

WRI-09-R018

**THE USE OF HAZ-FLOTE TO EFFICIENTLY REMOVE
MERCURY FROM CONTAMINATED MATERIALS**

TOPICAL REPORT

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**By
Terry Brown**

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Laramie, Wyoming**

**Kamalendu Das
Task 15**

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DISCLAIMER

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ABSTRACT

There are thousands of known contaminated sites in the United States, including Superfund sites (1500 to 2100 sites), RCRA corrective action sites (1500 to 3500 sites), underground storage tanks (295,000 sites), U.S. Department of Defense sites (7300 sites), U.S. Department of Energy sites (4,000 sites), mining refuse piles, and numerous other hazardous metals and organic contamination sites. Only a small percentage of these sites has been cleaned up. The development of innovative technologies to handle the various clean-up problems on a national and international scale is commonplace. Many innovative technologies have been developed that can be used to effectively remediate contaminated materials. Unfortunately, many of these technologies are only effective for materials coarser than approximately 200 mesh. In addition, these technologies usually require considerable investment in equipment, and the clean-up costs of soil material are relatively high – in excess of \$100 to \$500 per yd³. These costs result from the elaborate nature of the processes, the costs for power, and the chemical cost. The fine materials are disposed of or treated at considerable costs. As a result, the costs often associated with amelioration of contaminated sites are high.

Western Research institute is in the process of developing an innovative soil washing technology that addresses the removal of contaminants from the fine size-fraction materials located at many of the contaminated sites. This technology has numerous advantages over the other ex-situ soil washing techniques. It requires a low capital investment, low operating costs and results in high levels of re-emplacement of the cleaned material on site. The process has the capability to clean the fine fraction (<200 mesh) of the soil resulting in a replacement of 95+% of the material back on-site, reducing the costs of disposal. The Haz-Flote™ technology would expand the application of soil washing technology to heavy soils (clay-type should) to which current soil washing practices are not applied. WRI is not aware of any other soil washing technologies that demonstrate this ability at the expected cost on a per ton basis. The market for this technology is considered excellent for Superfund and other inorganic contaminated sites.

EXECUTIVE SUMMARY

The Haz-Flote technology was developed using materials collected from mercury-contaminated sites associated with monometer locations found along oil and gas pipelines in northeastern Canada and in the Silver Creek watershed near Helena, Montana. The bulk of the work on the Haz-Flote process was focused on removing mercury from contaminated soil fines. Samples of mercury contaminated soil were obtained, and the distribution of mercury within the various size fractions was determined. The -200 mesh (<75 μm) material contains the majority of the mercury contaminant. It also appeared that much of the mercury was associated with the organic matter when present in the contaminated material. For convenience, the samples were sieved through a 70 mesh screen. All of the testing was conducted on the -70 mesh (<212 μm) fraction. Some samples contained in excess of 100 mg Hg/kg of dry soil before processing. Mercury concentrations after processing were reduced to as low as 0.95 mg Hg/kg of dry soil. This represents >99% mercury reduction for the fine fraction. Processing of the scrubber solution with Phase II of Haz-Flote, the column flotation phase, removed >99.5% of the mercury. This process will extend the use of soil washing techniques to contaminated sites that are currently remediated using more costly technologies. The early phases of development of the Haz-Flote process were funded by the US Department of Energy Morgantown Energy Technology Center (now the Federal Energy Technology Center and the Montana Department of Environmental Quality).

INTRODUCTION

There are thousands of known contaminated sites in the United States, including Superfund sites (1500 to 2100 sites), RCRA corrective action sites (1,500-3,500 sites), underground storage tanks (295,000 sites), U.S. Department of Defense sites (7,300 sites), U.S. Department of Energy sites (4,000 sites), mining refuse piles, and numerous other hazardous metals and organic contamination sites. The development of innovative technologies to handle the various clean-up problems on a national and international scale is commonplace. Many innovative technologies have been developed that can be used to effectively remediate contaminated materials. Unfortunately, many of these technologies are only effective for coarse materials. In addition, these technologies usually require considerable investment in equipment, and the clean-up costs of soil material are relatively high often in excess of \$100 to \$500 per yd³. These costs result from the elaborate nature of the processes, the costs for power, and the chemical cost. The fine materials are disposed of or treated at considerable costs. As a result, the costs often associated with amelioration of contaminated sites are high.

Western Research Institute is developing an innovative soil washing technology that addresses the removal of contaminants from the fine size-fraction materials located at many of the contaminated sites. This technology has numerous advantages over other ex-situ soil washing techniques. It requires a low capital investment, low operating costs and results in high levels of re-emplacement of the cleaned material on site. The process has the capability to clean the fine fraction (< 200 mesh) of the soil resulting in a replacement of 95+% of the material back on-site, reducing the costs of disposal. The Haz-Flote technology would expand the application of soil washing technology to heavy soils (clay-type soils) to which current soil washing practices are not applied. WRI is not aware of any other soil washing technologies that demonstrate this ability at the expected cost on a per ton basis. The market for this technology is considered excellent for Superfund and other inorganic contaminated sites.

The Haz-Flote technology uses a carefully designed selection of chemicals in combination with an attrition scrubbing process followed by final cleanup or removal step using column flotation. The practice of using froth flotation for the beneficiation of mineral ores was first used in the late 1800's, with the process flourishing during the early 1900's. The use of a column in the flotation process was patented in the early 1960's by Boutin and Tremblay (Canadian patents 680,576 and 694,547). However, it was not until about 1981 that a surge in commercial interest for using column flotation in the ore beneficiation processing was seen. Worldwide application of column flotation systems include Cu, Mo, Pb, Zn, and Sn cleaning in the minerals mining industry, bulk sulfide roughing of Au ores, coal, graphite and phosphate flotation.

