SUBTASK 1.8 – MERCURY RELEASE FROM DISTURBED ANOXIC SOILS

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EXECUTIVE SUMMARY

The primary objectives of experiments conducted at the Energy & Environmental Research Center (EERC) were to provide information on the secondary release of mercury from contaminated anoxic sediments to an aqueous environment after disturbance/change of in situ physical conditions and to evaluate its migration and partitioning under controlled conditions, including implications of these processes for treatment of contaminated soils.

Experimental work included 1) characterization of the mercury-contaminated sediment; 2) field bench-scale dredging simulation; 3) laboratory column study to evaluate a longer-term response to sediment disturbance; 4) mercury volatilization from sediment during controlled drying; 5) resaturation experiments to evaluate the potential for secondary release of residual mercury after disturbance, transport, drying, and resaturation, which simulate a typical scenario during soil excavation and transport to waste disposal facilities; and 6) mercury speciation and potential for methylation during column incubation experiments.

The background mercury concentration in the sediment used for the experiment ranged between 323 to 377 mg/kg, with methylmercury ranging between 13 to 21 µg/kg. Results of the field bench-scale dredging simulation confirmed mercury release exceeding U.S. Environmental Protection Agency (EPA) standards. Observed mercury release during the mixing and resaturation test, however, appears to be only temporary. Binding to organic particles, sulfides, and likely iron oxides results in relatively fast capture of mercury released in early stages of disturbance. Only a minor increase of the mercury concentration in vapors was observed during the volatilization test. The results indicate that some mercury becomes available for volatilization in drier soils; however, all recorded levels and the calculated mercury concentration remain several orders of magnitude below regulatory limits of 0.1 mg/m^3.

Because of the high organic content in soils used in experiments, over 250% water content reduction was observed over the period of drying. Resulting mass reduction implies that sediment drying could translate into considerable reduction of costs associated with handling and transport of contaminated soils to final disposal locations. Incubation experiments indicate that the largest fraction of mercury in each sediment column was in the form of mercury sulfide, presumably cinnabar. The observed reaction rates indicate that mercury combined with sulfides in the sediment almost immediately.

The stability observed for mercury in undisturbed anoxic sediments may represent an opportunity for treating wastewater highly contaminated with mercury and other toxic metals in natural or engineered anoxic ponds (reactors).

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1.0 INTRODUCTION

The adverse impact of mercury on human health and the environment has been long recognized; however, only the relatively recent introduction of the risk-based concept in contaminant evaluation has accelerated scientifically based research to understand mercury's occurrence and cycling. Biogeochemical understanding of the global mercury cycle has changed dramatically since the U.S. Environmental Protection Agency (EPA) singled out mercury for special study in 1990 under Title III of the Clean Air Act Amendments. The improvement of sampling protocol and the development of more sensitive analytical techniques such as CVAAS (cold-vapor atomic absorption spectroscopy) resulted in more reliable detection of the contaminant source and its transport routes. The source of mercury in water, soil, and atmosphere has been most often traced to individual industrial units, and the frequent occurrence of organic forms of mercury in the food chain has been linked to behavioral abnormalities, impaired growth, reduced reproductive process, and death of organisms. At the same time, however, insufficient data and uncertainties associated with the secondary release of mercury, potentially resulting in its increased bioavailability, hindered the progress in remediation of mercury-contaminated sites. These problems typically pertain to contaminated sites exposed to both natural and human-induced disturbance, such as wetlands; resaturated, reclaimed, or abandoned mine land sites; tidal environments, etc. Many previously contaminated sites reach a state of geochemical equilibrium in which contaminants are bound to sediments and are immobilized. In some cases, however, disturbances associated with site cleanup may remobilize contaminants and cause unnecessary environmental damage. As a result, both the technical and regulatory community is challenged with decisions pertaining to the benefits and risks associated with cleanup strategy and technologies applied to design, justify, or approve a specific remedial approach.

2.0 OBJECTIVES

The primary research objectives were to provide information on the secondary release of mercury from contaminated anoxic sediments to an aqueous environment after disturbance or change of in situ physical conditions and to evaluate mercury's migration and partitioning under controlled conditions, including implications of these processes for treatment of contaminated soils. Interim results after the first year of experimental work indicated that project findings may apply to more than just investigation of mercury release mechanisms. The scope of the project was broadened to investigate release of other industrial metals from disturbed sediments, namely, those that may, potentially, be associated with acidic discharge from mining operations. In addition, the column experiments were designed to investigate the fate/speciation of mercury in organic- and sulfide-rich wetland sediments.

3.0 SCOPE OF WORK

The scope of work for this project entailed the following tasks:

- Selection of a mercury-contaminated site and sampling and analysis of sediment, pore water, and water in the affected environment.

- A field bench-scale experiment to evaluate mercury release from sediments to the aqueous environment after a simulated disturbance such as dredging.

- A laboratory column study to evaluate potential for a long-term mercury release to the water.

- A volatilization experiment to evaluate potential risks associated with mercury release from contaminated sediments during drying.

- Evaluation of the secondary release/leaching of mercury and selected metals from disturbed, transported, and dried sediments after resaturation.

- A respirometry experiment using spiked water to determine potential toxicity effects of $HgCl_2$ on the microbial population of wetland soil samples.

- A column incubation study to determine the efficiency of mercury capture on organic soils, mercury speciation, and distribution in sediment and water.

A detailed description of experimental activities including their results is provided in individual sections for each respective experiment.

4.0 EXPERIMENTAL METHODS AND RESULTS

4.1 Sediment Characterization

The project was initiated in April 1999, with early work focused on field sampling, detailed experimental design, and logistics. The field effort undertaken April 29 – May 7 consisted of water and sediment sampling from a publicly accessible mercury-contaminated lagoon at Berry's Creek, in the area of the Meadowland wetlands in New Jersey. The section of the estuarine Berry's Creek became severely polluted as a result of past refining and reprocessing activities upstream of the site. The sediment profile at the sampling location is characterized by about a 50-cm-thick layer of black, mucky, organic-rich sediment underlain by light gray silty sediments with remnants of well preserved/nondecayed organic debris. The interface between these two distinguished layers is very clear.

Sediment samples were collected using a Teflon spoon and were placed in a glass jar with a Teflon-coated lid prior to shipment to laboratory. Unless otherwise noted, water samples were filtered using 0.45-µm Geotech disposable filters, placed in Teflon bottles, and shipped without preservation to the laboratory for immediate processing. Sampling jars and bottles were always double-bagged and preserved in ice-filled coolers. All samples were always shipped overnight for immediate processing. Sampling procedures strictly adhered to standards described in Bloom (1994) with guidance kindly provided by Frontier Geosciences. All mercury and metal analyses were conducted by the same organization.

Water samples were collected during high tide at Berry's Creek. Sediment samples for pore water extraction were collected into 10-cm-long by 7-cm-diameter polycarbonate sleeves filled and capped under water to preserve anoxic conditions. Teflon seals were used as inserts to separate the sediment from the plastic lid. Samples arrived at the laboratory in perfectly preserved anoxic conditions. Sediment samples were placed in glass jars with Teflon-coated lids. A second set of sediment samples was collected in 7-cm-diameter by 50-cm-long polycarbonate sleeves pushed to a depth of 10–12 cm. This way only the bottom portion of the sampling column was filled to preserve the natural sediment profile, and the sampling sleeve became the experimental column to minimize sediment processing. Two sets of three samples/columns each were topped with ambient creek and deionized (DI) water on site, respectively. Columns were sealed with Teflon caps and double-bagged prior to transport to the EERC laboratory. Four additional columns were filled with sediment using the same method, sealed on-site, and topped with DI at the EERC upon arrival. Composite sediment for volatilization and resaturation experiments was loaded into 3.5-gallon plastic pails, sealed, and transported to the EERC. Finally, a reference column, indicated as GF in Table 1, filled with DI water was prepared from uncontaminated sediments collected from the English Coulee in Grand Forks using the same sampling technique.

The total mercury content in the reference sample (Table 1) is 3 orders of magnitude lower than that in sediment samples collected from the target location at Berry's Creek (BC).

Results of sediment analysis are provided in Table 1 and Appendix A5. Additional detailed sediment analyses were conducted prior to the resaturation experiment and are presented in Section 4.5. Mercury analysis for water is presented in Section 4.2, Table 2. The background mercury concentration in the sediment used for the experiment ranged between 323 to 377 mg/kg on a dry weight basis (102–119 mg/kg on an as-received basis) with methylmercury ranging between 13 to 21 µg/kg.

4.2 Field Bench-Scale Dredging Simulation

The field experiment consisted of a simulated disturbance (dredging simulation). Black, organic-rich sediment representing the upper portion of the investigated soil profile (interval of 10–15 cm from top) was loaded to a 20-L glass vessel filled with water from the creek and kept in suspension using a battery-powered rotor with Teflon-coated stirrer. The test started after

Table 1. Sediment Analysis, dry basis

Sample	Date	Location	Sediment Description	THg, mg/kg[1]	MeHg,[2] µg/kg	LOI,[3] %	Fe, mg/kg	Mn, mg/kg
ERC-1	5/2/99	BC	Black organic-rich	353	12.8	21.6	35,700	2200
ERC-2	5/2/99	BC	Gray sediment	20.4	1.03	18.6	34,100	489
ERC-3	5/2/99	BC	Gray sediment	66.2	9.77	23.9	52,000	739
FCS-1	5/2/99	BC	Sample for pore water extraction	377	18.8	21.1	41,800	1120
FCS-2	5/2/99	BC	Sample for pore water extraction	323	21.4	19.6	41,800	3003
ERC-4	6/29/99	BC	Naturally dried sediment	252	53	NA	47,500	805
ERC-5	5/20/99	GF	Reference	44.9 µg/kg	0.902	14.8	20,700	660

[1] Total mercury.
[2] Methylmercury.
[3] Loss on ignition.

Table 2. Water Analysis for Dredging Simulation

Sample	Description	Time	THg, ng/L	MeHg, ng/L	TOC,[1] mg/L	Fe, µg/L	Mn, µg/L
ERCW-2	Creek Water	12:16	10.2	0.367	8.4	<150	564
PCS -1	Pore water after extraction		14.5	0.172	20	190	3,840
PCS -2	Pore water after extraction		87.2	0.129	19	180	5,370
ERCW-6	Filled vessel, pretest sample	10:41	1,540	0.552	10	280	1,290
ERCW-3	Settled after 15 min of stirring	12:31	**2,810**	0.704	12	650	2,310
ERCW-5	Stirred	13:46	**2,550**	0.559	13	220	2,270
ERCW-8	Settled	16:01	413	0.053	14	<150	2,240
Unfiltered Samples							
ERCW-1	Creek	12:01	857	4.37	16	760	616
ERCW-4	Settled after 15 min of stirring	12:46	**40,800**	NR	44	68,000	3,420
ERCW-7	Stirred	14:11	**5,340**	NR	540	550,000	19,000

[1]Total organic carbon.

collection of Sample ERCW-6 at 10:42 and had to be suspended after 15 min of stirring because of rapid filter clogging during sampling. Samples ERCW-3 and ERCW-4 were collected from suspension prior to restarting the test. Only Samples ERCW-5 and ERCW-7 were collected during the second stirring that started at 13:15 and was terminated after 60 min. The last sample, ERCW-8, was collected about 100 min after stirring ceased. The original sampling plan based on regular sample collection intervals during mixing was not met because of high sediment load leading to rapid filter clogging.

The analytical results for water samples are provided in Table 2 and Appendix A5; field-measured parameters are in Table 3. Mercury content in filtered and unfiltered samples from ambient creek water used during the experiment was 10.2 and 857 ng/L, respectively. Two pore water samples extracted from sediment cores contained 14.5 and 87.2 ng/L of total mercury and 0.13 and 0.17 ng/L of MeHg. Shortly after initiation of intense disturbance of soils during the mixing test, the mercury content in the water increased to 1540, 2810, and 2550 ng/L in filtered samples and up to 40,800 ng/L in unfiltered ones, i.e., over 250 times the total mercury increase in unfiltered samples. The concentration of total suspended solids during the test was approximately 28,000 mg/L, which can be compared to a situation during dredging. After termination of mixing and settling of about 2 hours, the mercury content in the sampled water dropped to 413 ng/L.

The bolded results in Table 2 indicate that the mercury concentration in water exceeds EPA standards for drinking water (2 µg/L) in both filtered and unfiltered samples as a result of stirring. Replicate analysis for residual mercury in suspension after about 2 hours of settling, however, is different as evidenced from Samples ERCW-3 and ERCW-8 with mercury concentrations 2810 and 413 ng/L, respectively. This may indicate that the mercury concentrations during the first stirring when sampling failed because of filter clogging were higher. It also indicates that after original release, the mercury concentrations in an aqueous environment decline. This was confirmed by previous tests conducted by Lindberg and Harriss (1977).

Table 3. Field-Measured Parameters

Sample	Description	Time	pH	EC, mS/cm [1]	DO, % [1]	Eh, mV	Temp., °C
ERCW-2	Creek water	12:16	7.6	8.1	51.0	54.8	16.3
ERCW-6	Filled vessel, pretest sample	10:41	7.4	6.7	49.0	ND[1]	17.0
ERCW-3	Settled after 15 min of stirring	12:31	7.2	6.5	45.5	–15.2	17.7
ERCW-5	Stirred	13:46	7.4	6.7	42.6	–23.6	18.6
ERCW-8	Settled	16:01	7.1	6.8	37.4	–5.8	18.0
Unfiltered Samples							
ERCW-1	Creek water	12:01	7.8	8.2	59.0	–53.6	16.2
ERCW-4	Settled after 15 min of stirring	12:46	7.1	6.4	9.5	–12.9	17.0
ERCW-7	Stirred	14:11	7.3	6.4	11.8	45.6	18.0

[1] Electrical conductivity.
[2] Dissolved oxygen.

Results of the experiment confirmed mercury release from contaminated sediments even above regulatory limits for drinking water; however, observed release appears to be only temporary. Binding to organic particles, sulfide, and likely iron oxides results in relatively fast capture of released mercury. Similar findings are described by Bloom and Lasorsa (1999).

4.3 Column Study

Sediment sampling for the column study is described in Section 4.1. Samples were collected in 7-cm-diameter, 50-cm-long polycarbonate sleeves pushed to a depth of 10–12 cm. This way only the bottom portion of the sampling column was filled to preserve the natural sediment profile, and the sampling sleeve became the experimental column to minimize sediment processing. Two sets of three samples/columns each were topped with ambient creek (NW set) and deionized water (DI set) on-site, respectively. Column NW-1 remained undisturbed; column NW-2 was mildly disturbed (top 2 cm of the sediment); and column NW-3 was disturbed after initial sampling. Samples filled with DI water were mixed after initial sampling. Reference column RS-1 was collected from wetland near the English Coulee in Grand Forks, North Dakota, and filled with native water. A declining trend for redox potential was observed during the column study (Appendix B); see Table 4.

