
3	155	81
4	295	62
5	210	75
6	105	89
7	28	97
8	47	95
9	50	94
10	69	93
11	28	97
12	45	94

The higher residual mercury values in the first six cycles were due to inadequate iodine concentrations during leaching. This was caused by inadequate communication to new technicians. The last two tests, Cycles 11 and 12, were monitored very carefully. Figure 4-11 illustrates the effect of the second-stage iodine concentration on the residual mercury.

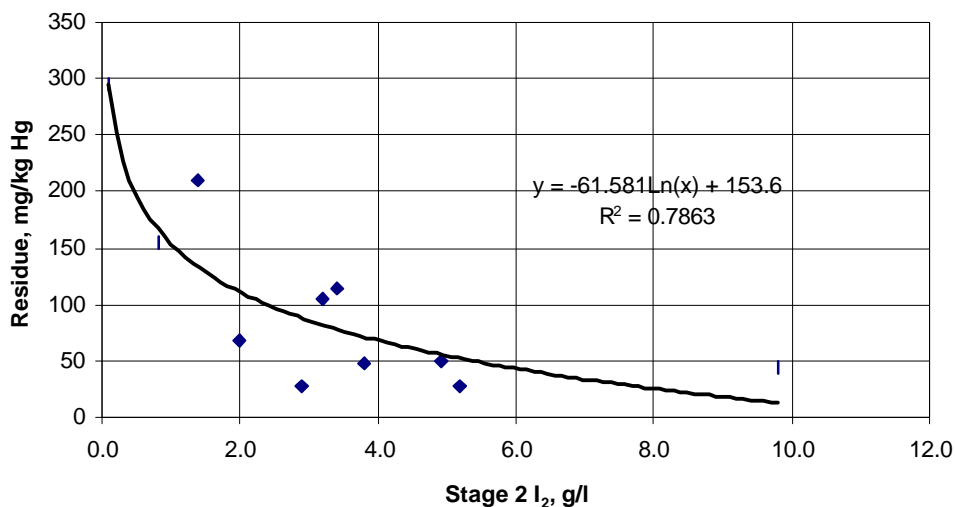


FIGURE 4-11. EFFECT OF SECOND STAGE IODINE CONCENTRATION ON RESIDUAL MERCURY

All residues were evaluated by the TCLP. The TCLP results are summarized in Table 4-15.

All the TCLP mercury results were below the TCLP limit of 0.2 mg/L (200 ppb). However, all but one result exceeded the Universal Treatment Standard of 0.025 mg/L (25 ppb) that will go into effect for mixed wastes on May 26, 2000.

TABLE 4-15. TCLP RESULTS OF INEEL LOCKED-CYCLE RESIDUES

<u>Cycle</u>	<u>Residual Mercury, mg/kg</u>	<u>TCLP Mercury, mg/L</u>
1	250	0.04
2	115	0.04
3	155	0.04
4	295	0.04
5	210	0.06
6	105	0.06
7	28	0.04
8	47	0.04
9	50	0.06
10	69	0.18

The mercury containing leach solution was adjusted to 4.0 ± 0.5 pH using dilute sulfuric acid prior to passing the solution through the columns of iron turnings. The adjustment required an average of 6 grams of 100% sulfuric acid/batch (12 lb H₂SO₄/ton material). The mercury cementation results are tabulated in Table 4-16.

TABLE 4-16. CEMENTATION OF MERCURY FROM INEEL LEACH SOLUTION USING IRON COLUMNS

<u>Cycle</u>	<u>Leach Solution Hg, mg/kg</u>	<u>Discharge Solution Hg, mg/kg</u>
1	405	5
2	430	9
3	405	12
4	405	26
5	385	45
6	375	64
7	385	22
8	415	5
9	410	7.4
10	420	7.3

The decreasing efficiency in the cementation through cycle 6 was due to deposition of leach residue solids in the columns. The columns were cleaned during the seventh cycle, but the

efficiency still was not as good as the tests with the lamp glass.

The effluent from the iron columns was adjusted to between 10 and 11 pH by the addition of calcium hydroxide. The hydrated lime addition averaged 12 grams per batch (24 lb/ton material). This adjustment precipitated the soluble iron and any residual mercury. Table 4-17 summarizes the precipitation data.

TABLE 4-17. IRON PRECIPITATION DATA, INEEL LOCKED-CYCLE TESTS

Cycle	Precipitate		Filtrate	Removal
	Grams	Hg, mg/kg	Hg, mg/kg	Efficiency, %
1	20.6	1120	0.9	99.8
2	24.7	600	0.5	99.9
3	21.7	1180	0.6	99.9
4	22.2	11,100	0.8	99.8
5	25.3	34,700	1.1	99.7
6	32.2	420	NA	--
7	30.0	680	1.0	99.7
8	52.8	385	1.0	99.8
9	35.6	520	3.9	99.0
10	20.0	520	0.7	99.8

The efficiency of mercury removal from the combined cementation and iron precipitation steps was 99.8% or greater in six of the ten cycles.

The potassium iodide content of the iron precipitation filtrate was determined and a portion of the filtrate that contained either sufficient potassium iodide or liquid volume for the next leach cycle was set aside. The balance of the solution, if any, was advanced to iodine regeneration. A total of nine iodine regenerations were performed. The efficiency of the regeneration tended to be proportional to the quantity of iodine. The range of conversion efficiency was 60 to 100% and averaged 93%.

Using the addition data subsequent to Cycle 3, the indicated iodine make-up requirement was 40

grams per leach (80 lb I₂/ton dry basis material). This requirement was larger than expected. It probably was due to leaching at 60°C. Reducing the temperature to 45 or 50°C would significantly reduce the amount of iodine volatilized during leaching. On a commercial scale, it may be practical to collect most of the volatilized iodine in a scrubber and recycle it to the process.

SECTION 5.0

CONCLUSIONS AND COMPARISON AGAINST SUCCESS CRITERIA

This section summarizes the results of both the laboratory-scale and the bench-scale testing performed on three different waste types, and compares the results to the success criteria that were defined in the Management Plan. A conceptual design, a mass balance, and costs for a GEMEP pilot-scale plant for treating the INEEL waste is also presented. A recommendation is made as to whether pilot-scale testing of the GEMEP process should be conducted for any of the wastes tested under the base program.

5.1 SUCCESS CRITERIA

The success criteria as defined in the Management Plan for the base program were as follows:

1. Selective separation and removal of elemental mercury and mercury compounds from at least one of the three waste types to be tested must be demonstrated.
2. Mercury removal from one or more of the waste types should be demonstrated such that the treated wastes, when subjected to TCLP extraction, yield extracts in which the mercury concentration does not exceed the TCLP limit of 0.2 mg/L (200 ppb) mercury. (NOTE: This objective was developed before EPA established a Universal Treatment Standard for mercury of 0.025 mg/L as TCLP. Please refer to Section 1.0 for further discussion of this issue).
3. Sufficient data to proceed to design of a pilot-scale unit should have been developed.
4. Projected treatment costs, based on the results of the bench-scale tests, should be competitive with baseline and alternative technologies.

The first two criteria are related to the effectiveness of the GEMEP process for selectively removing mercury from wastes. Results previously discussed in Sections 3.0 and 4.0 are summarized below in Section 5.2 to show that these criteria have been met for the waste types tested in this program.

The latter two criteria are related to cost and design issues. Further development of the results presented in Sections 3.0 and 4.0 is needed to determine if these latter criteria have been attained. Sections 5.3 and 5.4 present mass balances, a conceptual design of a pilot-scale system, and cost estimates for construction and operation of a pilot-scale system for one of the DOE wastes studied, the INEEL waste. Section 5.5 compares the cost estimate to literature values for costs for a competing technology (thermal treatment), and makes a recommendation as to whether the GEMEP technology should proceed to the pilot scale for any of the wastes tested in the base program.

5.2 GEMEP EFFECTIVENESS FOR MERCURY REMOVAL

Mercury was effectively extracted from all three waste types tested during this program, where the definition of effectiveness is that the treated solids pass the TCLP test. Hence, success criteria 1 and 2 were attained. (Evaluation of effectiveness does not include cost considerations; cost is considered in Sections 5.4 and 5.5). Results that demonstrate attainment of the first two success criteria are summarized below for each waste tested.

5.2.1 Fluorescent Lamps

Results that demonstrate the effectiveness of the GEMEP process for treating fluorescent lamp waste were obtained by GE-CRD (laboratory-scale results) and CMRI (bench-scale results), as summarized below.

Laboratory-Scale Results. The laboratory-scale tests performed by GE-CRD demonstrated that the optimal conditions for GEMEP extraction of whole lamps (minus the aluminum endcaps, which are separated before extraction) are 0.02 M iodine/0.04 M iodide, 45°C, 3/1 liquid-solid ratio, and one hour reaction time. These conditions yield, on average, 0.6 mg of mercury and 0.63 mg of iodine in the treated lamp solids. The average residual mercury level of 0.6 mg (for a 270 g lamp) is below the estimated limit of 1 mg needed to make it physically impossible to fail

the TCLP test, even if all the mercury were to dissolve during the test. The optimal conditions can also be practically maintained at the pilot scale. Pilot plant equipment for a one-hour reaction time will not be unreasonably large. The iodine and iodide concentrations are at the low end of those typically used for the GEMEP process, and iodine losses are less than 1% based on weight of treated solids. Heating can be provided by an electric pad heater around the outside of a steel extraction tank. Pilot testing would be needed to evaluate the economics of heating the extraction slurry vs. operating for a longer time at a lower temperature.

Further separation of whole lamps into different fractions results in a more economical process than using the minimal separation step of removing the aluminum endcaps. The laboratory-scale tests have shown that separation of crushed lamps by size into plus 30 and minus 30 mesh fractions yields a plus 30 mesh fraction that contains little mercury (less than that which could result in failure of TCLP) and represents 95% of the initial lamp mass. Due to the recent establishment of a Universal Treatment Standard for mercury of 0.025 mg/L TCLP (lower than the TCLP limit of 0.2 mg/L), however, TCLP testing of the plus 30 mesh fraction should be conducted to determine if this material would produce a TCLP extract with mercury concentrations less than the UTS. If so, this material would not need to be processed by GEMEP extraction to be acceptable for land disposal.

The minus 30 mesh fraction contains the bulk of the mercury and requires GEMEP extraction or some other form of treatment to render it non-hazardous. Laboratory-scale tests showed that it was possible to reduce the mercury content in the minus 30 mesh fraction to a level that passed the TCLP test. The recommended conditions for treatment of the minus 30 mesh fraction were 0.1 M iodine/0.2 M iodide, 45°C, and 4/1 liquid-solid ratio for one hour. TCLP testing of treated solids would need to be conducted to demonstrate that the UTS is attained, however, because the residual mercury remaining in the treated solids is high enough to theoretically cause the UTS to be exceeded, were it all to dissolve.

Removal of the plus 30 mesh fraction prior to GEMEP extraction will result in smaller equipment

and operating costs for the GEMEP process, since a much smaller mass of material (approximately 5% of the initial lamp mass) actually needs to be extracted. However, it is necessary to account for equipment and operating costs of the sizing (pre-segregation) process to assess the economics of this approach compared to treating whole lamps with only the endcaps removed. Pre-segregation was evaluated in more detail by CMRI in their bench-scale tests, as summarized further below. Costs are discussed further in Section 5.4.

Bench-Scale Results. Bench-scale tests on fluorescent lamps included evaluation of crushing and pre-segregation methods, bench-scale GEMEP extraction tests, and locked-cycle tests of the entire extraction, mercury recovery, reagent recycle, and iodine recovery processes.

It was possible to concentrate the mercury content in fluorescent lamps, after crushing, into the non-metal fraction that was finer than 65 mesh. This fraction contained about 425 mg Hg/kg. Bench-scale testing of minus 65-mesh glass reduced the residual mercury to concentrations ranging from 7 to 15 mg/kg, with corresponding mercury extraction efficiencies of 96.4 to 98.3%. The treated glass readily passed the 0.2 mg Hg/l TCLP limit in all samples tested. TCLP extraction results ranged from 0.01 to 0.10 mg Hg/L, with all but one of the results also below the UTS of 0.025 mg/L.

The mercury-laden extraction solution was passed through iron columns to reduce the mercury, and the solution was then made basic by the addition of lime to remove soluble iron. The efficiency of mercury removal from the combined iron column treatment and lime precipitation steps exceeded 99.9% in six of the ten test cycles. These results demonstrate that the GEMEP process can efficiently solubilize the mercury in fluorescent lamps, and that the solubilized mercury can subsequently be removed from the extraction solution.

5.2.2 East Fork Poplar Creek Sediment

Bench-scale GEMEP extraction testing of East Fork Poplar Creek sediment reduced the residual mercury from about 800 mg/kg in the raw waste to about 3 mg/kg. The solubilization of uranium present in the sediments was low, ranging from 0.01% to 0.13%. This result indicates that the GEMEP extraction solution would not become a radioactive waste, and that nearly all the uranium initially present in the sediments (99 to 99.99%) would remain with the treated sediments, while mercury would be effectively extracted.

Locked-cycle tests were not performed on the East Fork Poplar Creek sediment, because this waste has since been disposed by DOE and is no longer of concern for that reason. Hence, data on GEMEP process steps other than the extraction step were not generated, and no mass balance or cost calculations were performed.

5.2.3 INEEL Soil/Sludge

The INEEL waste was subjected to bench-scale testing of pre-segregation steps, GEMEP extraction, and locked-cycle testing of the entire GEMEP process (extraction, mercury recovery, iodine/iodide recycle, and iodine recovery). Pre-segregation by wet screening on sieves was able to effectively separate the INEEL waste into a plus 4 mesh fraction that passed TCLP and consisted of rock, asphalt, and a light colored tar/polymer. The minus 4 mesh material was a fine silt with a mercury concentration in the range of 700 to 800 mg/kg; this material underwent GEMEP extraction and locked-cycle testing.

Seventeen bench-scale extraction tests were run at varying conditions of iodine/iodide concentration, temperature, and pH. Mercury concentrations were reduced from an initial level of 700-800 mg/kg to 17 to 64 mg/kg. For nine of the 17 tests, the treated solids were also analyzed by TCLP. All of the extracts of treated solids contained mercury at concentrations less than the TCLP limit of 0.2 mg/L, while for four of the nine samples, the mercury extract concentration was

also less than the UTS of 0.025 mg/L. Hence, it was possible to conduct bench-scale extractions of the INEEL waste using conditions that would produce treated solids that could be directly land-disposed.

Results of the locked-cycle tests were less successful. The mercury concentrations in TCLP extracts of treated solids were all above the UTS of 0.025 mg/L, although they were below the TCLP limit of 0.2 mg/L. Because attaining the UTS was not an objective of this test program (since testing preceded its promulgation), no locked-cycle tests were performed to see if the UTS could be attained by adjustments in test conditions.

Locked-cycle testing is a better simulation of actual treatment conditions than bench-scale extraction tests alone, because it is necessary to recover and recycle iodine, iodide and water to make the GEMEP process economical. When extraction tests are conducted, fresh solutions are used, but in locked-cycle testing, extraction solution is recycled. Some reduction in the efficiency of mercury extraction is anticipated when locked-cycle test results are compared to simple extraction test results. Hence, it is not unusual that some of the treated solids from extraction tests passed the UTS, while none of the treated solids from the locked-cycle tests passed the UTS.

It was possible to efficiently remove solubilized mercury from the extraction solution during locked-cycle testing of the INEEL waste. The combined efficiency of mercury removal by the mercury precipitation and iron precipitation steps was 99.8% or greater in six of the ten cycles. Regeneration of iodine was less efficient, with a range of 60 to 100% and an average of 93%.

To summarize, the GEMEP process was able to effectively extract mercury from the INEEL waste, but not to the degree required to routinely produce treated solids that would pass the UTS. It may be possible to do so, but further testing would be necessary to define the conditions that would be needed.

5.3 PILOT-SCALE DESIGN AND OPERATION

Attainment of the third success criterion requires that a pilot-scale system be designed. A conceptual design for a pilot-scale GEMEP system sized to process 24 kg of waste per hour is presented in this section, along with a description of the stages of operation and a detailed mass balance for processing of INEEL waste in the pilot system. Simplified mass balances for treatment of INEEL waste and fluorescent lamps at a rate of 100 kg/hr are also presented.

The pilot-scale system is based on treatment of INEEL waste, although with some modifications (e.g., addition of equipment to crush lamps and remove magnetic components), it could also be used to treat fluorescent lamps. This conceptual design is used as a basis for the cost estimate for treating INEEL waste presented in Section 5.4.

5.3.1 Pilot System Equipment and Process Description

The flowsheet for a pilot-scale GEMEP system is shown in Figure 5-1. The system is based on a waste processing rate of 24 kg/hr. The equipment list for this flowsheet is presented in Appendix B-1. The process description presented below is specific to the INEEL waste and refers to the equipment shown in Figure 5-1.

GE MERCURY EXTRACTION PROCESS FLOWSHEET

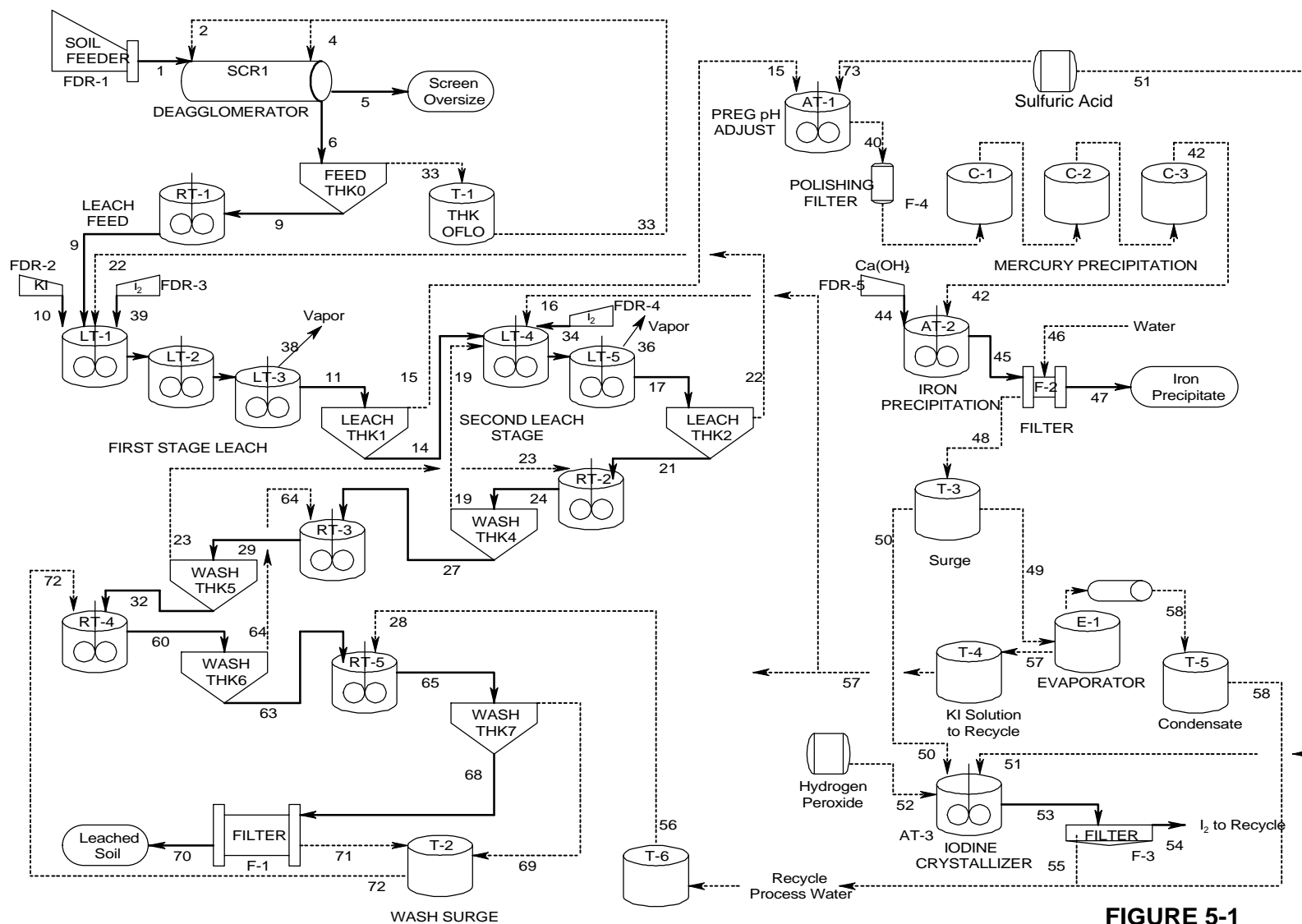


FIGURE 5-1

The pilot-scale system includes equipment for each stage of the GEMEP process, including pre-segregation, leaching (mercury extraction), washing, mercury recovery (precipitation), iron precipitation, and iodine recovery. Two stages of leaching and four stages of countercurrent washing are included. Effective washing is important both to remove solubilized mercury from the treated solids, and to remove iodine/iodide solution from the treated solids to maximize recovery and reuse of iodine. The process description below references the equipment names presented on Figure 5-1, and identifies when tanks are not shown (not all tanks and pumps appear on the figure, to reduce its complexity).

Pre-Segregation. The waste soil is charged to a belt feeder (FDR-1). The belt feeder discharges into a rotary scrubber or deagglomerator (SCR1), i.e., a rotating, baffled horizontal drum. Recirculating water is added to generate a 30 to 50% solids slurry. The scrubber serves to deagglomerate the particles and loosen any particles that are stuck to screen oversize, i.e., rock and debris. The discharge end of the scrubber is fitted with a 4-mesh screen that allows separation of the rock and debris from the remaining soil. A recirculating water spray is used to rinse soil from the rock and debris. The rock and debris discharge by a chute into a collection drum. This oversize material would be tested by TCLP, and if the concentration in the TCLP extract is below the UTS, it would not require further processing.

The minus 4-mesh slurry is pumped to a flocculation tank (in general, flocculation tanks are not shown as separate tanks, but are included with the thickeners) where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into a thickener (FEED THK0). The clarified, thickener overflow solution advances to a surge tank (T-1) that serves as a reservoir for the recirculating water. The thickener underflow is pumped to a surge tank (RT-1) as the leach circuit feed.

Leaching (Mercury Extraction). The feed slurry is metered to a leach tank (LT-1) where advancing hot solution from the second-stage leach thickener (LEACH THK2) is added. Iodine crystals and, if needed, potassium iodide are added using small screw feeders (FDR-2, FDR-3).

The first-stage leach slurry is heated to 50°C by immersion heaters in the three leach vessels. The vessels are vented to an eductor- type scrubber (not shown) operating on the KI recycle solution. The purpose of the scrubber is to reclaim vaporized iodine for subsequent recycle.

The first-stage leach slurry discharges to a flocculation tank where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into a thickener (LEACH THK1). The clarified, thickener overflow solution advances to the pregnant leach solution, pH adjustment tank AT-1. The thickener underflow is pumped to the second-stage leach (LT-4 and LT-5).

In the second-stage leach, advancing hot solution from the first wash-stage thickener (WASH THK4) and recycle KI solution are added. Iodine crystals are added using small screw feeders (FDR-4). The second-stage leach slurry is heated to 50°C by immersion heaters in the two leach vessels. The vessels are vented to an eductor- type scrubber operating on the KI recycle solution.

The second-stage leach slurry discharges to a flocculation tank where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into a thickener (LEACH THK2). The clarified, thickener overflow solution advances to first-stage leach.

Countercurrent Washing to Remove Solubilized Mercury and Recover Iodine/Iodide. The second-stage thickener underflow slurry is pumped a repulping tank (RT-2) where it is mixed with advancing overflow solution from the second wash thickener (WASH THK5). The repulped slurry advances to a flocculation tank where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into the first wash thickener (WASH THK4). The clarified, thickener overflow solution advances to second-stage leach.

The first wash-stage thickener (WASH THK4) underflow slurry is pumped to a repulping tank RT-3 where it is mixed with advancing overflow solution from the third wash thickener (WASH THK6). The repulped slurry advances to a flocculation tank where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into the second wash thickener

(WASH THK5). The clarified, thickener overflow solution advances to first wash repulping tank RT-2.

The second wash-stage thickener (WASH THK5) underflow slurry is pumped to a repulping tank RT-4 where it is mixed with advancing overflow solution from the fourth wash thickener WASH THK7. The repulped slurry advances to a flocculation tank where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into the third wash thickener WASH THK6. The clarified, thickener overflow solution advances to second wash repulping tank RT-3. The third wash-stage thickener (WASH THK6) underflow slurry is pumped to a repulping tank RT-5 where it is mixed with recycle process water from T-6. The repulped slurry advances to a flocculation tank where it is flocculated using a polyacrylamide polymer. The flocculated slurry overflows into the fourth wash thickener WASH THK7. The clarified, thickener overflow solution advances to the third wash repulping tank RT-4. The thickener underflow is pumped to a plate and frame pressure filter where the washed, leach residue is dewatered.

Mercury Precipitation. The pH of the pregnant leach solution is adjusted to approximately 4 using sulfuric acid (see tank AT-1). The sulfuric acid is added using a pH controlled metering pump. The pH-adjusted solution then is pumped through a polishing filter and three columns containing iron turnings, in series (C-1, C-2, and C-3). The iron turnings reduce the iodine in the leach solution to iodide, i.e., ferrous iodide. The iodide-mercury complex is reduced to elemental mercury and ferrous iodide. The elemental mercury remains in the columns.

Iron Precipitation. The mercury-barren solution, consisting of mostly calcium, potassium and ferrous iodide, advances to the iron precipitation stage (tank AT-2). The solution pH is adjusted to approximately 10 using calcium hydroxide added through a screw feeder (FDR-5). The iron readily precipitates at this pH. The precipitate slurry then is filtered using a multiple-cartridge filter F-2. The filtrate advances to a surge tank (T-3) and the cartridges are discarded after washing with water.