Similar foam separation techniques to remove ions, such as mercury, cadmium and copper, from aqueous systems has also been used by a number of investigators. Okamoto and Chou (1975) showed that the removal of mercury ions from aqueous solutions was almost

quantitative by chelating the ions with the surfactant 4-dodecyldiethylenetriamine. In another study, the cationic surfactant hexadecyltrimethylammonium bromide was used in a foam fractionation study to remove trace levels of mercury (II) nitro complexes (Miller and Sullivan, 1971). Another type of foam flotation process, colloid flotation, has been successfully used to remove mercury from sea water (Voyce and Zeitlin, 1974) and molybdenum from sea water (Kim and Zeitlin, 1971). In these applications, iron (III) hydroxide was used as a sorption surface for the target elements (elements targeted for removal). The resulting iron (III) hydroxide element complex was bound by sodium dodecylsulfate making it hydrophobic. This final complex could easily attach to an air bubble and could be removed from the solution via flotation. Huang and Wilson (1976) reported the use of adsorbing colloid flotation batch separations of mercury and cadmium from aqueous solutions down to concentrations of 20 ppb. The literature shows that the use of foam separation techniques to clean mineral ores and to remove potentially toxic elements and/or complexes from aqueous solutions can be successful. Therefore, it is very probable that these techniques can be used to separate elements such as mercury from soils.

Haz-Flote was initially developed for the removal of mercury and hydrocarbons from soil materials with some very good success. As a result, the State of Montana, Department of Environmental Quality decided to evaluate the use of the Haz-Flote technology to remove mercury from contaminated materials at several sites under their jurisdiction. If successful, the Haz-Flote technology would be scaled up to support the remediation of these contaminated sites. This report also discusses the use of Haz-Flote for the remediation of mercury contaminated materials associated with an oil and gas pipeline located in northeastern Canada.

OBJECTIVES

The objective of this research effort was to develop a low-cost method for remediation of contaminated soil materials. The process was developed as an ex-situ treatment technology for the removal of organics and metals from such materials. This research effort was conducted to determine the effectiveness of using Haz-Flote to remove mercury from fine materials such as sediments and tailings materials.

TECHNICAL APPROACH

Haz-Flote is an ex-situ treatment technology for metals-contaminated materials. A schematic of the process is shown in Figure 1. The process incorporates the use of specifically designed chemical reagents with a flotation separation device. The work described in this report addresses the refinement of the process for the remediation of mercury-contaminated soils. The evaluations were conducted using materials collected from manometer sites associated with an oil and gas pipeline in northeastern Canada and from the Silver Creek watershed sediments and associated tailings materials found adjacent to the stream channel. The mercury contamination was mostly found associated with the fine-textured (< 100 mesh) materials, which can be handled well using the Haz-Flote technology. The methods used to assess the usefulness of using the Haz-Flote technology are provided in the following discussions.

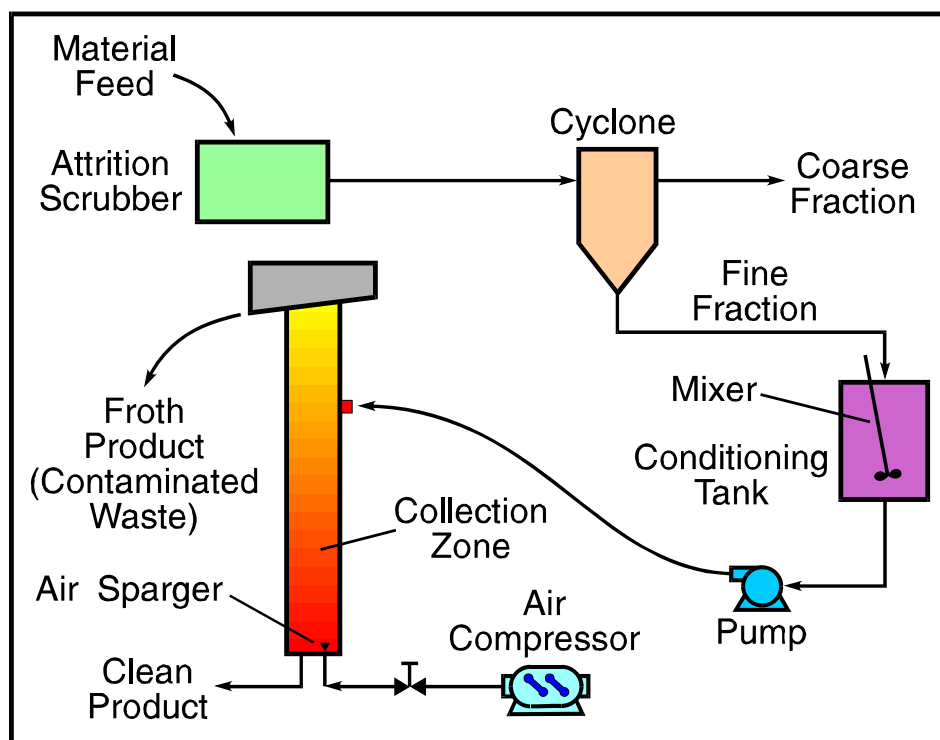


Figure 1 Schematic of the Haz-Flote™ Technology

Acquisition of Mercury Contaminated Samples

Samples used in this research effort were collected from oil and gas pipeline monometer sites located in northeastern Canada and from sediment and mill tailing materials in the Silver Creek drainage located near Helena, Montana. The mercury contaminated materials collected in Canada were taken from monometer installations while the location of the mercury-contaminated materials was not well documented in the Silver Creek drainage. Therefore, a number of

sediment samples were collected from the Silver Creek location to ensure that representative contaminated materials were acquired.

Mercury is a volatile species under many storage conditions, and samples may lose some mercury over time and under certain conditions. In addition, changes in speciation may also be an issue during storage. Because maintaining viable samples in storage may be a problem, samples were collected from the Silver Creek sites three times during the project so that "fresh" field materials were available for the research work. Care was taken to collect the minimum amount of material needed to complete the laboratory work, and samples not used in the study were disposed using a certified transporter to a licensed disposal facility. The contaminated materials collected from monometer sites in northeastern Canada were provided to the Western Research Institute by EERC. The sampling handling procedures were not known.

Characterization of Mercury Contaminated Material

Characterization of contaminated material was conducted to determine the chemical and physical nature of the materials. Total and extractable elemental analysis were determined using appropriate techniques. The particle size classification was determined for each material studied. In addition, the levels of contaminant were determined for each particle size class. In this study, mercury was found to be associated with the fine size fraction (< 100 mesh) and the organic matter found in the materials. Samples collected from two sites were evaluated in this study including a sample collected at manometer locations found along oil and gas pipelines in northeastern Canada and sediment and mill tailing samples collected from the Silver Creek site located near Helena, Montana. The material collected in northeastern Canada represented material containing high levels of organic matter while the Silver Creek materials represented stream sediments and mill tailings containing low levels of organic matter.