Analyses of samples collected during additional bench-scale experiments after transport to EERC laboratories indicate slight release of mercury to the native water column in both the undisturbed and disturbed experimental settings. Observed mercury enrichment in columns with DI water was over an order of magnitude higher relative to columns filled with native water. It is apparent that a long-term geochemical equilibrium established in a native anoxic environment between sediment and brine estuarine water provides conditions for relative mercury immobilization. Both EC (about 7 mS/cm) and pH of 7 remained stable during the experiment.

Table 4. Mercury Release – Column Study

Column	Date	Comment	THg, ng/L	MeHg, ng/L	TOC, mg/L	Fe, µg/L	Mn, µg/L
NW1	5/20/99	Undisturbed	37.7	0.065	13	51.9	1570
NW1	6/29/99	Undisturbed	22.5	0.032	17	ND	2210
NW-2	5/20/99	2-cm disturbed	64.4	0.014	16	81.0	2480
NW-2	6/29/99	Settled	53.4	0.049	18	ND	1550
NW-3	9/13/99	Initial	157	NA	NA	NA	NA
NW-3	9/13/99	MW-3 dup.	154	NA	NA	NA	NA
NW-3	9/22/99	Settled (mix 9/13)	344	NA	NA	NA	NA
DI-1	9/13/99	Initial	118	NA	NA	NA	NA
DI-1	9/22/99	Settled (mix 9/13)	3189	NA	NA	NA	NA
DI-2	5/20/99	Disturbed	5520	0.565	NA	NA	NA
DI-2	6/29/99	Settled	9190	NA	NA	NA	NA
RS-1	5/20/99	Reference GF	1.90	0.086	NA	211	1925

ND – Not detected.
NA – Not analyzed.

On the other hand, reaction of DI water observed in columns DI-1 and DI-2 resulted in mercury release with the most notable trends observed for column DI-2. While Eh values in DI-2 exhibited declining trends with a tendency to reach anoxic conditions, both pH (6.3 to 6.8) and EC (172 to 1381 µS/cm) increased. We speculate that this release of mercury is a result of dissolution of mineral salts and partial release from oxides in sediments that 1) initially captured mercury during their formation in brackish environment and 2) were in equilibrium with brine water prior to disturbance and exposure to DI water. Eh in water in column trends toward reestablishment of redox/anoxic conditions even after disturbance. The mercury released during the column experiment remains in aqueous solution above the disturbed sediment after colloidal particles have settled. This trend is in sharp contrast to dynamic experiments described in Section 4.5, where an abundance of particles present in solution and an ongoing oxidation process in an oxic environment contribute to the capture of mercury within early minutes of the experiment. This interesting trend has considerable implications for treatment and potential exposure of contaminated marine sediments to a freshwater environment and thus deserves further investigation.

4.4 Drying and Volatilization Experiment

This experiment consisted of the monitoring of mercury vapors from soil dried in laboratory conditions. The active flux chamber used for the experiment is an adaptation of the EPA isolation flux chamber used to measure volatile emissions from solid and liquid surfaces (EPA, 1986). The active flux chamber consists of a 30-cm-diameter cylindrical stainless steel shell that is covered with a tinted Plexiglas (acrylic) dome. A low-flow pump is used to deliver air into the flux chamber at a rate of 3 L/min. The air that is pumped into the flux chamber sweeps surface air into the dome where mercury vapor concentrations are measured. An iodated carbon trap prepared by Frontier Geosciences, Inc., was used to detect the average mercury concentration in the flux chamber.

The experiment was conducted in strictly controlled laboratory conditions. Soil was dried at a temperature of 25°C, and effluent vapors pumped from the flux chamber were collected in iodated carbon traps at a constant flow of 2 L/min for a period of 14 days. A minor increase in the mercury concentration in vapors was observed during the test. The results indicate that some mercury became available for volatilization in drier soils; however, all recorded levels and calculated mercury concentrations remained several orders of magnitude below the regulatory limits (Occupational Safety and Health Administration Permissible Exposure Limits [OSHA PEL]) of 0.1 mg/m^3. Data are provided in Table 5.

Table 5. Flux Calculated from Carbon Trap Data[1]

Sample	Time Elapsed, min	Moisture, %	Hg in Trap, ng/trap	Mercury, mg/m^3	Average Temp., °C	Flux, $mg/m^2/min$ Correction[2]	No Correction
ERCA-4	2,880	272	18.6	3.23E-06	25.0	1.8E-07	1.4E-07
ERCA-5	5,760	209	12.7	2.20E-06	25.0	1.2E-07	9.3E-08
ERCA-7	8,640	118	11.3	1.96E-06	25.0	1.1E-07	8.3E-08
ERCA-8	11,520	101	40.2	6.98E-06	25.0	3.8E-07	2.9E-07
ERCA-9	14,400	84	21.2	3.68E-06	24.0	2.0E-07	1.6E-07
ERCA-10	17,355	59	43.7	7.39E-06	25.0	4.0E-07	2.1E-07

[1] Inlet pumping rate = 3.0 L/min; outlet = 2.0 L/min; flux chamber area 0.071 m^2; pump time 2880 min.
[2] Evaporation correction normalizes flux for dry air at 25°C.

Because of the high organic content in soils used in experiments, over 250% water content reduction was observed over the period of drying (Figure 1). The attendant volume reduction may suggest a cleanup strategy prior to treatment of these organic-rich soils. If the treatment system is not on-site and provided that space for drying is available, the almost 80% mass reduction translates into considerable reduction of costs associated with transport of contaminated soils to a treatment plant or NDPES-approved disposal location. Another alternative is temporary drainage and drying of soils in place prior to transport off-site.

4.5 Secondary Release – Resaturation Experiment

A major goal of this specific test was the evaluation of the potential for secondary release of residual mercury after disturbance, transport, drying, and resaturation, which simulate a typical scenario during soil excavation and transport to waste disposal facilities. Ambient conditions such as temperature, pH, Eh, DO, and EC were measured during the experiment directly in suspension. A limited test conducted in June 1999 (Test 1) confirmed an inverse trend between mercury concentration and concentration of Fe, Mn, and TOC (Table 6, Figure 2). A replicate test (Test 2) was conducted in February 2001 to confirm trends observed during the first test and to further investigate the relationship for other metals.

Dried soil was loaded into a 20-L glass vessel for Test 1 and 15-L plastic pail for Test 2 and kept in suspension using a battery-powered rotor with a Teflon-coated stirrer. The mass-based ratio of dry sediment to water was about 1:35, or 3% for both tests. Sediment analyses for Test 2 are provided in Table 7 and Appendix A1. Water samples were collected at specific intervals during

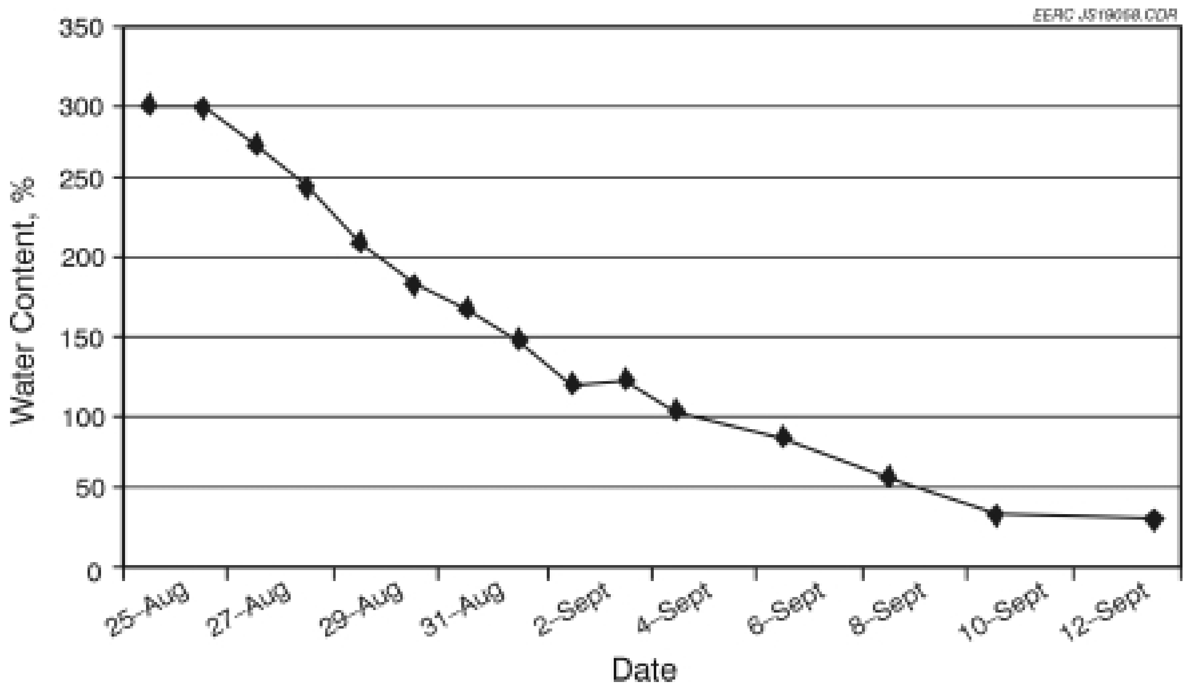

Figure 1. Water content during drying.

Table 6. Secondary Mercury Release – Test 1 (June 29, 1999)

Sample ID		ERCW MB	ERCW-23	ERCW-24	ERCW-25	ERCW-27
Note		Blank	Mix	Mix	Mix[1]	Settled
Time		13:10	13:25	13:45	14:05	15:35
Time Elapsed	min	0	15	35	55	145
THg	ng/L	0.80	1890	1050	896	97.1
Mn	µg/L	NA	5860	7060	8730	9120
Fe	µg/L	NA	4100	5230	5820	5920
TOC	mg/L	NA	15	19	24	25
pH		7.32	4.32	4.39	4.55	4.55
EC	mS/cm	0.01	1.44	1.38	1.23	1.27
DO	mg/L	37.8	8.33	8.17	7.49	5.47
Eh	mV	–13.6	156.6	152.4	143.2	142.7
T	°C	22	21.6	21.7	21.7	21.4

[1]Mixing stopped after sampling.

mixing and after the mixing was stopped. Analytical results from Test 1 are provided in Table 6 and Appendix A5; elemental trends are provided in Figure 2. Analytical results from Test 2 are summarized in Table 8 and Appendix A2, measured parameters are in Table 9, and elemental trends are provided in Figure 3.

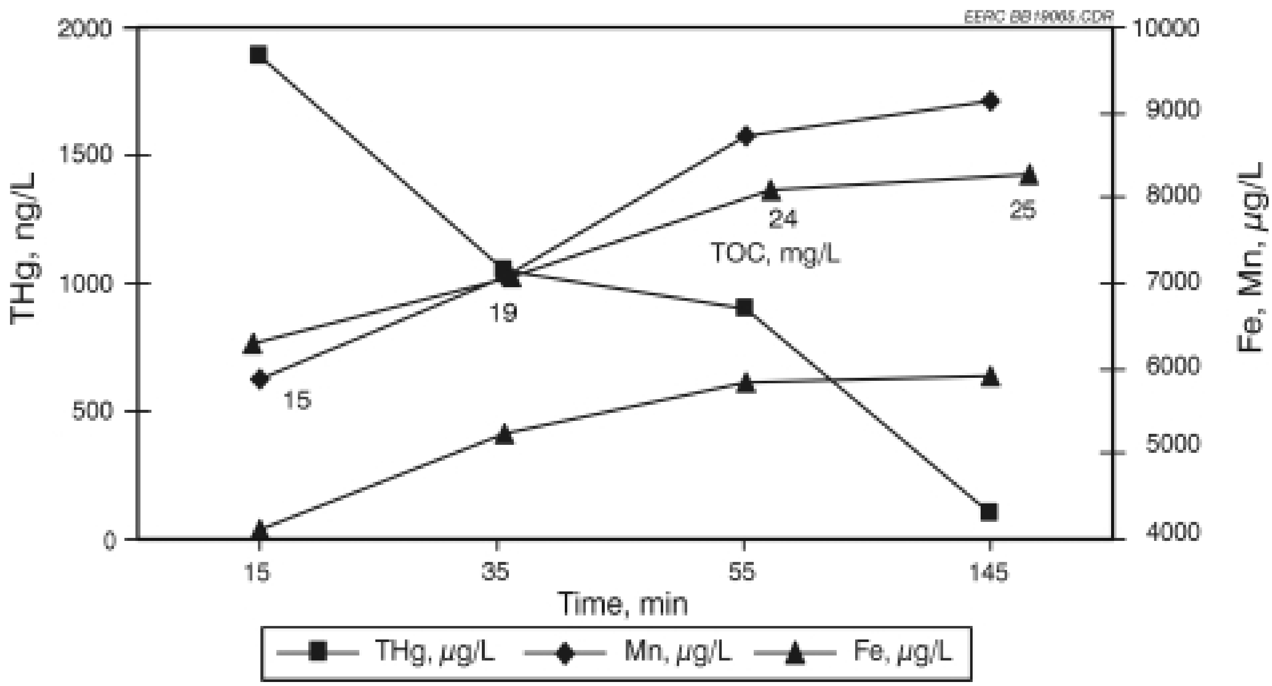

Figure 2. Total mercury, iron, manganese, and TOC trends during Test 1.

Table 7. Analysis of Dried Sediment for Test 2 (mg/kg)

Sample	Soil-1 DB	Soil-2 DB	Soil-1 AR	Soil-2 AR
Cr	1,185	1,193	1,302	1,277
Mn	577	529	634	566
Fe	26,806	25,549	29,457	27,355
Ni	121	128	133	137
Cu	430	433	473	464
Zn	2,556	2,639	2,809	2,826
Cd	21.9	23.4	24.1	25.1
Hg	193	224	212	240
Pb	338	350	371	375

DB – dry basis.
AR – as-received.