The iron precipitate is a secondary waste. It would be tested by TCLP, and if the mercury concentration in the TCLP extract is below the UTS, it could be disposed of without further processing to remove mercury. Iron precipitate was not tested by TCLP as part of this program, but would be tested in a pilot-scale program where a larger quantity would be produced.

Iodine Recovery and Recycle. A portion of iron filtrate containing sufficient iodide for make-up advances to an evaporator (E-1) where the volume is reduced, i.e., the iodide concentration is increased, such that when recycled the volume will not upset the process water balance.

The balance of the iron filtrate advances to an iodine regeneration step where hydrogen peroxide is metered in and the pH is controlled by addition of sulfuric acid. The iodine crystal slurry is filtered on a small filter. The filtrate, i.e., dilute sulfuric acid and soluble calcium sulfate, is recycled to the wash circuit. The iodine crystals are recycled to the leach circuit iodine feeders.

5.3.2 Mass Balances

A detailed mass balance for processing of the INEEL waste, based on a processing rate of 24 kg/hr and the flowsheet in Figure 5-1, is presented in Appendix B-2. Simplified mass balances for the INEEL waste and for fluorescent lamps, based on a processing rate of 100 kg/hr, are presented in Tables 5-1 and 5-2.

TABLE 5-1. SIMPLIFIED MASS BALANCE FOR FLUORESCENT LAMP GLASS

Stream	Total Mass Flow, kg/hr	Mercury Concentration, mg/kg	Material Description and Probable Characteristics⁽¹⁾
Fluorescent lamps	100	63	Feed to pre-segregation stage
Metal pieces from lamps	2.7	0	Residual waste, should meet TCLP and UTS limits
Plus 65-mesh glass (large fragments)	82.5	0.7	Residual waste, should meet TCLP and UTS limits
Minus 65-mesh glass (fine fragments and phosphor)	14.8	425	Feed to GEMEP extraction (leaching) stage
Leach slurry, 25% solids	59.2	--	Intermediate stream
Leach filtrate (extraction solution, mercury-laden)	44.4	138	Feed to mercury precipitation stage
Residual glass (glass that has been leached and washed)	14.8	12	Residual waste, meets TCLP and UTS limits
Mercury barren (solution exiting iron columns)	44.4	< 2	Feed to iron precipitation stage
Iron hydroxide waste	1.5	60	Secondary waste from iron precipitation, must test by TCLP to determine disposal
Iron filtrate (solution from iron precipitation stage)	44.4	< 0.1	Feed to iodine and iodide recovery/recycle stages

- (1) Evaluation based on bench-scale test results and TCLP extractions where available, or professional judgment when TCLP testing was not specifically performed. TCLP testing was not done on metal and large glass fragments, or on iron hydroxide waste.

TABLE 5-2. SIMPLIFIED MASS BALANCE FOR INEEL SOIL/SLUDGE WASTE

Stream	Total Mass Flow, kg/hr	Mercury Concentration, mg/kg	Material Description and Probable Characteristics⁽¹⁾
INEEL soil/sludge	100	733	Feed to pre-segregation stage
Over-size (rock, debris)	9	50	Residual waste, meets TCLP limits, may not meet UTS limit
Minus 4-mesh	91	800	Feed to GEMEP extraction (leaching) stage
Leach slurry, 19% solids	479	--	Intermediate stream
Leach filtrate (extraction solution, mercury-laden)	388	204	Feed to mercury precipitation stage
Residual soil (waste that has been leached and washed)	91	30	Residual waste, meets TCLP limits, need further testing to determine whether it will routinely meet UTS limit
Mercury barren (solution exiting iron columns)	388	7	Feed to iron precipitation stage
Iron hydroxide waste	15	180	Secondary waste from iron precipitation, must test by TCLP to determine disposal
Iron filtrate (solution from iron precipitation stage)	388	< 0.1	Feed to iodine and iodide recovery/recycle stages

- (1) Evaluation based on bench-scale test results and TCLP extractions where available, or professional judgment when TCLP testing was not specifically performed. TCLP testing was not done on iron hydroxide waste. For residual soil/sludge, some TCLP extract test results from extraction tests were below UTS limits, but UTS limits were not attained in locked-cycle testing.

5.4 COST ESTIMATE FOR PILOT-SCALE TREATMENT OF INEEL WASTE

A cost estimate for construction and operation of a pilot-scale system for GEMEP treatment of INEEL waste is presented in this section. The estimate assumes that the system will be constructed and operated at CMRI in Golden, Colorado, and does not consider costs for shipping wastes for pilot testing to CMRI. The primary purpose of the estimate is to provide DOE with information regarding the probable costs of constructing and operating a pilot-scale system at CMRI, should Option 1 of this contract be exercised. A secondary purpose of the estimate is to provide a basis for comparison of the GEMEP technology with a competing technology, thermal treatment, to determine if success criterion 4 was attained. Because of the limited quantity of INEEL waste in storage at the present time, the pilot-scale system design presented herein would be sufficiently large to treat all the INEEL waste. Full-scale treatment costs are hence estimated to be approximately the same as pilot-scale costs for this particular waste.

5.4.1 Cost Basis

To prepare a capital cost estimate, it is necessary to select a size for the treatment system, which in turn requires that one assume a treatment rate. One must also assume a duration of operation or volume of waste to be treated. A treatment rate of 24 kg/hr (53 lb/hr) was selected as appropriate for a pilot-scale system. A thirty day operational period was also assumed as a maximum duration of pilot operation. If the pilot-scale system were operated for 30 days, 24 hours per day at 24 kg/hr, this leads to a maximum waste volume treated of 17,000 kg (equivalent to seventy-five 55-gallon drums, each weighing approximately 500 lb). Because there is a limited quantity of INEEL waste in storage, this plant, if built for pilot-scale testing, would also serve as the full-scale plant for treatment of this waste. A smaller system could be constructed for pilot-scale testing, but the capital and operating cost savings of a smaller system would not be substantial. Also, for certain pieces of equipment, smaller versions are not readily available.

The flowsheet as costed is the same flowsheet that was shown in Figure 5-1. A detailed mass balance and equipment list for the flowsheet are shown in Appendix B. Each of the items of equipment is sized and costed. The total cost of the process equipment is estimated at \$56,990.

The cost for fabrication and assembly is based on the fabrication of four modules: 1) feed preparation, 2) leaching, 3) residue washing, and 4) solution treatment. Each of the modules is fabricated as a stand-alone assembly of equipment with its own power and control center/panel. The estimated costs for the fabricated modules are shown in Tables 5-3, 5-4, 5-5, and 5-6.

For operation in the field, the modules are mounted on low-boy trailers fitted with shed-type roofs. The total costs for the four modules plus trailers are summarized in Table 5-7. Briefly these costs are:

Total Equipment and Materials	\$ 95,140
Design and Procurement	7,200
Trailers (used)	30,000
Fabrication and Assembly	53,423
Contingency	<u>37,177</u>
Total Estimated Cost	\$ 223,060

TABLE 5-3. COST OF FEED PREPARATION MODULE

Item	Number	Costs
Purchased Equipment		\$7,935
Flowmeters	2	\$1,000
Electrical distribution 10 amp 220 volt NEMA 4	2	\$400
40 amp 110 volt NEMA 4	4	\$1,000
Steel for supports, skid base		\$1,200
Pipe, fittings, tubing		<u>\$500</u>
Total materials		\$12,035
Design, procurement		\$1,000
Fabrication, assembly		<u>\$9,000</u>
Total cost		\$22,035
Contingency	20%	<u>\$4,407</u>
Total Estimated Cost for Feed Preparation		\$26,442

TABLE 5-4. COST OF LEACH MODULE

Item	Number	Costs
Purchased Equipment		\$15,570
Flowmeters	3	\$1,500
Pyrometers	5	\$1,500
Electrical distribution 10 amp 220 volt NEMA 4	17	\$3,400
40 amp 110 volt NEMA 4	7	\$1,750
Steel for supports, skid base		\$1,200
Pipe, fittings, tubing		<u>\$1,000</u>
Total materials		\$25,920
Design, procurement		\$1,200
Fabrication, assembly		<u>\$14,000</u>
Total cost		\$41,120
Contingency	20%	<u>\$8,224</u>
Total Estimated Cost for Leach Module		\$49,344

TABLE 5-5. COST OF WASH MODULE

Item	Number	Costs
Purchased Equipment		\$20,115
Flowmeters	2	\$1,000
Electrical distribution 10 amp 220 volt NEMA 4	18	\$3,600
40 amp 110 volt NEMA 4	8	\$2,000
Steel for supports, skid base		\$2,500
Pipe, fittings, tubing		<u>\$2,000</u>
Total materials		\$31,215
Design, procurement		\$1,500
Fabrication, assembly		<u>\$20,000</u>
Total cost		\$52,715
Contingency	20%	<u>\$10,543</u>
Total Estimated Cost for Wash Module		\$63,258

TABLE 5-6. COST OF SOLUTION PROCESSING MODULE

Item	Number	Costs
Purchased Equipment		\$13,370
Flowmeters	4	\$2,000
Pyrometers	7	\$2,100
Electrical distribution 10 amp 220 volt NEMA 4	10	\$2,000
40 amp 110 volt NEMA 4	4	\$1,000
Steel for supports, skid base,		\$2,500
Pipe, fittings, tubing		<u>\$3,000</u>
Total materials		\$25,970
Design, procurement		\$3,500
Fabrication, assembly		<u>\$20,000</u>
Total cost		\$49,470
Contingency	20%	<u>\$9,894</u>
Total Estimated Cost for Solution Processing		\$59,364

TABLE 5-7. SUMMARY OF FABRICATED MODULE COSTS

Purchased Equipment		\$56,990
Flowmeters		\$5,500
Pyrometers		\$3,600
Electrical distribution	10 amp 220 volt NEMA 4	\$9,400
	40 amp 110 volt NEMA 4	\$5,750
Steel for supports, skid base,		\$7,400
Pipe, fittings, tubing		<u>\$6,500</u>
Total materials		\$95,140
Design, procurement		\$7,200
Trailers for equipment		\$30,000
Fabrication, assembly		<u>\$53,543</u>
Total cost		\$185,883
Contingency	20%	<u>\$37,177</u>
Total Estimated Cost		\$223,060

Operating costs for the processing of the 24 kg/hr of soil are shown in Table 5-8. The daily cost is estimated as follows:

	<u>Daily Cost</u>	<u>Cost per Metric Ton (1,000 kg)</u>
Reagents	\$928	\$1,637
Utilities	58	100
Rentals	90	156
Labor	<u>5,500</u>	<u>9,550</u>
Total	\$ 6,576	\$ 11,443

Disposal costs will be site dependent. For pilot operations, it was assumed that off-site disposal would be required for all waste materials, since the estimate assumes that the testing would be conducted at CMRI. These costs were assumed at \$4 per kg of rad waste, \$1 per gallon of liquid waste, and \$4 per kg of non-rad mercury waste. These assumed unit costs are highly variable, particularly the cost for disposal of rad waste. Also, costs for disposal of treated, radioactive (but no longer mercury-bearing) solids, and recovered mercury, would be incurred for the competing technology of thermal treatment as well.

For the processing of the 17 metric tons of soil, the cost for disposal of treated solids (assumed to be rad waste) and secondary wastes is estimated at \$91,000 or about \$5,400 per metric ton. The cost breakdown is presented in Table 5-9, Item 5: Allowance for Off-Site Disposal of Residuals.

TABLE 5-8. OPERATING COSTS

Reagents	kg/hr	Unit Cost	Daily Cost
Potassium Iodide	0.2	\$30.00	\$144.00
Iodine	1.5	\$20.00	\$720.00
Sulfuric acid	0.7	\$1.10	\$18.48
Hydrogen peroxide, 30%	0.6	\$2.20	\$31.68
Hydrated lime	0.6	\$0.30	\$4.32
Flocculant	0.05	\$6.60	\$7.92
Iron turnings	0.05	\$1.00	\$1.20
Total reagents			\$927.60
Utilities	hp	watts	
Motor power	12	9000	\$0.10 \$21.60
Heating power		12000	\$0.10 \$28.80
Miscellaneous power		3000	\$0.10 \$7.20
Total power			\$57.60
Labor	no	hrs	rate
Operators	9	8	\$50.00 \$3,600.00
Analysts	2	8	\$50.00 \$800.00
Supervision	1	8	\$85.00 \$680.00
Management	1	4	\$105.00 \$420.00
Total labor			\$5,500.00
Rentals	no	rate	
Feed scale	1	\$50.00	\$50.00
Office trailer	1	\$20.00	\$20.00
Decon trailer	1	\$20.00	\$20.00
Total rentals			\$90.00
Total daily cost			\$6,575.20

TABLE 5-9. TOTAL COSTS FOR PILOT PLANT OPERATION

1	Project Planning	Number	Unit Cost	Cost		
	Management Plan	1	\$8,600	\$8,600		
	Plot Plant Operation & Maintenance Plan	1	\$26,000	\$26,000		
	Permits and Licenses	1	\$17,200	\$17,200		
	Total Planning			\$51,800		
2	Mobilization and Site Preparation	Number	Unit Cost	Cost		
	Transport Plot Plant Trailers to Site	2	\$3,000	\$6,000		
	Slab at site, 400 sq ft	1	\$4,000	\$4,000		
	Office, decon trailers - 1st month rental			\$1,200		
	Start up Reagents					
	Iodine	50 kg	\$20	\$1,000		
	Potassium iodide	100 kg	\$30	\$3,000		
	Iron turnings	50 kg	\$1	\$50		
	Set-up on site	Number	Hours	Days	Rate	Cost
	Operators	4	8	5	\$50	\$8,000
	Analysts	1	8	5	\$50	\$2,000
	Electrician	2	8	5	\$50	\$4,000
	Supervision	1	8	5	\$85	\$3,400
	Total Mobilization and set-up					\$32,650
3	Equipment cost			Cost		
	Total equipment list			\$223,060		
4	Operations	Number	Unit Cost	Cost		
	Total cost	30	\$6,959	\$208,776		
5	Decontamination/disposal	Number	Hours	Days	Rate	Cost
	Operators	4	8	5	\$50	\$8,000
	Electrician	2	8	5	\$50	\$4,000
	Supervision	1	8	5	\$85	\$3,400
	Allowance for Off-Site Disposal of Residuals:	Number	Unit Cost	Cost		
	Oversize Material (rad, not RCRA); 0.1 x mass treated	1500 kg	\$4	\$6,000		
	Treated Solids (rad, not RCRA) = mass treated	17000 kg	\$4	\$68,000		
	Iron Hydroxide Sludge (rad, not RCRA) = Fe consumed	4000 kg	\$4	\$16,000		
	Process Water (rad, not RCRA) = batch size x 5	1000 gal	\$1	\$1,000		
	Mercury/Iron Turnings - Hg reclaim facility	50 kg	\$4	\$200		
	Total decontamination/disposal:			\$106,600		
	Total costs for program			\$622,886		

5.4.2 Pilot Testing Costs

As noted above, the cost estimate presented herein is for a pilot-scale operation. The number of DOE wastes available that are sufficiently low in radioactivity to allow off-site treatability testing at CMRI's facilities is limited. In this case it turns out that the quantity of the appropriate and currently available waste, the INEEL waste, also is low. For this reason, pilot-scale and full-scale treatment costs for the INEEL waste are roughly equivalent. The major costs for the processing are:

1. Labor, which can be reduced by increasing the scale of processing
2. Make-up iodine - the quantity estimated from the mass balance is based on the locked-cycle laboratory tests. In these tests, no effort was made to scrub iodine from vapors exiting vessels and because of the batch nature of the testing, there were other losses. The pilot plant would include a scrubbing system to recover iodine vapors. The unit cost for iodine is also high and variable, depending on market conditions and the quantity purchased. Estimates range from \$10/kg to \$40/kg; a value of \$20/kg was used in this report.

The estimated costs for a pilot program that would be carried out at CMRI are detailed in Table 5-9. The total costs (capital and operating, plus an allowance for off-site disposal of residuals) for a pilot program to process 17 metric tons of INEEL soil are about \$623,000. Costs for disposal of residuals are highly dependent on the waste type. Also, costs for disposal of the major residual waste (i.e., the treated solids with mercury removed, but still radioactive) would be incurred for a competing technology such as thermal treatment as well. Oversize material would also be removed before processing via a competing technology, and require some form of disposal. The residual wastes that are unique to the GEMEP process, and are appropriately classified as secondary wastes, include: process water, iron/mercury column residue, and iron hydroxide sludge.

5.5 COMPARISON WITH COMPETING TECHNOLOGIES & RECOMMENADATIONS

A summary of results and a comparison with competing technologies are presented for the INEEL waste and fluorescent lamp waste. A comparison has not been made for the East Fork Poplar Creek sediment, since this waste has since been disposed by DOE and is no longer of concern.

5.5.1 INEEL Soil/Sludge Waste

The GEMEP process was shown to be effective in removing mercury from the INEEL soil. Scrubbing and sizing removed about 9% of the weight as non-mercury waste. The balance of the soil contained about 800 mg Hg/kg. GEMEP processing of the INEEL soil reduced the total mercury content to less than 50 mg/kg. The soil after GEMEP treatment easily passes the 0.2 mg Hg/L TCLP limit. Values observed were 0.02 to 0.04 mg Hg/l. The UTS limit of 0.025 mg Hg/L was attained for some treated solids samples during bench-scale extraction tests. The UTS limit was not attained for any of the residues generated during locked-cycle tests. However, it is possible that the UTS limit could have been routinely attained by altering the process conditions. Additional locked-cycle tests were not performed to see if the UTS limit could be attained, because attainment of the UTS was not an objective of the test program. (The UTS was not promulgated until the tests were essentially completed).

Costs were determined for pilot-scale treatment of INEEL soil. These costs indicated a reagent and utility cost of about \$1,700 per metric ton, and were based on data from the locked-cycle tests. It is expected that reagent costs could be reduced by installing a system to recover iodine vapors. In the locked-cycle tests, there was no means of capturing iodine vapors, and it is suspected that the high iodine make-up requirement was primarily due to loss of iodine by volatilization.

Labor costs for the pilot-scale system are estimated at \$9,550 per metric ton. The total of reagent, utility, and labor costs is \$11,250 per metric ton. Labor costs on a per ton basis can be

reduced by building a treatment system with a greater throughput. The capital cost for the GEMEP pilot unit is estimated at \$223,000. This cost, amortized over the small volume of waste assumed for this estimate (17,000 kg or 17 metric tons), is \$13,100 per ton. This calculation does not consider the cost benefit of using the equipment for treating of other wastes, once the INEEL waste has been treated.

Full-scale costs for treatment of the INEEL waste are anticipated to be roughly equivalent to the pilot-scale estimated costs. Capital costs on a per ton basis are high primarily because of the small volume of waste assumed for the estimate. Labor costs on a per ton basis could be reduced to some degree if a larger-size treatment plant were designed and operated, since the number of operators is more directly related to the complexity and number of unit operations rather than the sizes of the equipment. However, a larger system is not warranted for treatment of the INEEL waste. Reagent costs, driven primarily by the iodine make-up requirement, can be potentially lowered by using scrubbers to recover volatilized iodine, but are not sensitive to the scale of the plant.

Thermal desorption is a competitive process for treating the INEEL soil. Costs for pilot-scale testing of the thermal treatment technology were estimated by consulting a firm active in thermal processing of mercury-bearing soils. The firm provided a monthly rental cost for their pilot equipment and one engineer. Other costs (labor, utilities, rental of ancillary equipment, planning document preparation, etc.) were estimated in a similar manner to the estimate developed for pilot-scale testing of the GEMEP process. The operating cost and total cost estimates for pilot-scale testing of thermal treatment are presented in Tables 5-10 and 5-11. The costs for pilot testing of thermal treatment are approximately one-half those estimated for pilot testing of the GEMEP process.

**TABLE 5-10. OPERATING COST ESTIMATE:
THERMAL TREATMENT PILOT TEST**

Reagents		kg/hr	Unit Cost	Daily Cost
<hr/>				
Utilities	hp	watts		
Motor power	12	9000	\$0.10	\$21.60
Heating power		12000	\$0.10	\$28.80
Miscellaneous power		3000	\$0.10	<u>\$7.20</u>
Total power				\$57.60
Labor	no	hrs	rate	
Operators	6	8	\$50.00	\$2,400.00
Analysts	1	8	\$50.00	\$400.00
Supervision	1	8	\$85.00	\$680.00
Management	1	4	\$105.00	<u>\$420.00</u>
Total labor				\$3,900.00
Rentals		no	rate	
Feed scale		1	\$50.00	\$50.00
Mercury thermal treatment unit		1	\$500.00	\$500.00
Office trailer		1	\$20.00	\$20.00
Decon trailer		1	\$20.00	<u>\$20.00</u>
Total rentals				\$590.00
 				<hr/>
Total daily cost				<u>\$4,547.60</u>

**TABLE 5-11 TOTAL COSTS FOR PILOT PLANT OPERATION:
THERMAL TREATMENT**

1	Project Planning	Number		Unit Cost	Cost	
	Management Plan	1		\$8,600	\$8,600	
	Pilot Plant Operation & Maintenance Plan	1		\$26,000	\$26,000	
	Permits and Licenses	1		\$17,200	\$17,200	
	Total Planning				\$51,800	
2	Mobilization and Site Preparation	Number		Unit Cost	Cost	
	Transport Pilot Plant to Site	1		\$5,000	\$5,000	
	Slab at site, 400 sq ft	1		\$4,000	\$4,000	
	Office, decon trailers - 1st month rental				\$1,200	
	Set-up on site	Number	Hours	Days	Rate	Cost
	Operators	4	8	5	\$50	\$8,000
	Analysts	1	8	2	\$50	\$800
	Electrician	1	8	2	\$50	\$800
	Supervision	1	8	5	\$85	\$3,400
	Total Mobilization and set-up					\$23,200
3	Equipment cost					Cost
	Rented; included in daily operations cost					
4	Operations	Number		Unit Cost	Cost	
	Total cost	30		\$4,548	\$136,428	
5	Decontamination/disposal	Number	Hours	Days	Rate	Cost
	Operators	2	8	5	\$50	\$4,000
	Electrician	1	8	2	\$50	\$800
	Supervision	1	8	5	\$85	\$3,400
	Allowance for Off-Site Disposal of Residuals:	Number		Unit Cost	Cost	
	Treated Solids (rad, not RCRA) = mass treated	20000 kg		\$4	\$80,000	
	Mercury - Hg reclaim facility	50 kg		\$4	\$200	
	Total decontamination					\$88,400
	Total costs for program					\$299,828

An attempt was made to locate costs for thermal treatment of other mercury-contaminated wastes at larger scales. An Internet search identified two full-scale soil treatment projects of approximately 1,000 tons, with costs on the order of \$400 to \$460 per U.S. ton (see Appendix B-3). The cost detail provided was not sufficient to determine whether costs for disposal of secondary wastes and treated solids were included.

The pilot-scale systems costed here were sized to process only 17,000 kg of waste (approximately 20 U.S. tons). While treating larger quantities offers an economy of scale with respect to capital and labor costs, the cost per ton of waste treated for reagents such as iodine would not be significantly reduced by going to a larger scale. Costs for iodine alone are projected to be on the order of \$1,000 per ton for GEMEP treatment of the INEEL waste, based on the iodine make-up requirements observed during the locked-cycle tests. As stated previously, it should be possible to significantly reduce iodine consumption by adding a scrubber system to capture and reclaim iodine vapor. However, in the absence of testing to determine the effectiveness of a scrubber system or other iodine recovery system, it is not possible to predict to what degree iodine consumption costs could be reduced. Based on the currently available data and cost projections, thermal treatment is likely to be the more cost effective option for treatment of INEEL waste. When one also considers the small quantity of INEEL waste in storage, further effort to develop the GEMEP technology for this particular waste is not recommended.

5.5.2 Fluorescent Lamps

The GEMEP process was shown to be effective in removing mercury from fluorescent lamp waste. The sizing and magnetic separation steps were able to separate from the crushed lamp waste a sizable percentage of recyclable glass and metal containing less than 1 mg/kg mercury. The balance of the glass (the minus 48-mesh fraction) can be treated by the GEMEP process to reduce the total mercury content to less than 10 mg/kg. The residual glass after GEMEP treatment easily passes the 0.2 mg Hg/L TCLP limit. Values observed were 0.01 to 0.10 mg Hg/L. Residual glass will also pass the UTS limit of 0.025 mg/L, depending on the extraction

conditions.

No estimate was made of the cost of full-scale treatment of fluorescent glass by the GEMEP process. However, the crushing and segregation steps produce recyclable metal and glass. The residue, after extraction at appropriate conditions, will no longer exceed the UTS limit for mercury, and if non-rad, it can be recycled or disposed at minimal cost. Costs for treatment of lamp glass on a unit mass basis (\$ per ton whole lamps) will be lower than those for INEEL waste estimated above, for two major reasons: 1) iodine losses are not as large, based on the bench-scale results, and 2) it is possible to separate out those lamp components that are relatively free of mercury via pre-segregation steps, thereby reducing the quantity of lamp waste that requires GEMEP extraction to reduce the mercury concentration to an acceptable level.

The competing technology for management of spent or off-spec, non-rad fluorescent lamps is to ship them to a lamp recycling facility, where thermal treatment is used to recover the mercury. According to estimates obtained by GE-CRD, recycling facilities charge \$0.25 per linear foot to accept fluorescent lamps. The lamps used in this testing program were four feet long and weigh approximately 286 grams. Using these figures and the \$0.25/linear foot value, the cost for recycling of fluorescent lamps on a mass basis is \$3,300 per U.S. ton whole lamps. If reagent and utility costs for pre-segregation and GEMEP treatment are on the order of \$1,000 per ton (a conservatively high estimate), it would be necessary to build a plant large enough to reduce labor costs to the \$2,000 per ton range, for the GEMEP process to be competitive with recycling. However, in the face of a competing technology it is probable that the recyclers would reduce their prices to some degree. Hence, it is difficult to predict whether the GEMEP process would be competitive with recycling (including thermal treatment) of fluorescent lamps.