Desorption and Complexing Determinations

HAZ-FLOTE testing required a significant effort to determine the parameters and the levels and/or concentrations required to successfully remove the contaminants from the materials and/or solutions studied. A combination of pH controls, oxidation requirements and complexing alternatives were assessed. Scrubbing or mixing tests and flotation tests were conducted using a bench scale unit and other less vigorous mixing techniques were used to provide initial information or screening tests for the various combinations of extractants used. The chemicals required for the extraction of mercury from the contaminated materials and for the removal of the mercury from the extraction fluid were optimized for each application through testing and experimentation. A range of oxidants, desorbers, and complexers were evaluated to determine their ability to liberate the contaminant from the matrix. The pregnant solution was removed from the conditioned material using a separation step that included leaching, filtration, centrifugation or settling. Following the extraction of the Hg solution from the soil materials, the pregnant solution can be treated with appropriate chemicals that will cause the formation of

colloidal material, which then sorbs the mercury compounds present in solution. The colloids containing the sorbed mercury will be separated from the solution using a flotation unit.

Column Flotation

Once mercury is removed from the contaminated materials and is complexed into a form that does not allow re-sorption to the materials, the contaminants will be removed from the system using a column flotation system. Laboratory tests were conducted using the adsorbing colloid flotation method described by Huang and Wilson (1976). Cupric sulfide was used as the adsorbing colloid with hexadecyltrimethylammonium bromide (HTA) as the collector. The method was found to remove mercury from aqueous solutions leaving residual mercury levels as low as 5 ppb and was also insensitive to pH variation from 1 to 8.

Flotation tests were conducted in a glass column of 2.54 cm inside diameter and 39 cm in height. Using the relatively small column significantly reduced the amount of mercury waste generated during this research effort. A medium porosity stainless steel frit was used as a gas sparger. A valve was provided at the bottom of the column through which samples were removed and additional collector was injected as the tests proceeded. Nitrogen gas was used to generate the foam in the column. The gas flow was measured and controlled with a mass flow controller before it passed through a saturator and into the column.

Initial flotation tests were conducted on a stock solution of mercuric chloride in water. In these tests the flotation removed the mercury efficiently. However, when the method was applied to a solution containing the scrubber reagents and mercuric chloride, separation was very poor. It was determined that the scrubber solution was oxidizing the sulfide ion to sulfate ion. The soluble sulfates did not precipitate, and recovery was limited by the availability of sulfide ion. This problem was overcome either by allowing the scrubber solution to age, providing time for the oxidant to disproportionate, or by adding an excess of sulfide. Once the problem of oxidizing the sulfide ion was resolved, flotation tests were conducted using the actual scrubber solution. Tests were also conducted where the precipitate was allowed to settle for several days. Ten minutes of flotation proved to be as effective as settling for 48 hours. Both methods removed about 99.5 % of the mercury from the scrubber solution.

RESULTS AND DISCUSSION

Characterization of Mercury Contaminated Materials

The level of mercury found in size-fractions of the materials collected for the study are presented in Tables 1 and 2 for the Canadian and Montana sites, respectively.

Table 1. Total concentrations of mercury in the contaminated soil by particle size fraction using dry-sieve and wet-sieve separation techniques.

Sieve Size	Dry Sieve Hg (mg/kg)	Wet Sieve Hg (mg/kg)	Dry Sieve Wt-g (g)	Wet Sieve Wt of Soil (g)	Tot-Hg Dry (mg)	Tot-Hg Wet (mg)
10 mesh	14	10	50	35	0.7	0.4
20 mesh	14	8.2	139	129	2	1
70 mesh	23	9.8	547	450	13	4
200 mesh	47	8.8	336	274	16	2
325 mesh	200	80	60	29	12	2
400 mesh	220	110	16	6	3	0.7
-400 mesh	220	250	73	129	16	32

As expected, the -200 mesh material contains about 86% of the mercury contaminant on a wet-sieve basis and about 76% on a dry-sieve basis. This difference is expected, as a wet-sieve analysis would separate fine particles that are attached to the coarse fraction, while the coarse fraction associated with the dry-sieve analysis would contain a portion of the fine size fraction attached.

Characterization of contaminated sediment material collected from Silver Creek was conducted to determine the chemical and physical nature of the sediment and tailings materials collected. The levels of contaminant were determined for each particle size class. Samples were divided into five (5) size classes and each size class was analyzed for total mercury levels. The size classes tested were (1) >20 mesh; (2) <20 mesh by >40 mesh; (3) <40 mesh by > 80mesh; (4) <80 mesh by >100 mesh; and (5) <100 mesh by >200 mesh. Percent solids were determined for each size class of materials. The data are presented in Table 2. For the most part, the data show higher amounts of mercury associated with the small particle size fraction, which compares well to the data collected for the northern Canada sample evaluations.

Table 2. Hg levels found in the size fractions for sediment samples collected in Silver Creek, Montana.