The results of resaturation and mixing experiments indicate that mercury concentrations drop considerably within early 10 minutes of mixing. This is contrary to the behavior of other observed metals that, as expected, increase their mobility in the aqueous solution with an attendant decline of pH. Trends observed for mercury are likely associated with one or a combination of physical and chemical reactions in response to stirring. We suggest that the reactions responsible for mercury retention are 1) sediment disturbance resulting in disintegration/dissolution of large particles and consequent mercury affinity to the fine-particle fraction in suspended sediment as observed in natural geological settings for mercury and other metals (Shilts, 1993, 1994); 2) increased content of TOC in response to disturbance, providing for the capture of dissolved mercury on organic particles; 3) decreased pH of the suspension

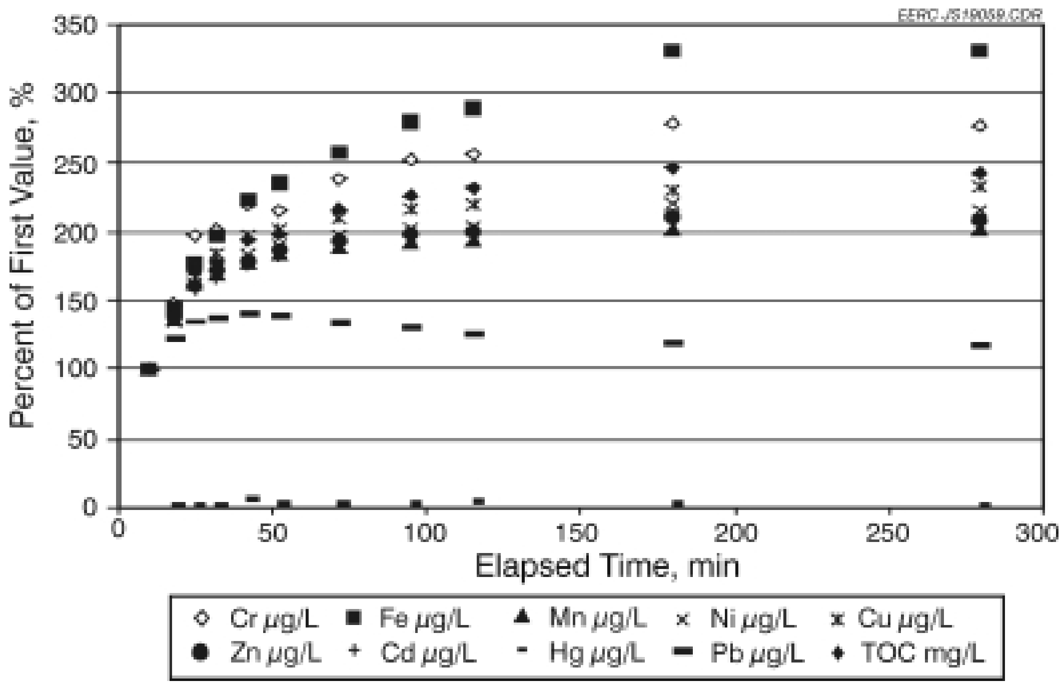

Figure 3. Elemental trends as percentage of the first value (100% = results from the first sample collected 10 min from test start-up).

Table 8. Secondary Mercury Release – Test 2 (February 7, 2001)

Sample	Elapsed Time (min)	Cr, µg/L	Fe, µg/L	Mn, µg/L	Ni, µg/L	Cu, µg/L	Zn, µg/L	Cd, µg/L	Hg, µg/L	Pb, µg/L
EERC-1	Blank	–0.1	1	0.22	0.16	2.86	5.83	0.01	0.001	2.49
EERC-2	10	65	4,456	6,301	949	1,030	33,385	231	11.29	110
EERC-3	18	96	6,450	8,566	1,287	1,484	46,539	314	0.157	135
EERC-4	25	128	7,943	10,212	1,547	1,774	53,703	366	0.197	149
EERC-5	32	131	8,772	10,656	1,642	1,898	57,353	383	0.208	152
EERC-6	42	142	9,945	11,168	1,738	2,030	59,957	407	0.813	154
EERC-7	52	140	10,524	11,572	1,812	2,078	62,705	420	0.236	152
EERC-8	72	156	11,489	11,861	1,874	2,160	65,010	444	0.260	147
EERC-10	95	164	12,445	12,193	1,928	2,227	66,358	453	0.260	143
EERC-11	115	167	12,899	12,251	1,939	2,253	66,822	458	0.573	137
EERC-12	180	181	14,726	12,686	2,095	2,375	70,541	489	0.296	131
EERC-14	280	180	14,746	12,771	2,034	2,391	69,526	473	0.175	128

resulting in destruction of metal oxide and metal sulfide bonds, allowing mercury to compete for the freed bond and replace/exchange other metallic species; and 4) sudden change of redox conditions providing reaction incentive for previously mentioned processes.

Table 9. Test Measured Parameters – Test 2 (February 7, 2001)

Sample	Elapsed Time, min	pH	EC, µS/cm	DO, mg/L	Eh, mV	TOC, mg/L	Temp., °C
EERC-1	Blank	7.03	22	2.22	−11.4	2.1	25.1
EERC-2	10	3.80	760	7.88	188.9	17.2	23.9
EERC-3	18	3.90	1170	6.65	197.0	23.8	23.2
EERC-4	25	3.89	1407	8.06	200.1	29.3	23.1
EERC-5	32	3.60	1669	8.07	199.3	30.6	23.2
EERC-6	42	3.62	1182	7.97	198.3	33.4	23.0
EERC-7	52	3.63	1092	7.93	197.6	34.2	22.9
EERC-8	72	3.65	1006	7.90	196.2	37.2	22.8
EERC-10	95	3.65	998	7.86	196.2	38.8	22.5
EERC-11	115	3.66	1008	8.07	195.8	39.8	22.4
EERC-12	180	3.67	1004	8.44	199.6	42.4	21.3
EERC-14	280	3.68	1015	9.54	193.5	41.6	20.7

While a resaturation test confirmed low potential for release of residual mercury to the aqueous environment, it did not provide information on which one of the noted processes is dominating mercury retention. Organic matter and sulfide bonding are, in general, considered primary factors in considerations on mercury immobilization. To investigate their relative efficiency, an incubation experiment described in Section 4.6 was conducted with a focus on mercury speciation in spiked sediment columns.

4.6 Column Incubation Study – Mercury Speciation

The main objective of this incubation experiment was to investigate the fate of inorganic mercury (Hg) in organic- and sulfide-rich wetland sediments. Results of experiments described in the preceding text confirmed that Hg remained tightly bound to sediments collected from a mercury-contaminated estuary at Berry's Creek, New Jersey. Two theories were postulated to explain the attenuation of Hg to the Berry's Creek sediment. One was that the Hg bound with sulfides in the sediment to form cinnabar (HgS), one of the most stable sulfide compounds. The other theory was that the Hg was bound to organic matter, which was abundant in the sediment. A combination of the theories would be that the Hg was distributed between the sulfide and organic matter in the sediment. This experiment was conducted to determine the fate of inorganic Hg (in the form of $HgCl_2$) when added to anoxic sediments that are rich in both organic carbon and sulfide. The expected result was that the Hg would bind to sulfides in the sediment; however, given the high concentrations of sulfate and organic carbon in the sediment, it is possible that sulfate reduction occurring within the sediment could result in the formation of MeHg, or alternatively, the high organic carbon content of the sediment could lead to the formation of organically bound Hg.

The experiment was carried out in columns of wetland sediment that were spiked with $HgCl_2$. The columns were incubated for 3 months, after which they were analyzed for a suite of Hg compounds, including total Hg, MeHg, and inorganic Hg species. In addition, sediment samples were collected for sulfide analysis.

An additional component of the research was to investigate the stability of the HgS or organically bound Hg compounds (if formed) when subjected to oxidizing conditions. A week before the columns were done incubating, half of the columns were transferred to open containers and mixed daily to encourage oxidation of the sediment. The water and sediment from these containers were analyzed for total Hg and MeHg, and the sediment was analyzed for inorganic Hg species.

The potential binding of Hg with iron or manganese oxides was also investigated to a small extent. One of the columns used for the sediment oxidation experiment was spiked with Fe_2O_3 and MnO_2, with the assumption that the mercury speciation analysis of this sediment would reveal any Hg bound to these compounds during oxidation.

4.6.1 Respirometry Experiment

Before the column experiments were initiated, a respirometry experiment was conducted using spiked water to determine the potential toxicity effects of $HgCl_2$ on the microbial population of wetland soil samples. This experiment was carried out to ensure that the addition of $HgCl_2$ to the soil did not inhibit sulfate-reducing microbes and, therefore, the potential for mercury methylation.

Ten bottles were filled with sediment from Kelly's Slough and spiked with various concentrations of $HgCl_2$. The headspace in each bottle was flushed twice a day and analyzed for CO_2 concentration with a Sable Systems infrared CO_2 analyzer. Results are shown in Table 10.

After 3 weeks, the cumulative concentration of CO_2 was calculated and averaged for each pair of duplicate bottles. The data presented in Figure 4 show that the sediment with the lowest concentration of Hg produced the highest concentration of CO_2, but all the Hg-spiked sediments were more biologically active than the controls (0 mg/L Hg). One explanation for this may be that the $HgCl_2$ was toxic to a portion of the microbial population, eliminating the competition for the surviving microbes (Gallagher, personal communication). Based on the results of the respirometry experiment, it was determined that the toxicity effects of $HgCl_2$ on the microbes within the sediment were probably not of concern for the column experiments.

4.6.2 Sediment Collection

The sediment used for the column studies was collected from a wetland in eastern North Dakota, 6 miles west of Grand Forks. The site, called Kelly's Slough, is an area from which saline water from the Dakota Sandstone percolates upward, forming several wetlands.

The samples were collected from the top 8 to 10 inches of sediment in a small area along the edge of the wetland. The top 2 inches of sediment was a dark brown in color and composed of silt and clay intermixed with an abundance of organics and plant material. The sediment from

Table 10. Respirometry Experiment Setup

Bottles	Hg Concentration (as $HgCl_2$), mg/kg
1 and 2	0
3 and 4	200
5 and 6	600
7 and 8	1200
9 and 10	1800

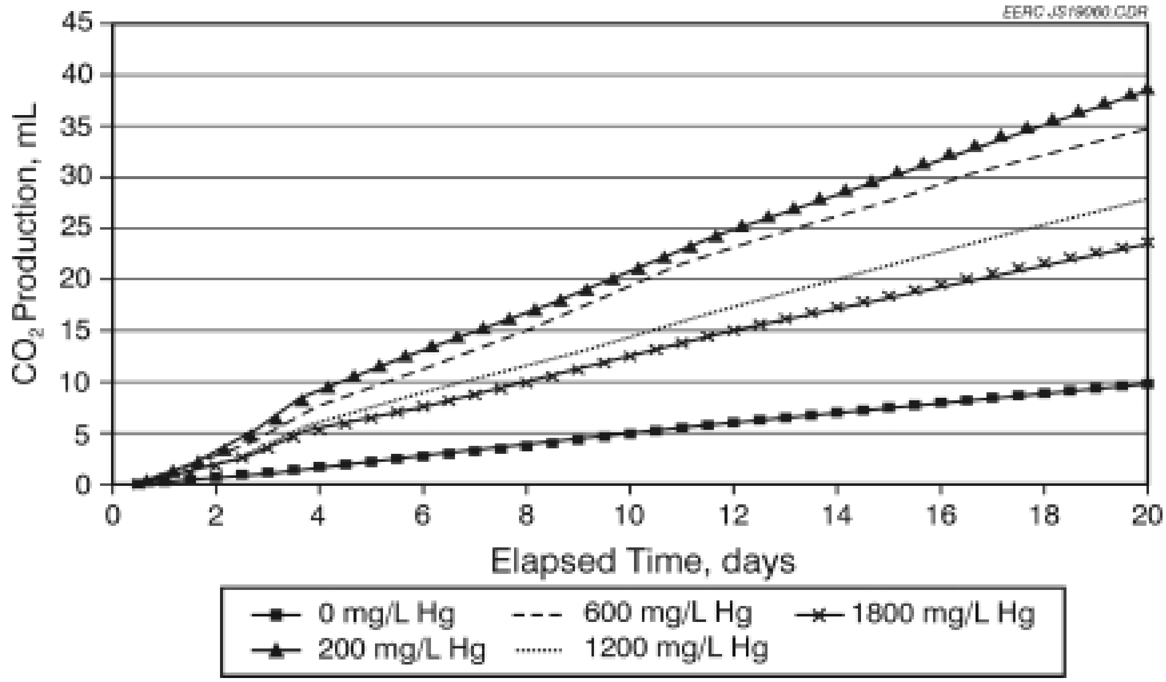

Figure 4. Results of the respirometry experiment.

2 to 10 inches depth was an organic-rich, black material containing a large silt and clay fraction of sediment, as well as some fine- to coarse-grained sand. Both layers of sediment smelled strongly of hydrogen sulfide (H_2S).

The sediment was collected using a Teflon shovel to transfer the sediment into two 5-gallon plastic buckets. Precautions were taken to minimize the disruption of the sediment during sampling. In addition, two 20-L polyethylene carboys were filled with water from the slough. All sampling items that came in contact with the sediment and/or water were first cleaned with Alconox and a 10% HCl solution.

The slough water was analyzed on-site for pH, conductivity, and temperature. The average pH was 8.79; average EC was 9.29 mS/cm; and temperature was 1.5°C. Sediment samples were collected for sulfide and carbon analyses to be conducted at the University of North Dakota Water Quality Laboratory (UNDWQL). The sulfide-sulfur content of the sediment was 0.26%, and the total carbon content was 5.24%. The sediment and water collected from Kelly's Slough were stored (airtight) at 4°C until ready for use in the column experiments.

4.6.3 Column Preparation

Before the sediment collected from Kelly's Slough was disturbed for use in the column experiments, the pH, conductivity, DO content, and temperature of the free-standing water at the top of each bucket were measured (Table 11). In addition, samples of this water were collected and sent to UNDWQL for analysis of major anions, cations, TOC, and total inorganic carbon (TIC). A water sample from each bucket was also collected for total Hg analysis. These results are listed in Table 11.

Table 11. Results of the Slough Water Analysis (units in mg/L unless otherwise noted)

Chemical Parameter	Water Sample from Bucket 1	Water Sample from Bucket 2
pH	7.35	7.38
EC, mS/cm	10.9	10.1
DO	0.01	0.01
Cl^-	3830	3810
NO_3-N	< 1.0	< 1.0
SO_4^{2-}	1170	1040
TOC	22.5	29.9
TIC	137.8	125.3
THg (ng/L)	1.61	3.28
Ca	384	360
K	74.9	68.4
Mg	190	180
Na	1970	1720
Fe	< 0.08	< 0.08
Mn	1.46	1.90

The columns used for the laboratory experiment were clear polycarbonate cylinders 3 inches in diameter and 24 inches in length (total volume = 2.78 liters), sealed on both ends by Teflon-lined caps. All equipment used in the laboratory experiments was cleaned with Alconox and soaked in a 10% HCl solution before use.

In order to fill the columns, enough unaltered wetland sediment to fill columns 1 through 6 was weighed and put in a large plastic container that was cleaned with Alconox and 10% HCl. The sediment was mixed by hand (with gloves on) until it appeared uniform in consistency. A portion of this sediment was taken out and used to fill columns 1 and 2. The remaining sediment was reweighed and then spiked to a concentration of 1000 mg/kg Hg as $HgCl_2$. The $HgCl_2$ was mixed into the sediment by hand, using two layers of chemical-resistant gloves. Once the sediment was thoroughly mixed, a portion was removed and used to fill columns 3 and 4. The remaining sediment was reweighed and then spiked with 1000 mg/kg Fe_2O_3 and 1000 mg/kg MnO_2. The sediment was mixed again and then used to fill columns 5 and 6. The sediment used to fill columns 7 and 8 was collected from a separate container that consisted of unaltered wetland sediment into which cattail "tops" were mixed as a source of fresh organic carbon. The sediment–cattail mixture was spiked with 1000 mg/kg Hg as $HgCl_2$ before being used to fill the columns. A summary of the column contents is presented in Table 12.

A total of four sediment samples were collected during the column preparation for analysis by Frontier Geosciences, Inc. Two samples were collected from the container of mixed sediment before it was spiked with Hg. This was the same sediment used to fill columns 1 and 2 (the control columns). These samples were sent to Frontier Geosciences, Inc., immediately for total Hg analysis (Table 13). A sample was collected from the sediment used to fill columns 3 and 4, and a sample was collected from the sediment used to fill columns 5 and 6. These samples were frozen immediately after collection and were analyzed for inorganic Hg species at a later date.