With respect to radioactive fluorescent lamp waste stored at DOE sites, it appears that there is no longer a need for development of an alternative treatment technology to handle these wastes. The DOE sites have been able to dispose of this type of waste recently, and hence no sites were interested in providing lamp waste for this project. It is not anticipated that there will be future

interest in using the GEMEP technology for this particular waste, since the DOE sites are currently disposing of it without undue difficulty. While further pilot testing of the GEMEP technology may be of interest to manufacturers of lamps such as General Electric, it is not recommended that DOE pursue further testing, due to limited interest at the DOE sites.

SECTION 6.0

REFERENCES

- [1] D.F. Foust, U.S. patent 5,226,545 (July 13, 1993)
- [2] D.F. Foust, "Hydrometallurgical Removal of Mercury from Contaminated Soil 1. Selection of Extracting Agent", 92CRD216 (October 1992).
- [3] D.F. Foust, "Hydrometallurgical Removal of Mercury from Contaminated Soil 2. Use of KI/I₂ as an Extracting Agent", 92CRD222 (November 1992).
- [4] D.F. Foust, "The Removal of Soluble Mercury from an Aqueous Iodide/Iodine Solution", 96CRD071 (May 1996).
- [5] D.F. Foust, E. Barren, "The Removal of Soluble Mercury from an Aqueous Iodide/Iodine Solution 2. Cementation by Iron or Zinc", 96CRD141 (October 1996).
- [6] The TCLP (Toxicity Characteristic Leaching Procedure) test is defined by EPA Method 1311.
- [7] D.F. Foust, D.A. Haitko, D.K. Dietrich, "Protocol for the Determination of Leachable Mercury in Fluorescent Lamps" 97CRD031, (March 1997).
- [8] The current EPA limit for soluble mercury in solution or leached from solids during the TCLP test is 200 ppb.
- [9] Federal Register, May 26, 1998. 40 CFR Parts 148, 261, 266, 268, and 271: Land Disposal Restrictions Phase IV: Final Rule Promulgating Treatment Standards for Metal Wastes and Mineral Processing Wastes; Mineral Processing Secondary Materials and Bevill Exclusion Issues; Treatment Standards for Hazardous Soils; and Exclusion of Recycled Wood Preserving Wastewaters; Final Rule.
- [10] D.D. Gates, K.K. Chao, P.A. Cameron, "The Removal of Mercury from Solid Mixed Waste using Chemical Leaching Processes", Environmental Sciences Division No. 4361 Oak Ridge National Laboratory, July 1995.
- [11] R.P. Traver, "Development and Use of EPA's Synthetic Soil Matrix (SSM/SARM)", U.S. EPA Risk Reduction Engineering Lab, Edison, NJ February 1989.
- [12] D.W. Whisenhunt Jr., M. J. Brennan, "Determination of Iodide and Iodine in the Presence of Mercury by the Oxidation of Iodide to Iodine Followed by Extraction into Toluene", (manuscript in preparation).

- [13] For a detailed procedure on extracting mercury from fluorescent lamps using HNO_3 and HF see D.W. Whisenhunt Jr., "Evaluation of the Aicher Test as Performed at the Bucyrus Lamp Plant", 97CRD072 (May 1997).

Appendix A

Data Sheets for Bench-Scale Testing

Appendix A-1

Data Sheets for Bench-Scale Extractions of East Fork Poplar Creek Sediment

COLORADO MINERALS RESEARCH INSTITUTE		CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		
Metallurgical & Mineral Processing Consultants			Project No.:	971026
5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-1

Feed:	<u> 200 </u> dry grams			Hg,		U	
		% moisture	Damp wt.	mg/kg	Hg, mg	mg/kg	U, mg
		50 x 100	0.0	11.8	880	10	66
		100 x 200	0.0	25.9	830	21	40
		<200	42.6	283.0	760	123	21
							3.4

Leach:	25% solids							
			Liquid		Potassium Iodide		Iodine	
		dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
	Solids feed:	200.14	121	480	0.1	10.0	0.05	7.6
	Total I required							
Water:								

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
11:00	start	48	6.5	0
11:08	0	48	4.1	2
11:38	30	48	2.5	0.66
12:06	60	48	3.9	0.15
12:36	90	49	4.07	0.17
1:06	120	49.4	4.36	0

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-1-1	Residue		200	2.7	1	0.3	38	8	99.87
		mL		mg/L			mg/L		
A-1-2	Filtrate	650		300	195	100	0.015	0.010	0.13
	Balance, versus feed					126			146

COLORADO MINERALS RESEARCH INSTITUTE		CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET	Project No.: 971026	
Metallurgical & Mineral Processing Consultants			Date: 10/7/97	
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Test No: A-2

Feed:	dry grams	% moisture		Damp wt.	Hg,	Hg, mg	U	U, mg
		50 x 100	0.0		mg/kg		mg/kg	
		100 x 200	0.0	25.9	830	21	40	1.0
		<200	42.6	283.0	760	123	21	3.4

Leach:		25% solids		Liquid		Potassium Iodide		Iodine	
	dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g		
Solids feed:	200	121							
Total I required				0.2	19.9	0.1	15.2		
Water:			480						

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
10:10	start	49	6.58	0
10:20	0	50	3.9	2
10:50	30	51	4.04	0.9
11:20	60	50	3.26	1
11:50	90	49	3.94	0.15
12:20	120	50.4	4.31	0

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-2-1	Residue		205	6.6	1	0.7	40	8	99.87
		mL		mg/L			mg/L		
A-2-2	Filtrate	649		305	198	99	0.016	0.010	0.13
	Balance, versus feed					128			157

COLORADO MINERALS RESEARCH INSTITUTE		CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		
Metallurgical & Mineral Processing Consultants			Project No.:	971026
5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-3

Feed:	<u> 200 </u> dry grams			Hg,		U	
		% moisture	Damp wt.	mg/kg	Hg, mg	mg/kg	U, mg
		50 x 100	0.0	11.8	880	10	66
		100 x 200	0.0	25.9	830	21	40
		<200	42.6	283.0	760	123	21
							3.4

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
Solids feed:	200	121					
Total I required				0.4	39.9	0.2	30.5
Water:			480				

Heat to 50°C, adjust pH to 5.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
2:30	start	49	6.34	0
2:34	0	48.5	4.93	1.28
3:05	30	49.9	4.2	0.4
3:35	60	48	5	0
4:04	90	47.4	5.12	0
4:35	120	47.8	5.2	0

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-3-1	Residue		200	3.1	1	0.3	34	7	99.94
		mL		mg/L			mg/L		
A-3-2	Filtrate	850		220	187	100	0.005	0.004	0.06
	Balance, versus feed					121			130

COLORADO MINERALS RESEARCH INSTITUTE		CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		
Metallurgical & Mineral Processing Consultants			Project No.:	971026
5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-4

Feed:	<u> 200 </u> dry grams			Hg,		U	
		% moisture	Damp wt.	mg/kg	Hg, mg	mg/kg	U, mg
		50 x 100	0.0	11.8	880	10	66
		100 x 200	0.0	25.9	830	21	40
		<200	42.6	283.0	760	123	21
							3.4

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
Solids feed:	200	121					
Total I required				0.4	39.9	0.2	30.5
Water:			480				

Heat to 50°C, adjust pH to 7.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
2:20	start	48.6	6.3	0
2:34	0	48.7	5.8	0
3:04	30	50.5	6	0
3:34	60	48.6	6.1	0
4:04	90	47.3	6.12	0
4:34	120	47.6	6.1	0

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-4-1	Residue		200	2.8	1	0.3	40	8	99.94
		mL		mg/L			mg/L		
A-4-2	Filtrate	645		345	223	100	0.007	0.005	0.056
	Balance, versus feed					144			153

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Metallurgical & Mineral Processing Consultants			Project No.:	971026
5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-5

Feed:	<u> 200 </u> dry grams			Hg,		U	
		% moisture	Damp wt.	mg/kg	Hg, mg	mg/kg	U, mg
		50 x 100	0.0	11.8	880	10	66
		100 x 200	0.0	25.9	830	21	40
		<200	42.6	283.0	760	123	21
							3.4

Leach:	25% solids							
			Liquid		Potassium Iodide		Iodine	
		dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
	Solids feed:	200.14	121	480	0.025	2.5	0.0125	1.9
	Total I required							
Water:								

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
10:05	start	48	6.40	0.0
10:10	0	48	4.00	2.5
10:40	30	50	5.02	0.5
11:10	60	52	5.05	0.4
11:40	90	52	4.78	0.2
12:05	120	54	4.40	

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-5-1	Residue		205	140	29	19.6	32	7	99.99
		mL		mg/L			mg/L		
A-5-2	Filtrate	735		160	118	80	0.001	0.001	0.011
	Balance, versus feed					94			126

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5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-6

Feed:	dry grams	% moisture		Damp wt.	Hg,	Hg, mg	U	U, mg
		50 x 100	0.0		mg/kg		mg/kg	
		100 x 200	0.0	25.9	830	21	40	1.0
		<200	42.6	283.0	760	123	21	3.4

Leach:		25% solids		Liquid		Potassium Iodide		Iodine	
	dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g		
Solids feed:	200	121							
Total I required				0.05	5.0	0.025	3.8		
Water:			480						

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
10:05	start	48	6.08	0.0
10:10	0	48	4.00	1.5
10:40	30	50	5.00	0.5
11:10	60	52	5.18	0.5
11:40	90	52	4.72	0.3
12:10	120	54	4.50	

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-6-1	Residue		212	73	15	5.9	32	7	99.99
		mL		mg/L			mg/L		
A-6-2	Filtrate	720		340	245	94	0.001	0	0.01
	Balance, versus feed					168			130

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Metallurgical & Mineral Processing Consultants			Project No.:	971026
5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-7

Feed:	<u> 200 </u> dry grams			Hg,		U	
		% moisture	Damp wt.	mg/kg	Hg, mg	mg/kg	U, mg
		50 x 100	0.0	11.8	880	10	66
		100 x 200	0.0	25.9	830	21	40
		<200	42.6	283.0	760	123	21
							3.4

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
Solids feed:	200	121					
Total I required				0.1	10.0	0.05	7.6
Water:			480				

Heat to 50°C, adjust pH to 5.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
10:10	start	48	6.97	0.0
10:15	0	48	4.97	1.0
10:45	30	49	5.75	0.2
11:15	60	50	5.60	0.2
11:45	90	50	5.43	0.2
12:15	120	50	5.30	

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-7-1	Residue		206	39	8	5.0	39	8	99.91
		mL		mg/L			mg/L		
A-7-2	Filtrate	1390		110	153	95	0.005	0.007	0.09
	Balance, versus feed					104			154

COLORADO MINERALS RESEARCH INSTITUTE		CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		
Metallurgical & Mineral Processing Consultants			Project No.:	971026
5906 McIntyre Street	(303) 279-2581		Date:	10/7/97
Golden, CO 80403	FAX 279-6061			

Test No: A-8

Feed:	<u> 200 </u> dry grams			Hg,		U	
		% moisture	Damp wt.	mg/kg	Hg, mg	mg/kg	U, mg
		50 x 100	0.0	11.8	880	10	66
		100 x 200	0.0	25.9	830	21	40
		<200	42.6	283.0	760	123	21
							3.4

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry, g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
Solids feed:	200	121					
Total I required				0.1	10.0	0.05	7.6
Water:			480				

Heat to 50°C, adjust pH to 7.0 with 1000 g/L H₂SO₄

Leach 2.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H ₂ SO ₄
10:10	start	48	6.97	
10:15	0	48	6.97	0
10:45	30	49	6.05	0
11:15	60	50	6.00	0
11:45	90	50	6.00	0
12:15	120	50	5.96	

Sample ID		Total wet grams	Dry Wt. grams	Hg mg/kg	Hg mg	Hg, % Distn	U mg/kg	U mg	U, % Distn
A-8-1	Residue		202	42	8	4.8	38	8	99.03
		mL		mg/L			mg/L		
A-8-2	Filtrate	1535		110	169	95	0.049	0.1	0.97
	Balance, versus feed					114			148

Appendix A-2

Data Sheets for Bench-Scale Extractions of Fluorescent Lamps

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Test No: G-1 2 Hours 20 °C

		% moisture	Damp wt.
Feed:	<u> 50 </u> dry grams	<u><65-mesh</u> 15.0	59.0

Leach:		25% solids					
Solids feed: Total I required Water:	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
	<u> 50 </u>	<u> 9 </u>		0.04	1.00	0.02	0.76
			141				

Heat to target temperature, add KI and I₂

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:40	start	17	9.7
9:45	0		9.4
10:15	30	16	6.8
11:00	60	15	7.1
11:15	90	15	7.2
11:45	120	15	7.2

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.5	420	21.21		
G-1-1	Residue	59	50.45	19.8	1.00	4.7%	
		mL		mg/L			
G-1-2	Filtrate	305		66.6	20.31	95.3%	0.0058
Balance, versus feed						100.5%	

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Test No: G-2 6 Hours 20 °C

	% moisture	Damp wt.
Feed: 50 dry grams	<65-mesh	15.0
		59.0

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	50	9	141	0.04	1.00	0.02	0.76
Total I required							
Water:							

Heat to target temperature, add KI and I₂
Leach for desired time
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:40	start	17	9.7
9:45	0		9.5
10:15	30	16	6.8
11:00	60	15	7.1
11:15	90	15	7.2
	120		
	180		
13:30	240	15	7.4
14:45	300	16	7.5
15:50	360	16	7.5

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.2	420	21.08		
G-2-1	Residue	59	50.16	12.6	0.63	2.9%	
		mL	mg/L				
G-2-2	Filtrate	300		70.2	21.06	97.1%	0.0030
Balance, versus feed							102.9%

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Test No: G-3 24 Hours 20 °C

		% moisture	Damp wt.
Feed:	50 dry grams	<65-mesh 15.0	59.0

Leach: 25% solids

		Liquid	Potassium Iodide	Iodine			
	dry g	grams	mL	KL, M	KL, g	I2, M	I2, g
Solids feed:	50	9		0.04	1.00	0.02	0.76
Total I required			141				
Water:							

Heat to target temperature, add KI and I2
Leach for desired time
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:40	start	17	9.7
9:45	0		9.5
10:15	30	16	6.8
11:00	60	15	7.1
11:15	90	15	7.2
	120		
	180		
13:30	240	15	7.4
14:45	300	16	7.5
15:50	360	16	7.5
17:00	480	18	7.4
9:30	1440	18	7.6

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.5	420	21.21		
G-3-1	Residue	58	50.46	14.3	0.72	3.4%	
		mL	mg/L				
G-3-2	Filtrate	275		74.8	20.57	96.6%	0.0005
Balance, versus feed						100.4%	

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Test No: G-4 2 Hours 35 °C

		% moisture	Damp wt.
Feed:	<u> 50 </u> dry grams	<u><65-mesh</u>	<u> 15.0 </u>
			<u> 59.0 </u>

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	<u> 50 </u>	<u> 9 </u>					
Total I required				<u> 0.04 </u>	<u> 1.00 </u>	<u> 0.02 </u>	<u> 0.76 </u>
Water:			<u> 141 </u>				

Heat to target temperature, add KI and I2

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:15	start	40	9.7
9:20	0	39	6.7
9:50	30	40	6.8
10:20	60	35	7.2
10:50	90	33	7.4
11:20	120	31	7.4

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	<u> 59 </u>	<u> 50.8 </u>	<u> 420 </u>	<u> 21.34 </u>		
G-4-1	Residue	<u> 57 </u>	<u> 50.77 </u>	<u> 14.4 </u>	<u> 0.73 </u>	<u> 4% </u>	
		<u> mL </u>	<u> mg/L </u>				
G-4-2	Filtrate	<u> 275 </u>		<u> 71.4 </u>	<u> 19.64 </u>	<u> 96% </u>	<u> 0.0014 </u>
Balance, versus feed						<u> 95.5% </u>	

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Test No: G-5 6 Hours 35 °C

		% moisture	Damp wt.
Feed:	<u> 50 </u> dry grams	<u><65-mesh</u>	<u> 15.0 </u>
			<u> 59.0 </u>

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
		dry g	grams	mL	KI, M	KI, g	I2, M
Solids feed:	<u> 50 </u>	<u> 9 </u>					
Total I required				0.04	1.00	0.02	0.76
Water:			141				

Heat to target temperature, add KI and I2

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:15	start	40	9.7
9:20	0	39	7.4
9:50	30	40	6.9
10:20	60	35	7.2
10:50	90	33	7.4
11:20	120	31	7.4
12:00	180	38	7.4
13:30	240	32	7.6
14:45	300	36	7.6
15:25	360	32	7.7

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.8	420	21.34		
G-5-1	Residue	58	50.81	22.4	1.14	6%	
		mL		mg/L			
G-5-2	Filtrate	290		67.0	19.43	94%	0.0002
Balance, versus feed						96.4%	

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Test No: G-6 6 Hours 35 °C

		% moisture	Damp wt.
Feed:	<u> 50 </u> dry grams	<u><65-mesh</u>	<u> 15.0 </u>
			<u> 59.0 </u>

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
		dry g	grams	mL	KI, M	KI, g	I2, M
Solids feed:	<u> 50 </u>	<u> 9 </u>					
Total I required				<u> 0.04 </u>	<u> 1.00 </u>	<u> 0.02 </u>	<u> 0.76 </u>
Water:			<u> 141 </u>				

Heat to target temperature, add KI and I2

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:15	start	40	9.7
9:20	0	39	7.4
9:50	30	40	6.9
10:20	60	35	7.2
10:50	90	33	7.4
11:20	120	31	7.4
12:00	180	38	7.4
13:30	240	32	7.6
14:45	300	36	7.6
15:25	360	32	7.7

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	<u> 59 </u>	<u> 50.9 </u>	<u> 420 </u>	<u> 21.38 </u>		
G-6-1	Residue	<u> 57 </u>	<u> 50.86 </u>	<u> 27.4 </u>	<u> 1.39 </u>	<u> 7% </u>	
		<u> mL </u>		<u> mg/L </u>			
G-6-2	Filtrate	<u> 295 </u>		<u> 65.6 </u>	<u> 19.35 </u>	<u> 93% </u>	<u> 0.0002 </u>
Balance, versus feed						<u> 97.0% </u>	

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Test No: G-7 2 Hours 50 °C

			% moisture	Damp wt.
Feed:	<u> 50 </u>	dry grams	<u><65-mesh</u>	<u> 15.0 </u>
				<u> 59.0 </u>

Leach:		25% solids				
		Liquid		Potassium Iodide		Iodine
	dry g	grams	mL	KI, M	KI, g	I2, M
Solids feed:	<u> 50 </u>	<u> 9 </u>				
Total I required				0.04	1.00	0.02
Water:			141			0.76

Heat to target temperature, add KI and I2

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:20	start	46	7.8
	0		
9:50	30	52	6.8
10:20	60	52	7.2
10:50	90	52	7.5
11:20	120	52	7.6

Checked with Another Meter

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.9	420	21.38		
G-7-1	Residue	58	50.91	25.6	1.30	6%	
		mL		mg/L			
G-7-2	Filtrate	275		72.6	19.97	94%	0.0001
	Balance, versus feed					99.5%	

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Test No: G-8 6 Hours 50 °C

		% moisture	Damp wt.
Feed:	<u> 50 </u> dry grams	<u><65-mesh</u>	<u> 15.0 </u>
			<u> 59.0 </u>

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
Solids feed:	<u> 50 </u>	<u> 9 </u>					
Total I required				<u> 0.04 </u>	<u> 1.00 </u>	<u> 0.02 </u>	<u> 0.76 </u>
Water:			<u> 141 </u>				

Heat to target temperature, add KI and I₂

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:20	start	46	7.8
9:20	0		
9:50	30	52	6.9
10:20	60	52	7.2
10:50	90	52	7.5
11:20	120	52	7.6
12:00	180	50	8
13:20	240	50	8.6
14:45	300	50	8.7
15:25	360	50	8.8

Checked with Another Meter

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	<u> 59 </u>	<u> 49.8 </u>	<u> 420 </u>	<u> 20.92 </u>		
G-8-1	Residue	<u> 61 </u>	<u> 49.81 </u>	<u> 47.6 </u>	<u> 2.37 </u>	<u> 12% </u>	
		<u> mL </u>		<u> mg/L </u>			
G-8-2	Filtrate	<u> 275 </u>		<u> 64.2 </u>	<u> 17.66 </u>	<u> 88% </u>	<u> 0 </u>
Balance, versus feed						<u> 95.7% </u>	

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET	Project No.: 971026 Date: 2/9/98
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Test No: G-9 2 Hours 20 °C

		% moisture		Damp wt.
Feed:	<u> 50 </u> dry grams	<u><65-mesh</u>	<u> 15.0 </u>	<u> 59.0 </u>

		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g
Solids feed:	<u> 50 </u>	<u> 9 </u>					
Total I required				0.08	1.99	0.04	1.52
Water:			<u> 141 </u>				

Heat to target temperature, add KI and I₂

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:30	start	20	10.1
10:00	0	20	6.7
	30		
11:00	60	20	7.1
	90		
12:00	120	19	7.2

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	<u> 59 </u>	<u> 50.6 </u>	<u> 420 </u>	<u> 21.25 </u>		
G-9-1	Residue	<u> 57.1 </u>	<u> 50.63 </u>	<u> 8.5 </u>	<u> 0.43 </u>	<u> 1.9% </u>	
		<u> mL </u>	<u> mg/L </u>				
G-9-2	Filtrate	<u> 265 </u>		<u> 85.0 </u>	<u> 22.53 </u>	<u> 98.1% </u>	<u> 0.0125 </u>
Balance, versus feed						<u> 108.0% </u>	

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Test No: G-10 4 Hours 20 °C

			% moisture	Damp wt.	
Feed:	50	dry grams	<65-mesh	15.0	59.0

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	50	9	141	0.08	1.99	0.04	1.52
Total I required							
Water:							

Heat to target temperature, add KI and I₂

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:30	start	20	10.1
10:00	0		6.7
	30		
11:00	60	20	7.1
	90		
12:00	120	19	7.2
13:15	180	20	7.25
14:00	240	20	7.2

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	51.1	420	21.46		
G-10-1	Residue	58	51.04	6.7	0.34	1.5%	
		mL	mg/L				
G-10-2	Filtrate	257		86.6	22.26	98.5%	0.0106
Balance, versus feed						105.3%	

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		Project No.: 971026 Date: 2/9/98
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Test No: G-11 6 Hours 20 °C

		% moisture	Damp wt.
Feed:	50 dry grams	<65-mesh 15.0	59.0

Leach:		25% solids				
		Liquid		Potassium Iodide		Iodine
		grams	mL	KI, M	KI, g	I ₂ , M I ₂ , g
Solids feed:	50	9				
Total I required				0.08	1.99	0.04 1.52
Water:			141			

Heat to target temperature, add KI and I₂
Leach for desired time
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH
9:30	start	20	10.1
10:00	0		6.7
	30		
11:00	60	20	7.1
	90		
12:00	120	19	7.2
13:15	180	20	7.25
14:00	240	20	7.2
	300	20	
15:55	360	20	7.3

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.8	420	21.34		
G-11-1	Residue	58.2	50.8	7.3	0.37	1.7%	
		mL		mg/L			
G-11-2	Filtrate	255		86.4	22.03	98.3%	0.0094
Balance, versus feed						105.0%	

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Test No: G-12 2 Hours 35 °C

		% moisture	Damp wt.
Feed:	50 dry grams	<65-mesh 15.0	59.0

Leach:		25% solids				
Solids feed: Total I required Water:	dry g	Liquid		Potassium Iodide		Iodine
		grams	mL	KI, M	KI, g	I ₂ , M
	50	9		0.08	1.99	0.04
			141			1.52

Heat to target temperature, add KI and I₂
Leach for desired time
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH
10:00	start	35	10.1
10:15	0	34	6.4
	30		
11:00	60	34	7.0
	90		
12:15	120	34	7.1

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.3	420	21.13		
G-12-1	Residue	58	50.31	8.4	0.42	1.8%	
		mL	mg/L				
G-12-2	Filtrate	262		86.6	22.69	98.2%	0.0087
Balance, versus feed		109.4%					

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Test No: G-13 4 Hours 35 °C

		% moisture	Damp wt.
Feed:	<u>50</u> dry grams	<u><65-mesh</u>	<u>15.0</u>
			<u>59.0</u>

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
		dry g	grams	mL	KI, M	KI, g	I ₂ , M
Solids feed:	<u>50</u>	<u>9</u>					
Total I required				<u>0.08</u>	<u>1.99</u>	<u>0.04</u>	<u>1.52</u>
Water:			<u>141</u>				

Heat to target temperature, add KI and I₂

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
10:00	start	35	10.1
10:15	0	34	6.4
	30		
11:00	60	34	7.0
	90		
12:15	120	34	7.1
13:15	180	34	7.15
14:15	240	34	7.2

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	<u>59</u>	<u>50.6</u>	<u>420</u>	<u>21.25</u>		
G-13-1	Residue	<u>55.8</u>	<u>50.59</u>	<u>9.2</u>	<u>0.47</u>	<u>2.0%</u>	
		<u>mL</u>		<u>mg/L</u>			
G-13-2	Filtrate	<u>270</u>		<u>83.8</u>	<u>22.63</u>	<u>98.0%</u>	<u>0.0061</u>
Balance, versus feed					<u>108.7%</u>		

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Test No: G-14 2 Hours 20 °C

		% moisture	Damp wt.
Feed:	50 dry grams	<65-mesh 15.0	59.0

Leach:		25% solids				
Solids feed: Total I required Water:	dry g	Liquid		Potassium Iodide		Iodine
		grams	mL	KI, M	KI, g	I ₂ , M
	50	9				I ₂ , g
			141	0.12	2.99	0.06
						2.28