Sample ID	Hg mg/Kg	Solids %	Sample ID	Hg mg/Kg	Solids %
Site 1a (+20)	1.3	74.8	Site 3b (+20)	2.0	46.4
Site 1a (-20+40)	2.3	68.7	Site 3b (-20+40)	2.2	59.0
Site 1a (-40+80)	4.8	70.6	Site 3b (-40+80)	4.3	65.0
Site 1a (-80+100)	9.1	64.6	Site 3b (-80+100)	7.2	66.5
Site 1a (-100+200)	9.2	69.8	Site 3b (-100+200)	5.9	62.5
Site 1b (+20)	1.1	76.6	Site 6a (+20)	0.9	49.0
Site 1b (-20+40)	2.1	69.0	Site 6a (-20+40)	<0.1	49.0
Site 1b (-40+80)	4.8	66.1	Site 6a (-40+80)	0.2	38.2
Site 1b (-80+100)	8.6	65.0	Site 6a (-80+100)	0.3	42.5
Site 1b (-100+200)	6.7	63.8	Site 6a (-100+200)	0.4	37.4
Site 2 (+20)	3.1	26.3	Site 6b (+20)	<0.1	47.3
Site 2 (-20+40)	1.2	46.3	Site 6b (-20+40)	0.3	41.0
Site 2 (-40+80)	2.3	49.7	Site 6b (-40+80)	0.6	37.3
Site 2 (-80+100)	1.8	56.6	Site 6b (-80+100)	0.7	31.8
Site 2 (-100+200)	2.6	57.0	Site 6b (-100+200)	1.0	50.1
Site 3a (+20)	0.6	75.0	Goldfil Tailings A - +200 mesh	99.5	91
Site 3a (-20+40)	1.8	69.3			
Site 3a (-40+80)	4.6	67.6	Goldfil Tailings B - +200 mesh	114	78.8
Site 3a (-80+100)	6.3	66.7			
Site 3a (-100+200)	6.5	67.4	Drumlummon Tailings A - +200 m	1.6	87.6
			Drumlummon Tailings B - + 200 m	0.9	96.6

Scrubber Tests for Mercury Contaminated Soils Contaminated Collected from Monometer Sites located in northeastern Canada

These tests were conducted to find a reagent or reagent combination capable of separating mercury from a fine soil and containing the element in the liquid phase where it is amenable to flotation. After suitable reagents were identified, testing was conducted to reduce reagent consumption in the scrubber while maintaining or increasing mercury removal efficiency. Test variables included the pulp density, pH reagents and reagent dosages, temperature, residence time, multiple stage washing, and rinse water volume. Samples passing a 70 mesh sieve were used in this testing. The results of these tests are presented in Table 3. The reduction in mercury content was calculated by subtracting the mercury content of the cleaned

soil from that of the feed material and dividing by the mercury content of the feed. Each data point presented in the table is the average of four replicates.

Table 3. Mercury removal efficiency determined with NaOCl solution as the extractant.

Test Number	Reagent (ml)	Reagent (ml/g)	Treatment	Efficiency (%)
1-C	120	4.69	Shake 10m, ambient T, Closed	93.2
1-1	120	5.04	Shake 90m, amb T, Closed	85.6
1-2	60	2.32	Shake 90m, amb T, Closed	90.5
1-3	120	5.11	Shake 10m, amb T, open	85.5
1-4	120	4.98	Shake 90m, amb T, open	84.4
1-5	60	2.62	Shake 90m, amb T, open	93.6
2-C	120	4.49	Shake 10m, amb T, Closed	93.9
2-1	60	2.20	Shake 10m, amb T, Closed	90.5
2-2	120	5.31	Shake 10m, 35 C, Closed	96.8
2-3	60	2.06	Shake 10m, 35 C, Closed	93
2-4	120	4.41	Shake 10m, 50 C, Closed	OR
2-5	60	2.45	Shake 10m, 50 C, Closed	97.1
3-1	60	1.65	Shake 10m, amb T, water rinse	84.0
3-2	110	3.67	2 nd wash, of 3-1, water rinse	94.6
3-3	150	6.25	3 rd wash of 3-1, CaCl ₂ rinse	95.9
3-4	40	0.89	Shake 10m, amb T, water rinse	83.4
3-5	73.3	1.88	2 nd wash of 3-4, water rinse	94.7
3-6	100	3.03	3 rd wash of 3-4, CaCl ₂ rinse	96.9
4-1	60	2.30	2.9% NaOCl, 10m, 50C, CaCl ₂ rinse	92.1
4-2	30	1.36	1.4% NaOCl, 10m, 50C, CaCl ₂ rinse	90.0
4-3	15	0.57	0.7% NaOCl, 10m, 50C, CaCl ₂ rinse	84.1
4-4	60	2.40	5.8% NaOCl, 10m, 50C, CaCl ₂ rinse	97.7
4-5	30	1.34	5.8% NaOCl, 10m, 50C, CaCl ₂ rinse	97.0
4-6	15	0.54	5.8% NaOCl, 10m, 50C, CaCl ₂ rinse	89.0
7-C	120	4.38	Shake 10 m, amb T, closed	94.7
7-1	15	0.72	5.8% NaOCl, 10m, 50C, CaCl ₂ rinse	OR
7-2	15	0.66	5.8% NaOCl, 10m, 50C, CaCl ₂ rinse, open	OR
7-3	30	1.31	2 washes w/15ml, no rinse	93.9
7-4	30	1.12	2 washes w/15ml, rinse between	93.1
7-5	30	1.18	1 wash w/30 ml	93.3

OR = samples out of range for the analyzer

Table 4 Mercury removal efficiency determined with Ca(OCl)₂ (solid) as the extractant.