Table 12. Summary of Laboratory Column Contents

Column	Spiked Hg Concentration, mg/kg Hg as $HgCl_2$	Additional Components
1	0	None
2	0	None
3	1000	None
4	1000	None
5	1000	1000 mg/kg Fe_2O_3 1000 mg/kg MnO_2
6	1000	1000 mg/kg Fe_2O_3 1000 mg/kg MnO_2
7	1000	Cattails
8	1000	Cattails

Table 13. Results of Total Hg Analysis on the Sediment Used to Fill Columns 1 and 2

		Total Hg, ng/g	
Sample	Dry Fraction	Wet Basis	Dry Basis
Unspiked Sediment	0.52	14.7	28.4
Duplicate	0.50	16.5	32.9

4.6.4 Column Incubation and Analysis

The columns were left to incubate, undisturbed, at room temperature for approximately 3 months. One week prior to the end of the incubation, four of the columns (the duplicate in each set) were used to conduct oxidation experiments. The saturated sediments in columns 2, 3, 5, and 7 were each emptied into rectangular plastic containers. Measurements for pH, temperature, DO, and volatile Hg were taken from the saturated sediments immediately after they were put into the plastic containers (Table 14). Volatile Hg was measured with a portable Jerome Hg analyzer, but all readings were below detection and, therefore, not listed in the table.

After the above measurements were taken, 1 L of DI water was added to each container of column sediment and mixed. The sediments were mixed daily and rehydrated as necessary for 7 days. At the end of the seventh day, several samples were taken of the exposed column sediments and sent to Frontier Geosciences, Inc., for various mercury analyses, including total Hg and MeHg within the sediment and water, and Hg speciation analysis of the sediment. Sediment samples were also collected for sulfide analyses by UNDWQL. Standard QA/QC

Table 14. Measurement of pH, Temperature, and DO from the Saturated Sediment in Columns 2, 3, 5, and 7 Just Before Oxidation Experiments

Column	pH	Temp., °C	DO, mg/L
2	7.16	22.6	0.05
3	6.91	23.1	0.01
5	7.03	23.9	0.01
7	6.82	23.9	0.01

procedures were used throughout sampling and analysis of the column sediments. Several blank and duplicate samples were taken and submitted to UNDWQL and Frontier Geosciences, Inc.

The undisturbed columns (columns 4, 6, and 8) were sent to Frontier Geosciences, Inc., for analysis at the same time as the exposed column sediments. Once at the laboratory, the pore water was separated from the column sediments in a nitrogen-filled glove box. A mercury speciation analysis was conducted on the column sediment, and total Hg and MeHg analyses were conducted on both the column sediment and water. For a complete and detailed explanation of the analyses and QA/QC procedures conducted at Frontier Geosciences, see Appendix A.

4.6.5 Results and Discussion

The results of the mercury analyses of the column sediments conducted at Frontier Geosciences are shown in Table 15. The first two columns of data represent the sediment used to fill columns 3, 4, 5, and 6 before column incubation. Columns 3, 5, and 7 were mixed and exposed to air for a week before analysis, while columns 4, 6, and 8 remained sealed until

Table 15. Concentration of Various Mercury Species Within the Saturated Column Sediment, mg/kg Hg

Mercury Species	Column							
	3 and 4 (T = 0)	5 and 6 (T = 0)	3	4	5	6	7	8
Water-Soluble								
Hg	6.507	6.419	8.550	10.541	8.365	5.827	2.934	5.424
HgO	0.0089	0.0049	0.0153	0.0106	0.0314	0.0299	0.0279	0.0257
Organo-Complexed Hg (Hg humics)	1.956	4.285	3.385	4.736	2.724	3.146	5.419	7.690
Strongly Complexed Hg (Hg_2Cl_2; Hg^0)	53.734	66.101	48.673	78.507	43.530	62.745	45.442	116.143
Cinnabar Hg (HgS; HgSe; HgAu)	929.078	782.271	838.999	733.833	832.132	881.544	445.619	523.571
Total Hg 1[1]	991.3	859.1	899.6	827.6	895.8	953.3	499.4	652.9
Total Hg 2[2]	NA[3]	NA	920.4	913.7	949.6	936.7	565.1	736.8
MeHg	0.0043	0.0037	0.1883	0.1502	0.1568	0.1664	0.1781	0.3249
Pore Water Mercury, total	NA	NA	0.0432	0.0068	0.0060	0.0199	0.0276	0.0048

[1] Total Hg 1 represents the total concentration of mercury based on the sum of the individual mercury species.
[2] Total Hg 2 is the concentration of mercury determined by a separate analysis specifically for total mercury.
[3] Not applicable.

analysis. All columns shown in the table were spiked with 1000 mg/kg Hg as $HgCl_2$; however, columns 5 and 6 each contain 1000 mg/kg Fe_2O_3 and MnO_2, and the sediment from columns 7 and 8 were mixed with fresh organic matter from cattail plants immediately before the column incubation. The data for columns 1 and 2 (the control columns) are not reported or discussed, since mercury concentrations in the sediment were insignificant compared to the mercury concentrations in the other columns.

The most obvious characteristic about the data is that the largest fraction of mercury in each sediment column was in the form of mercury sulfide, presumably cinnabar. It appears as though the Hg combined with sulfides in the sediment almost immediately, based on the results of the Hg speciation analysis of the sediment used to fill columns 3, 4, 5, and 6 prior to column incubation.

Concentrations of total Hg and MeHg in the column pore waters (Table 15) were insignificant compared to the overall concentrations of mercury in the system. These data did not exhibit any obvious trends between the columns.

Since the total concentrations of mercury in columns 3 through 6 vary slightly and mercury concentrations in columns 7 and 8 appear much lower than the rest, a separate table was compiled that compares the percentage that each species contributes to the total mercury concentration (Table 16). The reason for the low Hg concentrations in columns 7 and 8 is not known; however, there was a significant buildup in pressure in these two columns during incubation. It is possible that volatile mercury could have escaped from the top or bottom of the columns since the caps were not designed to withhold positive pressures. This could also be supported by the fact that there are relatively high concentrations of the strongly complexed Hg fraction (Hg_2Cl_2 or Hg^o) within columns 7 and 8 (Figure 3). This increase is most likely a result

Table 16. Percent Concentrations of Various Mercury Species Within the Column Sediment

	Column ID							
Mercury Species	3 and 4 (T = 0)	5 and 6 (T = 0)	3	4	5	6	7	8
Water-Soluble Hg	0.656	0.747	0.950	1.273	0.949	0.611	0.587	0.830
HgO	0.001	0.001	0.002	0.001	0.004	0.003	0.006	0.004
Organo-Complexed Hg (Hg humics)	0.197	0.499	0.376	0.572	0.321	0.330	1.085	1.177
Strongly complexed Hg (Hg_2Cl_2; Hg^0)	5.421	7.694	5.409	9.484	4.867	6.581	9.095	17.781
Cinnabar Hg (HgS; HgSe; HgAu)	93.724	91.059	93.242	88.651	93.842	92.458	89.192	80.157
Methylmercury	0.0004	0.0004	0.021	0.018	0.017	0.017	0.036	0.050

of increased microbial activity due to the addition of labile organic matter to the sediment. The stimulation of the microbial activity probably created a more reducing environment, resulting in an increased rate of Hg(II) reduction to Hg^0. The addition of organic matter to columns 7 and 8 also resulted in an increase in the percentage of organically bound mercury within the two columns.

Another interesting trend in the data is the formation of methylmercury in all columns during incubation (Figure 5). The methylation of Hg is mediated by sulfate-reducing bacteria (Gilmour et al., 1998; Benoit et al., 1999). There is no doubt that the sediment columns were conducive to sulfate reduction, given the high sulfate concentrations in the sediment pore water and the highly reducing environment of the sediments. This is also supported by high sulfide concentrations in the sediment, as well as a strong odor of hydrogen sulfide. Generally, the presence of sulfides inhibits methylmercury formation (Jackson, 1993; Gilmour et al., 1998; Benoit et al., 1999); however, given the large concentrations of mercury added to the columns, it is not surprising that a small fraction of the mercury was methylated by the active sulfate-reducing environment. The increased amount of methylmercury in columns 7 and 8 is supported by previous research that has documented increased methylmercury production as a result of stimulation of the microbial population due to addition of labile organic carbon in freshwater environments (Jackson, 1993).

The only overwhelming trend between the columns that were exposed to air for a week (columns 3, 5, and 7) and the columns that remained anoxic until analysis (columns 4, 6, and 8) was in the distribution of strongly complexed Hg species (Figure 6). The columns that were exposed to air contain less of this mercury species than the anoxic columns. If the strongly complexed mercury was in the form of elemental mercury (Hg^0) as discussed previously, then the loss in the exposed sediments may have been a result of volatilization.

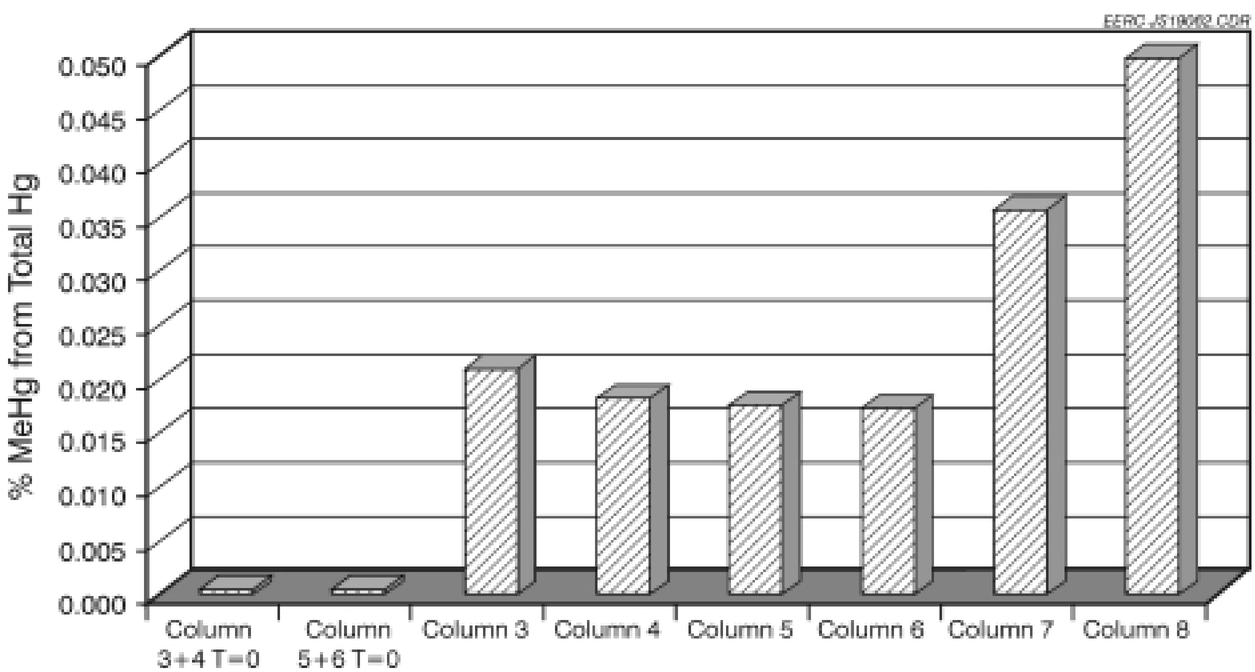

Figure 5. Percentage of methylmercury from total Hg concentrations in each column.

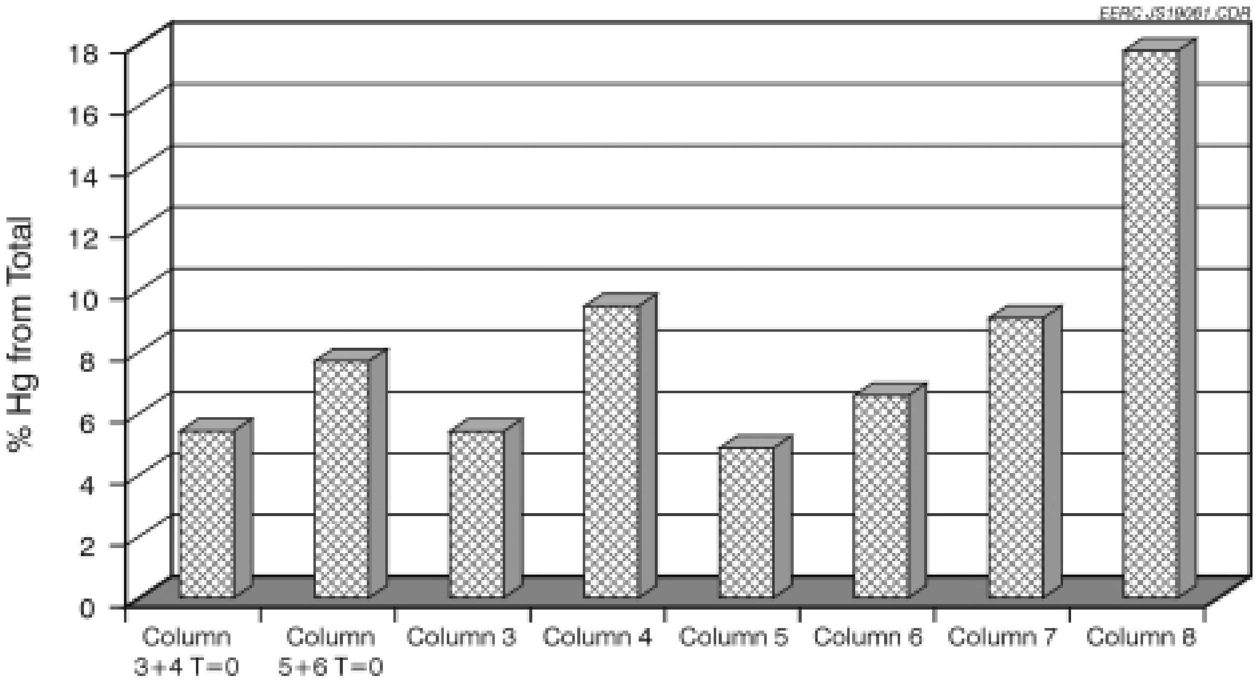

Figure 6. Distribution of strongly complexed mercury in the column sediments.

No trends were seen in the cinnabar mercury (HgS) species between the exposed and unexposed columns. This might suggest that there was not a strong tendency for the sulfide to oxidize; however, even after mixing on a daily basis, the exposed sediments still tended to revert back to anoxic conditions beneath the top few centimeters of sediment and, therefore, were not fully oxidized.

5.0 SUMMARY OF CONCLUSIONS

The data collected during the EERC study on mercury release from disturbed anoxic soils indicate that a sulfide bond in anaerobic conditions of the targeted aquatic environment results in immobilization of mercury. The primary research findings are as follows:

- The background mercury concentration in sediment used for the experiment ranged between 323 to 377 mg/kg on dry weight basis (102–119 mg/kg on as-received basis) with methylmercury ranging between 13 to 21 µg/kg.