Heat to target temperature, add KI and I₂
Leach for desired time
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH
12:00	start	13	11.0
	0	13	6.8
12:30	30	13	6.8
13:00	60	23	7.2
13:30	90	22	7.2
14:00	120	22	7.3

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.4	420	21.17		
G-14-1	Residue		50.4	10.8	0.54	2.6%	
		mL	mg/L				
G-14-2	Filtrate	297		70.0	20.79	97.4%	0.0002
Balance, versus feed						100.8%	

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Test No: G-15 4 Hours 50 °C

		% moisture		Damp wt.	
Feed:	50	dry grams	<65-mesh	15.0	59.0

Leach:		25% solids					
Solids feed: Total I required Water:		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g
	50	9	141	0.12	2.99	0.06	2.28

Heat to target temperature, add KI and I₂

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
12:00	start	13	11.0
12:00	0	13	6.8
12:30	30	13	6.8
13:00	60	20	7.1
13:30	90	20	7.3
14:00	120	19	7.3
15:00	180	19	7.5
16:00	240	19	7.7

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
G-15-1	Feed	59	51.2	420	21.50		
	Residue		51.2	8.5	0.44	2.3%	
		mL	mg/L				
G-15-2	Filtrate	300		62.0	18.60	97.7%	0.0001
Balance, versus feed						88.5%	

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET	Project No.: 971026 Date: 2/19/98
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Test No: G-16 2 Hours 20 °C

		% moisture	Damp wt.
Feed:	50 dry grams	<65-mesh 15.0	59.0

Leach:		25% solids				
Solids feed: Total I required Water:	dry g	Liquid		Potassium Iodide		Iodine
		grams	mL	KI, M	KI, g	I ₂ , M
	50	9		0.08	1.99	0.06
			141			2.28

Heat to target temperature, add KI and I₂
Leach for desired time
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH
12:00	start	13	11.0
12:00	0	13	6.8
12:30	30	13	6.8
13:00	60	20	7.1
13:30	90	20	7.2
14:00	120	19	7.3

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn	I ₂ , M
	Feed	59	50.7	420	21.29		
G-16-1	Residue		50.7	12.8	0.65	3.2%	
		mL	mg/L				
G-16-2	Filtrate	290		68.0	19.72	96.8%	0
Balance, versus feed						95.7%	

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		Project No.: 971026 Date: 2/19/98
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Test No: G-17 4 Hours 20 °C

		% moisture		Damp wt.	
Feed:	50	dry grams	<35-mesh	15.0	59.0

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	50	9	141	0.08	1.99	0.06	2.28
Total I required							
Water:							

Heat to target temperature, add KI and I2

Leach for desired time

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH
12:00	start	13	11.0
12:00	0	13	6.8
12:30	30	13	6.8
13:00	60	20	7.1
13:30	90	20	7.3
14:00	120	19	7.3
15:00	180	19	7.5
16:00	240	19	7.5

Residue rinse

Sample ID		Total wet grams	Calc dry grams	Hg mg/kg	Hg mg	Hg, % Distn
G-17-1	Feed	59	51.1	420	21.46	
	Residue		51.1	11.4	0.58	2.7%
G-17-2		mL	mg/L			
	Filtrate	288		72.0	20.74	97.3%
Balance, versus feed						99.3%

Appendix A-3

Data Sheets for Locked-Cycle Testing of Fluorescent Lamps

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date: 2/24/98
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Run No: 1 Date Started: 24-Feb-98
Cycle No: 1 Date Finished: _____
Test No. 1-1

Feed: 458.5 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	580	420	193

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I ₂ , M	I ₂ , g	I ₂ , g	KI, g
Solids feed:	458.5	122							
Total KI reqd:				0.08	18.3				18.3
Recycle KI Soln:					0				
Total Iodine reqd:						0.04	14.0	14.0	
Recycled Iodine									
Water:		1254							

Adjust pH to 7.6 with 500 g/L sulfuric acid
Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
Transfer to vacuum filter, filter, wash with 550 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

		pH		500 g/L H ₂ SO ₄	
Time	Minutes	read	adjust	mV	mL
11:30	start	11.9	7.6		1.5
11:40	0	7.15		386	
12:00	60	7.33			
13:40	120	7.36			
15:40	240	7.53			

		Hg				I ₂		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-1-1	Filtrate	1620	130	210.6	98.3%				
1-1-2	Residue	460.2	7.8	3.6	1.7%				
Filtrate sample		78	Balance=		111%				

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	2/24/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 1 Date Started: 24-Feb-98
 Cycle No: 1 Date Finished: _____
 Test No. 1-1

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1560 mL
 Adjust solution pH to _____ with _____ mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
9:30	start		20		
10:15	advance		20		
10:45	30		20		
11:15	60		20		
11:35	90		20		
	120				
	Composite				

		Hg		I ₂		KI	
Sample ID	vol	mg/L	mg	g/L	g	g/L	g
1-1-3	Discharge	1520	2.3	3.5			
Discharge sample		50					

Iron Precipitation

Charge precipitation vessel 1470 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
	start				
	0				
	10				
	20				
	30				

			Hg			I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-1-4	Filtrate	1500		0.0	0%			18.3	
1-1-5	Precipitate	2.3	855	2.0	100%				
Filtrate sample									

1-1-5 is 30% Fe

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No: 971026-6 Date: 2/24/98
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Run No: 1 Date Started: 24-Feb-98
 Cycle No: 1 Date Finished: _____
 Test No. 1-1

Volume of iron precipitation filtrate available to advance mL g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain grams potassium iodide to use in next cycle
 Or a maximum volume of mL
 Volume advanced to recycle mL containing g KI

Iodine Recovery

Measure sufficient filtrate to contain grams potassium iodide for oxidation to I₂
 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

g H₂SO₄/g KI
 g H₂SO₄
 mL 1000 g/L
 g H₂O₂/g KI
 g H₂O₂
 mL 35% H₂O₂

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH read	adjusted
0						
0						
15						
30						

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-1-6	Filtrate						18.3	
1-1-7	Precipitate							
Filtrate sample								

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc 971026-6 Date: 2/24/98
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Run No: 1 Date Started: 25-Feb-98
Cycle No: 2 Date Finished: 26-Feb-98
Test No. 1-2

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	420	168

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				-4.1
Recycle KI Soln:			1094	18.3	20.0				
Total Iodine reqd:						0.04	12.2	12.2	
Recycled Iodine									
Water:		0							

Adjust pH to 6.8 with 500 g/L sulfuric acid
Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Time	Minutes	pH		mV	500 g/L H2SO4
		read	adjust		mL
	start	8.5	6.8		1.1
8:30	0	6.8		240	
9:30	60	6.5		356	
	120				
12:30	240	6.9		390	

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-2-1	Filtrate	2165	48	103.9	96.6%				
1-2-2	Residue	402.5	9.0	3.6	3.4%				
Filtrate sample		180	Balance=		64%				

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No: 971026-6 Date: 2/24/98
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Run No: 1
 Cycle No: 2
 Test No: 1-2

Date Started: 25-Feb-98
 Date Finished: 26-Feb-98

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 2000 mL
 Adjust solution pH to **4.0** with 0.2 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
	start				
	advance				
	30				
	60				
	90				
	120				
	Composite				

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-2-3	Discharge			0.0				
Discharge sample								

Iron Precipitation

Charge precipitation vessel mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
16:20	start		4.4		0.73
16:30	0		6.3	11.6	4.13
	10				
	20				
17:05	30		12.0		

Sample ID		wt/vol	Hg		Distn	I ₂		KI	
			mg/kg-L	mg		g/L	g	g/L	g
1-2-4	Filtrate	1930	0.05	0.1	4%				
1-2-5	Precipitate	5.8	405	2.3	96%				
Filtrate sample		82							

1-2-5 is 30% Fe

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date: 2/27/98
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Run No: 1 Date Started: 27-Feb-98
Cycle No: 3 Date Finished: _____
Test No. 1-3

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	420	168

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I ₂ , M	I ₂ , g	I ₂ , g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				-3.4
Recycle KI Soln:			860	22.5	19.4				
Total Iodine reqd:						0.04	12.2	-0.7	
Recycled Iodine							12.9		
Water:		234							

Adjust pH to 7.0 with 500 g/L sulfuric acid
Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

		pH		500 g/L H ₂ SO ₄	
Time	Minutes	read	adjust	mV	mL
10:45	start	12.17	6.6		2.6
10:50	0	5.99			
11:40	60	6.88		410	
13:00	120	7.17		416	
15:00	240	7.23		409	

DI water wash, 400 mL

		Hg				I ₂		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-3-1	Filtrate	1600	100	160.0	97.9%				
1-3-2	Residue	402.6	8.5	3.4	2.1%				
Filtrate sample		100	Balance=		97%				

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	2/27/98
5906 McIntyre Street Golden, CO 80403	(303) 279-2581 FAX 279-6061			

Run No: 1 Date Started: 27-Feb-98
 Cycle No: 3 Date Finished: _____
 Test No. 1-3

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1500 mL
 Adjust solution pH to **4.0** with 0.2 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
10:20	start		25	7.0/3.3	
10:55	advance		21	3.4	3.57
11:30	30		22	3.2	
	60				
	90				
	120				
12:15	Composite			4.9	293

		Hg		I ₂		KI	
Sample ID	vol	mg/L	mg	g/L	g	g/L	g
1-3-3	Discharge	1400	0.05	0.1			
Discharge sample		60					

Iron Precipitation

Charge precipitation vessel 1340 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes
 Transfer to vacuum filter, filter, and wash 20 mL
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
	start		4.9	11.3	2.8
12:30	0		11.3		
	10				
12:50	20		12.1		
13:00	30		12.1		

		Hg				I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-3-4	Filtrate	1360	0.05	0.1	2%				
1-3-5	Precipitate	4.2	745	3.1	98%				
Filtrate sample		60							

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No: 971026-6 Date: 2/27/98
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Run No: 1 Date Started: 27-Feb-98
 Cycle No: 3 Date Finished: _____
 Test No: 1-3

Volume of iron precipitation filtrate available to advance 1300 mL 17.0 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 935 mL containing 15.9 g KI

Iodine Recovery

Measure sufficient filtrate to contain 6.2 grams potassium iodide for oxidation to I₂
365 mL neutralization filtrate to iodine recovery
6.2 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI
2.6 g H₂SO₄
2.6 mL 1000 g/L
 Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction 0.23 g H₂O₂/g KI
1.43 g H₂O₂
3.60 mL 35% H₂O₂
 g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
 Stir for 60 minutes, settle, decant, filter solids, do not wash
 Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
 Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
14:01	0	2.6			12.1	0.83
14:40	0		3.6		1.2	
	15					
15:10	30				1.5	

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-3-6	Filtrate	375	0.05					
1-3-7	Precipitate	3.7						
Filtrate sample		75						

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date: 3/3/98
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Run No: 1 Date Started: 3-Mar-98
Cycle No: 4 Date Finished: 4-Mar-98
Test No. 1-4

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	420	168

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				0.0
Recycle KI Soln:			935	17.0	15.9				
Total Iodine reqd:						0.04	12.2	8.5	
Recycled Iodine							3.7		
Water:		159							

Adjust pH to 6.9 with 500 g/L sulfuric acid
Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Time	Minutes	pH		mV	500 g/L H2SO4
		read	adjust		mL
	start				
8:30	0	10.4	6.9	394	1.4
9:20	60	6.7		396	
11:00	120	7.1		398	
12:30	240	7.3		398	

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-4-1	Filtrate	2365	76	179.7	98.1%				
1-4-2	Residue	398	8.8	3.5	1.9%				
Filtrate sample		100	Balance=		109%				

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No: 971026-6 Date: 3/3/98
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Run No: 1
 Cycle No: 4
 Test No: 1-4

Date Started: 3-Mar-98
 Date Finished: 4-Mar-98

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 2264 mL
 Adjust solution pH to **4.0** with 0.6 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
13:15	start		23	3.7	410
13:45	advance		21	3.85	405
	30				
	60				
	90				
15:45	120			4.0	385
	Composite				

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-4-3	Discharge	2230	0.05	0.1				
	Discharge sample	100						

Iron Precipitation

Charge precipitation vessel 2130 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
16:00	start		3.9	8.9	2.5
16:05	0		8.9		
16:15	10		8.5	10.3	0.4
	20				
16:35	30		11.1		

Sample ID		wt/vol	Hg		Distn	I ₂		KI	
			mg/kg-L	mg		g/L	g	g/L	g
1-4-4	Filtrate	2170	0.05	0.1	1%				
1-4-5	Precipitate	6.6	1125	7.4	99%				
	Filtrate sample	70							

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No: 971026-6 Date: 3/3/98
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Run No: 1
 Cycle No: 4
 Test No: 1-4

Date Started: 3-Mar-98
 Date Finished: 4-Mar-98

Volume of iron precipitation filtrate available to advance 2100 mL 14.0 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 1094 mL containing 15.3 g KI

Iodine Recovery

Measure sufficient filtrate to contain 14.1 grams potassium iodide for oxidation to I₂
1006 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42	g H ₂ SO ₄ /g KI
5.9	g H ₂ SO ₄
5.9	mL 1000 g/L
0.23	g H ₂ O ₂ /g KI
3.24	g H ₂ O ₂
8.2	mL 35% H ₂ O ₂

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
9:15	0	5.9	8.2		10.2	1.0
9:20	0				1.0	
	15					
10:35	30				1.4	

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-4-6	Filtrate	1005	0.05					
1-4-7	Precipitate	10.4						
Filtrate sample		100						

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 4-Mar-98
 Cycle No: 5 Date Finished: 6-Mar-98
 Test No: 1-5

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	400	400	160

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	0							
Total KI reqd:				0.08	15.9				0.6
Recycle KI Soln:			1094	14.0	15.3				
Total Iodine reqd:						0.04	12.2	1.8	
Recycled Iodine							10		
Water:		106							

Adjust pH to 7.0 with 500 g/L sulfuric acid
 Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
 Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
 Measure volume of filtrate plus wash, transfer to cementation
 Take sample of filtrate for analysis

Time	Minutes	pH		mV	500 g/L H2SO4
		read	adjust		mL
11:30	start	8.9	6.9	412	0.5
11:35	0	6.9		401	
	60				
14:00	120	7.4		382	0.1
15:30	240	7.2		382	

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-5-1	Filtrate	1760	100	176.0	97.8%				
1-5-2	Residue	405	9.7	3.9	2.2%				
Filtrate sample		60	Balance=		112%				

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date:
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Run No: 1 Date Started: 4-Mar-98
 Cycle No: 5 Date Finished: 6-Mar-98
 Test No. 1-5

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1700 mL
 Adjust solution pH to **4.0** with mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
8:25	start			3.2	
9:00	advance			3.3	
	30				
	60				
	90				
11:00	120			3.9	
	Composite				

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-5-3	Discharge	1700	0.05	0.1				
	Discharge sample	50						

Iron Precipitation

Charge precipitation vessel 1650 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
11:10	start		3.9	10.7	2.62
11:17	0		10.7		
	10				
	20				
12:05	30		11.3		

Sample ID		wt/vol	Hg		Distn	I ₂		KI	
			mg/kg-L	mg		g/L	g	g/L	g
1-5-4	Filtrate	1660	0.05	0.1	1%				
1-5-5	Precipitate	3.95	1430	5.6	99%				
	Filtrate sample	60							

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 4-Mar-98
 Cycle No: 5 Date Finished: 6-Mar-98
 Test No. 1-5

Volume of iron precipitation filtrate available to advance 1600 mL 17.5 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 909 mL containing 15.9 g KI

Iodine Recovery

Measure sufficient filtrate to contain 12.1 grams potassium iodide for oxidation to I₂
691 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI
5.1 g H₂SO₄
5.0 mL 1000 g/L
 Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction 0.23 g H₂O₂/g KI
2.8 g H₂O₂
7.0 mL 35% H₂O₂
 g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
 Stir for 60 minutes, settle, decant, filter solids, do not wash
 Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
 Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
8:35	0	5.0	7.0	16	9.7	1.1
8:40	0			15	1.2	
8:55	15			15	1.2	
9:10	30			15	1.3	

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-5-6	Filtrate	690	0.6				0	
1-5-7	Precipitate	8.6						
Filtrate sample		40						

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date:
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Run No: 1 Date Started: 20-Mar-98
Cycle No: 6 Date Finished: 24-Mar-98
Test No. 1-6

Feed: 400 dry grams % moisture 21 Damp wt. 506 Hg, mg/kg 400 Hg, mg 160

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				0.0
Recycle KI Soln:			909	17.5	15.9				
Total Iodine reqd:						0.04	12.2	3.6	
Recycled Iodine							9		
Water:		185					Total iodine		24.4

Adjust pH to 7.0 with 500 g/L sulfuric acid
Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

		pH		500 g/L H2SO4	
Time	Minutes	read	adjust	mV	mL
11:10	start	9.7	6.9	405	1.0
11:15	0	6.9		404	
12:15	60	7.2		400	
13:15	120	7.4	6.9	401	0.2
14:30	240	7.3	6.8	398	0.1

		Hg				I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-6-1	Filtrate	1500	130	195.0	97.6%	2.8	4.2	11	16.5
1-6-2	Residue	404	11.8	4.8	2.4%				
Filtrate sample		50	Balance=		125%	Total iodine		16.8	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No. 971026-6
Metallurgical & Mineral Processing Consultants			Date:
5906 McIntyre Street Golden, CO 80403	(303) 279-2581 FAX 279-6061		

Run No: 1 Date Started: 20-Mar-98
 Cycle No: 6 Date Finished: 24-Mar-98
 Test No. 1-6

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1450 mL
 Adjust solution pH to **4.0** with 0.6 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
8:15	start	19	20	3.9	410
8:40	advance	19	20	3.9	410
8:55	30	19	20	3.9	398
9:15	60	19	20	3.7	392
9:45	90	19	20	3.3	392
10:25	120	19	20	3.3	392
	Composite	22		4.8	310

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-6-3	Discharge	1460	3.5	5.1	0	0	21.5	31.4
	Discharge sample	60					Total iodine	24.0

Iron Precipitation

Charge precipitation vessel 1400 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes
 Transfer to vacuum filter, filter, and wash Added 5 mL flocculant
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
11:00	start	22	4.85	8.6	1.0
11:05	0	21	7.6	11.1	0.5
11:15	10	21	11.3		
11:25	20	20	11.3		
11:35	30	20	11.1		

Sample ID		wt/vol	Hg		Distn	I ₂		KI	
			mg/kg-L	mg		g/L	g	g/L	g
1-6-4	Filtrate	1420	0.4	0.6	17%	0	0	13	18.5
1-6-5	Precipitate	2.6	1105	2.9	83%				
	Filtrate sample	60						Total iodine	14.1

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 20-Mar-98
 Cycle No: 6 Date Finished: 24-Mar-98
 Test No. 1-6

Volume of iron precipitation filtrate available to advance 1360 mL 13.5 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 1094 mL containing 14.8 g KI

Iodine Recovery

Measure sufficient filtrate to contain 3.6 grams potassium iodide for oxidation to I₂
266 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI
1.5 g H₂SO₄
1.5 mL 1000 g/L
 Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction 0.23 g H₂O₂/g KI
0.8 g H₂O₂
2.1 mL 35% H₂O₂
 g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
 Stir for 60 minutes, settle, decant, filter solids, do not wash
 Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
 Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
14:30	0	1.5	2.1	22	10.7	1.1
14:35	0			22	1.1	
14:50	15			22	1.3	
15:05	30			21	1.4	

			Hg		I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-6-6	Filtrate	350	0.5		0	0	0	0
1-6-7	Precipitate	1.15						
Filtrate sample		50	Total iodine 0.0					

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 24-Mar-98
 Cycle No: 7 Date Finished: 27-Mar-98
 Test No: 1-7

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	400	160

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				2.3
Recycle KI Soln:			1094	12.5	13.7				
Total Iodine reqd:						0.04	12.2	11.0	
Recycled Iodine							1.2		
Water:		0					Total iodine		24.4

Adjust pH to 7.0 with 500 g/L sulfuric acid
 Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
 Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
 Measure volume of filtrate plus wash, transfer to cementation
 Take sample of filtrate for analysis

Time	Minutes	pH		mV	500 g/L H2SO4
		read	adjust		mL
8:30	start	9.65	6.9	399	1.0
8:40	0	6.9		405	
9:40	60	7.0		412	
10:40	120	7.0		411	
12:40	240	7.15		415	

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-7-1	Filtrate	1570	120	188.4	97.5%	5.8	9.1	10.5	16.5
1-7-2	Residue	404	11.8	4.8	2.5%				
Filtrate sample		70	Balance=		121%	Total iodine		21.7	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No. 971026-6
Metallurgical & Mineral Processing Consultants			Date:
5906 McIntyre Street Golden, CO 80403	(303) 279-2581 FAX 279-6061		

Run No: 1 Date Started: 24-Mar-98
 Cycle No: 7 Date Finished: 27-Mar-98
 Test No. 1-7

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1500 mL
 Adjust solution pH to **4.0** with 0.5 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
13:15	start	22	20	3.5	424
13:45	advance	22	20	3.7	414
14:00	30	22	20	3.4	413
14:30	60	22	20	3.3	413
15:00	90	22	20	3.2	413
15:15	120	25	20	4.3	331
	Composite	25		4.3	331

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-7-3	Discharge	1550	3.1	4.8	0	0	18	27.9
Discharge sample		50						
Total iodine								21.3

Iron Precipitation

Charge precipitation vessel 1500 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes Added 5 mL flocculant
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
15:30	start	25	4.3	11.1	2.6
15:40	0	24	11.1		
15:50	10	24	11.1		
16:00	20	23	11.1		
16:10	30	23	11.1		

			Hg			I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-7-4	Filtrate	1540	0.4	0.6	13%	0	0	15	23.1
1-7-5	Precipitate	5.2	815	4.2	87%				
Filtrate sample		40	Total iodine						17.7

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 24-Mar-98
 Cycle No: 7 Date Finished: 27-Mar-98
 Test No. 1-7

Volume of iron precipitation filtrate available to advance 1500 mL 12.5 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 1094 mL containing 13.7 g KI

Iodine Recovery

Measure sufficient filtrate to contain 5.0 grams potassium iodide for oxidation to I₂
406 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI
2.1 g H₂SO₄
2.1 mL 1000 g/L
 Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction 0.23 g H₂O₂/g KI
1.15 g H₂O₂
2.9 mL 35% H₂O₂
 g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
 Stir for 60 minutes, settle, decant, filter solids, do not wash
 Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
 Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
8:10	0	2.1	2.9	19	10.2	1.2
8:20	0			20	1.4	
8:35	15			20	1.4	
8:50	30			19	1.7	

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-7-6	Filtrate	500	0.5		4.8	2.4	0	0
1-7-7	Precipitate	4.5						
Filtrate sample		100	Total iodine				2.4	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 27-Mar-98
 Cycle No: 8 Date Finished: 30-Mar-98
 Test No: 1-8

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	400	160

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				2.3
Recycle KI Soln:			1094	12.5	13.7				
Total Iodine reqd:						0.04	12.2	7.7	
Recycled Iodine							4.5		
Water:		0					Total iodine		24.4

Adjust pH to 7.0 with 500 g/L sulfuric acid
 Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
 Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
 Measure volume of filtrate plus wash, transfer to cementation
 Take sample of filtrate for analysis

Time	Minutes	pH		mV	500 g/L H2SO4
		read	adjust		mL
9:35	start	10.2	7.0	377	1.1
9:55	0	7.0		386	
10:55	60	6.9		386	
11:55	120	6.8		388	
13:55	240	7.1		390	

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-8-1	Filtrate	1660	100	166.0	96.4%	3.3	5.5	12.5	20.8
1-8-2	Residue	389.2	15.8	6.1	3.6%				
Filtrate sample		60	Balance=		108%	Total iodine		21.3	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date:
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Run No: 1 Date Started: 27-Mar-98
 Cycle No: 8 Date Finished: 30-Mar-98
 Test No. 1-8

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1600 mL
 Adjust solution pH to **4.0** with 0.6 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
7:25	start	20	20	3.6	385
8:00	advance	16	20	2.9	378
8:30	30	16	20	2.9	377
9:30	60	15	20	2.8	378
9:30	90	15	20	2.8	375
	120				
	Composite	15		5.0	275

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-8-3	Discharge	1580	1.9	3.0	0	0	18.5	29.2
Discharge sample		80	Total iodine					
			22.3					

Iron Precipitation

Charge precipitation vessel 1500 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes Added 3 mL flocculant
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
10:50	start	15	4.9	10.8	2.1
11:15	0	15	10.8		
11:25	10	15	10.9		
11:35	20	15	10.9		
11:45	30	15	10.9		

			Hg			I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-8-4	Filtrate	1500	0.05	0.1	4%	0	0	15	22.5
1-8-5	Precipitate	3.5	520	1.8	96%				
Filtrate sample		60	Total iodine						17.2

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 27-Mar-98
 Cycle No: 8 Date Finished: 30-Mar-98
 Test No. 1-8

Volume of iron precipitation filtrate available to advance 1440 mL 19 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 840 mL containing 16.0 g KI

Iodine Recovery

Measure sufficient filtrate to contain 11.4 grams potassium iodide for oxidation to I₂
600 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI
4.78 g H₂SO₄
4.8 mL 1000 g/L
 Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction 0.23 g H₂O₂/g KI
2.6 g H₂O₂
6.6 mL 35% H₂O₂
 g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
 Stir for 60 minutes, settle, decant, filter solids, do not wash
 Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
 Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
15:35	0	4.8	6.6	19	9.8	1.3
15:40	0			19	1.3	
15:55	15			20	1.3	
16:10	30			20	1.3	

			Hg		I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-8-6	Filtrate	660	0.3	0.2	0	0	0	0
1-8-7	Precipitate	9.5						
Filtrate sample		60	Total iodine 0.0					

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date:
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Run No: 1
Cycle No: 9
Test No. 1-9