Test Number	Reagent (g)	Reagent (g/g)	Treatment	Efficiency (%)
5-C			5.75% NaOCl, 10m, ambT, CaCl ₂ rinse	94.5
5-1	6	0.26	Ca(OCl) ₂ dry, H ₂ O act., CaCl ₂ rinse	87.8
5-2	6	0.26	Ca(OCl) ₂ dry, 5% HCl act., CaCl ₂ rinse	97.7
5-3	6	0.22	Ca(OCl) ₂ dry, 10% HCl act., CaCl ₂ rinse	92.8
5-4	6	0.23	Ca(OCl) ₂ dry, 25% HCl act., CaCl ₂ rinse	95.9
5-5	6	0.22	Ca(OCl) ₂ dry, 50% HCl act., CaCl ₂ rinse	95.7
6-C			5.75% NaOCl, 10m, ambT, CaCl ₂ rinse	94
6-1	5.88	0.25	Ca(OCl) ₂ dry, 5% HCl act., CaCl ₂ rinse	97.1
6-2	6.04	0.23	Ca(OCl) ₂ dry, H ₂ O act., 50C, CaCl ₂ rinse	96.3
6-3	2.97	0.12	Ca(OCl) ₂ dry, H ₂ O act., 50C, CaCl ₂ rinse	91.9
6-4	3.05	0.14	Ca(OCl) ₂ dry, 5% HCl act., CaCl ₂ rinse	97.9
8-C	3.1	0.12	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	95.2
8-1	2.12	0.07	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	95.2
8-2	0.99	0.04	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	95.3
8-3	0.55	0.02	Ca(OCl) ₂ dry, H ₂ O act., 50C, 30m	OR
8-4	0.89	0.03	Ca(OCl) ₂ dry, 10% HCl act., 30m, H ₂ O rinse	95.8
8-5	0.96	0.03	Ca(OCl) ₂ dry, 5% HCl act., 50C, 30 min	92.8
9-C	1.13	0.04	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	94.6
9-1	1.01	0.04	Ca(OCl) ₂ dry, 5% HNO ₃ act., 30m, H ₂ O rinse	95.4
9-2	1.14	0.04	Ca(OCl) ₂ dry, 5% HCl act., 10m, H ₂ O rinse	91.8
9-3	0.51	0.02	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	OR
9-4	0.54	0.02	Ca(OCl) ₂ dry, H ₂ O act., 50C, 30m, H ₂ O rinse	OR
9-5	0.5	0.02	Ca(OCl) ₂ dry, 5% HNO ₃ act., 30m, H ₂ O rinse	92.1
10-1	0.77	0.034	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	93.0
10-2	0.74	0.032	Ca(OCl) ₂ dry, 5% HNO ₃ act., 30m, H ₂ O rinse	89.7
10-3	0.74	0.030	Ca(OCl) ₂ dry, 10% HCl act., 30m, H ₂ O rinse	92.0
10-4	0.49	0.019	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	93.9
10-5	0.49	0.019	Ca(OCl) ₂ dry, 5% HNO ₃ act., 30m, H ₂ O rinse	96.1
10-6	0.56	0.023	Ca(OCl) ₂ dry, 10% HCl act., 30m, H ₂ O rinse	94.6
11-1	0.75	0.031	5% HCl, 30m pretreat, Ca(OCl) ₂ dry 30m	93.9
11-2	0.75	0.030	5% HNO ₃ , 30m pretreat, Ca(OCl) ₂ dry 30m	93.0
11-3	0.75	0.029	10% HCl, 30m pretreat, Ca(OCl) ₂ dry 30m	96.1
11-4	0.5	0.020	5% HCl, 30m pretreat, Ca(OCl) ₂ dry 30m	90.2
11-5	0.5	0.018	5% HNO ₃ , 30m pretreat, Ca(OCl) ₂ dry 30m	90.7
11-6	0.5	0.020	10% HCl, 30m pretreat, Ca(OCl) ₂ dry 30m	93.9
12-1	0.81	0.023	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	92.1
12-2	0.82	0.032	Ca(OCl) ₂ dry, 5% HNO ₃ act., 30m, H ₂ O rinse	95.0
12-3	0.81	0.032	Ca(OCl) ₂ dry, 10% HCl act., 30m, H ₂ O rinse	96.1

12-4	0.55	0.022	Ca(OCl) ₂ dry, 5% HCl act., 30m, H ₂ O rinse	93.4
12-5	0.52	0.022	Ca(OCl) ₂ dry, 5% HNO ₃ act., 30m, H ₂ O rinse	93.1
12-6	0.59	0.026	Ca(OCl) ₂ dry, 10% HCl act., 30m, H ₂ O rinse	94.1
13-C	0.68	0.027	Ca(OCl) ₂ dry, 120 ml 5% HCl	94.6
13-1	0.61	0.022	Ca(OCl) ₂ dry, 60 ml 5% HCl	86.8
13-2	0.61	0.023	Ca(OCl) ₂ dry, 30 ml 5% HCl	83.3
13-3	0.4	0.016	Ca(OCl) ₂ dry, 60 ml 5% HCl	92.1
13-4	0.44	0.020	Ca(OCl) ₂ dry, 30 ml 5% HCl	82.0
13-5	0.43	0.019	Ca(OCl) ₂ dry, 30 ml 10% HCl	83.7
14-1	1.44	0.030	Ca(OCl) ₂ dry, 5% HCl	94.3
14-2	0.71	0.056	Test 14-1 solids, wash w/ Ca(OCl) ₂ dry, 5% HCl	>98.2
14-3	1.05	0.021	Ca(OCl) ₂ dry, 5% HCl	90.0
14-4	0.54	0.037	Test 14-3 solids, wash w/ Ca(OCl) ₂ dry, 5% HCl	97.9
14-5	0.77	0.030	Ca(OCl) ₂ dry, 5% HCl	94.3
14-6	0.24	0.009	Washed w/ liquid from test 14-5 + Ca(OCl) ₂ dry	72.0
15-1	1.51	0.031	Ca(OCl) ₂ dry, 5% HCl	92.7
15-2	0.74	0.057	Test 15-1 solids, wash w/ Ca(OCl) ₂ dry, 5% HCl	98.6
15-3	1.01	0.021	Ca(OCl) ₂ dry, 5% HCl	93.5
15-4	0.47	0.036	Test 15-3 solids, wash w/ Ca(OCl) ₂ dry, 5% HCl	98.1
15-5	0.7	0.033	Ca(OCl) ₂ dry, 5% HCl	97.4
15-6	0.18	0.007	Washed w/ liquid from test 14-5 + Ca(OCl) ₂ dry	73.3
16-1	2.8	0.058	2 washes and rinses, fresh reagents (ox+acid)	98.6
16-2	NA		Washed with reagent from test 16-1 and wash 2	77.6
16-3	0.69	0.023	2 nd wash of test 16-2 solids + fresh reagents	98.1
16-4	0.67	0.028	Reagent from test 16-3 + fresh added	97.1
17-1	2	0.041	2 washes and rinses, fresh reagents (ox+acid)	98.8
17-2	NA		Washed with reagent from test 17-1 and wash 2	81.4
17-3	0.49	0.018	2 nd wash of test 17-2 solids + fresh reagents	97.0
17-4	0.49	0.020	Reagent from test 17-3 + fresh added	96.0
18-1	1.46	0.061	2 washes and rinses, multiple rinses	99.2
18-2	0.75	0.030	3 washes and rinses, small doses + fresh reagents	98.5
18-3	1.01	0.040	2 washes/rinses, fresh reagents (ox+acid), 10m.	98.6
18-4	1.00	0.042	2 washes/rinses, fresh reagents (ox+acid), 30m.	99.0
18-5	0.76	0.030	1 wash and rinse, 60 min.	96.7

The efficiency values associated with the various combinations of reagents and experimental conditions show that many of the treatments provide very efficient removal of mercury from the soil materials. The most effective treatments resulted in removal efficiencies in the 95 percent range. The use of NaOCl and Ca(OCl)₂ for oxidation and mercury complexation seemed to be equally effective. However, the influence of Na on aggregate slaking and clay dispersion tends to reduce its usefulness as the treated product may require a CaCl₂ wash or gypsum treatment to support plant growth after the mercury has been removed.