- Results of the field bench-scale dredging simulation confirmed mercury releases from sediment as high as 2.8 µg/L in filtered water samples and 40 µg/L in unfiltered, both of which exceed EPA standards. Observed release, however, appears to be only temporary. Binding to organic particles, sulfide and likely iron oxides result in relatively fast capture of released mercury after settling.

- A slight release of mercury to the native water column in both the undisturbed and disturbed experimental setting was documented by column study. Observed mercury enrichment in columns with DI water was over an order of magnitude higher relative to columns filled with native water.

- A minor increase of the mercury concentration in vapors was observed during the volatilization test. The results indicate that some mercury becomes available for volatilization in drier soils; however, all recorded levels and calculated mercury concentrations remain several orders of magnitude below regulatory limits (OSHA PEL) of 0.1 mg/m^3.

- Because of the high organic content in soils used in experiments, over 250% water content reduction was observed over the period of drying. Attendant volume reduction may suggest a cleanup strategy for treatment of these organic-rich soils. Almost 80% mass reduction translates into considerable reduction of costs associated with handling and transport of contaminated soils to a reatment plant or NDPES-approved disposal location.

- The results of resaturation and mixing experiments indicate that mercury concentration considerably drops during early stages of mixing. The primary processes responsible for mercury retention are 1) sediment disturbance resulting in disintegration/dissolution of large particles and subsequent mercury affinity to the fine-particle fraction in suspended sediment, 2) the presence of sulfides, 3) an increased content of TOC in response to disturbance providing for the capture of dissolved mercury on organic particles, 3) decreased pH of the suspension resulting in destruction of metal oxide and metal sulfide bonds allowing mercury to compete for freed bond and replace/exchange other metallic species, and 4) sudden change of redox conditions providing reaction incentives for previously mentioned processes.

- Results of the respirometry experiment using organic-rich wetland sediment spiked with various concentrations of $HgCl_2$ indicate that the sediment with the lowest concentration of Hg produced the highest concentration of CO_2, but all of the Hg-spiked sediments were more biologically active than the controls (0 ppm Hg).

- The results of the incubation experiment indicate that the largest fraction of mercury in each sediment column was in the form of mercury sulfide, presumably cinnabar. It appears as though the Hg combined with sulfides in the sediment almost immediately. Concentrations of total Hg and MeHg in the column pore waters were insignificant compared to the overall concentrations of mercury in the system.

- MeHg was formed in all columns during incubation.

- The columns that were exposed to air contain less strongly complexed Hg species than the anoxic columns. If the strongly complexed mercury was in the form of elemental mercury (Hg^0), then the loss in the exposed sediments may have been a result of volatilization.

- No trends were seen in the cinnabar mercury (HgS) species between the exposed and unexposed columns.

- The stability observed for mercury in undisturbed anoxic soil may represent an opportunity for treating wastewater highly contaminated with mercury and other toxic metals in natural or engineered anoxic ponds (reactors).

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APPENDIX A

ANALYTICAL DATA AND CHAIN-OF-CUSTODY FORMS

A-1

Trace Metals in Soils

Trace Metals in Soils (EERC)												
analyzed by												
Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109												
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com												
		dry	**blank corrected trace metals concentrations, µg/g (ppm) *as received* basis**									
sample	**dilution**	**fraction**	**Cr**	**Mn**	**Fe**	**Ni**	**Cu**	**Zn**	**Cd**	**Hg**	**Pb**
SOIL-1	500x	0.910	1,185	577	26,806	121	430	2,556	21.9	193	338
dry weight basis			1,302	634	29,457	133	473	2,809	24.1	212	371
SOIL-2	500x	0.934	1,193	529	25,549	128	433	2,639	23.4	224	350
dry weight basis			1,277	566	27,355	137	464	2,826	25.1	240	375
COLUMN-4	50x	0.578	15.16	324	3,828	8.97	8.94	40.4	0.235	864	6.60
dry weight basis			26.22	561	6,623	15.52	15.46	69.9	0.407	1,495	11.43
COLUMN-6	50x	0.498	13.88	940	4,871	9.60	8.94	37.1	0.230	937	6.25
dry weight basis			27.87	1,887	9,780	19.28	17.96	74.6	0.462	1,881	12.56
50x digestion blank-1	50x		0.26	0.25	5	0.07	0.02	0.13	-0.05	0.0000	-0.010
50x digestion blank-2	50x		0.19	0.31	-3	0.10	0.01	0.14	-0.02	0.0001	-0.014
50x digestion blank-3	50x		0.23	0.27	3	0.06	0.00	0.10	-0.04	0.0002	-0.013
mean	50x		0.23	0.27	2	0.08	0.01	0.12	-0.03	0.0001	-0.012
SD	50x		0.03	0.03	4	0.03	0.01	0.02	0.02	0.0001	0.002
eMDL	50x		0.10	0.09	12	0.08	0.02	0.05	0.05	0.0002	0.006
500x blank-1	500x		-78	12	3881	6	-10	-399	1.9	0.0	-6.5
500x blank-2	500x		-79	12	3855	5	-10	-399	1.9	0.1	-6.5
500x blank-3	500x		-79	12	3851	5	-10	-399	2.0	0.1	-6.5
mean	500x		-79	12	3863	5	-10	-399	1.9	0.1	-6.5
SD	500x		1	0	16	0	0	0	0.0	0.1	0.0
eMDL	500x		2	0.1	49	1	0.2	0.4	0.1	0.2	0.03
NIST-2710	500x		23.8	9,290	22,445	17.2	2,751	6,524	22.8	32.93	5,341

Trace Metals in Soils (EERC)

analyzed by

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com

sample	dilution	dry fraction	Cr	Mn	Fe	Ni	Cu	Zn	Cd	Hg	Pb
			blank corrected trace metals concentrations, mg/g (ppm) *as received* basis								
certified soil value	500x		39.0	10,100	33,800	14.3	2,950	6,952	21.8	32.6	5,532
% recovery	500x		61.0	92.0	66.4	120.2	93.2	93.8	104.6	101.0	96.6
matrix spike level	500x		258.1	258.1	258.1	258.1	258.1	258.1	258.1	985.2	258.1
SOIL-2 + 258.1 ug/g MS	500x		1,628	773	25,435	392	682	3,029	291	1,184	591
net	500x		spk too low	244	spk too low	264	249	spk too low	267	960	241
% recovery	500x		spk too low	94.6	spk too low	102.2	96.4	spk too low	103.6	97.5	93.3
matrix spike duplicate level	500x		246.6	246.6	246.6	246.6	246.6	246.6	246.6	1,051	246.6
SOIL-2 + 246.6 ug/g MSD	500x		1,469	830	26,973	397	726	3,399	298	1,263	625
net	500x		spk too low	301	spk too low	269	292	spk too low	274	1,039	275
% recovery	500x		spk too low	122.2	spk too low	109.2	118.5	spk too low	111.3	98.9	111.4
SOIL-2 r1	500x		1,148	522	24,949	125	413	2,535	23	244.0	343
SOIL-2 r2	500x		1,237	536	26,149	131	453	2,744	24	203.0	358
average	500x		1,193	529	25,549	128	433	2,639	23	224.0	350
RPD (%)	500x		7.4	2.7	4.7	4.1	9.2	7.9	5.8	18.1	4.4
analytical method			ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	CVAFS	ICP/MS
date analyzed			18-Feb-01	18-Feb-01	18-Feb-01	18-Feb-01	18-Feb-01	########	########	########	18-Feb-01
notes:	"spiked too low" means the chosen spiking level was less than ambient, making assessment of spike recoveries impossible										
	mercury samples were analyzed at 1000x dilution										
	all ICP/MS data were corrected by the method of standard additions on sample SOIL-1										

A-2

Trace Metals in Water

Trace Metals in Water Samples (EERC)

analyzed by

Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109

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		blank corrected trace metals comcentrations, μg/L (ppb)								
sample ID	**dilution**	**Cr**	**Fe**	**Mn**	**Ni**	**Cu**	**Zn**	**Cd**	**Hg**	**Pb**
1M	5x	-0.1	1	0.22	0.16	2.86	5.83	0.01	0.001	2.49
2M	100x	65	4,456	6,301	949	1,030	33,385	231	11.29	110
3M	100x	96	6,450	8,566	1,287	1,484	46,539	314	0.157	135
4M	100x	128	7,943	10,212	1,547	1,774	53,703	366	0.197	149
5M	100x	131	8,772	10,656	1,642	1,898	57,353	383	0.208	152
6M	100x	142	9,945	11,168	1,738	2,030	59,957	407	0.813	154
7M	100x	140	10,524	11,572	1,812	2,078	62,705	420	0.236	152
8M	100x	156	11,489	11,861	1,874	2,160	65,010	444	0.260	147
9M	100x	167	11,576	11,839	1,865	2,171	64,911	444	0.290	145
10M	100x	164	12,445	12,193	1,928	2,227	66,358	453	0.260	143
11M	100x	167	12,899	12,251	1,939	2,253	66,822	458	0.573	137
12M	100x	181	14,726	12,686	2,095	2,375	70,541	489	0.296	131
13M	1000x	61,291	1,842,409	30,712	6,208	21,429	131,875	953	8,795	16,768
14M	100x	180	14,746	12,771	2,034	2,391	69,526	473	0.175	128
15M	100x	181	14,416	12,733	2,070	2,408	71,371	487	0.167	128
COLUMN-4 porewater	5x	10.4	59	2,252	5.5	1.27	3.49	0.02	6.82	0.13
COLUMN-6 porewater	5x	11.2	233	13,104	10.1	1.28	2.25	0.00	19.87	0.16
COLUMN-8 porewater	5x	17.5	9,858	5,891	17.8	1.39	3.77	0.00	4.87	0.33
4M rep 1	100x	131	7,872	10,249	1,557	1,773	53,823	369	0.197	149
4M rep 2	100x	126	8,014	10,175	1,537	1,774	53,583	362	*nd*	148
mean	100x	128	7,943	10,212	1,547	1,774	53,703	366	*nd*	149
RPD (%)	100x	4.0	1.8	0.7	1.3	0.0	0.4	2.0	*nd*	1.0

Trace Metals in Water Samples (EERC)										
analyzed by										
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phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com										
		blank corrected trace metals comcentrations, μg/L (ppb)								
sample ID	**dilution**	**Cr**	**Fe**	**Mn**	**Ni**	**Cu**	**Zn**	**Cd**	**Hg**	**Pb**
spike level		51.5	103	103	103	103	103	103	0.673	103
4M matrix spike	100x	188.0	8,164	10,435	1,660	1,922	55,006	470.3	0.924	251.3
net	100x	59.8	spk too low	spk too low	spk too low	spk too low	spk too low	104.6	0.727	102.8
% recovery	100x	116.0	spk too low	spk too low	spk too low	spk too low	spk too low	101.5	108.0	99.8
4M + matrix spike dup	100x	197.4	8,274	10,320	1,689	1,907	54,915	470.4	0.924	252.7
net	100x	69.2	spk too low	spk too low	spk too low	spk too low	spk too low	104.7	0.727	104.1
% recovery	100x	134.3	spk too low	spk too low	spk too low	spk too low	spk too low	101.6	108.0	101.1
mean result	100x	192.7	8,219	10,378	1,674	1,915	54,961	470.4	0.924	252.0
RPD (%)	100x	4.9	1.3	1.1	1.8	0.8	0.2	0.0	0.0	0.5
blank-1	100x	-405	1250	212	-58	26	-48	-36	*nd*	4
blank-2	100x	-406	950	212	-58	25	-46	-36	*nd*	4
blank-3	100x	-406	1064	212	-58	25	-47	-36	*nd*	4
mean	100x	-406	1088	212	-58	25	-47	-36	*nd*	4
SD	100x	0.9	152	0.3	0.2	0.1	0.8	0.2	*nd*	0.0
eMDL	100x	2.6	455	0.8	0.7	0.3	2.3	0.7	*nd*	0.0
Blank-1	5x	-2.7	-21	1.64	-0.06	0.82	1.08	-0.17	0.0002	0.19
Blank-2	5x	-3.0	-26	1.65	-0.01	0.79	0.98	-0.16	0.0001	0.21
Blank-3	5x	-3.0	-24	1.71	-0.06	0.66	1.09	-0.17	0.0001	0.25
mean	5x	-2.9	-23	1.67	-0.04	0.76	1.05	-0.16	0.0001	0.22

Trace Metals in Water Samples (EERC)

analyzed by

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		blank corrected trace metals comcentrations, mg/L (ppb)								
sample ID	**dilution**	**Cr**	**Fe**	**Mn**	**Ni**	**Cu**	**Zn**	**Cd**	**Hg**	**Pb**
SD	5x	0.1	3	0.04	0.03	0.08	0.06	0.00	0.0001	0.03
eMDL	5x	0.4	8	0.12	0.08	0.25	0.17	0.01	0.0001	0.09
NIST-1643d rep 1	5x	20.41	93.76	43.94	61.65	23.55	82.07	6.28	1,539	17.91
NIST-1643d rep 2	5x	20.26	91.61	43.32	62.67	23.53	81.05	6.35	1,453	17.92
mean	5x	20.34	92.68	43.63	62.16	23.54	81.56	6.32	1,496	17.92
RPD (%)	5x	0.7	2.3	1.4	1.6	0.1	1.2	1.1	5.6	0.1
certified aqueous value	5x	18.53	91.2	37.66	58.1	20.5	72.48	6.47	1,590	18.15
% recovery	5x	109.7	101.6	115.9	107.0	114.8	112.5	97.6	94.1	98.7
method		ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	ICP/MS	CVAFS	ICP/MS
date analyzed		18-Feb-01	18-Feb-01	18-Feb-01	18-Feb-01	18-Feb-01	18-Feb-01	18-Feb-01	11-Feb-01	18-Feb-01
notes:	all chromium results except 13M are reported from 5x dilution									
	sample 13M contained large amounts of solids									
	mercury samples were analyzed without previous dilution									
	the CRM for mercury is NIST-1641d									
	all ICP/MS results are corrected by the method of matrix spike additions									
	"spiked too low" means he chosen spiking level was less than ambient, making calculation of recoveries impossible.									
	"nd" means not determined									

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Mercury Speciation in Water

Mercury Speciation in Water Samples (EERC)

analyzed by

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	Hg concentrations, mg/L		percent	
sample ID	total	methyl	methyl	comments
EERC-1	0.001			
EERC-2	11.29			
EERC-3	0.157			
EERC-4	0.197			QC sample
EERC-5	0.208			
EERC-6	0.813			
EERC-7	0.236			
EERC-8	0.260			
EERC-9	0.290			
EERC-10	0.260			
EERC-11	0.573			
EERC-12	0.296			
EERC-13	8,795			
EERC-14	0.175			
EERC-15	0.167			
porewater C4	6.82	0.0635	0.93	QC sample
porewater C6	19.87	0.1312	0.66	QC sample
porewater C8	4.87	0.3027	6.21	QC sample
2 Aq 0x A	0.002			
3 Aq 0x A1	11.30	0.0334	0.30	
3 Aq 0x A2	75.08			
5 Aq 0x A1	7.31	0.0093	0.13	QC sample
5 Aq 0x A2	4.73			
7 Aq 0x A1	27.62	0.0702	0.25	