Date Started: 31-Mar-98
Date Finished: 1-Apr-98

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	400	160

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I ₂ , M	I ₂ , g	I ₂ , g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				0.0
Recycle KI Soln:			840	19	16.0				
Total Iodine reqd:						0.04	12.2	2.7	
Recycled Iodine							9.5		
Water:		254					Total iodine		24.4

Adjust pH to 7.0 with 500 g/L sulfuric acid
Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

		pH		500 g/L H ₂ SO ₄	
Time	Minutes	read	adjust	mV	mL
8:30	start	8.9	6.9	407	0.7
8:45	0	6.9		406	
9:45	60	6.9		404	
10:45	120	7.0		406	
12:45	240	7.2		403	

		Hg				I ₂		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-9-1	Filtrate	1620	105	170.1	96.5%	4.1	6.6	10	16.2
1-9-2	Residue	403	15.2	6.1	3.5%				
Filtrate sample		80	Balance=		110%	Total iodine		19.0	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No. 971026-6
Metallurgical & Mineral Processing Consultants			Date:
5906 McIntyre Street Golden, CO 80403	(303) 279-2581 FAX 279-6061		

Run No: 1 Date Started: 31-Mar-98
 Cycle No: 9 Date Finished: 1-Apr-98
 Test No. 1-9

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1540 mL
 Adjust solution pH to **4.0** with 1.5 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
13:45	start	17	20	3.9	439
14:15	advance	18	20	3.7	414
14:45	30	18	20	3.4	414
15:15	60	18	20	3.2	414
15:45	90	18	20	3.2	414
	120				
	Composite	21		4.7	330

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-9-3	Discharge	1600	4.0	6.4	0	0	15	24.0
	Discharge sample	50					Total iodine	18.3

Iron Precipitation

Charge precipitation vessel 1550 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes added 3 mL flocculant
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
15:50	start	21	4.7	11.0	2.5
16:05	0	21	11.0		
16:15	10	20	11.0		
	20				
	30				

Sample ID		wt/vol	Hg		Distn	I ₂		KI	
			mg/kg-L	mg		g/L	g	g/L	g
1-9-4	Filtrate	1560	0.4	0.6	13%	0	0	16	25.0
1-9-5	Precipitate	5.2	820	4.3	87%				
	Filtrate sample	60						Total iodine	19.1

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 31-Mar-98
 Cycle No: 9 Date Finished: 1-Apr-98
 Test No: 1-9

Volume of iron precipitation filtrate available to advance 1500 mL 14 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 1094 mL containing 15.3 g KI

Iodine Recovery

Measure sufficient filtrate to contain 5.6 grams potassium iodide for oxidation to I₂
400 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI
2.35 g H₂SO₄
2.4 mL 1000 g/L
 Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction 0.23 g H₂O₂/g KI
1.3 g H₂O₂
3.3 mL 35% H₂O₂
 g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
 Stir for 60 minutes, settle, decant, filter solids, do not wash
 Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
 Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
8:45	0	2.4	3.3	17	10.5	1.0
8:55	0			18		
9:10	15			18		
9:25	30			17		

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-9-6	Filtrate	460	0.2	0.1	8.4	3.9	0	0
1-9-7	Precipitate	2.1						
Filtrate sample		60				Total iodine	3.9	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6
		Date:

Run No: 1 Date Started: 1-Apr-98
 Cycle No: 10 Date Finished: 2-Apr-98
 Test No: 1-10

Feed: 400 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
21.0	506	400	160

Leach: 25% solids 4 hours ambient temperature

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, M/g/L	KI, g	I2, M	I2, g	I2, g	KI, g
Solids feed:	400	106							
Total KI reqd:				0.08	15.9				0.6
Recycle KI Soln:			1094	14	15.3				
Total Iodine reqd:						0.04	12.2	10.1	
Recycled Iodine							2.1		
Water:		0					Total iodine		24.4

Adjust pH to 7.0 with 500 g/L sulfuric acid
 Leach 4 hours, measure pH and emf at 60, 120, and 240 minutes
 Transfer to vacuum filter, filter, wash with 400 mL water or iodine recovery wash filtrate
 Measure volume of filtrate plus wash, transfer to cementation
 Take sample of filtrate for analysis

Time	Minutes	pH		mV	500 g/L H2SO4
		read	adjust		mL
9:45	start	9.7	7.0	386	0.9
10:00	0	7.0		386	
11:00	60	6.8		408	
12:00	120	7.1		403	
14:00	240	7.1		401	

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-10-1	Filtrate	1580	105	165.9	97.0%	4.8	7.6	11.8	18.6
1-10-2	Residue	404	12.8	5.2	3.0%				
Filtrate sample		80	Balance=		107%	Total iodine		21.8	

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No. 971026-6
Metallurgical & Mineral Processing Consultants			Date:
5906 McIntyre Street Golden, CO 80403	(303) 279-2581 FAX 279-6061		

Run No: 1 Date Started: 1-Apr-98
 Cycle No: 10 Date Finished: 2-Apr-98
 Test No. 1-10

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 1500 mL
 Adjust solution pH to **4.0** with 0.2 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
14:20	start	20	20	3.7	409
14:50	advance	20	20	3.2	402
15:20	30	20	20	3.0	402
15:50	60	19	20	3.0	402
	90				
	120				
	Composite	21		4.3	335

Sample ID		vol	Hg		I ₂		KI	
			mg/L	mg	g/L	g	g/L	g
1-10-3	Discharge	1480	4.7	7.0	0	0	17.5	25.9
Discharge sample		80	Total iodine					
			19.8					

Iron Precipitation

Charge precipitation vessel 1400 mL discharge
 Adjust to 10-11 pH by addition of dry hydrated lime
 Maintain at 10-11 pH for 30 minutes 2 mL floc added
 Transfer to vacuum filter, filter, and wash
 Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
16:30	start	21	4.3	11.4	2.2
16:35	0	21	11.4		
16:45	10	21	11.6		
	20				
	30				

			Hg			I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
1-10-4	Filtrate	1420	0.2	0.3	5%	0	0	13	18.5
1-10-5	Precipitate	5.8	915	5.3	95%				
Filtrate sample		80	Total iodine						14.1

Signed: _____

Date: _____

Witness: _____

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No: 971026-6 Date:
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Run No: 1
 Cycle No: 10
 Test No: 1-10

Date Started: 1-Apr-98
 Date Finished: 2-Apr-98

Volume of iron precipitation filtrate available to advance 1340 mL 17 g/L KI

Iodide Recycle

Set aside sufficient filtrate to contain 15.9 grams potassium iodide to use in next cycle
 Or a maximum volume of 1094 mL
 Volume advanced to recycle 980 mL containing 16.7 g KI

Iodine Recovery

Measure sufficient filtrate to contain grams potassium iodide for oxidation to I₂
154 mL neutralization filtrate to iodine recovery
 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction 0.42 g H₂SO₄/g KI

g H₂SO₄
 mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23 g H₂O₂/g KI
 g H₂O₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

mL 35% H₂O₂

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
	0					
	0					
	15					
	30					

Sample ID		Hg			I ₂		KI	
		wt/vol	mg/kg-L	mg	g/L	g	g/L	g
1-10-6	Filtrate					0		0
1-10-7	Precipitate							
Filtrate sample					Total iodine		0.0	

Signed: _____

Date: _____

Witness: _____

Appendix A-4

Data Sheets for Bench-Scale Extractions of INEEL Soil/Sludge

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061		CHEMICAL EXTRACTION OF MERCURY-BEARING WASTE DATA SHEET		Project No.: 971026-06
				Date: 5/8/98

Test No: I-1

25°C

0.1 M KI

0.05 M I2

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
		0.0			0
		0.0			0
		0.0	100.0	495	50

Leach:

25% solids

	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0					
Total I required				0.1	5.0	0.05	3.8
Water:			300				

Heat to 25°C, do not adjust pH

Leach 4.0 hours

Transfer to vacuum filter, filter, wash with 100 mL water

Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H2SO4
8:15	start	26	8.2	
8:20	0	26	8.2	
8:45	30	25	8.0	
9:15	60	25	7.7	
9:45	90	25	7.5	
10:15	120	25	7.4	
11:15	180	26	7.4	
12:15	240	26	7.2	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn
I-1-1	Residue		97.2	60	5.8	0.008
I-1-2	Filtrate		mL	mg/L		
			420	1820	764	99.99
Balance, versus feed						1556

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Test No: I-4 45°C 0.2 M KI
0.1 M I2

Feed:	dry grams	Hg,			
		% moisture	Damp wt.	mg/kg	Hg, mg
		0.0			0
		0.0			0
		0.0	100.0	495	50

Leach:		25% solids					
Solids feed:	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
	100	0					
Total I required				0.2	10.0	0.1	7.6
Water:			300				

Heat to 45°C, adjust pH to 4.0 with 1000 g/L H2SO4
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	Read pH	Adjusted pH	mL 1000g/L H2SO4
11:25	start	42	8.1	4.8	12.6
11:30	0	44	5.4	4.3	3.7
12:00	30	44	4.8	3.7	2.4
12:30	60	42	5.6	4.0	4.5
13:00	90	46	4.6		
13:30	120	46	5.7		
14:30	180	46	5.8		
15:30	240	45	5.7		

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn
I-4-1	Residue		106.5	31	3.3	0.004
I-4-2	Filtrate		mL	mg/L	795	100.00
			350	2270		
Balance, versus feed						1612

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Test No: I-5 45°C 0.1 M KI
0.05 M I2

Feed:	dry grams	Hg,			
		% moisture	Damp wt.	mg/kg	Hg, mg
		0.0			0
		0.0			0
		0.0	100.0	495	50

Leach:		25% solids					
Solids feed:	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
	100	0					
Total I required				0.1	5.0	0.05	3.8
Water:			300				

Heat to 45°C, adjust pH to 4.0 with 1000 g/L H2SO4
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	Read pH	Adjusted pH	mL 1000g/L H2SO4
11:25	start	42	8.0	4.4	12.6
11:30	0	44	5.3	3.7	2.5
12:00	30	44	5.4	4.0	1.9
12:30	60	47	4.8	4.0	5.3
13:00	90	46	5.3		
13:30	120	47	5.7		
14:30	180	46	5.4		
15:30	240	46	5.6		

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn
I-5-1	Residue		106.6	23	2.5	0.049
I-5-2	Filtrate		mL	mg/L	48	99.95
			397	120		
Balance, versus feed						101

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				Date: 5/8/98

Test No: I-7 45°C 0.1 M KI
0.05 M I2

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
		0.0			0
		0.0			0
		0.0	100.0	495	50

Leach:		25% solids					
	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0					
Total I required				0.1	5.0	0.05	3.8
Water:			300				

Heat to 45°C, do not adjust pH
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H2SO4
10:10	start	43	8.1	
10:20	0	43	7.5	
10:40	30	45	7.2	
11:10	60	45	6.9	
11:40	90	45	6.9	
12:10	120	44	6.8	
13:10	180	45	6.8	
14:10	240	45	6.8	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn
I-7-1	Residue		97.8	35	3.4	0.005
I-7-2	Filtrate		mL	mg/L	746	100.00
			350	2130		
Balance, versus feed						1513

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Test No: I-8 45°C 0.2 M KI
0.1 M I2

Feed:	dry grams	Hg,			
		% moisture	Damp wt.	mg/kg	Hg, mg
		0.0			0
		0.0			0
		0.0	100.0	495	50

Leach:		25% solids					
	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0					
Total I required				0.2	10.0	0.1	7.6
Water:			300				

Heat to 45°C, do not adjust pH
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H2SO4
10:10	start	42	8.2	
10:20	0	43	7.5	
10:40	30	45	7.3	
11:10	60	45	7.0	
11:40	90	45	6.8	
12:10	120	46	6.7	
13:10	180	46	6.7	
14:10	240	46	6.7	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn
I-8-1	Residue		98.1	34	3.3	0.004
I-8-2	Filtrate		mL	mg/L	764	100.00
			398	1920		
Balance, versus feed						1550

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Test No: I-9 45°C 0.4 M KI
0.2 M I2

Feed:	dry grams	Hg,			
		% moisture	Damp wt.	mg/kg	Hg, mg
		0.0			0
		0.0			0
		0.0	100.0	495	50

Leach:		25% solids					
Solids feed:	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
	100	0					
Total I required				0.4	19.9	0.2	15.2
Water:			300				

Heat to 45°C, do not adjust pH
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash

Time	Minutes	°C	pH	mL 1000g/L H2SO4
10:10	start	43	8.1	
10:20	0	43	7.5	
10:40	30	45	7.3	
11:10	60	45	7.1	
11:40	90	45	6.8	
12:10	120	46	6.7	
13:10	180	46	6.7	
14:10	240	46	6.7	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn
I-9-1	Residue		98.0	26	2.5	0.048
I-9-2	Filtrate		mL	mg/L	51	99.95
			440	115		
Balance, versus feed						107

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				Date: 5/15/98

Test No: I-10 45°C 0.2 M KI
0.1 M I₂

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:		25% solids						
		Liquid		Potassium Iodide		Iodine		Total
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g	I, g
Solids feed:	100	0	300	0.2	10.0	0.1	7.6	15.2
Total I required								
Water:								

Heat to 45°C, do not adjust pH
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
11:30	start	21	8.0	
11:45	0	42	6.9	
12:15	30	46	6.7	
12:45	60	46	6.7	
13:15	90	46	6.7	
13:45	120	46	6.7	
14:45	180	46	6.7	
15:45	240	46	6.7	

Residue rinse

Sample ID		Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI	KI	I ₂	I ₂	Total I
I-10-1	Residue	95.6	48.8	4.7	5.2					
		mL	mg/L			g/L	g	g/L	g	g
I-10-2	Filtrate	687	125	86	94.8	21.5	14.8	6.5	4.5	15.8
Balance, versus feed					179	103%				

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				Date: 5/15/98

Test No: I-11 45°C 0.3 M KI
0.15 M I₂

Feed:				Hg,	
		% moisture	Damp wt.	mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:	25% solids							
			Liquid	Potassium Iodide		Iodine		Total
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g	I, g
Solids feed:	100	0	300	0.3	14.9	0.15	11.4	22.8
Total I required								
Water:								

Heat to 45°C, do not adjust pH
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
11:30	start	21	8.1	
11:45	0	42	6.8	
12:15	30	46	6.7	
12:45	60	46	6.6	
13:15	90	46	6.6	
13:45	120	46	6.6	
14:45	180	46	6.6	
15:45	240	45		

Residue rinse

Sample ID		Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI g/L	KI g	I ₂ g/L	I ₂ g	Total I g
I-11-1	Residue	97.4	48.4	4.7	5.2					
		mL	mg/L			g/L	g	g/L	g	g
I-11-2	Filtrate	688	125	86	94.8	31.4	21.6	8.8	6.1	22.6
Balance, versus feed					179	99%				

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				Date: 5/15/98

Test No: I-13 35°C 0.3 M KI
0.15 M I₂

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:		25% solids						Total I, g
		Liquid		Potassium Iodide		Iodine		
	dry g	grams	mL	KI, M	KI, g	I ₂ , M	I ₂ , g	
Solids feed:	100	0	300	0.3	14.9	0.15	11.4	22.8
Total I required Water:								

Heat to 35°C, do not adjust pH
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
11:30	start	21	8.1	
11:45	0	30	6.9	
12:15	30	33	6.7	
12:45	60	34	6.7	
13:15	90	33	6.7	
13:45	120	34	6.6	
14:45	180	34	6.6	
15:45	240	36	6.6	

Residue rinse

Sample ID		Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI	KI	I ₂	I ₂	Total I
I-13-1	Residue	97.7	46.6	4.6	4.8					
		mL	mg/L			g/L	g	g/L	g	g
I-13-2	Filtrate	745	120	89	95.2	25.0	18.6	11.8	8.8	23.0
Balance, versus feed					186	101%				

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				Date: 5/21/98

Test No: I-14 45 0.2 M KI
0.1 M I2

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:		25% solids							
Stage 1	dry g	Liquid		Potassium Iodide		Iodine		Total I, g	
		grams	mL	KI, M	KI, g	I2, M	I2, g		
Solids feed:		100	0		0.2	10.0	0.1	7.6	15.2
Total I required									
Water:			300						
Stage 2									
Total I required				0.2	10.0	0.1	7.6	15.2	
Water:			300						

Heat to 45°C, do not adjust pH
Leach 2.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Releach cake in 300 mL water containing the above iodine and iodide additions
Heat to 45°C, do not adjust pH
Leach 2.0 hours
Filter and wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
	0			
	30			
	60			
	120			
	0			
	30			
	60			
	120			

Residue rinse

Sample ID		Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI	KI	I2	I2	Total I
I-14-1	Residue			0.0	#DIV/0!					
		mL	mg/L			g/L	g	g/L	g	g
I-14-2	Filtrate			0	#DIV/0!		0.0		0.0	0.0
Balance, versus feed					0	0%				

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				Date: 5/21/98

Test No: I-15 45 0.2 M KI
0.1 M I2

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:		25% solids								
Stage 1	dry g	Liquid		Potassium Iodide		Iodine		Total I, g		
		grams	mL	KI, M	KI, g	I2, M	I2, g			
Solids feed:		100	0		0.2	10.0	0.1	7.6	15.2	
Total I required										
Water:			300							
Stage 2										
Total I required				0.1	5.0	0.05	3.8	7.6		
Water:			300							

Heat to 45°C, do not adjust pH
Leach 2.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Releach cake in 300 mL water containing the above iodine and iodide additions
Heat to 45°C, do not adjust pH
Leach 2.0 hours
Filter and wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
	0			
	30			
	60			
	120			
	0			
	30			
	60			
	120			

Residue rinse

Sample ID		Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI	KI	I2	I2	Total I
I-15-1	Residue			0.0	#DIV/0!					
		mL	mg/L			g/L	g	g/L	g	g
I-15-2	Filtrate			0	#DIV/0!		0.0		0.0	0.0
Balance, versus feed						0		0%		

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				Date: 5/21/98

Test No: I-16 55°C 0.2 M KI
0.1 M I2

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:		25% solids								
Stage 1	dry g	Liquid		Potassium Iodide		Iodine		Total I, g		
		grams	mL	KI, M	KI, g	I2, M	I2, g			
Solids feed:		100	0		0.2	10.0	0.1	7.6	15.2	
Total I required										
Water:			300							
Stage 2										
Total I required				0.2	10.0	0.1	7.6	15.2		
Water:			300							

Heat to 45°C, do not adjust pH
Leach 2.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Releach cake in 300 mL water containing the above iodine and iodide additions
Heat to 45°C, do not adjust pH
Leach 2.0 hours
Filter and wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
	0			
	30			
	60			
	120			
	0			
	30			
	60			
	120			

Residue rinse

Sample ID		Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI	KI	I2	I2	Total I
I-16-1	Residue			0.0	#DIV/0!					
		mL	mg/L			g/L	g	g/L	g	g
I-16-2	Filtrate			0	#DIV/0!		0.0		0.0	0.0
Balance, versus feed					0	0%				

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				Date: 5/21/98

Test No: I-17 55°C 0.2 M KI
0.1 M I2

Feed: <u>100</u> dry grams		% moisture	Damp wt.	Hg, mg/kg	Hg, mg
	4- x 16-mesh	0.0	5.2	210	1
	16- x 100-mesh	0.0	8.4	806	7
	<100-mesh	0.0	86.4	495	43

Leach:		25% solids							
Stage 1	dry g	Liquid		Potassium Iodide		Iodine		Total I, g	
		grams	mL	KI, M	KI, g	I2, M	I2, g		
Solids feed:	100	0							
Total I required				0.2	10.0	0.1	7.6	15.2	
Water:			300						
Stage 2									
Total I required				0.1	5.0	0.05	3.8	7.6	
Water:			300						

Heat to 45°C, do not adjust pH
Leach 2.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Releach cake in 300 mL water containing the above iodine and iodide additions
Heat to 45°C, do not adjust pH
Leach 2.0 hours
Filter and wash with 100 mL water
Repulp with 200 mL water containing 5 grams potassium iodide
Refilter and wash with 100 mL water
Measure volume of combined filtrates plus washes

Time	Minutes	°C	pH	
	0			
	30			
	60			
	120			
	0			
	30			
	60			
	120			

Residue rinse

Sample ID			Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI	KI	I2	I2	Total I
I-17-1	Residue				0.0	#DIV/0!					
			mL	mg/L			g/L	g	g/L	g	g
I-17-2	Filtrate				0	#DIV/0!		0.0		0.0	0.0
Balance, versus feed						0	0%				

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Test No: I-18 50°C 0.3 M KI
0.15 M I2

Feed:	100	dry grams	Hg,			
			% moisture	Damp wt.	mg/kg	Hg, mg
			0.0			0
			0.0			0
			0.0	100.0	815	82

Leach:		25% solids					
	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0					
Total I required				0.3 g/l	-2.7	0.15	11.4
Leach KI solution			300	58.9	17.7		
Repulp KI solution			300	58.9	17.7		

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H2SO4
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Repulp in 300 ml of the same solution as used for leaching, heat to 50°C for 1 hour
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Dry and sample solids

Time	Minutes	°C	pH	mL 1000g/L H2SO4
	start			
9:00	0	55	5.3/5.0	3
	30	53	5.3	
10:00	60	52	5.5	
	90			
11:00	120	53	5.7/5.1	1
12:00	180	53	5.7	
13:00	240	53	5.7	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI g/l	KI g	I2 M	I2 g
I-18-1	Residue		98.5	77	7.6	6.9				
I-18-2	Filtrate-1		mL	mg/L						
			340	300	102	92.3	68.8	23.4	0.141	12.17
I-18-3	Filtrate-2		395	2.4	0.9	0.9	48.5	19.2	0	0
Balance, versus feed						136		1.20		0.53

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Test No: I-19 50°C 0.4 M KI
0.2 M I2

Feed:	100	dry grams	Hg,			
			% moisture	Damp wt.	mg/kg	Hg, mg
			0.0			0
			0.0			0
			0.0	100.0	815	82

Leach:		25% solids					
	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0					
Total I required				0.4 g/l	2.3	0.2	15.2
Leach KI solution			300	58.9	17.7		
Repulp KI solution			300	58.9	17.7		

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H2SO4
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Repulp in 300 ml of the same solution as used for leaching, heat to 50°C for 1 hour
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Dry and sample solids

Time	Minutes	°C	pH	mL 1000g/L H2SO4
	start			
9:00	0	55	5.3/5.0	3
	30	53	5.3	
10:00	60	52	5.6	
	90			
11:00	120	53	5.8/5.1	1
12:00	180	53	5.7	
13:00	240	53	5.7	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI g/l	KI g	I2 M	I2 g
I-19-1	Residue		98.3	26.4	2.6	2.5				
I-19-2	Filtrate-1		mL	mg/L						
			335	300	100.5	96.6	55.7	18.66	0.07	6.16
I-19-3	Filtrate-2		395	2.3	0.9	0.9	47.2	18.64	0.00	0.00
Balance, versus feed						128		0.94		0.20

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Test No: I-20 50°C 0.6 M KI
0.3 M I2

Feed:	100	dry grams	Hg,			
			% moisture	Damp wt.	mg/kg	Hg, mg
			0.0			0
			0.0			0
			0.0	100.0	815	82

Leach:		25% solids					
	dry g	Liquid		Potassium Iodide		Iodine	
		grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0					
Total I required				0.6 g/l	12.2	0.3	22.8
Leach KI solution			300	58.9	17.7		
Repulp KI solution			300	58.9	17.7		

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H2SO4
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Repulp in 300 ml of the same solution as used for leaching, heat to 50°C for 1 hour
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Dry and sample solids

Time	Minutes	°C	pH	mL 1000g/L H2SO4
	start			
9:00	0	53	5.3/5.0	3
	30	57	5.4	
10:00	60	53	5.5	
	90			
11:00	120	53	5.7/5.1	1
12:00	180	51	5.7	
13:00	240	53	5.7	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI g/l	KI g	I2 M	I2 g
I-20-1	Residue		97.7	21.2	2.1	2.0				
I-20-2	Filtrate-1		mL	mg/L						
			340	300	102	97.0	55.7	18.94	0.05	4.49
I-20-3	Filtrate-2		400	2.7	1.1	1.0	47.2	18.88	0.00	0.00
Balance, versus feed						129		0.63		0.10

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Test No: I-21 50°C 0.8 M KI
0.4 M I2

Feed:	100	dry grams	Hg,			
			% moisture	Damp wt.	mg/kg	Hg, mg
			0.0			0
			0.0			0
			0.0	100.0	815	82

Leach:		25% solids					
		Liquid		Potassium Iodide		Iodine	
	dry g	grams	mL	KI, M	KI, g	I2, M	I2, g
Solids feed:	100	0		0.8	22.2	0.4	30.5
Total I required				g/l			
Leach KI solution				58.9	17.7		
Repulp KI solution			300	58.9	17.7		

Heat to 50°C, adjust pH to 4.0 with 1000 g/L H2SO4
Leach 4.0 hours
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Repulp in 300 ml of the same solution as used for leaching, heat to 50°C for 1 hour
Transfer to vacuum filter, filter, wash with 100 mL water
Measure volume of filtrate plus wash
Dry and sample solids

Time	Minutes	°C	pH	mL 1000g/L H2SO4
	start			
9:00	0	53	5.3/5.0	3
	30	57	5.4	
10:00	60	53	5.6	
	90			
11:00	120	53	5.7/5.1	1
12:00	180	51	5.7	
13:00	240	53	5.7	

Residue rinse

Sample ID		Wet grams	Dry grams	Hg mg/kg	Hg mg	Hg, % Distn	KI g/l	KI g	I2 M	I2 g
I-21-1	Residue		97.9	19.8	1.9	1.7				
I-21-2	Filtrate-1		mL	mg/L	108.5	97.5	98.2	34.4	0.2	19.3
			350	310						
I-21-3	Filtrate-2		400	2.1	0.8	0.8	48.5	19.4	0.0	0.0
Balance, versus feed						137		0.7		0.3

Appendix A-5

Data Sheets for Locked-Cycle Testing of INEEL Soil/Sludge

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 1
Test No. 2-1

Date Started: 24-Jun-98

Feed:	<u>1000</u> dry grams	% moisture	Damp wt.	Hg, mg/kg	Hg, mg
		5.0	1053	800	800