Therefore, the use of $\text{Ca}(\text{OCl})_2$ for oxidation/complexation was deemed the more appropriate reagent for the removal of mercury from soil materials. The most effective treatment was found to be a combination of $\text{Ca}(\text{OCl})_2$ for oxidation/complexation and either HCl or HNO_3 to acidify the system.

The question that dictates potential application of this technology is associated with economics. How much does each treatment cost assuming that scale-up can be accomplished maintaining the efficiencies experienced in the laboratory. The estimated cost for reagents on a per ton of material basis is provided in Table 5. These costs do not include the capital costs required to build the remediation system.

Table 5. Estimated costs for reagents to remediate contaminated materials collect from northeastern Canada locations (based on 1999 costs).

Treatment #	Amount. –Reagent (gal/ton)	Cost (\$/ton)	Treatment #	Amount. –Reagent (gal/ton)	Cost (\$/ton)
1-C	1124	337	1-1	1209	363
1-2	556	167	1-3	1224	367
1-4	1194	358	1-5	628	188
2-C	1077	323	2-1	527	158
2-2	1273	382	2-3	494	148
2-4	1058	317	2-5	587	176
3-1	395	119	3-2	879	264
3-3	1498	449	3-4	213	64
3-5	451	135	3-6	726	218
4-1	551	165	4-2	327	98
4-3	137	41	4-4	575	173
4-5	321	96	4-6	128	39
7-C	1050	315	7-1	174	52
7-2	157	48	7-3	314	94
7-4	267	80	7-5	282	85
5-C			5-1	518	492
5-2	519	493	5-3	444	422
5-4	457	434	5-5	432	410
6-C			6-1	492	468
6-2	456	433	6-3	234	222
6-4	275	261			
8-C	238	226	8-1	149	142
8-2	78	74	8-3	39	37
8-4	67	63	8-5	69	65

9-C	84	80	9-1	77	73
9-2	84	79	9-3	37	35
9-4	40	38	9-5	42	40
10-1	68	64	10-2	63	60
10-3	59	57	10-4	38	36
10-5	38	36	10-6	45	43
11-1	61	58	11-2	60	57
11-3	57	54	11-4	39	37
11-5	37	35	11-6	40	38
12-1	47	44	12-2	65	62
12-3	64	61	12-4	44	42
12-5	44	42	12-6	51	49
13-C	53	50	13-1	45	43
13-2	47	45	13-3	32	30
13-4	39	38	13-5	37	36
14-1	60	57	14-2	112	106
14-3	43	40	14-4	74	70
14-5	61	58	14-6	18	17
15-1	63	60	15-2	114	108
15-3	42	40	15-4	72	68
15-5	66	63	15-6	15	14
16-1	117	111	16-2		
16-3	47	45	16-4	57	54
17-1	83	78	17-2		
17-3	36	34	17-4	40	38
18-1	121	115	18-2	60	57
18-3	80	76	18-4	84	80
18-5	61	58			

The most cost effective reagent treatment combination for remediating this material appears to be the $\text{Ca}(\text{OCl})_2$ and acid reagents. The oxidizing agent applied at a rate of .02g/g of material (2%) with a 5% HCl will result in a reagent cost in the vicinity of \$40/ton of material. These values do not include the capital costs. In addition, these costs do not reflect potentially improved efficiencies that may result from designing a system that uses counter-current flow and other processes that will make the system more efficient.

Mercury Flotation Tests

The results of the flotation/precipitation tests in terms of mercury removal are summarized in Table 6. The reduction in mercury content was calculated by subtracting the mercury content of the liquid after foaming from that of the column feed and dividing by the mercury content of the feed. This result was multiplied by 100 to give percentage mercury reduction. The removal efficiencies of 99+ % compared very well to the results published by Huang and Wilson (1976). This test was very successful and proved to be a very useful component of the Haz-Flote technology.

Table 6. Effect of precipitation/flotation on mercury concentration in the scrubber solution.

Sample ID	Treatment	Initial [Hg] (mg/L)	Final [Hg] (mg/L)	Efficiency (%)
Simulated Scrubber Solution	10 m flotation	16.69	0.025	99.8
Scrubber Solution Test 72-1	10 m flotation	11.88	0.059	99.5
Scrubber Solution Test 72-2	Precipitation	8.64	0.042	99.5
Scrubber Solution Test 72-3	Precipitation	9.4	0.043	99.5

Scrubber Tests for Mercury Contaminated Soils Collected from Sediments and Mill Tailings Sites Located Along Silver Creek, Helena, Montana

The portion of the research is an extension of the development of the Haz-Flote process looking at materials having different characteristics from those collected from northeastern Canada. The materials collected from the Silver Creek drainage contain much less organic matter than the materials previously evaluated. The contaminated material is associated with mercury spillage from an abandoned gold mine milling operation.

Samples were collected from the Silver Creek site near Helena, Montana, during the fall of 1999. Duplicate sediment samples were collected from four locations in Silver Creek and from two sites associated with tailing ponds along the creek. Characterization of contaminated sediment material collected from Silver Creek was conducted to determine the chemical and physical nature of the sediment materials collected. This data was previously provided in Table 2.

The data show that higher amounts of Hg are associated with the small particle size fraction (Table 2). All samples evaluated showed the highest levels in the <80-mesh size. These results are similar to those found in the previous work done using samples collected from

monometer sites along pipelines in Canada. For the sediment materials collected in Silver Creek, mercury levels ranged from less than 1 mg/kg in the mesh fraction to about 9 mg/kg in the 100 mesh fraction. Mine tailings contained mercury levels of about 100 mg/kg.