Mercury Speciation in Water Samples (EERC)

analyzed by

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	Hg concentrations, mg/L		**percent**	
sample ID	**total**	**methyl**	**methyl**	**comments**
porewater C4 rep 1	6.98			
porewater C4 rep 2	6.67			
mean	6.82			
RPD (%)	4.5			
porewater C6 rep 1	20.13			
porewater C6 rep 2	19.61			
mean	19.87			
RPD (%)	2.6			
porewater C8 rep 1	4.62			
porewater C8 rep 2	5.13			
mean	4.87			
RPD (%)	10.4			
5AQ 0X A1 rep 1		0.0091		
5AQ 0X A1 rep 2		0.0093		
mean		0.0092		
RPD (%)		1.1		

Mercury Speciation in Water Samples (EERC)

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sample ID	**Hg concentrations, mg/L** **total**	**methyl**	**percent methyl**	**comments**
spiked sample	EERC-4	column-4		
spiking level	0.673	1.000		
sample + MS	0.924	1.160		
% recovery	108.0	109.6		
sample + MSD	0.924	1.126		
% recovery	108.0	109.8		
mean	0.924	1.143		
RPD (%)	0.0	0.2		
blank-1	0.00016	0.00012		
blank-2	0.00007	0.00003		
blank-3	0.00014	-0.00003		
mean	0.00012	0.00004		
SD	0.00005	0.00008		
estimated MDL	0.00014	0.00023		
reference material	NIST-1641d	DORM-2		DORM-2 is a digested dogfish tissue
CRM rep 1	1,539	4,972		
CRM rep 2	1,453			
mean	1,496			
RPD (%)	5.6			
certified value	1,590	4,470		
% recovery	94.1	111.2		

Mercury Speciation in Water Samples (EERC)				
analyzed by				
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phone: 206-62-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com				
	Hg concentrations, mg/L		**percent**	
sample ID	**total**	**methyl**	**methyl**	**comments**
analytical dates	11-Feb-01	23-Feb-01		
	12-Feb-01			

A-4

Mercury Speciation in Sediments and Mercury Speciation by Selective Sequential Extractions

Mercury Speciation in EERC Sediments

analyzed by

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	dry	percent	Hg concentration, mg/g		percent	
sample ID	fraction	LOI	total	methyl	methyl	comment
EERC soil-1	0.910		192.6			
dry weight basis			211.6			
EERC soil-2	0.934		223.6			
dry weight basis			239.4			
2S 0x A	0.511		0.0175			
dry weight basis			0.0342			
3S 0x A1	0.475	10.58	899.6	0.1893	0.0210	CH_3Hg from refrigerated (1 week) SSE samples
dry weight basis			1,894	0.3985		total Hg from sum of SSE speciation
3S 0x B1	0.524		902.2	0.1444	0.0160	CH_3Hg sample frozen until extraction
dry weight basis			1,722	0.2756		
3S 0x B2	0.497		959.3			
dry weight basis			1,930			
5S 0x A1	0.474	9.79	818.9	0.1429	0.0175	CH_3Hg from refrigerated (1 week) SSE samples
dry weight basis			1,728	0.3015		total Hg from sum of SSE speciation
5S 0x A2	0.445	10.79	972.6	0.1727	0.0178	CH_3Hg from refrigerated (1 week) SSE samples
dry weight basis			2,186	0.3881		total Hg from sum of SSE speciation
5S 0x B1	0.455		949.6	0.1808	0.0190	CH_3Hg sample frozen until extraction
dry weight basis			2,087	0.3974		

Mercury Speciation in EERC Sediments

analyzed by

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sample ID	dry fraction	percent LOI	Hg concentration, mg/g total	methyl	percent methyl	comment
7S 0x A1	0.355	13.01	499.4	0.1791	0.0359	CH_3Hg from refrigerated (1 week) SSE samples
dry weight basis			1,407	0.5045		total Hg from sum of SSE speciation
7S 0x B1	0.400		589.1	0.1846	0.0313	CH_3Hg sample frozen until extraction
dry weight basis			1,473	0.4615		total Hg from sum of SSE speciation
7S 0x B2	0.427		606.8			
dry weight basis			1,421			
Column 4	0.578	10.60	864.1	0.1503	0.0174	CH_3Hg sample frozen until extraction
dry weight basis			1,495	0.2600		
Column 6	0.489	12.26	936.7	0.1644	0.0176	CH_3Hg sample frozen until extraction
dry weight basis			1,916	0.3362		
Column 8	0.508	12.00	736.8	0.3249	0.0441	CH_3Hg sample frozen until extraction
dry weight basis			1,450	0.6396		
Column 3+4 T=0	0.511	10.84	991.3	0.0053	0.0005	CH_3Hg from refrigerated (1 week) SSE samples
dry weight basis			1,939	0.0103		total Hg from sum of SSE speciation
Column 5+6 T=0	0.476	11.32	859.1	0.0047	0.0005	CH_3Hg from refrigerated (1 week) SSE samples
dry weight basis			1,797	0.0098		total Hg from sum of SSE speciation
blank-1			0.0000	-0.000007		
blank-2			0.0001	-0.000009		
blank-3			0.0002	-0.000006		
mean			0.0001	-0.000007		
estimated MDL			0.0002	0.000006		

Mercury Speciation in EERC Sediments

analyzed by

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sample ID	dry fraction	percent LOI	Hg concentration, mg/g total	Hg concentration, mg/g methyl	percent methyl	comment
NIST-2710			32.93			NIST certified soil sample
certified			32.60			101.0% recovery
EERC Soil 2 rep 1			243.8			
EERC Soil 2 rep 2			203.4			
RPD (%)			18.1			
MS spike level			985	1.390		spiked samples: soil-2 for total, and
EERC Soil + MS			1,184	1.398		column-4 for methyl
% recovery			97.5	89.7		
MSD spike level			1,051	1.379		
EERC Soil + MSD			1,263	1.509		
% recovery			98.9	98.3		
RPD (%)			1.4	9.1		
dates analyzed	19-Feb-01	24-Feb-01	14-Feb-01	21-Feb-01		

Mercury Speciation by Sequential Selective Extractions (EERC)									
analyzed by									
Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109									
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com									
	dry			**mercury concentrations, ng/g (ppb)**					
sample ID	**fraction**	**F1**	**F2**	**F3**	**F4**	**F5**	**sum**	**methyl**	**comment**
3S 0x A1	0.475	8,550	15.3	3,385	48,673	838,999	899,622	188.3	methyl Hg on unfrozen samples
dry weight basis		18,000	32.2	7,126	102,469	1,766,314	1,893,942	396.4	
% in fraction		0.95	0.002	0.38	5.41	93.3	100.0	0.021	
5S 0x A1	0.474	9,275	30.2	4,249	40,653	764,716	818,923	141.9	methyl Hg on unfrozen samples
dry weight basis		19,568	63.7	8,964	85,766	1,613,325	1,727,686	299.4	
% in fraction		1.13	0.004	0.52	4.96	93.4	100.0	0.017	
5S 0x A2	0.445	7,455	32.6	1,198	46,407	917,549	972,642	171.7	methyl Hg on unfrozen samples
dry weight basis		16,753	73.3	2,692	104,285	2,061,908	2,185,711	385.8	
% in fraction		0.77	0.003	0.12	4.77	94.3	100.0	0.018	
7S 0x A1	0.355	2,934	27.9	5,419	45,442	445,619	499,442	178.1	
dry weight basis		8,265	78.6	15,265	128,006	1,255,265	1,406,879	501.7	
% in fraction		0.59	0.006	1.08	9.10	89.2	100.0	0.036	
Column 4	0.558	10,541	10.6	4,736	78,507	733,833	827,628	150.2	
dry weight basis		18,891	19.0	8,487	140,694	1,315,113	1,483,204	269.2	
% in fraction		1.27	0.001	0.57	9.49	88.7	100.0	0.018	
Column 6	0.484	5,827	29.9	3,146	62,745	881,544	953,292	164.4	
dry weight basis		12,039	61.8	6,500	129,638	1,821,372	1,969,611	339.7	
% in fraction		0.61	0.003	0.33	6.58	92.5	100.0	0.017	
Column 8	0.503	5,424	25.7	7,690	116,143	523,571	652,854	324.9	
dry weight basis		10,783	51.1	15,288	230,901	1,040,897	1,297,920	645.9	
% in fraction		0.83	0.004	1.18	17.8	80.2	100.0	0.050	
Column 3+4 T=0	0.511	6,507	8.9	1,956	53,734	929,078	991,284	4.27	methyl Hg on unfrozen samples
dry weight basis		12,734	17.4	3,828	105,155	1,818,157	1,939,890	8.36	
% in fraction		0.66	0.001	0.20	5.42	93.7	100.0	0.0004	
Column 5+6 T=0	0.478	6,419	4.9	4,285	66,101	782,271	859,081	3.65	methyl Hg on unfrozen samples
dry weight basis		13,429	10.3	8,964	138,287	1,636,550	1,797,240	7.64	
% in fraction		0.75	0.001	0.50	7.69	91.1	100.0	0.0004	

Mercury Speciation by Sequential Selective Extractions (EERC)

analyzed by

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sample ID	dry fraction	F1	F2	F3	F4	F5	sum	methyl	comment
		mercury concentrations, ng/g (ppb)							
Cosumnes R. @ Moke	0.534	-0.6	0.3	71.4	8.4	4.2	84	0.887	
dry weight basis		-1.2	0.5	133.7	15.8	7.9	157	1.661	
% in fraction		-0.77	0.32	85.4	10.1	5.02	100.0	1.061	
blank-1		0.1	0.66	0.67	1.6	0.15		-0.007	
blank-2		0.9	0.36	0.48	1.0	0.08		-0.009	
blank-3		-0.1	0.30	0.57	0.6	0.05		-0.006	
mean		0.3	0.44	0.57	1.1	0.09		-0.007	
SD		0.5	0.19	0.10	0.5	0.05		0.002	
eMDL		1.6	0.58	0.29	1.5	0.15		0.005	
HgS + kaolin		344	232	19	44,346	2,916,175	2,961,116		
$HgCl_2$ + kaolin		2,415,242	24,020	1,684	10,865	250	2,452,061		
NIST-2710		122	21	547	14,005	12,617	27,312		
Column 4 rep 1	0.578	12,396	6.0	5,042	63,688	724,626	805,758		
Column 4 rep 2	0.537	8,686	15.2	4,429	93,326	743,139	849,595		
mean	0.558	10,541	10.6	4,736	78,507	733,883	827,677		
RPD (%)	7.4	35.2	86.1	12.9	37.8	2.5	5.3		
Column 6 rep 1	0.489	5,098	35.9	2,250	68,718	912,509	988,611		
Column 6 rep 2	0.478	6,556	23.9	4,041	56,722	850,578	917,921		
mean	0.484	5,827	29.9	3,146	62,745	881,544	953,291		
RPD (%)	2.3	25.0	40.2	56.9	19.0	7.0	7.4		
Column 8 rep 1	0.508	5,384	33.7	8,824	135,325	565,816	715,383		
Column 8 rep 2	0.498	5,463	17.6	6,556	99,961	481,686	593,684		
mean	0.503	5,424	25.7	7,690	116,143	523,751	653,034		
RPD (%)	2.0	1.4	62.6	29.5	33.0	16.1	18.6		
Cosumnes R. @ Moke r1								1.057	
Cosumnes R. @ Moke r2								0.717	
mean								0.887	
RPD (%)								38.3	

Mercury Speciation by Sequential Selective Extractions (EERC)									
analyzed by									
Frontier Geosciences Inc. 414 Pontius North, Suite B Seattle, WA 98109									
phone: 206-622-6960 fax: 206-622-6870 e-mail: nicolasb@frontier.wa.com									
	dry	**mercury concentrations, ng/g (ppb)**							
sample ID	**fraction**	**F1**	**F2**	**F3**	**F4**	**F5**	**sum**	**methyl**	**comment**
spiked sample		7S-0X-A1	7S-0X-A1	7S-0X-A1	7S-0X-A1	7S-0X-A1		column 4	
MS level		6,105	30.5	12,210	61,050	976,800		1,389	
sample + MS		8,651	56.3	18,139	109,534	1,517,415		1,397	
% recovery		93.6	93.1	104.2	104.9	109.6		89.7	
MSD level		6,105	30.5	12,210	61,050	976,800		1,379	
sample + MSD		8,782	57.5	18,217	110,003	1,523,048		1,506	
% recovery		95.8	97.0	104.8	105.8	110.2		98.3	
RPD (%)		2.2	3.9	0.7	0.4	0.6		9.1	
date analyzed	23-Feb-01	20-Feb-01	21-Feb-01	22-Feb-01	23-Feb-01	26-Feb-01		21-Feb-01	21-Feb-01

A-5

Mercury Analyses Pertaining to Experiments 1–5

FRONTIER GEOSCIENCES INC.
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SENT VIA FACSIMILE AND U.S. MAIL

October 25, 1999

Mr. Jarda Solc
EERC-UND
15 N 23rd Street
Grand Forks, ND 58203

RE: **Water and Soils Results**

Dear Mr. Solc,

Attached please find results for the iodated carbon traps and water samples submitted to Frontier Geosciences on September 14, 1999. Briefly, all of the associated quality control analyses were within acceptable limits and looked very good.

IC Traps

All of the associated quality control analyses looked very good. Analysis of two "B" traps (not reported) indicated that there was no breakthrough for these samples.

Waters

All of the associated QA/QC analyses looked good. There were no analytical issues of note.

Please call or e-mail me (jamesk@frontier.wa.com) if you have any questions or concerns.

Sincerely,

James Keithly
cc: Ralph Turner
project files

Trace Metals Results for EERC - Water Samples

Reported October 25, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (µg/L)	ERCW 19	ERCW 20	ERCW 21
Hg	0.154	0.157	0.118

NR= Not reported; these analytes were not requested.

ND = not detected

Trace Metals Results for EERC - Water Samples

Reported October 25, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Matrix Spike / Matrix Spike Duplicate Report

Analyte (µg/L)	Sample QC'd	Sample Value	Spike Level	MS	% Rec.	MSD	% Rec.	RPD
Hg	another client	0.005	0.010	0.015	96.6	0.014	87.8	6.2

NR= Not reported

Quality Control Data - Preparation Blank Report

Analyte (µg/L)	PBW1	PBW2	PBW3	Mean	Std Dev	Est. MDL
Hg	0.00006	0.00007	0.00007	0.00007	0.00001	0.0005

Est. MDL = Estimated method detection limit

Std Dev = Standard deviation

NR= Not reportable

Quality Control Data - Standard Reference Material Report

Analyte (µg/L)	SRM Identity	Cert. Value	Obs. Value	% Rec.
Hg	DORM-2	4.640	4.445	95.8

SRM Identity = Standard reference material identity

Cert. Value = Certified value

Obs. Value = Experimental result

% Rec. = Percent recovery

Total Mercury Results for EERC - Iodated Carbon Traps

Reported October 25, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (ng/trap)	ERCA-4	ERCA-5	ERCA-6	ERCA-7
Hg	18.6	12.7	95.9	11.3

Analyte (ng/trap)	ERCA-8	ERCA-9	ERCA-10
Hg	40.2	21.2	43.7

Total Mercury Results for EERC - Iodated Carbon Traps

Reported October 25, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Duplicate Report

Analyte (ng/trap)	Sample QC'd	Rep. 1	Rep. 2	Mean	RPD
Hg	another client	10.7	11.4	11.1	6.1

Quality Control Data - Matrix Spike Report

Analyte (ng/trap)	Sample QC'd	Mean	Spike Level	MS	% Rec.
Hg	another client	11.1	40.0	51.0	99.8

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414 Pontius North · Seattle, WA 98109

October 25, 1999

Jarda Solc
EERC-UND
15 N 23rd St
Grand Forks, ND 58202

Re: Hg Release

Dear Jarda:

Enclosed are the analytical results for the water sample (4) received from you on September 23, 1999. The samples arrived in good shape and were immediately treated to 2% bromine monochloride and allowed to oxidize overnight prior to analysis. The samples were analyzed using EPA Method 1631. No analytical difficulties were encountered and all QA indices were with acceptable limits.