Leach: 30% solids 2 hours 60°C
Stage 1: (Note: there were two Stage 1 leaches)

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				77.5
Total Iodine reqd:						25.4	59.3	59.3	
Water:		2281						Total iodine	118.5

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Sample 1

Stage 1

		pH		
Time	Minutes	read	mV	
8:15	0	unstable	411	
8:45	30	6.3	412	
9:15	60	unstable	414	
10:45	120	unstable	403	

Sample 1

Stage 2

		pH		
Time	Minutes	read	mV	
	0	6.0	399	
	30	6.0	408	
	60	unstable	415	
	120	unstable	404	

Sample 2

Stage 1

		pH		
Time	Minutes	read	mV	
8:15	0	6.6	399	
8:45	30	6.0	408	
9:15	60	5.8	415	
10:45	120	5.0	404	

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				38.7
Total Iodine reqd:						12.7	29.6	29.6	
Water:		2333						Total iodine	59.2

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 1
Test No. 2-1

Date Started: 24-Jun-98

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, hold for next cycle, first stage leach
Take sample of filtrate for analysis

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-1-1	1st Filtrate	3840	405	1555	78.7%	7.4	28.4	47.2	181.2
2-1-2	2nd Filtrate	2333	89	208	10.5%	3.5	8.2	26.9	62.8
2-1-3	Residue	853.6	250	213	10.8%			0.47%	4.0
				Balance=		247%		Total iodine	
								226.2	

Cementation:

Solution volume advancing to cementation 3840 mL
Adjust solution pH to **4.0** with 2 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
18:48	start	28	30-35		
	advance				
	Composite				

Sample ID		Hg		
		vol	mg/L	mg
2-1-4	Discharge	2500	5	12.5

Iron Precipitation

Charge precipitation vessel 2500 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Added 2 mL flocculant

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date: 8/14/98
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Run No: 2
Cycle No: 1
Test No. 2-1

Date Started: 24-Jun-98

Time	Minutes	°C	pH		g Ca(OH)2
			Read	Adjust	
	start	25	4		
	0	25	11		
	10	25	11		
	20	25	11		
	30	25	11		~10

Sample ID		Hg			
		wt/vol	mg/kg-L	mg	Distn
2-1-5	Filtrate	2500	0.9	2.3	9%
2-1-6	Precipitate	20.6	1120	23.1	91%

KI	
g/L	g
49.7	124.3

Volume of iron precipitation filtrate available to advance

2500 mL

Total iodine 95.0
49.7 g/L KI
124.25 g KI

Iodide Recycle

Volume advanced to recycle

1000 mL

containing

49.7 g KI

Iodine Recovery

Volume advanced to iodine

1500

mL neutralization filtrate to iodine recovery

74.6

total grams potassium iodide

Acidify with 140% of stoich. H2SO4 for iodide oxidation reaction

<u>0.42</u>	g H2SO4/g KI
<u>31.3</u>	g H2SO4
<u>31.3</u>	mL 1000 g/L
<u>0.23</u>	g H2O2/g KI
<u>17.1</u>	g H2O2
<u>42.2</u>	mL 35% H2O2

Add 120% of stoichiometric H2O2 for iodide oxidation reaction

$$\text{g 100\% H}_2\text{O}_2 / 35\% / 1.13 = \text{mL 35\%}$$

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H2SO4	mL H2O2	°C	pH	
					read	adjusted
	0	31.3	43.4	25	1.04	

Sample ID		Hg			I2
		wt/vol	mg/kg-L	mg	g
2-1-7	Filtrate	1500			
2-1-8	Precipitate	57			57

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 2
Test No. 2-2

Date Started: 26-Jun-98

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				77.5
Total Iodine reqd:						25.4	59.3	59.3	
Water:		2281						Total iodine	118.5

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
8:10	0	6.5	375
8:40	30	~6	404
9:10	60	~6	405
10:10	120	~5.8	400

Stage 2

		pH	
Time	Minutes	read	mV
11:00	0	-	385
11:30	30		428
12:00	60		435
1:00	120	~6.14	423

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				0.0
Total Iodine reqd:						12.7	29.6	0.0	
Recycle KI Soln:			780	49.7	38.8				
Recycle I ₂ :							29.6		
Water:		1553						Total iodine	59.3

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, hold for next cycle, first stage leach

Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 2
Test No. 2-2

Date Started: 26-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-2-1	1st Filtrate	1990	430	855.7	83.4%	11.9	23.7	77.5	154.2
2-2-2	2nd Filtrate	1940	34	66.0	6.4%	3.4	6.6	20.4	39.6
2-2-3	Residue	902.8	115	103.8	10.1%			0.32%	2.9
				Balance=	128%	Total iodine 180.6			

Cementation:

Solution volume advancing to cementation 2330 mL
Adjust solution pH to **4.0** with 1.6 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
	start				
	Composite				

Sample ID		Hg			
		vol	mg/L	mg	
2-2-4	Discharge	2115	9	19.0	

Iron Precipitation

Charge precipitation vessel 2095 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Added 2 mL flocculant

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
	start				
13:30	0	26	10.08		
13:40	10	26	10.24		
13:50	20	25	10.38		
14:00	30	25	10.43		13

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 2
Test No. 2-2

Date Started: 26-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-2-5	Filtrate	2060	0.5	1.0	7%			60.8	125.2
2-2-6	Precipitate	24.68	600	14.8	93%				

Total iodine 95.7

Volume of iron precipitation filtrate available to advance 2060 mL 60.8 g/L KI
125.25 g KI

Iodide Recycle

Volume advanced to recycle 0 mL containing 0.0 g KI

Iodine Recovery

Volume advanced to iodine recycle 2060 mL neutralization filtrate to iodine recovery
125 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42 g H₂SO₄/g KI

52.604 g H₂SO₄

52.604 mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23 g H₂O₂/g KI

28.807 g H₂O₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

71.0 mL 35% H₂O₂

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
9:40	0	102	157	40.2	0.36	
9:55	15				0.72	
10:10	30				1.5	

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-2-7	Filtrate	2258			
2-2-8	Precipitate	86			86

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Run No: 2
Cycle No: 3
Test No. 2-3

Date Started: 26-Jun-98

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				7
Total Iodine reqd:						25.4	59.3	0	
Advance Leach Soln:			2300	26.9	61.9	3.5	8.1		
Advance Leach Soln:			400	20.4	8.2	3.4	1.4		
Recycle Iodine:							51.0		
Water:		0						Total iodine 119.6	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
1:50	0		409
2:20	30		444
2:50	60		444
3:50	120	6.1	424

Stage 2

		pH	
Time	Minutes	read	mV
5:00	0		418
5:30	30		423
6:00	60		433
7:00	120	6.1	418

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				
Total Iodine reqd:						12.7	29.6	0.0	
Recycle I ₂ :							0.0		
Water:		2333						Total iodine 0.0	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, hold for next cycle, first stage leach
Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE		Project No	971026-6
Metallurgical & Mineral Processing Consultants		DATA SHEET		Date:	8/14/98
5906 McIntyre Street	(303) 279-2581				
Golden, CO 80403	FAX 279-6061				

Run No: 2
Cycle No: 3
Test No. 2-3

Date Started: 26-Jun-98

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-3-1	1st Filtrate	2405	405	974.0	83.0%	6.1	14.7	48.2	115.9
2-3-2	2nd Filtrate	2435	20	48.7	4.1%	0.8	1.9	4.7	11.4
2-3-3	Residue	977.3	155	151.5	12.9%			0.37%	3.6
				Balance=	147%	Total iodine 116.7			

Cementation:

Solution volume advancing to cementation 2405 mL
Adjust solution pH to **4.0** with 6.5 mL 1000 g/L H2SO4

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
	start	25.6	30-35	4.1	160
17:00	advance	25.6	30-35	4.1	160
17:15		27	32	4.9	118
17:30		27	32	4.7	155
17:45		27	32	4.2	162
18:00		27	32	3.7	184
18:15	Composite	27		3.6	188

Sample ID		Hg		
		vol	mg/L	mg
2-3-4	Discharge	2375	12	28.5

Iron Precipitation

Charge precipitation vessel 2375 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes Added 2 mL flocculant
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH)2
			Read	Adjust	
	start				
18:50	0	27.5	10.13		14.5
19:00	10	27.2	10.27	0.5	15.0
19:10	20	27	10.48		
19:20	30	26.7	10.67		

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 3
Test No. 2-3

Date Started: 26-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-3-5	Filtrate	2600	0.6	1.6	6%		0	48	124.8
2-3-6	Precipitate	21.65	1180	25.5	94%				

Total iodine 95.4

Volume of iron precipitation filtrate available to advance 2600 mL 48.0 g/L KI
124.8 g KI

Iodide Recycle

Volume advanced to recycle 1240 mL containing 59.5 g KI

Iodine Recovery

Volume advanced to iodine recycle 1240 mL neutralization filtrate to iodine recovery
59.52 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42 g H₂SO₄/g KI
24.998 g H₂SO₄
24.998 mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23 g H₂O₂/g KI
13.69 g H₂O₂
33.7 mL 35% H₂O₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
9:00	0	44	69	38.4	0.32	
9:20	15			48.5	0.25	
9:35	30			41.2	0.71	

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-3-7	Filtrate	965	0.4		
2-3-8	Precipitate	32			32

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET		
Metallurgical & Mineral Processing Consultants			Project Nc	971026-6
5906 McIntyre Street	(303) 279-2581		Date:	8/14/98
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 4
Test No. 2-4

Date Started: 26-Jun-98

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				33.2	77.5				42.3
Total Iodine reqd:						25.4	59.3	0	
Advance Leach Soln:			1540	20.4	31.4	3.4	5.2		
Advance Leach Soln:			800	4.7	3.8	0.8	0.6		
Recycle Iodine:							53.0		
Water:		0						Total iodine 118.5	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Stage 1

		pH		°C
Time	Minutes	read	mV	
8:40	0	7	3	30
9:10	30	5.9	41	60
9:40	60	5.7	52	69
10:40	120	6.3	45	65

Stage 2

		pH		°C
Time	Minutes	read	mV	
12:00	0	6.5	10 -50	43
12:30	30	6.75	0 -50	50
13:00	60	6.6	30	60
14:00	120	7.0	35	65

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				0.0
Total Iodine reqd:						12.7	29.6	0.0	
Recycle I ₂ :									
Water:		2333						Total iodine 0.0	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, hold for next cycle, first stage leach

Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 4
Test No. 2-4

Date Started: 26-Jun-98

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-4-1	1st Filtrate	2415	405	978.1	74.8%	7.5	18.1	41.2	99.5
2-4-2	2nd Filtrate	2530	11	27.8	2.1%	0.1	0.3	5.4	13.7
2-4-3	Residue	1025	295	302.4	23.1%			0.42%	4.3
Balance= 164%						Total iodine 108.2			

Cementation:

Solution volume advancing to cementation 2415 mL
Adjust solution pH to **4.0** with 3 mL 1000 g/L H2SO4

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
11:40	start	25	29	4.5	163
12:10	advance	27		3.7	182
12:40		28		3.6	188
13:00		29		3.6	190
	Composite				

Sample ID		Hg		
		vol	mg/L	mg
2-4-4	Discharge	2320	25.6	59.4

Iron Precipitation

Charge precipitation vessel 2300 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes Added 2 mL flocculant
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH)2
			Read	Adjust	
13:50	start	29.5	10.41		10.71
	0				
14:00	10	27.7	10.42		
14:10	20	27	10.12		
14:20	30	27	9.9		

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 4
Test No. 2-4

Date Started: 26-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-4-5	Filtrate	2200	0.8	1.8	1%	0.0634	0.1395	60	132.0
2-4-6	Precipitate	22.2	11100	246.4	99%				

Total iodine 101.0

Volume of iron precipitation filtrate available to advance 2200 mL 60.0 g/L KI
132 g KI

Iodide Recycle

Volume advanced to recycle 640 mL containing 38.4 g KI

Iodine Recovery

Volume advanced to iodine recycle 1540 mL neutralization filtrate to iodine recovery
92.4 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42 g H₂SO₄/g KI
38.808 g H₂SO₄
38.808 mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23 g H₂O₂/g KI
21.252 g H₂O₂
52.3 mL 35% H₂O₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
	0	59	60			

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-4-7	Filtrate	1600	1	1.6	
2-4-8	Precipitate	42			42

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 5
Test No. 2-5

Date Started: 29-Jun-98

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				62
Total Iodine reqd:						25.4	59.3	0	
Advance Leach Soln:			1600	4.7	7.5	0.8	1.3		
Advance Leach Soln:			1500	5.4	8.1	0.1	0.2		
Recycle Iodine:							58		
Water:		0						Total iodine 118.5	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
14:15	0		440
14:45	30		469
15:15	60		449
16:15	120	6.3	447

Stage 2

		pH	
Time	Minutes	read	mV
17:00	0		497
17:30	30		454
18:00	60		455
19:00	120	6.5	449

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				0.0
Total Iodine reqd:						12.7	29.6	0.0	
Recycle KI Soln:			800	48	38				
Recycle I ₂ :									
Water:		1533						Total iodine 29.4	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, hold for next cycle, first stage leach
Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 5
Test No. 2-5

Date Started: 29-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-5-1	1st Filtrate	2480	385	954.8	79.0%	4.7	11.7	26.6	66.0
2-5-2	2nd Filtrate	2680	19	50.9	4.2%	1.4	3.8	13.9	37.3
2-5-3	Residue	964	210	202.4	16.8%			0.42%	4.0
Balance=						151%	Total iodine		97.4

Cementation:

Solution volume advancing to cementation 2460 mL
Adjust solution pH to **4.0** with 6.7 mL g 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
16:50	start	25	38	4	449
18:00	advance				
	Composite				

Sample ID		Hg		
		vol	mg/L	mg
2-5-4	Discharge	2440	45	109.8

Iron Precipitation

Charge precipitation vessel 2420 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes Added 6 mL flocculant
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
18:10	start	25	3.4	10	12
	0				
18:20	10	25		10.5	
18:30	20	25		11	
18:40	30	25		11.5	

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 5
Test No. 2-5

Date Started: 29-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-5-5	Filtrate	2440	1.1	2.7	0%		0	46	112.2
2-5-6	Precipitate	25.3	34700	877.9	100%				

Total iodine 85.8

Volume of iron precipitation filtrate available to advance 2400 mL 46.0 g/L KI
110.4 g KI

Iodide Recycle

Volume advanced to recycle 0 mL containing 0.0 g KI

Iodine Recovery

2400 mL neutralization filtrate to iodine recovery
110.4 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42 g H₂SO₄/g KI
30.2 g H₂SO₄
30.2 mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23 g H₂O₂/g KI
16.6 g H₂O₂
46.1 mL 35% H₂O₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
	0	30	46			

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-5-7	Filtrate	2041			
2-5-8	Precipitate	90			90

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc 971026-6 Date: 8/14/98
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Run No: 2
Cycle No: 6
Test No. 2-6

Date Started: 30-Jun-98

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				44
Total Iodine reqd:						25.4	59.3	0	
Advance Leach Soln:			1000	5.4	5.4	0.1	0.1		
Advance Leach Soln:			2000	13.9	27.8	1.4	2.8		
Recycle Iodine:							58		
Water:								Total iodine 119.9	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
7:15	0		394
7:45	30		393
8:15	60	7.0	394
9:15	120	7.0	396

Stage 2

		pH	
Time	Minutes	read	mV
10:15	0	6.6	410
10:45	30	6.5	420
11:15	60	6.5	420
12:15	120	6.4	409

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				0.0
Total Iodine reqd:						12.7	29.6	0.0	
Recycle KI Soln:			400	48	19				
Recycle KI Soln:			640	38	24				
Recycle I ₂ :							22		
Water:		1293						Total iodine 55.3	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, hold for next cycle, first stage leach
Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 6
Test No. 2-6

Date Started: 30-Jun-98

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-6-1	1st Filtrate	3025	375	1134.4	89.6%	5.1	15.4	36.5	110.4
2-6-2	2nd Filtrate	2345	19	44.6	3.5%	3.2	7.5	21.3	49.9
2-6-3	Residue	826	105	86.7	6.9%			0.26%	2.1
				Balance=		158%		Total iodine	
								147.2	

Cementation:

Solution volume advancing to cementation 3025 mL
Adjust solution pH to **4.0** with 26 mL 1000 g/L H2SO4

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
10:00	start	25	33	4.8	420
10:30	advance	25		4.6	439
11:45		25		5.1	370
12:15				5	
	Composite				

Sample ID		Hg			
		vol	mg/L	mg	
2-6-4	Discharge	2700	64	173	

Iron Precipitation

Charge precipitation vessel 2700 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes Added 2 mL flocculant
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH)2
			Read	Adjust	
12:30	start	27	11		11
12:30	0	27	11.5		
12:40	10	27	11.3		
12:50	20	27	11.3		
13:00	30	27	11.3		

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 6
Test No. 2-6

Date Started: 30-Jun-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-6-5	Filtrate	2780	62	172.4	93%	0	0	28	77.8
2-6-6	Precipitate	32.2	420	13.5	7%				
Total iodine									59.5

Volume of iron precipitation filtrate available to advance 2780 mL 28.0 g/L KI
77.84 g KI

Iodide Recycle

Volume advanced to recycle 1970 mL containing 55.2 g KI

Iodine Recovery

Volume advanced to iodine recycle 810 mL neutralization filtrate to iodine recovery
22.7 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42 g H₂SO₄/g KI
9.5256 g H₂SO₄
9.5256 mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23 g H₂O₂/g KI
5.2164 g H₂O₂
12.8 mL 35% H₂O₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
13:45	0	10	15	25	1.79	
14:00	15			27	2	
14:30	30			29	2.32	

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-6-7	Filtrate	840	35		
2-6-8	Precipitate	14.6			14.6

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc 971026-6 Date: 8/14/98
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Run No: 2 Date Started: _____
Cycle No: 7
Test No. 2-7

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				26
Total Iodine reqd:						25.4	59.3	2	
Advance Leach Soln:			200	13.9	2.8	1.4	0.3		
Advance Leach Soln:			2300	21.3	49.0	3.2	7.4		
Recycle Iodine:							50		
Water:								Total iodine 118.5	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
13:00	0	6.83	393
13:30	30	6.6	394
14:00	60	6.2	395
15:00	120	6.3	397

Stage 2

		pH	
Time	Minutes	read	mV
15:55	0		433
16:25	30		451
16:55	60		459
17:55	120	6.3	450

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				
Total Iodine reqd:						12.7	29.6		
Recycle KI Soln:			1700	22.7	39				
Recycle I ₂ :							30		
Water:		633						Total iodine 59.5	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, hold for next cycle, first stage leach
Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 7
Test No. 2-7

Date Started: _____

			Hg			I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-7-1	1st Filtrate	2460	385	947.1	81.3%	10.3	25.3	51.9	127.7
2-7-2	2nd Filtrate	2860	67	191.6	16.4%	2.9	8.3	13.9	39.8
2-7-3	Residue	958	28	26.8	2.3%			0.39%	3.7
Balance=					146%	Total iodine			164.5

Cementation:

Solution volume advancing to cementation 2460 mL
Adjust solution pH to **4.0** with 13 mL 1000 g/L H2SO4

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
	start				
15:40	advance		30 -35		
	Composite				

Column was plugged with solids, removed and cleaned turnings

Hg			
Sample ID	wt	mg/kg	mg
7 FeCleanout	120	17000	2040

Hg				
Sample ID	vol	mg/L	mg	
2-7-4	Discharge	3420	22	75.2

Iron Precipitation

Charge precipitation vessel 2440 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes Added 50 mL flocculant
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

		pH		g	
Time	Minutes	°C	Read	Adjust	Ca(OH)2
18:30	start	25.9	3.4		31
20:00	0		10		

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 7
Test No. 2-7

Date Started: _____

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-7-5	Filtrate	3265	1	3.3	14%	0.0634	0.207	37.5	122.4
2-7-6	Precipitate	30	680	20.4	86%				

Total iodine 93.8

Volume of iron precipitation filtrate available to advance 3250 mL 37.5 g/L KI
122 g KI

Iodide Recycle

Volume advanced to recycle 1600 mL containing 60.0 g KI

Iodine Recovery

Measure sufficient filtrate to contain 1650 mL neutralization filtrate to iodine recovery
62 grams potassium iodide for oxidation to I₂
62 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42	g H ₂ SO ₄ /g KI
25.988	g H ₂ SO ₄
25.988	mL 1000 g/L
0.23	g H ₂ O ₂ /g KI
14.231	g H ₂ O ₂
35.1	mL 35% H ₂ O ₂

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash

Adjust pH to less than 4.0, if necessary at 0 and 15 minutes

Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
8:40	0	41	63	25	1.09	
8:55	15			32	1.13	
9:25	30			32.7	1.22	

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-7-7	Filtrate	1740	0.5		
2-7-8	Precipitate	58			58

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2 Date Started: 1-Jul-98
Cycle No: 8
Test No. 2-8

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I2, g/l	I2, g	I2, g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				39
Total Iodine reqd:						25.4	59.3	0	
Advance Leach Soln:			2800	13.9	38.9	2.9	8.1		
Recycle Iodine:							51		
Water:		0						Total iodine 118.4	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Stage 1

		pH		°C
Time	Minutes	read	mV	
7:00	0	6.4	426	35
7:30	30	6.4	432	40
8:00	60	6.5	441	
9:00	120	6.3	440	65

Stage 2

		pH		°C
Time	Minutes	read	mV	
9:20	0	6.2	412	35
9:50	30	6.7	453	50
10:20	60	6.1	452	70
11:20	120	6.3	444	60

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I2, g/l	I2, g	I2, g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				
Total Iodine reqd:						12.7	29.6		
Recycle KI Soln:			270	22.7	6				
Recycle KI Soln:			1600	37.5	60				
Recycle I2:							7		
Water:		463						Total iodine 57.6	

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, hold for next cycle, first stage leach

Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 8
Test No. 2-8

Date Started: 1-Jul-98

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-8-1	1st Filtrate	2600	415	1079.0	93.7%	9.0	23.4	31.4	81.6
2-8-2	2nd Filtrate	2385	13	31.0	2.7%	3.8	9.1	7.6	18.1
2-8-3	Residue	872	47	41.0	3.6%			0.57%	5.0
Balance= 144%						Total iodine 112.5			

Cementation:

Solution volume advancing to cementation 2600 mL
Adjust solution pH to **4.0** with 9 mL 1000 g/L H2SO4

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
	start				
9:45	advance	25	30 -35	4.4	155
10:00		25	30 -35	4	91
10:15		25	31	4.2	69
10:30		25	32	4	152
10:45		25	32	3.9	151
11:00	Composite	25	32	3.8	150

Sample ID		Hg		
		vol	mg/L	mg
2-8-4	Discharge	2600	5	13.0

Iron Precipitation

Charge precipitation vessel 2600 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes
Transfer to vacuum filter, filter, and wash
Take sample of filtrate
Added 50 mL flocculant

Time	Minutes	°C	pH		g Ca(OH)2
			Read	Adjust	
	start				11.5
11:25	0	26	10.1		11.5
11:35	10	26	10.8	0.5	12
11:45	20	26	10.8		12
11:55	30	26	10.7		12

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date: 8/14/98
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Run No: 2
Cycle No: 8
Test No. 2-8

Date Started: 1-Jul-98

Sample ID		Hg				I ₂		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-8-5	Filtrate	2408	1	2.4	11%	0	0	30	72.2
2-8-6	Precipitate	52.8	385	20.3	89%				

Total iodine 55.2

Volume of iron precipitation filtrate available to advance 2270 mL 30.0 g/L KI
68 g KI

Iodide Recycle

Volume advanced to recycle 1695 mL containing 50.9 g KI

Iodine Recovery

Measure sufficient filtrate to contain _____ grams potassium iodide for oxidation to I₂
Volume advanced to iodine recycle 575 mL neutralization filtrate to iodine recovery
17 total grams potassium iodide

Acidify with 140% of stoich. H₂SO₄ for iodide oxidation reaction

0.42	g H ₂ SO ₄ /g KI
7.245	g H ₂ SO ₄
7.245	mL 1000 g/L

Add 120% of stoichiometric H₂O₂ for iodide oxidation reaction

0.23	g H ₂ O ₂ /g KI
3.9675	g H ₂ O ₂
9.8	mL 35% H ₂ O ₂

g 100% H₂O₂ / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.