Mercury removal efficiency data are provided in Table 7. Overall the removal rates appear to be relatively low compared to the previous work conducted with soil materials containing high levels of organic matter. However, several treatments appeared to successfully remove mercury from the sediments and mill tailing materials. Mercury removal rates of 99% were reached using treatment 13, which consisted of treating the same soil with $\text{Ca}(\text{OCl})_2$ and HCl reagents three (3) times. Modifying the sequence of attractants and increasing contact time also accomplished higher removal rates.

Table 7. Mercury removal efficiency determined with $\text{Ca}(\text{OCl})_2$ (solid) as the extractant for contaminated samples collect in the Silver Creek drainage.

Treatment #	Treatment	Initial [Hg]	Final [Hg]	Efficiency
		(mg/L)	(mg/L)	(%)
1	$\text{Ca}(\text{OCl})_2$ + 5% HCl, shake 30m, H_2O rinse,	18.93	6.53	65.5
2	$\text{Ca}(\text{OCl})_2$ + 10% HCl, shake 30m, H_2O rinse,	18.93	15.90	16.0
3	$\text{Ca}(\text{OCl})_2$ + 5% HNO_3 , shake 30m, H_2O rinse,	18.93	15.03	20.6
4	Part A – $\text{Ca}(\text{OCl})_2$ shake 30m, H_2O rinse. Part B – 100ml 5% HCl, shake 30 m, H_2O rinse.	18.93	14.40	23.9
5	Run treatment 2 times	18.93	1.91	89.9
6	$\text{Ca}(\text{OCl})_2$ + 5% HCl, shake 30m, H_2O rinse, run a second time.	18.93	20.05	0
7	Treatment 4 Plus – extract with HCl first, and $\text{Ca}(\text{OCl})_2$ second.	18.93	14.10	25.5
8	Treatment 6 with 60m reaction time, no 2 nd run	18.93	17.85	5.7
9	Treatment 6 with 90 min reaction time, no 2 nd run.	18.93	10.25	45.9
10	$\text{Ca}(\text{OCl})_2$, shake 30m, H_2O rinse,	18.93	15.95	15.7
11	5% HCl, shake 30 m, H_2O rinse.	18.93	17.35	8.3
12	Part A - 5% HCl, shake 30 min, Solids go to Part B - $\text{Ca}(\text{OCl})_2$, shake 30m,	19.35	10.05	48.06
13	Treatment 4 – run 3 times.	19.20	0.2	98.9

A combination of $\text{Ca}(\text{OCl})_2$ and HCl appeared to provide the best reagent combination to remove mercury from contaminated materials. A question that was addressed in this set of trials was whether or not the sequence of reagent application impacts the results. The data presented in Table 7 show that the most effective sequence for mercury removal is to initially oxidize the system followed by acidification. The reason for the difference is not known at this time. Apparently, low pH reduces the oxidation process associated with the conversion of Hg^0 to Hg^{+2} . The data collected show that the oxidants and competitive sorbents remove some mercury from the mill tailings and the stream sediments. However, the removal rates appear to be relatively low unless repetitive treatments are prescribed such as used in Treatment 13.

The column flotation experiments were not conducted for these materials. The testing program for the northern Canada materials showed that the flotation method for removing mercury from the extraction solution was very successful. The use of flotation to remove solution mercury from the extractions taken from the Silver Creek samples is also expected to work well with minimal modifications to the reagent solutions. Therefore, column flotation testing was not conducted for these materials.

CONCLUSIONS

The Haz-Flote technology was shown to be an effective treatment for the removal of mercury from contaminated materials. This effort also demonstrated that the technology works very well on the soil materials taken from northeastern Canada, which were characterized with high organic matter. More extreme measures, such as the use of repetitive treatments, were necessary to remove mercury from soils and mill tailings collected from the Silver Creek drainage located near Helena, Montana. However, Haz-Flote still resulted in a 99 % removal rate. The application of Haz-Flote in a counter-current design will likely remedy any issues such as the requirement for repetitive treatment applications.

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APPENDIX I

SAFETY PLAN FOR MERCURY SCRUBBING AND FLOTATION EXPERIMENTS

BACKGROUND

The Haz-Flote™ process is conceived as an economical and environmentally sound method for removing heavy metals from contaminated soils. Because of the relatively large number of sites with significant mercury contamination, a mercury contaminated soil sample was selected to evaluate the technical feasibility of the process. The soil (sediment) samples were obtained from Silver Creek located north of Helena, Montana. Samples collected from the site ranged from less than 1 ppm to about 100 ppm Hg.

Sample characterization will be conducted by Intermountain Laboratories (IML) located in Sheridan, Wyoming.

OBJECTIVE

The objective of this document is to establish the procedures that will be followed in order to minimize the exposure of laboratory personnel to the potential hazards associated with the mercury contaminated samples. Through the application of engineering controls, appropriate personal protective equipment, medical surveillance, and workplace environmental monitoring, employee exposure to Hg should be kept very near zero.

GENERAL SAFETY PROCEDURES

Four strategies will be employed in order to minimize hazardous exposures: (1) the smallest quantities of the material that can practicably be used for each phase of the testing will be used; (2) sample containers and process vessels will be open for the minimum time needed to complete each operation, containers will remain sealed at all other times; (3) appropriate engineering controls and personal protective equipment (e.g. fume hood, gloves, respirators etc.) will be used. Strict adherence to WRI policies prohibiting eating, smoking etc. in laboratories will be required. The work area will be kept clean and uncluttered. All work will be performed in designated areas, and all people with access to this area will be informed that toxic materials may be present in the area; (4) air monitoring for Hg concentration will be conducted during all phases of the operation. Engineering controls will be evaluated and improved if any quantity of mercury vapor is detected; and (5) protective gear used during the experiments will be placed in an appropriate container and disposed at an appropriate landfill.

The experimental work can be conveniently divided into four tasks. The laboratory methods for each task and the safety measures to be used during implementation of each task are described in the following paragraphs.