If you have any questions or concerns about the data or this report please do not hesitate to call me.

Best regards,

Ralph Turner

Ralph Turner

Total Hg in Water Samples

EERC-UND

Hg Release

Samples Received 9/23/99

Bottle #	Sample ID	Date Collected	Total Hg (UF) ng/L	Notes
CENT-891	ERCW 29	9/22/99	3189	
NIC-109	ERCW 30	9/22/99	344	
TS-28	ERCW 31	9/22/99	43.4	
CENT-600	ERCW 32	9/22/99	36.3	

Quality Assurance Summary

EERC-UND

Hg Release

Samples Received 9/23/99

Method Blanks (2% BrCl)				
	Rep 1		0.10	
	Rep 2		0.10	
	Rep 3		0.15	
	mean		0.12	
	SD		0.03	
	Est MDL		**0.09**	
Matrix Duplicates (MD)				
Matrix Spikes (MS/MSD)				
	FGS-B-624 (MS)		12.6	Not EERC
	Spike		10.1	
	Net		9.1	
	% Recovery		**90.1**	
	FGS-B-624 (MSD)		12.4	Not EERC
	Spike		10.1	
	Net		8.86	
	% Recovery		**87.7**	**1.9%RPD**
SRMs				
	DORM-2		4432	
	certified value		4640 ± 260	
	% Recovery		**95.5**	
Datasets			THG81-991014	

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SENT VIA FACSIMILE AND U.S. MAIL

August 19, 1999

Mr. Jarda Solc
EERC-UND
15 N 23rd Street
Grand Forks, ND 58203

RE: Water, Soil and Iodated Carbon Trap Results

Dear Mr. Solc,

Attached please find results for the water, soil and iodated carbon trap samples submitted to Frontier Geosciences on June 30, 1999. Briefly, all of the associated quality control analyses were within acceptable limits and looked very good. Specific instances are discussed by matrix below.

Water

All of the associated QA/QC analyses looked good. There were no analytical issues of note.

Soil

All of the associated quality control analyses looked very good. The matrix spike results for iron were not within control limits, but this is because the spike added was much smaller than the concentration present in the sample.

Iodated Carbon Traps

All of the associated QA/QC looks very good. Because results for the "B" trap analyzed are greater than the "A" trap results it appears likely that the flow direction was reversed for this sample.

Please call or e-mail me (jamesk@frontier.wa.com) if you have any questions or concerns.

Sincerely,

James Keithly

cc: Ralph Turner

Trace Metals Results for EERC - Water Samples

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (µg/L)	ERCW 10	ERCW 12	ERCW 17	ERCW 23
Mn	2210	1550	NR	5860
Fe	ND	ND	NR	4100
Hg	0.0225	0.0534	9.19	1.89
MeHg	0.000032	0.000049	NR	NR
TOC	17000	18000	NR	15000

Analyte (µg/L)	ERCW 24	ERCW 25	ERCW 26	ERCW 27
Mn	7060	8730	23800	9120
Fe	5230	5820	929000	5920
Hg	1.05	0.896	5370	0.0971
MeHg	NR	NR	NR	NR
TOC	19000	24000	89000	25000

Analyte (µg/L)	ERCW MB
Mn	NR
Fe	NR
Hg	0.0008
MeHg	NR
TOC	NR

NR= Not reported; these analytes were not requested.

ND = not detected

Trace Metals Results for EERC - Water Samples

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Duplicate Report

Analyte (µg/L)	Sample QC'd	Rep. 1	Rep. 2	Mean	RPD
Mn	ERCW-12	1545	1551	1548	0.4
Fe	ERCW-12	ND	ND	ND	N/C
Hg	ERCW-17	9.191	9.926	9.558	7.7
MeHg	LVW129	0.001066	0.001138	0.001102	6.5
TOC	ERCW-26/1	8900	9000	8950	1.1

N/C = Not calculated.

ND = Not detected.

Quality Control Data - Matrix Spike / Matrix Spike Duplicate Report

Analyte (µg/L)	Sample QC'd	Sample Mean	Spike Level	MS	% Rec.	MSD	% Rec.	RPD
Mn	ERCW-12	1548	1000	1732	18.4	1672	12.4	3.5
Fe	ERCW-12	ND	2000	1965	90.9	1901	87.7	3.3
Hg	ERCW-23	1.888	5.051	7.260	106.4	6.991	101.0	3.8
MeHg	LVW193	0.001102	0.002000	0.004759	182.9	0.004563	173.1	4.2
TOC	NR	NR	NR	NR	N/C	NR	N/C	NR

NR= Not reported

Trace Metals Results for EERC - Water Samples

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Preparation Blank Report

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	Std Dev	Est. MDL
Mn	0.1	0.1	0.1	0.1	0.1	0.01	0.02
Fe	-0.5	0.0	-0.1	-1.0	-0.4	0.5	1.4
Hg	0.0005	0.0002	0.0003	N/AV	0.0003	0.0002	0.0005
MeHg	0.0000003	0.0000002	0.0000006	N/AV	0.0000004	0.0000002	0.000001
TOC	ND	ND	NR	NR	NR	NR	1500

Est. MDL = Estimated method detection limit

Std Dev = Standard deviation

NR= Not reportable

Quality Control Data - Standard Reference Material Report

Analyte (µg/L)	SRM Identit	Cert. Value	Obs. Value	% Rec.	Obs. Value	% Rec.
Mn	NIST 1643d	37.7	38.0	101.0	38.4	102.1
Fe	NIST 1643d	91.2	104.3	114.4	105.8	116.0
Hg	DORM-2	4640	4366	94.1	N/AV	N/AV
MeHg	DORM-2	4470	3926	87.8	N/AV	N/AV
TOC	SPEX #16-104	20.0	19.1	95.5	20.6	103.0

SRM Identity = Standard reference material identity

Cert. Value = Certified value

Obs. Value = Experimental result

% Rec. = Percent recovery

Trace Metals Results for EERC

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (mg/Kg)	ERC-4 AR	ERC-4-DW
Total Solid	93.2%	NR
LOI	22.7%	NR
Mn	750	805
Fe	44300	47500
Hg	235	252
MeHg	0.049	0.053

AR= As received basis

DW= Dry weight basis

NR= Not reported

Trace Metals Results for EERC

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Duplicate Report

Analyte (mg/Kg)	Sample QC'd	Rep. 1	Rep. 2	Mean	RPD
Mn	another client	568	596	582	4.9
Fe	another client	18000	19510	18750	8.1
Hg	another client	6.506	5.883	6.195	10.1
MeHg	another client	0.953	0.867	0.910	9.5

*All results for Fe and Mn reported on a Dry Weight basis.

Quality Control Data - Matrix Spike / Matrix Spike Duplicate Report

Analyte (mg/Kg)	Sample QC'd	Mean	Spike Level	MS	% Rec.	Spike Level	MSD	% Rec.	RPD
Mn	another client	582	196	652	35.7	219	779	90	17.7
Fe*	another client	18750	392	21340	660.7	438	24440	1299	13.5
Hg	another client	6.195	12.84	19.08	100.4	12.84	19.64	105	2.9
MeHg	another client	0.910	24.60	28.51	112.2	24.60	28.94	114	1.5

* The spike added is much smaller than the concentration in the sample. Therefore, these results are not considered significant.

Trace Metals Results for EERC

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Preparation Blank Report

Analyte (mg/Kg)	PBS1	PBS2	PBS3	PBS4	Mean	Std Dev	Est. MDL
Mn	0.20	0.28	0.30	0.29	0.26	0.05	0.14
Fe	20	35	40	39	33	9	10
Hg	0.00000	0.00002	0.00004	NR	0.00002	0.00002	0.00005
MeHg	0.000006	0.000005	0.000006	NR	0.000006	0.000001	0.000002

Est. MDL = Estimated method detection limit

Std Dev = Standard deviation

NR= Not reported

Quality Control Data - Standard Reference Material Report

Analyte (mg/Kg)	SRM	Cert. Value	Obs. Value	% Rec.	SRM	Cert. Value	Obs. Value	% Rec.
Mn	NIST 2709	538	505	94	NIST 2710	10100	6434	63.7
Fe	NIST 2709	35000	21420	61	NIST 2710	33800	21650	64.1
Hg	DORM 2	4.640	3.955	85	NIST2709	1.400	1.45	103.6
MeHg	DORM 2	4.470	5.260	118	IAEA-356	0.00546	0.00707	129.5

SRM Identity = Standard reference material identity

Cert. Value = Certified value

Obs. Value = Experimental result

% Rec. = Percent recovery

N/C = not calculated

Total Mercury Results for EERC - Iodated Carbon Traps

ReportedAugust 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (ng/trap)	IC-A-ERCA2	IC-A-ERCA3	IC-B-ERCA3
Hg	42.0	0.10	8.29

* It appears that airflow for this trap was reversed. Please see case narrative.

Total Mercury Results for EERC - Iodated Carbon Traps

Reported August 19, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Duplicate Report

Analyte (ng/trap)	Sample QC'd	Rep. 1	Rep. 2	Mean	RPD
Hg	another client	104	96	100	8.0

Quality Control Data - Matrix Spike Report

Analyte (ng/trap)	Sample QC'd	Mean	Spike Level	MS	% Rec.
Hg	another client	104	400	437	83.3

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Jarda Polc
EERC – UND
15 N. 23rd Street
Grand Forks, ND 58203
(701) 777-5217
Fax (701) 777-5181

June 25, 1999

Dear Mr. Polc,

Enclosed please find the report for the samples submitted to our laboratory on May 21, 1999. I apologize for the lateness of this report. As you know, your Project Manager, Ralph Turner, is currently in the field. Also, your back-up Project Manager, James Kiethly, is on vacation, therefore there was some delay in getting this report out. I have prepared a summary report with all of the sample results and table with the quality control data summaries. Dr. Turner may wish to follow this report up with a more detailed interpretation of your results.

The methods used for the analysis of your samples and the dates of analysis are summarized in the table below:

Analyte	Matrix	Preparation Method	Analysis Method	Date of Analysis
THg	IC Trap	HNO_3/H_2SO_4 Dig.	CV-AFS (1631 mod.)	6/4/99
Fe	Sediment	HNO_3/HF Bomb	ICP-MS (1638 mod.)	6/14/99
Mn	Sediment	HNO_3/HF Bomb	ICP-MS (1638 mod.)	6/14/99
THg	Sediment	Cold Aqua Regia	CV-AFS (1631 mod.)	6/22/99
MeHg	Sediment	Me_2Cl_2 Extraction	CV-AFS (1630 mod.)	6/10/99
%TS	Sediment	N/A	Grav. (160.3 mod.)	6/3/99
%LOI	Sediment	N/A	Grav. (160.4 mod.)	6/3/99
Fe	Waters	1% HNO_3, Oven	ICP-MS (1638 mod.)	6/9/99
Mn	Waters	1% HNO_3, Oven	ICP-MS (1638 mod.)	6/2/99
THg	Waters	1% BrCl	CV-AFS (1631 mod.)	6/21/99
MeHg	Waters	Distillation	CV-AFS (1630 mod.)	6/11/99
TOC	Waters	N/A	EPA Method 415.1	5/28/99

There were no analytical issues with the analysis of your samples, and all of the quality control samples were within the acceptable control limits.

Please feel free to give me a call if you have any questions.

Sincerely,

Michelle L. Gauthier
Laboratory Manager

Sample Results for EERC (Jarda Polc)			
Reported by Frontier Geosciences (Michelle Gauthier)			
June 25, 1999			
Analyte (Units)	**ERCA-1A**	**Analyte (Units)**	**ERCA-1B**
THg (ng)	7.90	THg (ng)	1.31
Analyte (Units)	**ERCW-9**	**Analyte (Units)**	**ERC-5**
Fe (µg/L)	51.9	Fe (mg/kg DB)	20,700
Mn (µg/L)	1570	Mn (mg/kg DB)	660
THg (ng/L)	37.7	THg (ng/g DB)	44.9
TOC (mg/L)	13	MeHg (ng/g DB)	0.902
		Total Solids (%)	53.7
Analyte (Units)	**ERCW-11**	Loss on Ignition (%)	14.8
Fe (µg/L)	81.0		
Mn (µg/L)	2480	**Analyte (Units)**	**ERCW-18**
THg (ng/L)	64.4	Fe (µg/L)	211
MeHg (ng/L)	0.014	Mn (µg/L)	1925
TOC (mg/L)	16	THg (ng/L)	1.90
		MeHg (ng/L)	0.086
Analyte (Units)	**ERCW-16**	TOC (mg/L)	16
THg (ng/L)	5520		

QC Summary Report for EERC (Jarda Polc)

Reported by Frontier Geosciences (Michelle Gauthier)

June 25, 1999

Sediment

Method Blanks	Fe (mg/kg)	Mn (mg/kg)	THg (ng/g)	MeHg (ng/g)		
Mean	-1	0.02	0.11	0.002		
Std. Dev.	22	0.09	0.11	0.001		
Est. MDL	66	0.26	0.35	0.003		
n	4	4	3	3		
SRM	**Fe (mg/kg)**	**Mn (mg/kg)**	**THg (ng/g)**	**MeHg (ng/g)**		
Source	NIST 2710	NIST 2710	NIST 2709	IAEA 356		
% Rec.	77.3	87.9	90.0	99.4		
Matrix QC	**Fe (mg/kg DB)**	**Mn (mg/kg DB)**	**THg (ng/g DB)**	**MeHg (ng/g DB)**	**TS (%)**	**LOI (%)**
Sample ID	ERC-5	ERC-5	ERC-5	Different Client	ERC-5	ERC-5
RPD	3.4	3.1	9.0	2.2	0.5	1.0
MS % Rec.	98.8	99.3	94.5	84.5	N/A	N/A
MSD % Rec.	102.8	96.2	100.6	80.0	N/A	N/A
MS/MSD RPD	4.0	3.2	6.4	5.5	N/A	N/A

QC Summary Report for EERC (Jarda Polc)			
Reported by Frontier Geosciences (Michelle Gauthier)			
June 25, 1999			
<u>IC Trap</u>			
Method Blanks	**THg (ng)**		
Mean	0.01		
Std. Dev.	0.02		
Est. MDL	0.10		
n	2		
SRM	**THg (ng)**		
Source	NIST 1630a		
% Rec.	104.5		
Matrix QC	**THg (ng)**		
Sample ID	ERCA-1A		
RPD	1.7		

QC Summary Report for EERC (Jarda Polc)

Reported by Frontier Geosciences (Michelle Gauthier)

June 25, 1999

Water

Method Blanks	Fe (µg/L)	Mn (µg/L)	THg (ng/L)	MeHg (ng/L)	TOC (mg/L)
Mean	-18.6	-0.20	0.05	0.003	< 1.5
Std. Dev.	1.3	0.08	0.01	0.003	N A
Est. MDL	3.9	0.25	0.04	0.009	1.5
n	4	4	3	3	1

SRM	Fe (µg/L)	Mn (µg/L)	THg (ng/L)	MeHg (ng/L)	TOC (mg/L)
Source	NIST 1643d	100.9	DORM-2	DORM-2	SPEX 16-104
% Rec.	102.0		88.1	93.3	102

Matrix QC	Fe (µg/L)	Mn (µg/L)	THg (ng/L)	MeHg (ng/L)	TOC (mg/L)
Sample ID	ERCW-9	ERCW-9	Different Client	ERCW-18	ERCW-9
RPD	13.4	1.3	2.8	19.9	8.0
MS % Rec.	88.9	96.7	103.8	85.2	95.0
MSD % Rec.	89.6	93.6	103.8	76.6	N/A
MS/MSD RPD	0.7	1.8	0.0	9.5	N/A

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SENT VIA FACSIMILE AND U.S. MAIL

Mr. Jarda Solc
EERC-UND
15 N 23rd Street
Grand Forks, ND 58203

June 15, 1999

RE: **Water and Soils Results**

Dear Mr. Solc,

Attached please find results for the soil and water samples submitted to Frontier Geosciences on May 4, 1999. Briefly, all of the associated quality control analyses were within acceptable limits and looked very good. Specific instances are discussed by matrix below.