Stir for 60 minutes, settle, decant, filter solids, do not wash
Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
Check filtrate for iodide and iodine

Time	Minutes	mL H ₂ SO ₄	mL H ₂ O ₂	°C	pH	
					read	adjusted
14:55	0	8	12	25	1.2	

Sample ID		Hg			I ₂
		wt/vol	mg/kg-L	mg	g
2-8-7	Filtrate	700			
2-8-8	Precipitate	13			13

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultant 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project I	971026-6
		Date:	8/14/98

Run No: 2
Cycle No: 9
Test No: 2-9

Date Started: _____

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
<u>5.0</u>	<u>1053</u>	<u>800</u>	<u>800</u>

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				60
Total Iodine reqd:						25.4	59.3	38	
Advance Leach Soln:			2300	7.6	17.5	3.8	8.7		
Recycle Iodine:							13		
Water:		0						Total iodine	118.5

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
11:50	0	7.1	422
12:20	30	6.2	476
12:50	60	6.3	429
13:50	120	6.2	425

Stage 2

		pH	
Time	Minutes	read	mV
15:15	0		413
15:45	30		413
16:15	60		439
17:15	120	6.4	444

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				
Total Iodine reqd:						12.7	29.6	29.6	
Recycle KI Soln:			1700	30	51				
Recycle I ₂ :									
Water:		633						Total iodine	68.6

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, hold for next cycle, first stage leach

Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUT Metallurgical & Mineral Processing Consultant 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project I 971026-6 Date: 8/14/98
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Run No: 2
Cycle No: 9
Test No. 2-9

Date Started: _____

			Hg			I2		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-9-1	1st Filtrate	2650	410	1086.5	88.5%	7.8	20.7	29.7	78.7
2-9-2	2nd Filtrate	2940	32	94.1	7.7%	4.9	14.4	16.5	48.5
2-9-3	Residue	947	50	47.4	3.9%			0.43%	4.1
Balance=					153%	Total iodine			135.4

Cementation:

Solution volume advancing to cementation 2630 mL
Adjust solution pH to **4.0** with 6.5 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
15:15	start	25		3.8	
17:20	advance				
	Composite				

Sample ID		Hg		
		vol	mg/L	mg
2-9-4	Discharge	2690	7.4	19.9

Iron Precipitation

Charge precipitation vessel 2670 mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes
Transfer to vacuum filter, filter, and wash
Take sample of filtrate
Added 2 mL flocculant

Time	Minutes	°C	pH		g Ca(OH) ₂
			Read	Adjust	
17:25	start	27	3.7		10
17:35	0		7.8		12
17:45	10		10		12
	20				
	30				

COLORADO MINERALS RESEARCH INSTITUT Metallurgical & Mineral Processing Consultant 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project I 971026-6 Date: 8/14/98
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Run No: 2 Date Started: _____
Cycle No: 9
Test No. 2-9

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-9-5	Filtrate	2550	3.9	9.9	35%	0	0	37.3	95.1
2-9-6	Precipitate	35.6	520	18.5	65%				
Total iodine									72.7

Volume of iron precipitation filtrate available to advance 2530 mL 37.3 g/L KI
94 g KI

Iodide Recycle

Volume advanced to recycle 1030 mL containing 38.4 g KI

Iodine Recovery

Volume advanced to iodine recycle 1500 mL neutralization filtrate to iodine recovery
56 total grams potassium iodide

Acidify with 140% of stoich. H2SO4 for iodide oxidation reaction 0.42 g H2SO4/g KI
23.499 g H2SO4
23.499 mL 1000 g/L
Add 120% of stoichiometric H2O2 for iodide oxidation reaction 0.23 g H2O2/g KI
12.869 g H2O2
45.0 mL 35% H2O2
g 100% H2O2 / 35%/1.13 = mL 35%

If insufficient solution, use what is available and ratio reagent additions.
Stir for 60 minutes, settle, decant, filter solids, do not wash
Adjust pH to less than 4.0, if necessary at 0 and 15 minutes
Check filtrate for iodide and iodine

Time	Minutes	mL H2SO4	mL H2O2	°C	pH	
					read	adjusted
19:00	0	29	45		1.35	
	0			28	1.35	

Sample ID		Hg			I2
		wt/vol	mg/kg-L	mg	g
2-9-7	Filtrate	2000	0.7		
2-9-8	Precipitate	48.9			48.9

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2 Date Started: _____
Cycle No: 10
Test No. 2-10

Feed: 1000 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	1053	800	800

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000	53							
Total KI reqd:				33.2	77.5				30
Total Iodine reqd:						25.4	59.3	0	
Advance Leach Soln:			2900	16.5	47.9	4.9	14.2		
Recycle Iodine:							45		
Water:		0						Total iodine	118.5

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, transfer to cementation
Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
7:20	0		409
7:40	30		419
8:20	60		415
9:20	120	6.2	417

Stage 2

		pH	
Time	Minutes	read	mV
10:15	0		450
10:45	30		460
11:15	60		454
12:15	120	6.4	446

Stage 2:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	1000								
Total KI reqd:				16.6	38.7				
Total Iodine reqd:						12.7	29.6	25.6	
Recycle KI Soln:			1030	38.4	40				
Recycle I ₂ :							4		
Water:		1303						Total iodine	59.9

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes
Transfer to vacuum filter, filter, wash with 500 mL water or iodine recovery wash filtrate
Measure volume of filtrate plus wash, hold for next cycle, first stage leach
Take sample of filtrate for analysis

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 10
Test No. 2-10

Date Started: _____

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-10-1	1st Filtrate	2650	420	1113.0	90.0%	11.1	29.4	41.7	110.5
2-10-2	2nd Filtrate	2760	24	66.2	5.4%	2.0	5.5	12.4	34.2
2-10-3	Residue	823	69	56.8	4.6%			0.53%	4.4
Balance= 155%						Total iodine 148.9			

Cementation:

Solution volume advancing to cementation mL
Adjust solution pH to **4.0** with mL 1000 g/L H2SO4
Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Time	Minutes	°C	cc/min	pH	mV
	start				
	advance				
10:20		30	35	4.05	-25
10:40		30	40	3.8	105
11:00		28	38	3.4	250
11:20			34	3.3	303
12:00	Composite	25		3.3	320

Sample ID		Hg		
		vol	mg/L	mg
2-10-4	Discharge	3970	7.3	29.0
			mg/kg	
Clean out	Iron solids	20	110000	2200

Iron Precipitation

Charge precipitation vessel mL discharge solution
Adjust to 10-11 pH by addition of dry hydrated lime
Maintain at 10-11 pH for 30 minutes Added _____ mL flocculant
Transfer to vacuum filter, filter, and wash
Take sample of filtrate

Time	Minutes	°C	pH		g Ca(OH)2
			Read	Adjust	
12:10	start				24.6
12:10	0	27	10.1		
12:20	10	27	10.6		
12:30	20	27	10.6		
12:40	30	27	10.6		

Sample ID		Hg				I2		KI	
		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-10-5	Filtrate	3600	0.7	2.5	6%		0	59	212.4
2-10-6	Precipitate	70	520	36.4	94%				
						Total iodine 162.4			

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET		
Metallurgical & Mineral Processing Consultants			Project Nc	971026-6
5906 McIntyre Street	(303) 279-2581		Date:	8/14/98
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 11
Test No. 2-11

Date Started: 10-Aug-98

Feed: 200 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	211	800	160

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	200	11							
Total KI reqd:				66.4	31.0				23.3
Total Iodine reqd:						50.8	23.7	24	
Stage 2 Leach Soln:			456	16.9	7.7		0.0		
Water:									
								Total iodine	47.4

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
8:45	0		
	30		
9:45	60		
10:45	120	6.3	387

Stage 2

		pH	
Time	Minutes	read	mV
11:40	0		
	30		
12:40	60		
13:40	120		

Stage 2:

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	200								
Total KI reqd:				33.4	15.6				0
Total Iodine reqd:						25.4	11.9	12	
Recycle KI Soln:			239	62.85	15.0		0.0		
Water:		228						Total iodine	23.5

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, hold for next cycle, first stage leach

Take sample of filtrate for analysis

		Hg				I ₂		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-11-1	1st Filtrate	475	505	239.9	90.4%	23.54	11.2	39.4	18.7
2-11-2	2nd Filtrate	622	32	19.9	7.5%	5.2	3.2	20.1	12.5
2-11-3	Residue	191.9	28.4	5.4	2.1%			0.39%	0.7

Balance= 166%

Total iodine 38.9

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No. 971026-6
		Date: 8/14/98

Run No: 2 Date Started: 10-Aug-98
 Cycle No: 11
 Test No. 2-11

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation 450 mL
 Adjust solution pH to **4.0** with 0 mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
 Recirculate for 30 minutes then advance until all solution is processed
 Take solution sample for analysis

		Hg			I ₂		KI	
Sample ID		vol	mg/L	mg	g/L	g	g/L	g
2-11-4	Discharge	450	8.1	3.6	0	0	70.0	31.5
Discharge sample			Total iodine					
			24.1					

COLORADO MINERALS RESEARCH INSTITUTE		LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project Nc	971026-6
Metallurgical & Mineral Processing Consultants			Date:	8/14/98
5906 McIntyre Street	(303) 279-2581			
Golden, CO 80403	FAX 279-6061			

Run No: 2
Cycle No: 12
Test No. 2-12

Date Started: 10-Aug-98

Feed: 200 dry grams

% moisture	Damp wt.	Hg, mg/kg	Hg, mg
5.0	211	800	160

Leach: 30% solids 2 hours 60°C

Stage 1:

		Liquid		Iodide		Iodine		Reagent Added	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	200	11							
Total KI reqd:				83.0	38.7				31
Total Iodine reqd:						63.5	29.6	29.6	
Stage 2 Leach Soln:			456	16.9	7.7		0.0		
Water									
								Total iodine	59.2

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, transfer to cementation

Take sample of filtrate for analysis

Stage 1

		pH	
Time	Minutes	read	mV
	0		
	30		
	60		
	120		

Stage 2

		pH	
Time	Minutes	read	mV
	0		
	30		
	60		
	120		

Stage 2:

		Liquid		Iodide		Iodine		Reagent To Add	
	dry g	grams	mL	KI, g/l	KI, g	I ₂ , g/l	I ₂ , g	I ₂ , g	KI, g
Solids feed:	200								
Total KI reqd:				41.7	19.5				0
Total Iodine reqd:						31.8	14.8	15	
Recycle KI Soln:			302	62.85	19.0		0.0		
Water:		165						Total iodine	29.5

Leach 2 hours, measure pH and emf at 30, 60, and 120 minutes

Transfer to vacuum filter, filter, wash with 200 mL water or iodine recovery wash filtrate

Measure volume of filtrate plus wash, hold for next cycle, first stage leach

Take sample of filtrate for analysis

		Hg				I ₂		KI	
Sample ID		wt/vol	mg/kg-L	mg	Distn	g/L	g	g/L	g
2-12-1	1st Filtrate	675	365	246.4	90.3%	21.8	14.7	50	33.8
2-12-2	2nd Filtrate	690	25	17.3	6.3%	9.8	6.8	29.9	20.6
2-12-3	Residue	202.6	45	9.1	3.34%			0.62%	1.3
				Balance=	170%	Total iodine 64.0			

COLORADO MINERALS RESEARCH INSTITUTE Metallurgical & Mineral Processing Consultants 5906 McIntyre Street (303) 279-2581 Golden, CO 80403 FAX 279-6061	LOCKED-CYCLE CHEMICAL EXTRACTION OF MERCURY- BEARING WASTE DATA SHEET	Project No 971026-6 Date: 8/14/98
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Run No: 2 Date Started: 10-Aug-98
Cycle No: 12
Test No. 2-12

Cementation:

Charge precipitation column with 500 grams iron turnings

Solution volume advancing to cementation mL
Adjust solution pH to **4.0** with mL 1000 g/L H₂SO₄

Start leach solution circulating through column of iron turnings
Recirculate for 30 minutes then advance until all solution is processed
Take solution sample for analysis

Sample ID		vol	Hg			I ₂		KI	
			mg/L	mg		g/L	g	g/L	g
2-12-4	Discharge	620	11.6	7.2		0.01	0.0062	69.1	42.8
Discharge sample			Total iodine						
			32.8						

Appendix B

Equipment List and Mass Balance

Appendix B-1

Equipment List for Pilot-Scale GEMEP System

EQUIPMENT LIST FOR GEMEP PROCESS

ITEM No.	NAME	DESCRIPTION	CRITERIA	MATERIALS OF CONSTRUCTION	VENDOR	HP	UNIT COST
FDR-1	Soil Feeder	6-cu ft bin, belt, sheaves, drive	30 kg/hr	steel, rubber	CMRI	0.25	2,750
SCR-1	De-agglomerator	Rotary scrubber, 3-dia, 2.5' scrubber, 6" screen	30 kg/hr 15 min	steel	CMRI	0.5	1,750
THK-0	Feed Thickener	Minus 4-mesh feed thickener	30 kg/hr	polypro, stainless			
		Includes: flocculant metering pump			Fisher	0.12	160
		flocculation tank and mixer	8" x 8": x 8", 3" dia		US Plastic	0.12	250
		thickener tank and drive	24" dia	polypro, stainless	CMRI	0.25	1,000
		underflow positive displacement pump	60 lph, var speed		Moyno	0.25	1,100
		overflow pump, magnetic coupled centr	1 lpm		Little Giant	0.12	150
T-1	Thickener Overflow Tank	Surge tank for overflow recycle solution	180 l, 55-gal	poly	US Plastic		75
P-1	Circulation Pump	magnetic coupled centr	1 lpm		Little Giant	0.12	150
RT-1	Feed repulper	18" x 18" x 18" agitated feed tank	40 min	poly	US Plastic		150
		drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
Feed preparation module subtotal						2.0	7,935
FDR-2	Potassium iodide feeder	3/8-inch diameter screw feeder, variable speed	2 kg/hr capacity	stainless	CMRI	0.12	2,000
FDR-3	Iodine feeder	3/8-inch diameter screw feeder, variable speed	2 kg/hr capacity	stainless	CMRI	0.12	2,000
LT-1	Leach Tank 1	18" x 18" x 18" agitated leach tank	40 min	poly	US Plastic		150
		includes: drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
		heater	1500 watt	stainless	Cole Parm		300
LT-2	Leach Tank 2	18" x 18" x 18" agitated leach tank	40 min	poly	US Plastic		150
		includes: drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
		heater	1500 watt	stainless	Cole Parm		300
LT-3	Leach Tank 3	18" x 18" x 18" agitated leach tank	40 min	poly	US Plastic		150
		includes: drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
		heater	1500 watt	stainless	Cole Parm		300

EQUIPMENT LIST FOR GEMEP PROCESS

ITEM No.	NAME	DESCRIPTION	CRITERIA	MATERIALS OF CONSTRUCTION	VENDOR	HP	UNIT COST
THK-1	Leach Thickener 1	Minus 4-mesh feed thickener Includes: flocculant metering pump floculation tank and mixer thickener tank and drive underflow positive displacement pump overflow pump, magnetic coupled centr	30 kg/hr 8" x 8": x 8", 3" dia 24" dia 60 lph, var speed 1 lpm	polypro, stainless polypro, stainless	Fisher US Plastic CMRI Moyno Little Giant	0.12 0.12 0.25 0.25 0.12	160 250 1,000 1,100 150
FDR-4	Iodine feeder	3/8-inch diameter screw feeder, variable speed	2 kg/hr capacity	stainless		0.12	2,000
LT-4	Leach Tank 4	18" x 18" x 18" agitated leach tank drill press drive, agitator heater	40 min 4" dia impeller 1500 watt	poly stainless stainless	US Plastic Grainger Cole Parm	0.25	150 400 300
LT-5	Leach Tank 5	18" x 18" x 18" agitated leach tank drill press drive, agitator heater	40 min 4" dia impeller 1500 watt	poly stainless stainless	US Plastic Grainger Cole Parm	0.25	150 400 300
THK-2	Leach Thickener 2	Minus 4-mesh feed thickener Includes: flocculant metering pump floculation tank and mixer thickener tank and drive underflow positive displacement pump overflow pump, magnetic coupled centr	30 kg/hr 8" x 8": x 8", 3" dia 24" dia 60 lph, var speed 1 lpm	polypro, stainless polypro, stainless	Fisher US Plastic CMRI Moyno Little Giant	0.12 0.12 0.25 0.25 0.12	160 250 1,000 1,100 150
Leaching module subtotal						3.3	15,570
RT-2	Wash Repulper 1	18" x 18" x 18" agitated leach tank drill press drive, agitator	40 min 4" dia impeller	poly stainless	US Plastic Grainger	0.25	150 400
THK-3	Wash Thickener 1	Minus 4-mesh feed thickener Includes: flocculant metering pump floculation tank and mixer thickener tank and drive underflow positive displacement pump	30 kg/hr 8" x 8": x 8", 3" dia 24" dia 60 lph, var speed	polypro, stainless polypro, stainless	Fisher US Plastic CMRI Moyno	0.12 0.12 0.25 0.25	160 250 1,000 1,100

EQUIPMENT LIST FOR GEMEP PROCESS

ITEM No.	NAME	DESCRIPTION	CRITERIA	MATERIALS OF CONSTRUCTION	VENDOR	HP	UNIT COST
		overflow pump, magnetic coupled centr	1 lpm		Little Giant	0.12	150
RT-3	Wash Repulper 2	18" x 18" x 18" agitated leach tank	40 min	poly	US Plastic		150
		drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
THK-4	Wash Thickener 2	Minus 4-mesh feed thickener	30 kg/hr	polypro, stainless			
		Includes: flocculant metering pump			Fisher	0.12	160
		flocculation tank and mixer	8" x 8": x 8", 3" dia		US Plastic	0.12	250
		thickener tank and drive	24" dia	polypro, stainless	CMRI	0.25	1,000
		underflow positive displacement pump	60 lph, var speed		Moyno	0.25	1,100
		overflow pump, magnetic coupled centr	1 lpm		Little Giant	0.12	150
RT-3	Wash Repulper 3	18" x 18" x 18" agitated leach tank	40 min	poly	US Plastic		150
		drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
THK-5	Wash Thickener 3	Minus 4-mesh feed thickener	30 kg/hr	polypro, stainless			
		Includes: flocculant metering pump			Fisher	0.12	160
		flocculation tank and mixer	8" x 8": x 8", 3" dia		US Plastic	0.12	250
		thickener tank and drive	24" dia	polypro, stainless	CMRI	0.25	1,000
		underflow positive displacement pump	60 lph, var speed		Moyno	0.25	1,100
		overflow pump, magnetic coupled centr	1 lpm		Little Giant	0.12	150
RT-4	Wash Repulper 4	18" x 18" x 18" agitated leach tank	40 min	poly	US Plastic		150
		drill press drive, agitator	4" dia impeller	stainless	Grainger	0.25	400
THK-6	Wash Thickener 4	Minus 4-mesh feed thickener	30 kg/hr	polypro, stainless			
		Includes: flocculant metering pump			Fisher	0.12	160
		flocculation tank and mixer	8" x 8": x 8", 3" dia		US Plastic	0.12	250
		thickener tank and drive	24" dia	polypro, stainless	CMRI	0.25	1,000
		underflow positive displacement pump	60 lph, var speed		Moyno	0.25	1,100
		overflow pump, magnetic coupled centr	1 lpm		Little Giant	0.12	150
FIL-1	Residue Filter	Plate and frame pressure filter	2 cu ft	poly, stainless	Hoesch		7,050
T-2	Thickener Overflow Tank	Surge tank for wash recycle solution	180 l, 55-gal	poly	US Plastic		75

EQUIPMENT LIST FOR GEMEP PROCESS

ITEM No.	NAME	DESCRIPTION	CRITERIA	MATERIALS OF CONSTRUCTION	VENDOR	HP	UNIT COST
P-2	Circulation Pump	magnetic coupled centr	1 lpm		Little Giant	0.12	150
Residue washing module subtotal						4.6	20,115
AT-1	Preg pH Adjustment	18" x 18" x 18" agitated tank drill press drive, agitator	40 min 4" dia impeller	poly stainless	US Plastic Grainger	0.25	150 400
P-3	Sulfuric acid metering	Peristaltic metering pump includes pH controller	0.06 lph		Masterflex Cole Parmer	0.12	350 600
P-4	Mercury recovery feed	underflow positive displacement pump	60 lph, var speed		Moyno	0.25	1,100
F-4	Polishing filter	Cartridge filter	5 micron	poly	US Plastic		50
C-1, 2, 3	Mercury precipitation	Glass columns containing iron turnings	4" dia x 4' tall	Pyrex glass	Ace Glass		600
FDR-5	Lime feeder	3/8-inch diameter screw feeder, variable speed includes pH controller	2 kg/hr capacity	stainless	CMRI Cole Parmer	0.12	2,000 600
AT-2	Iron precipitation	18" x 18" x 18" agitated tank drill press drive, agitator	40 min 4" dia impeller	poly stainless	US Plastic Grainger	0.25	150 400
P-5	Iron filter pump	magnetic coupled centr	1 lpm		Little Giant	0.12	150
F-2	Iron filter	Multiple cartridge	5 micron		US Plastic		220
T-3	Iron filtrate surge	Surge tank for iron filtrate	180 l, 55-gal	poly	US Plastic		75
P-6	Circulation Pump	magnetic coupled centr	1 lpm		Little Giant	0.12	150
E-1	Evaporator	Potassium iodide solutioin concentrator	20 kg/hr evaporate 9 kw	stainless, glass	CMRI Grainger		4,000
T-4	Recycle KI solution	Surge tank for concentrated KI soslution	180 l, 55-gal	poly	US Plastic		75

EQUIPMENT LIST FOR GEMEP PROCESS

ITEM No.	NAME	DESCRIPTION	CRITERIA	MATERIALS OF CONSTRUCTION	VENDOR	HP	UNIT COST
P-7	Circulation Pump	magnetic coupled centr	1 lpm		Little Giant	0.12	150
T-5	Recycle condensate	Surge tank for condensate	180 l, 55-gal	poly	US Plastic		75
AT-1	Preg pH Adjustment	18" x 18" x 18" agitated tank drill press drive, agitator	40 min 4" dia impeller	poly stainless	US Plastic Grainger	0.25	150 400
P-8	Sulfuric acid metering	Peristaltic metering pump includes pH controller	0.06 lph		Masterflex Cole Parmer	0.12	350 600
P-9	Hydrogen peroxide	Peristaltic metering pump	0.06 lph		Masterflex	0.12	350
T-6	Process water surge	Surge tank for process water	180 l, 55-gal	poly	US Plastic		75
P-9	Circulation Pump	magnetic coupled centr	1 lpm		Little Giant	0.12	150
Solution treatment module						2.0	13,370
Total Equipment						11.8	56,990

Appendix B-2

Mass Balance for GEMEP Treatment of INEEL Waste

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	% solids	Deg C	%-g/L	K2Hgl4 kg/hr	geq/hr	%-g/L	Hg kg/hr	geq/hr	%-g/L	Potassium Iodide kg/hr	kgeq/hr	%-g/L	Iodine kg/hr	kgeq/hr
1	Soil	Solid	22.7	9.5	2.40	95%	20				0.09%	0.0204	0.204						
		Liquid	1.2	1.2	1.00														
		Total	23.9	10.7	2.24							0.0204	0.00						
2	Deaglom liquid	Liquid	51.8	51.8	1.00		20	0.00	0.00005	0.0001									
3	Deaglom slurry	Solid	22.7	9.5	2.40	30%	20				0.09%	0.0204	0.204						
		Liquid	53.0	53.0	1.00			0.00	0.00005	0.0001									
		Total	75.7	62.4	1.21				0.00005			0.0204	0.00						
4	Screen wash	Liquid	23.1	23.1	1.00		20	0.00	0.00000	0.0000									
5	Screen oversize	Solid	2.0	0.9	2.40	95%	20				0.002%	0.0000	0.000						
		Liquid	0.1	0.1	1.00			0.00	0.00000	0.0000									
		Total	2.2	1.0	2.24				0.00000			0.0000	0.00						
6	Screen undersize	Solid	20.7	8.6	2.40	21%	20				0.099%	0.0204	0.203						
		Liquid	76.0	76.0	1.00			0.00	0.00005	0.0001									
		Total	96.7	84.6	1.14				0.00005			0.02	0.00						
7	Flocculant	Liquid	2.1	2.1	1.00		20	0.00	0.00000	0.0000									
8	Thickener feed	Solid	20.7	8.6	2.4	21%	20				0.099%	0.0204	0.203						
		Liquid	78.1	78.1	1.00			0.00	0.00005	0.0001									
		Total	98.7	86.7	1.14				0.00005			0.02	0.00						
9	Feed Thickener uflo	Solid	20.7	8.6	2.40	44%	20				0.10%	0.0204	0.203						
		Liquid	26.3	26.3	1.00			0.00	0.00000	0.0000									
		Total	46.9	34.9	1.35				0.00000			0.02	0.00						
33	Feed THK oflo	Liquid	51.8	51.8	1.00		20	0.00	0.00005	0.0001									
22	Leach THK 2 oflo	Liquid	79.7	77.5	1.00		50	0.43	0.03390					42.2	3.3	0.020	14.4	1.1	0.009
10	Potassium Iodide	Liquid	0.0	0.0	1.00		20	0.24	0.00000	0.0000					0.2	0.001			
39	Iodine make-up	Solid	1.5	0.3	4.60													1.5	0.012

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	% solids	Deg C	%-g/L	K2Hgl4 kg/hr	geq/hr	%-g/L	Hg kg/hr	geq/hr	%-g/L	Potassium Iodide kg/hr	kgeq/hr	%-g/L	Iodine kg/hr	kgeq/hr													
37	Leach 1	Solid	20.7	8.6	2.40	16%	50	0.33	0.03390	0.0862	0.099%	0.02	0.203	33.2	3.4	0.021	25.4	2.63	0.021													
		Liquid	107.5	104	1.04							0.02	0.00																			
		Total	128.2	112	1.14																											
38	Vapor					16%	50	1.02	0.10584	0.2692	0.010%	0.0020	0.020	36.5	3.8	0.023	21.2	2.20	0.017													
		Liquid	107.5	104	1.04							0.00	0.00																			
		Total	128.2	112	1.14																											
12	Flocculant	Liquid	5.2	5.2	1.00	15%	50	0.97	0.10584	0.2692	0.01%	0.0020	0.020	90.7	0.5	0.003	20.2	2.2	0.017													
		Liquid	112.7	109	1.03							0.00	0.00																			
		Total	133.3	118	1.13																											
14	Leach THK 1 uflo	Solid	20.7	8.6	2.40	42%	50	0.97	0.02771	0.0705	0.01%	0.0020	0.020	39.1	1.1	0.007	20.2	0.58	0.005													
		Liquid	28.5	28.5	1.00							0.00	0.00																			
		Total	49.2	37.1	1.32																											
15	THK 1 oflo Preg Soln	Liquid	84.1	80	1.05	17%	50	0.40	0.04081	0.1038	0.01%	0.0020	0.020	39.1	3.1	0.019	20.2	1.6	0.013													
		Liquid	54.8	54.8	1.00							0.00	0.00																			
		Liquid	17.5	17.5	1.00																											
19	Wash THK 1 oflo Soln B	Liquid	54.8	54.8	1.00	17%	50	0.40	0.04081	0.1038	0.01%	0.0020	0.020	39.7	4.0	0.024	17.8	1.79	0.014													
		Liquid	17.5	17.5	1.00							0.00	0.00																			
		Total	123.7	109	1.13																											
36	Vapor					17%	50	0.46	0.04638	0.1179	0.003%	0.0006	0.006	39.7	4.0	0.024	15.1	1.52	0.012													
		Liquid	103.1	101	1.02							0.00	0.00																			
		Total	123.7	109	1.13																											
18	Flocculant	Liquid	5.2	5.2	1.00	40	0.00	0.00000	0.0000					90.7	0.5	0.003																