Task 1. Sample Preparation

In this task representative samples of the contaminated soil are prepared for use in subsequent tasks. A portion of the soil sample will be removed from the bulk container and sieved. The -100 mesh fraction will then be divided into representative samples. The samples will be placed in 50mL Teflon centrifuge tubes or other appropriate containers, and the tubes will be capped and labeled with hazard code, and notebook and page numbers.

Laboratory Procedures

The sample of contaminated soil will be placed onto a -100 mesh stainless steel sieve provided with a matching bottom pan. Place a cover on the sieve and pan then gently shake them by hand. Mix and quarter the -100 mesh fraction from the pan three times. Place the resultant fractions in appropriate containers and cap and label the tubes. Place the +100 mesh solids from the sieve in a 5-gallon bucket that is clearly labeled as composite mercury contaminated sediment material. The bucket will be sealed immediately and held for later disposal using an appropriate licensed contractor.

Safety Procedures

When ever the bulk sample is opened, disposable Tyvec coveralls, heavy gauge nitrile gloves, and a half-mask respirator with Merc Sorb indicator cartridges will be worn. The sample container will be opened in a hood

All operations must be carried out in a fume hood. The sash will be set so that a minimum face velocity of 150 fpm is maintained when ever the sample container/containers are open. Nitrile gloves, Tyvec sleeves, and a laboratory apron will be worn when working with samples in the hood. Air monitoring will be conducted using a Jerome 431-X (or equivalent) mercury vapor analyzer (refer to the attached operating instructions). If any detectable concentration of mercury vapor is found outside the hood, the work will stop and engineering controls will be modified to eliminate the vapor.

Task 2. Scrubber Tests

The purpose of the scrubber tests is to identify the least severe conditions under which mercury compounds can be completely removed from the solids sample. In this task the soil samples described above will be mixed with various extraction solutions. The mixtures will then be filtered and the resultant liquid and solid fractions analyzed to determine the distribution of Hg between the two phases.

Laboratory Procedures

For a typical test, securely mount the flasks containing the treated samples in a wrist action shaker. Shake the flasks for the prescribed amount of time then allow them to stand for 1 hour before filtering in the hood.

After filtration measure and record the pH of the liquids before sealing the containers. Wipe the outside surface of the containers clean before removing the containers from the hood and submitting them for analysis.

Safety Procedures

Scrubber tests will be carried out in the hood. Air monitoring and personal protective equipment as described under Task 1 will be employed. The concentrated reagents from which the stock scrubber solutions will be prepared are very corrosive and require careful handling. All stock solutions will be prepared, stored, and used in the hood.

Task 3. Flotation Tests

In this task mercury is separated from the scrubber solution using a batch-type adsorbing colloid flotation technique. The flotation process will concentrate the mercury from the scrubber solution into a small volume of collapsed foam, and thus complete the soil cleaning cycle. Testing will be conducted to determine the effects of such process variables as suspended solids, ionic strength, pH, foaming rate, interfering ions, collector, and collector concentration on the separation efficiency.

Laboratory Procedures

The feed solution will be prepared in the hood, and all safety precautions listed for the scrubber tests above will be observed.

For a typical flotation test, place the bulk scrubber solution (prepared in task 2) in a 1L beaker. Save a sample of the flotation feed liquid for analysis. Add the appropriate metallic salts. While mixing gently add sodium sulfide solution. Add sodium nitrate to adjust ionic strength to the desired level. Add sodium hydroxide or nitric acid to adjust pH as needed. Add hexadecyltrimethylammonium bromide (HTA) to coagulate the colloid then allow the solution to stand for ten minutes.

Pour the solution and the floc into the flotation column. Begin air flow at the prescribed rate. At six minute intervals inject 10 ppm HTA solution to maintain steady foaming conditions. After foaming for the prescribed time, collect and weigh the collapsed foam. Save a sample of the collapsed foam and the column liquid for analysis. The collapsed foam may contain high levels of mercury, and should therefore be handled with care.

Safety Procedures

Flotation tests will be conducted in the hood, and all safety precautions established for the scrubber tests will be observed.

RESPONSIBILITIES

WRI has a fundamental responsibility to provide the facilities, equipment, and maintenance to provide a safe working environment, and to establish and implement policies and procedures that minimize the exposure of its employees to potential workplace hazards.

It is the responsibility of the program manager to be aware of potential hazards and to institute an appropriate safety plan that addresses these hazards. WRI's safety officer will review and approve safety plans for all laboratory activities. The safety officer will be responsible for checking equipment and maintenance to assure that it is in safe working order. The safety officer will assist with environmental monitoring, and establish procedures for the safe disposal of hazardous wastes generated by laboratory activities.

Scientists and technicians will be aware of the potential hazards associated with this program, and will work in accordance with the precautions established in the safety plan. They will have the opportunity to review this plan and provide input regarding its validity and implementation. All operations involving the contaminated soil samples will be documented in a WRI notebook. Any deviation from the prescribed plan will be documented and must be approved by the program manager. Scientists and technicians working on the project will report any acute exposure to the soil sample or failure of safety equipment or engineering controls to the program manager or WRI safety officer immediately.

MEDICAL MONITORING

All employees working with mercury contaminated samples will be subjected to a background physical examination to establish a baseline for comparison. The examination will consist of urine and blood tests on a routine basis. A baseline test will be performed prior to initiation of the project. Follow-up examinations will be conducted on a bi-weekly basis.

Acute Exposure

In the event of any acute exposure to the sample or to the concentrated reagents used in the testing, emergency medical attention will be sought immediately. Information regarding the nature of the sample and reagents will be available to help with emergency response.

Chronic Exposure

WRI will retain the services of a laboratory to assist in the assemblage of a medical monitoring program designed to detect signs of chronic exposure to the mercury contained in the soil sample.

EMERGENCY RESPONSE

The laboratory working area including the hood and bench areas will be tested for mercury vapor using the mercury vapor analyzer before work commences and after work is completed each day. Additional readings will be taken during laboratory activities every hour. Each test result will be documented in the laboratory notebook. If mercury is detected at high levels or a spill of solutions containing high levels of Hg occurs the technician and/or scientist should leave the working area, close the door, and contact the health and safety administrator and supervisor as soon as possible. If these contacts are not available the emergency response team should be employed.