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	% solids	Deg C	%-g/L	K2Hgl4 kg/hr	geq/hr	%-g/L	Hg kg/hr	geq/hr	Potassium Iodide %-g/L	kg/hr	kgeq/hr	Iodine %-g/L	kg/hr	kgeq/hr
20	LeachTHK 2 mix	Solid	20.7	8.6	2.40	16%	50				0.003%	0.0006	0.006						
		Liquid	108.2	106	1.02			0.44	0.04638	0.1179				42.2	4.5	0.027	14.4	1.52	0.012
		Total	128.9	115	1.12				0.04638			0.00	0.00						
21	Leach THK 2 uflo	Solid	20.7	8.6	2.40	42%	50				0.003%	0.0006	0.006						
		Liquid	28.5	28.5	1.00			0.44	0.01248	0.0317	0.11			42.2	1.2	0.007	14.37	0.41	0.003
		Total	49.2	37.1	1.32				0.01248			0.00	0.00						
22	Leach THK 2 oflo	Liquid	79.7	77.5	1.00		50	0.43	0.03390	0.0862				42.2	3.3	0.020	14.37	1.11	0.009
23	Wash THK 2 oflo	Liquid	49.5	49.5	1.00		34	0.15	0.00720	0.0183				15.0	0.7	0.004	4.0	0.20	0.002
24	Repulper 1	Solid	20.7	8.6	2.40	21%	40				0.003%	0.0006	0.006						
		Liquid	78.0	78.0	1.00			0.25	0.01968	0.0500				15.0					
		Total	98.7	86.6	1.14				0.01968			0.00	0.00	24.9	1.95	0.012	7.8	0.61	0.005
25	Flocculant	Liquid	4.1	4.1	1.00		40	0.00	0.00000	0.0000				0.0	0.0	0.000			
26	Flocculated slurry	Solid	20.7	8.6	2.40	20%	40				0.003%	0.0006	0.006						
		Liquid	82.2	82.2	1.00			0.24	0.01968	0.0500				23.7	1.95	0.012	7.4	0.61	0.005
		Total	102.8	90.8	1.13				0.01968			0.00	0.00						
27	WashTHK 1 uflo	Solid	20.7	8.6	2.40	43%	40				0.003%	0.00062	0.006						
		Liquid	27.4	27.4	1.00			0.24	0.00656	0.0167	0.061			23.7	0.65	0.004	7.40	0.20	0.002
		Total	48.0	36.0	1.33				0.00656			0.00	0.00						
19	Wash THK 1 oflo	Liquid	54.8	54.8	1.00		40	0.24	0.01312	0.0334				23.7	1.30	0.008	7.40	0.41	0.003
64	Wash THK 3 oflo	Liquid	45.3	45.3	1.00		29	0.10	0.00440	0.0112				10.8	0.49	0.003	2.2	0.10	0.001
29	Repulper 2	Solid	20.7	8.6	2.40	22%	34				0.003%	0.0006	0.006						
		Liquid	72.7	72.7	1.00			0.15	0.01096	0.0279				15.7	1.14	0.007	4.2	0.30	0.002
		Total	93.4	81.3	1.15				0.01096			0.00	0.00						
30	Flocculant	Liquid	3.1	3.1	1.00		40	0.00	0.00000	0.0000				0.0	0.0	0.000			
31	Flocculated slurry	Solid	20.7	8.6	2.40	21%	34				0.003%	0.0006	0.006						
		Liquid	75.8	75.8	1.00			0.14	0.01096	0.0279				15.0	1.14	0.007	4.0	0.30	0.002
		Total	96.5	84.4	1.14				0.01096			0.00	0.00						

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	% solids	Deg C	%-g/L	K2Hgl4 kg/hr	geq/hr	%-g/L	Hg kg/hr	geq/hr	%-g/L	Potassium Iodide kg/hr	kgeq/hr	%-g/L	Iodine kg/hr	kgeq/hr	
32	WashTHK 2 uflo	Solid	20.7	8.6	2.40	44%	34				0.003%	0.0006	0.006							
		Liquid	26.3	26.3	1.00			0.14	0.00380	0.0097	0.037			15.0	0.39	0.002	3.99	0.10	0.001	
		Total	46.9	34.9	1.35				0.00380			0.00	0.00							
23	Wash THK 2 oflo	Liquid	49.5	49.5	1.00		34	0.14	0.00716	0.0182					15.0	0.74	0.004	3.99	0.20	0.002
72	Wash THK 4 oflo + filt	Liquid	41.2	41.2	1.00		25	0.03	0.00120	0.0031					5.7	0.23	0.001	0.7	0.03	0.000
60	Repulper 3	Solid	20.7	8.6	2.40	23%	29				0.003%	0.0006	0.006							
		Liquid	67.5	67.5	1.00			0.07	0.00500	0.0127			9.3	0.63	0.004	2.0	0.13	0.001		
		Total	88.1	76.1	1.16				0.00500			0.00	0.00							
61	Flocculant	Liquid	4.1	4.1	1.00		40	0.00	0.00000	0.0000					0.0	0.0	0.000			
62	Flocculated slurry	Solid	20.7	8.6	2.40	22%	29				0.003%	0.0006	0.006							
		Liquid	71.6	71.6	1.00			0.07	0.00500	0.0127			8.8	0.63	0.004	1.8	0.13	0.001		
		Total	92.3	80.2	1.15				0.00500			0.00	0.00							
63	WashTHK 3 uflo	Solid	20.7	8.6	2.40	44%	29				0.003%	0.0006	0.006							
		Liquid	26.3	26.3	1.00			0.07	0.00184	0.0047	0.018			8.8	0.23	0.001	1.84	0.05	0.000	
		Total	46.9	34.9	1.35				0.00184			0.00	0.00							
64	Wash THK 3 oflo	Liquid	45.3	45.3	1.00		29	0.00	0.00316	0.0080					8.8	0.40	0.002	1.84	0.08	0.001
28	Process water	Liquid	20.7	20.7	1.00		40	0.00	0.00000	0.0000					0.0	0.0	0.000			
65	Repulper 4	Solid	20.7	8.6	2.40	31%	34				0.003%	0.0006	0.006							
		Liquid	46.9	46.9	1.00			0.04	0.00184	0.0047			4.9	0.23	0.001	1.0	0.05	0.000		
		Total	67.6	55.6	1.22				0.00184			0.00	0.00							
66	Flocculant	Liquid	3.1	3.1	1.00		40	0.00	0.00000	0.0000					0.0	0.0	0.000			
67	Flocculated slurry	Solid	20.7	8.6	2.40	29%	34				0.003%	0.0006	0.006							
		Liquid	50.0	50.0	1.00			0.04	0.00184	0.0047			4.6	0.23	0.001	1.0	0.05	0.000		
		Total	70.7	58.7	1.21				0.00184			0.00	0.00							

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	% solids	Deg C	%-g/L	K2Hgl4 kg/hr	geq/hr	%-g/L	Hg kg/hr	geq/hr	%-g/L	Potassium Iodide kg/hr	kgeq/hr	%-g/L	Iodine kg/hr	kgeq/hr													
68	WashTHK 4 uflo	Solid	20.7	8.6	2.40	44%	34	0.04	0.00096	0.0025	0.003%	0.0006	0.006	0.009	4.6	0.12	0.001	0.97	0.03	0.000												
		Liquid	26.3	26.3	1.00																											
		Total	46.9	34.9	1.35																											
69	Wash THK 4 oflo	Liquid	23.8	23.8	1.00	70%	34	0.04	0.00087	0.0022	0.003%	0.0006	0.006	0.000	4.6	0.11	0.001	0.97	0.023	0.000												
70	Final filter cake	Solid	20.7	34.9	2.40																											
Liquid		8.9	8.9	1.00																												
		Total	29.5	43.8	0.67			0.00000			0.000	0.000	0.000	4.6	0.04	0.000	1.0	0.01	0.000													
								0.00000			0.00		0.00																			
71	Filtrate	Liquid	17.4	17.4	1.00		34	0.00	0.00000	0.0000				4.6	0.08	0.000	1.0	0.02	0.000													
15	THK 1 oflo Preg Soln	Liquid	84.1	80.4	1.0		50	0.97	0.07813	0.1987				39.1	3.1	0.019	20.2	1.6	0.013													
73	Sulfuric Acid		0.10	0.06	1.83															20												
40	Feed to Hg removal	Liquid	84.3	80.5	1.0																											
41	Iron to columns	Solid																														
42	Mercury barrens	Liquid	84.3	80.5	1.0	3%	45	0.01	0.00078	0.0020	0.002		0.0197	0.196	65.5	5.3	0.032															
43	Mercury in column	Liquid	0.02	0.00	13.5																											
44	Hydrated lime to precip	Solid	0.6	0.3	2.2																											
45	Iron slurry	Solid	2.9	1.9	1.50																											
		Liquid	82.0	78.9	1.04																											
		Total	84.9	80.8	1.05																											
46	Filter cake wash	Liquid	8.8	8.8	1	50%	38							1.8	0.00	0.000																
47	Iron filter cake	Solid	2.9	1.9	1.50																											
		Liquid	2.9	1.0	1.00																											
		Total	5.8	2.9	1.98																											
48	Iron Precip filtrate	Liquid	87.8	86.6	1.01	38								60.8		5.3		0.032														

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	% solids	Deg C	%-g/L	K ₂ HgI ₄ kg/hr	geq/hr	%-g/L	Hg kg/hr	geq/hr	%-g/L	Potassium Iodide kg/hr	kgeq/hr	%-g/L	Iodine kg/hr	kgeq/hr
49	KI to evaporation	Liquid	40.8	40.3	1.01		38								60.8	2.45	0.015		
50	KI to iodine recovery	Liquid	47.0	46.4	1.01		38								60.8	2.8	0.017		
51	98% sulfuric acid	Liquid	0.6	0.3	1.83		20												
52	30% hydrogen peroxide	Liquid	0.6	0.5	1.19		20												
53	Iodine slurry	Solid	2.2	0.5	4.60	4%	40										100%	2.16	0.017
		Liquid	46.0	46.7	0.99										0.0	0.0	0.000		
		Total	48.2	47.2	1.02														
54	Iodine crystals	Solid	2.2	0.5	4.6		40										100%	2.16	0.017
55	Filtrate	Liquid	46.0	46.7	1.0		40								0.0	0.0	0.000		
57	Evaporated KI	Liquid	27.9	27.0	1.03		70								90.7	2.5	0.015		
58	Condensate	Liquid	12.9	13.3	0.0										0.0	0.0	0.0		
56	Process water	Liquid	59.0	60.0	1.0		40								0.0	0.0	0.000		
	Solution balance																		
	To feed prep		26.4																
	To leach		26.3																
	Make-up water to prep		25.2																
	Preg out		84.1																
	KI in		27.9																
	Process water in		60.2																
	Make-up water to leach		3.9																

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	Iron % kg/hr kgeq/hr	Sulfuric Acid kg/hr kgeq/hr	Hydrogen Peroxide %-g/L kg/hr kgeq/hr	Calcium kg/hr kgeq/hr	Value	Control	Value	Control
1	Soil	Solid	22.7	9.5	2.40					5% moisture		20	ambient temp
		Liquid	1.2	1.2	1.00					0.09% Hg			
		Total	23.9	10.7	2.24								
2	Deaglom liquid	Liquid	51.8	51.8	1.00					0.001 wash g/L K2Hgl4			
3	Deaglom slurry	Solid	22.7	9.5	2.40					70% moisture			
		Liquid	53.0	53.0	1.00								
		Total	75.7	62.4	1.21								
4	Screen wash	Liquid	23.1	23.1	1.00					make-up			
5	Screen oversize	Solid	2.0	0.9	2.40					9% of solids			
		Liquid	0.1	0.1	1.00					5% moisture			
		Total	2.2	1.0	2.24					0.002% Hg			
6	Screen undersize	Solid	20.7	8.6	2.40					0 g/L K2Hgl4			
		Liquid	76.0	76.0	1.00								
		Total	96.7	84.6	1.14								
7	Flocculant	Liquid	2.1	2.1	1.00					0.2 lb/ton in water			
										0.10% concentration			
8	Thickener feed	Solid	20.7	8.6	2.4					0.000 g/L K2Hgl4			
		Liquid	78.1	78.1	1.00								
		Total	98.7	86.7	1.14								
9	Feed Thickener uflo	Solid	20.7	8.6	2.40					44% solids in ulfo			
		Liquid	26.3	26.3	1.00								
		Total	46.9	34.9	1.35								
33	Feed THK oflo	Liquid	51.8	51.8	1.00								
22	Leach THK 2 oflo	Liquid	79.7	77.5	1.00					0.034 calc			
										0.0339 force			
10	Potassium Iodide	Liquid	0.0	0.0	1.00								
39	Iodine make-up	Solid	1.5	0.3	4.60								
												39.46 °C Feed to Leach 1	
												2192 BTU/hr Required	
												657.5 watts	

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	Iron % kg/hr kgeq/hr	Sulfuric Acid kg/hr kgeq/hr	Hydrogen Peroxide %-g/L kg/hr kgeq/hr	Calcium kg/hr kgeq/hr	Value	Control	Value	Control
37	Leach 1	Solid	20.7	8.6	2.40					19% solids in leach		50 °C	
		Liquid	107.5	104	1.04					0.2 molar KI		33.2 g/l KI	
		Total	128.2	112	1.14					0.1 molar I2		25.38 g/l I2	
38	Vapor									5% iodine volatile			
11	Leach 1 slurry	Solid	20.7	8.6	2.40					90% of Hg solubilized			
		Liquid	107.5	104	1.04					10% iodine to iodide			
		Total	128.2	112	1.14								
12	Flocculant	Liquid	5.2	5.2	1.00					0.5 lb/ton in KI recycle			
13	LeachTHK 1 mix	Solid	20.7	8.6	2.40					0.10% solution			
		Liquid	112.7	109	1.03								
		Total	133.3	118	1.13								
14	Leach THK 1 uflo	Solid	20.7	8.6	2.40					42% solids in ulfo			
		Liquid	28.5	28.5	1.00								
		Total	49.2	37.1	1.32								
15	THK 1 oflo Preg Soln	Liquid	84.1	80	1.05							40 °C calc WTHK 1	
19	Wash THK 1 oflo	Liquid	54.8	54.8	1.00					0.013 calculated		20 g/l KI force	
16	Soln B	Liquid	17.5	17.5	1.00					0.0131 force			
34	Iodine make-up	Solid	2.2									46 °C Feed to Leach 1	
35	Leach 2A	Solid	20.7	8.6	2.40					0 Hg in recycle		764.1 BTU/hr Required	
		Liquid	103.1	101	1.02					17% solids in Leach 2A		229.2 watts	
		Total	123.7	109	1.13					0.07 molar I2		50 °C	
												17.77 g/l I2	
36	Vapor									5% iodine volatile			
17	Leach 2B Discharge	Solid	20.7	8.6	2.40					30 ppm Hg in solids			
		Liquid	103.1	101	1.02					10% iodine to iodide			
		Total	123.7	109	1.13								
18	Flocculant	Liquid	5.2	5.2	1.00					0.5 lb/ton in KI recycle			
										0.10% solution			

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	Iron % kg/hr kgeq/hr	Sulfuric Acid kg/hr kgeq/hr	Hydrogen Peroxide %-g/L kg/hr kgeq/hr	Calcium kg/hr kgeq/hr	Value	Control	Value	Control
20	LeachTHK 2 mix	Solid	20.7	8.6	2.40								
		Liquid	108.2	106	1.02								
		Total	128.9	115	1.12								
21	Leach THK 2 uflo	Solid	20.7	8.6	2.40					42% solids in ulfo			
		Liquid	28.5	28.5	1.00								
		Total	49.2	37.1	1.32								
22	Leach THK 2 oflo	Liquid	79.7	77.5	1.00								
23	Wash THK 2 oflo	Liquid	49.5	49.5	1.00					0.001 calculated		34 °C	
24	Repulper 1	Solid	20.7	8.6	2.40					0.007 force		34 °C calculated	
		Liquid	78.0	78.0	1.00								
		Total	98.7	86.6	1.14								
25	Flocculant	Liquid	4.1	4.1	1.00					0.4 lb/ton in process water			
26	Flocculated slurry	Solid	20.7	8.6	2.40					0.10% solution			
		Liquid	82.2	82.2	1.00					80% of Hg solubilized			
		Total	102.8	90.8	1.13								
27	WashTHK 1 uflo	Solid	20.7	8.6	2.40					43% solids in ulfo			
		Liquid	27.4	27.4	1.00								
		Total	48.0	36.0	1.33								
19	Wash THK 1 oflo	Liquid	54.8	54.8	1.00								
64	Wash THK 3 oflo	Liquid	45.3	45.3	1.00					0.003 calc ulated		29 °C	
29	Repulper 2	Solid	20.7	8.6	2.40							29 °C calculated	
		Liquid	72.7	72.7	1.00								
		Total	93.4	81.3	1.15								
30	Flocculant	Liquid	3.1	3.1	1.00					0.3 lb/ton in process water			
31	Flocculated slurry	Solid	20.7	8.6	2.40					0.10% solution			
		Liquid	75.8	75.8	1.00								
		Total	96.5	84.4	1.14								

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	Iron % kg/hr kgeq/hr	Sulfuric Acid kg/hr kgeq/hr	Hydrogen Peroxide %-g/L kg/hr kgeq/hr	Calcium kg/hr kgeq/hr	Value	Control	Value	Control
32	WashTHK 2 uflo	Solid	20.7	8.6	2.40					44% solids in ulfo			
		Liquid	26.3	26.3	1.00								
		Total	46.9	34.9	1.35								
23	Wash THK 2 oflo	Liquid	49.5	49.5	1.00								
72	Wash THK 4 oflo + filt	Liquid	41.2	41.2	1.00					0.001 calculated		25 °C	
60	Repulper 3	Solid	20.7	8.6	2.40							34 °C calculated	
		Liquid	67.5	67.5	1.00								
		Total	88.1	76.1	1.16								
61	Flocculant	Liquid	4.1	4.1	1.00					0.4 lb/ton in process water			
62	Flocculated slurry	Solid	20.7	8.6	2.40					0.10% solution			
		Liquid	71.6	71.6	1.00					80% of Hg solubilized			
		Total	92.3	80.2	1.15								
63	WashTHK 3 uflo	Solid	20.7	8.6	2.40					44% solids in ulfo			
		Liquid	26.3	26.3	1.00								
		Total	46.9	34.9	1.35								
64	Wash THK 3 oflo	Liquid	45.3	45.3	1.00								
28	Process water	Liquid	20.7	20.7	1.00					1.00 kg/kg solids			
65	Repulper 4	Solid	20.7	8.6	2.40					0.000 Hg in water			
		Liquid	46.9	46.9	1.00								
		Total	67.6	55.6	1.22								
66	Flocculant	Liquid	3.1	3.1	1.00					0.3 lb/ton in process water			
67	Flocculated slurry	Solid	20.7	8.6	2.40					0.10% solution			
		Liquid	50.0	50.0	1.00								
		Total	70.7	58.7	1.21								

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream No.	Description	Phase	kg/hr	L/hr	SpG	Iron		Sulfuric Acid		Hydrogen Peroxide		Calcium		Value Control		Value Control	
						%	kg/hr	kgeq/hr	kg/hr	kgeq/hr	%-g/L	kg/hr	kgeq/hr	kg/hr	kgeq/hr		
68	WashTHK 4 ulfo	Solid	20.7	8.6	2.40									44% solids in ulfo			
		Liquid	26.3	26.3	1.00												
		Total	46.9	34.9	1.35												
69	Wash THK 4 oflo	Liquid	23.8	23.8	1.00												
70	Final filter cake	Solid	20.7	34.9	2.40									70% solids in ulfo			
		Liquid	8.9	8.9	1.00												
		Total	29.5	43.8	0.67												
71	Filtrate	Liquid	17.4	17.4	1.00												
15	THK 1 oflo Preg Soln	Liquid	84.1	80.4	1.0												
73	Sulfuric Acid		0.10	0.06	1.83				0.10	0.002				5.000 g/kg soil			
40	Feed to Hg removal	Liquid	84.3	80.5	1.0												
41	Iron to columns	Solid					0.44	0.016						120% stoic for I2 and Hg			
42	Mercury barrens	Liquid	84.3	80.5	1.0		0.44	0.016						99% removal of Hg 100% reduction of iodine		5 °C loss in Hg removal	
43	Mercury in column	Liquid	0.02	0.00	13.5												
44	Hydrated lime to precip	Solid	0.6	0.3	2.2							0.34	0.017	110% stoichiometric for Fe 54% calcium			
45	Iron slurry	Solid	2.9	1.9	1.50	15%	0.44	0.016						15% iron in ppt			
		Liquid	82.0	78.9	1.04									1.500 SG of precipitate			
		Total	84.9	80.8	1.05												
46	Filter cake wash	Liquid	8.8	8.8	1									3.000 liquid displacements			
47	Iron filter cake	Solid	2.9	1.9	1.50	15%	0.44	0.016						50% solids filter cake		5 °C loss in filtration	
		Liquid	2.9	1.0	1.00									97% wash efficiency			
		Total	5.8	2.9	1.98												
48	Iron Precip filtrate	Liquid	87.8	86.6	1.01												

PILOT SCALE MASS BALANCE BASED ON INEEL CONTAMINATED SOIL

Stream		Phase	kg/hr	L/hr	SpG	Iron		Sulfuric Acid		Hydrogen Peroxide		Calcium		Value		Value	
No.	Description					%	kg/hr kgeq/hr	kg/hr kgeq/hr	%-g/L	kg/hr kgeq/hr	kg/hr kgeq/hr	kg/hr kgeq/hr	kg/hr kgeq/hr	Control	Control	Control	Control
49	KI to evaporation	Liquid	40.8	40.3	1.01									60.8 calc 2.5 KI force			
50	KI to iodine recovery	Liquid	47.0	46.4	1.01												
51	98% sulfuric acid	Liquid	0.6	0.3	1.83			0.60 0.012						140% stoichiometric			
52	30% hydrogen peroxide	Liquid	0.6	0.5	1.19					0.58 0.010				120% stoichiometric			
53	Iodine slurry	Solid	2.2	0.5	4.60									40 °C reaction			
		Liquid	46.0	46.7	0.99												
		Total	48.2	47.2	1.02												
54	Iodine crystals	Solid	2.2	0.5	4.6												
55	Filtrate	Liquid	46.0	46.7	1.0												
57	Evaporated KI	Liquid	27.9	27.0	1.03									70 °C			
58	Condensate	Liquid	12.9	13.3	0.0									28483 BTU/hr 8545 watts			
56	Process water	Liquid	59.0	60.0	1.0												
	Solution balance																
	To feed prep		26.4														
	To leach		26.3														
	Make-up water to prep		25.2														
	Preg out		84.1														
	KI in		27.9														
	Process water in		60.2														
	Make-up water to leach		3.9														

Appendix B-3

Thermal Treatment Cost Information



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ETCAP compendium search results

Found 2 matches.

Searched for (Activated Sludge *or* Thermal Desorption) *AND* (mercury *or* Hg) *AND* (California)

Treatment Technology	Cost Elements	Cost	Site Characteristics/Comments
Thermal desorption	Total cost	\$593,000	<p><u>Borg-Warner Instrument Manufacturing Site, Vernon, California (1996)</u></p> <p><u>Media:</u> 1,500 tons soil and sludge.</p> <p><u>Contaminants:</u> 20 - 6,200 mg/kg mercury.</p> <p><u>Details:</u> Cleanup completed from 1/96 to 2/96. For the thermal desorption unit used, infrared radiation is used to heat the soil, and a purge gas transports the contaminants to an emissions control system. Soil is loaded 1-foot deep onto two 8 feet by 8 feet trays, and the trays are loaded into the unit's chamber. The unit is sealed, and 25 to 28 inches of mercury vacuum (or 50 to 100 torr) is attained. Subsequently, 1.4 million Btu/hr of heat is introduced to the system with the infrared heaters. The unit's thermal energy source uses propane-fired combustion chambers to directly heat an array of aluminized steel tubes to a temperature of 1,100deg.F. At this temperature, the tubing emits electromagnetic energy in the infrared spectrum. The soil is treated for 2 to 3 hours until the target temperature is attained. Mercury treated from 1 - 20 mg/kg.</p>
Thermal desorption	Total cost	\$461,000	<p><u>Clorox Site, Oakland, California (1996)</u></p>
	Total cost/unit	\$350/ton	<p><u>Media:</u> 1,000 tons soil, sludge, and solids.</p> <p><u>Contaminants:</u> 20 - 8,500 mg/kg mercury.</p> <p><u>Details:</u> Cleanup completed from 12/95 to 7/96. For the thermal desorption unit used, infrared radiation is used to heat the soil, and a purge gas transports the contaminants to an emissions control system. Soil is loaded 1-foot deep onto two 8 feet by 8 feet trays, and the trays are loaded into the unit's chamber. The unit is sealed, and 25 to 28 inches of mercury vacuum (or 50 to 100 torr) is attained. Subsequently, 1.4 million Btu/hr of heat is introduced to the system with the infrared heaters. The unit's thermal energy</p>

		source uses propane-fired combustion chambers to directly heat an array of aluminized steel tubes to a temperature of 1,100deg.F. At this temperature, the tubing emits electromagnetic energy in the infrared spectrum. The soil is treated for 2 to 3 hours until the target temperature is attained. Mercury treated from 1 - 20 mg/kg.
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