Soils

All of the associated quality control analyses looked very good. The matrix spike results for iron were not within control limits, but this is because the spike added was much smaller than the concentration present in the sample.

Waters

All of the associated QA/QC analyses looked good. There were no analytical issues of note.

Please call or e-mail me (jamesk@frontier.wa.com) if you have any questions or concerns.

Sincerely,

James Keithly
cc: Ralph Turner
project files

Trace Metals Results for EERC - Soils

Reported June 15, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (mg/Kg)	ERCS-1 AR	ERCS-1 DW	ERCS-2 AR	ERCS-2 DW	ERCS-3 AR	ERCS-3 DW
Total Solid	0.32	0.32	0.39	0.39	0.29	0.29
LOI	21.6%	21.6%	18.6%	18.6%	23.9%	23.9%
Mn	694	2200	189	489	211	739
Fe	11300	35700	13200	34100	14800	52000
Hg	112	353	7.89	20.4	18.9	66.2
MeHg	0.00404	0.0128	0.000396	0.00103	0.00279	0.00977

Analyte (mg/Kg)	FSC-1 AR	FSC-1 DW	FSC-2 AR	FSC-2 DW
Total Solid	0.37	0.37	0.39	0.39
LOI	21.1%	21.1%	19.6%	19.6%
Mn	417	1120	949	3003
Fe	13200	41800	13200	41800
Hg	119	377	102	323
MeHg	0.00594	0.0188	0.00677	0.0214

AR = as received basis

DW = dry weight basis

Trace Metals Results for EERC - Soils

Reported June 15, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Duplicate Report

Analyte (mg/Kg)	Sample QC'd	Rep. 1	Rep. 2	Mean	RPD
Mn	PCS-2	2470	2555	2513	3.4
Fe	PCS-2	34300	38430	36370	11.4
Hg	FSC-1	119	104.4	111.7	13.2
MeHg	FSC-1	0.005939	NR	N/C	N/C

N/C = Not calculated.
NR = not run

Quality Control Data - Matrix Spike/Matrix Spike Duplicate Report

Analyte (mg/Kg)	Sample QC'd	Mean	Spike Level	MS	% Rec.	Spike Level	MSD	% Rec.	RPD
Mn	PCS-2	2514	403.5	2944	106.6	436.9	2912	91.1	1.1
Fe	PCS-2	36370	807.0	36860	61*	873.9	38460	239*	4.2
Hg	FSC-1	111.7	167.9	266.8	92.4	179.4	284.6	96.3	6.4
MeHg	FSC-1	0.005939	0.025000	0.027420	85.9	0.025000	0.025930	80.0	5.6

* The spike added is much smaller than the concentration in the sample. Therefore, these results are not considered significant.

Trace Metals Results for EERC - Soils

Reported June 15, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Preparation Blank Report

Analyte (mg/Kg)	PBS1	PBS2	PBS3	PBS4	Mean	Std Dev	Est. MDL
Mn (50x)	0.39	0.35	0.32	0.33	0.35	0.03	0.10
Fe (50x)	48	54	48	46	49	3	10
Hg	0.0005	0.0003	0.0005	N/AV	0.0004	0.0001	0.0003
MeHg	0.000002	0.000002	0.000001	N/AV	0.000002	0.000001	0.000002

Est. MDL = Estimated method detection limit

Std Dev = Standard deviation

N/AV = Not available. Only three blanks were used for calculation of the estimated MDL.

Quality Control Data - Standard Reference Material Report

Analyte (mg/Kg)	SRM	Cert. Value	Obs. Value	% Rec.	SRM	Cert. Value	Obs. Value	% Rec.
Mn	NIST 2710	10100	9093	90	NIST 2709	538	471	4.7
Fe	NIST 2710	33800	30090	89	NIST 2709	35000	30540	90.4
Hg	DORM 2	4.640	4.277	92	NIST 1643d	4.640	N/AV	N/C
MeHg	DORM 2	4.470	4.117	92	DORM 2	27.70	N/AV	N/C

SRM Identity = Standard reference material identity

Cert. Value = Certified value

Obs. Value = Experimental result

% Rec. = Percent recovery

N/AV = not available

N/C = not calculated

Trace Metals Results for EERC - Water

Reported June 10, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Sample Results

Analyte (µg/L)	ERCW1-2	ERCW2-2	ERCW3-2	ERCW5-2
Mn	616	564	2310	2270
Fe	760	<150	650	220
Hg	0.857	0.0102	2.81	2.55
MeHg	0.00437	0.000367	0.000704	0.000559
TOC	16	8.4	12	13

Analyte (µg/L)	ERCW6-2	ERCW8-2	ERCWX-2	ERCW4-2
Mn	1290	2240	<1.2	3420
Fe	280	<150	<150	68000
Hg	1.54	0.413	0.001	40.8
MeHg	0.000552	0.000053	NR	NR
TOC	10	14	1.9	44

Analyte (µg/L)	ERCW7-2	PCS1	PCS2
Mn	19000	3840	5370
Fe	550000	190	180
Hg	5.34	0.0145	0.0872
MeHg	NR	0.000172	0.000129
TOC	540	20	19

The TOC values are supposed to have units of mg/L. This was confirmed by Frontier on 2/17/00. - BB

Trace Metals Results for EERC - Water

Reported June 10, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Duplicate Report

Analyte (µg/L)	Sample QC'd	Rep. 1	Rep. 2	Mean	RPD
Mn	ERCW2-2	564	580	572	2.9
Fe	ERCW2-2	ND	ND	N/C	N/C
Hg	ERCW3-2	2.807	2.815	2.811	0.3
MeHg	ERCW2-2	0.000367	0.000370	0.000369	0.8
TOC	ERCW2-2	8.400	8.900	8.650	5.8

ND = not detected

N/C = Not calculated.

Quality Control Data - Matrix Spike / Matrix Spike Duplicate Report

Analyte (µg/L)	Sample QC'd	Mean	Spike Level	MS	% Rec.	MSD	% Rec.	RPD
Mn	ERCW2-2	572	10000	10100	95.3	10100	95.3	0.0
Fe	ERCW2-2	ND*	2000	2220	103.7	2040	94.7	8.5
Hg	ERCW3-2	2.811	2.020	2.935	6.1	2.874	3.1	2.1
MeHg	ERCW2-2	0.000367	0.001110	0.001749	124.5	0.001675	117.8	4.3
TOC	ERCW2-2	8.4	20.0	28.9	102.5	N/AV	N/C	N/C

* Results are less than the MDL so recovery calculations use a zero.

N/AV = not available

N/C = not calculated

Trace Metals Results for EERC - Water

Reported June 10, 1999

Frontier Geosciences Inc., 414 Pontius Ave. N, Seattle WA 98109

Quality Control Data - Preparation Blank Report

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	Std Dev	Est. MDL
Mn	-0.4	-1.0	-1.2	-1.3	-1.0	0.4	1.2
Fe	20	122	115	55	78	49	150
Hg	0.0000	-0.0001	-0.0001	N/AV	-0.0001	0.0001	0.0002
MeHg	0.000002	0.000002	0.000000	N/AV	0.000001	0.000001	0.000003
TOC	ND	ND	ND	ND	N/C	N/C	1.5

Est. MDL = Estimated method detection limit

Std Dev = Standard deviation

N/AV = Not available. Only three blanks were used for calculation of the estimated MDL.

ND = not detected

NC = not calculated

Quality Control Data - Standard Reference Material Report

Analyte (µg/L)	SRM Identit	Cert. Value	Obs. Value	% Rec.	Obs. Value	% Rec.
Mn	NIST 1643d	37.7	38.0	100.8	38.3	101.6
Fe	NIST 1643d	91.2	98.9	108.4	102.8	112.7
Hg	NIST 1643d	4640	4147	89.4	-	N/C
MeHg	NIST 2976	27.70	20.8	74.9	-	N/C
TOC	Spex 14-122	20.0	19.5	97.5	21.8	109.0

SRM Identity = Standard reference material identity

Cert. Value = Certified value

Obs. Value = Experimental result

% Rec. = Percent recovery

Frontier Geosciences Inc.
Environmental Research & Specialty Analytical Laboratory

414 Pontius Avenue North Seattle WA 98109
(206) 622-6960 fax (206) 622-6870 Info@Frontier.WA.Com

Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 1 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N. 23rd St.
Grand Forks, ND 58201-9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK 10/10/00 after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 1Z X57 189 01 4243

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
•	EERC 1	water	2/7 10:55	NRC/Bolles	NO	TOTAL MERCURY THg
•	EERC 2		2/7 11:15			THg
•	EERC 3		2/7 11:23			THg
•	EERC 4		2/7 11:30			THg
•	EERC 5		2/7 11:37			THg
•	EERC 6		2/7 11:47			THg
•	EERC 7		2/7 11:57			THg
•	EERC 8		2/7 12:17			THg
•	EERC 9		2/7			THg
•	EERC 10	water	2/7 12:40			THg

Sample Receipt
C.O.C. Seal Intact? ☒ YES ☐ NO
Cooler Temperature: 1.6 °C
Comments:
COC seal opened @ 1030
VTSR: 900

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 15:00	Date: Time:	Date: Time:
Received by: Caroline Christy	Received by:	Received by:
Print name: CAROLINE CHRISTY	Print name:	Print name:
Company: FGS	Company:	Company:
Date: 2·8·01 Time: 1245	Date: Time:	Date: Time:

Frontier Geosciences Inc.

Environmental Research & Specialty Analytical Laboratory
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(206) 622-6960 fax (206) 622-6870 Info@Frontier.WA.Com

Chain of Custody Record & Laboratory Analysis Reques

Date: 2/7/01 Page: 2 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd ST, Grand Forks, ND 58201-9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK 12/12/00 after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 1Z X57 189 01 42435

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
•	EERC 11 1	water	2/7 13:00	Poll/Bolles	NO	TOTAL MERCURY THg
•	EERC 12		2/7 14:05			THg
•	EERC 13		2/7 14:15			THg
•	EERC 14		2/7 15:45			THg
•	EERC 15		2/7 16:45			TOTAL MERCURY THg
•	EERC-SOIL 1	SOIL	2/7		—	THg, Fe, Mn, Cr, Ni, Zn, Cu, Pb, Cd,
•	EERC-SOIL 2		2/7		—	— " —

Sample Receipt
C.O.C. Seal Intact? ☒ YES ☐ NO
Cooler Temperature: 1.6 °C
Comments: COC seal opened @ 1030
VTSR: 900

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 15:00	Date: Time:	Date: Time:
Received by: Caroline Christy	Received by:	Received by:
Print name: CAROLINE CHRISTY	Print name:	Print name:
Company: FGS	Company:	Company:
Date: 2-8-01 Time: 1245	Date: Time:	Date: Time:

Frontier Geosciences Inc.

Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Reques

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201-9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 1Z X57 189 01 42435

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
•	EERC - 1M	water	2/7	Sou/Bolles	NO	Fe, Mn, Cr, Ni, Zn, Cu, Pb, Cd
•	EERC - 2M					"
•	EERC - 3M					"
•	EERC - 4M					"
•	EERC - 5M					"
•	EERC - 6M					"
•	EERC - 7M					"
•	EERC - 8M					"
•	EERC - 9M					"
•	EERC - 10M		2/7			"

Sample Receipt
C.O.C. Seal Intact? ☒ YES ☐ NO
Cooler Temperature: 1.6 °C
Comments:
COC seal opened @ 1030
VTSR: 900

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 1500	Date: Time:	Date: Time:
Received by: Caroline Christy	Received by:	Received by:
Print name: CAROLINE CHRISTY	Print name:	Print name:
Company: FGS	Company:	Company:
Date: 2·8·01 Time: 1245	Date: Time:	Date: Time:

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Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Reques

Date: 2/7/01 Page: 4 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201-9018
CONTACT: Bethany Belles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbelles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days after date of receipt. JK 10/10/00
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 12 X57 189 01 42435

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	EERC - 11M	water	2/7	Boles/Volc	NO	Fe, Mn, Cr, Ni, Zn, Cu, Pb, Cd
	EERC - 12M	}	}	}		— " —
	EERC - 13M	}	}	}		— " —
	EERC - 14M	}	}	}		— " —
	EERC - 15M	water	2/7			— " —

Sample Receipt
C.O.C. Seal Intact? ☒ YES ☐ NO
Cooler Temperature: 16 °C
Comments:
COC seal opened @ 1030
VTSR: 900

Relinquished by: Bethany Belles	Relinquished by:	Relinquished by:
Print name: Bethany Belles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 1500	Date: Time:	Date: Time:
Received by: Caroline Christy	Received by:	Received by:
Print name: CAROLINE CHRISTY	Print name:	Print name:
Company: FGS	Company:	Company:
Date: 2-8-01 Time: 1245	Date: Time:	Date: Time:

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Chain of Custody Record & Laboratory Analysis Reques

Date: 2/7/01 Page: 85 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201 9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK 10/10/00 after date of receipt.
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Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 1Z X57 189 01 4243501[illegible]

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
•	2S/0x/A	Soil	7/2/01	B. Bolles	None	Total Hg
•	2Aq/0x/A	H2O	7/2/01	↓	↓	Total Hg
•	3S/0x/A1	Soil	↓	↓	↓	Hg Speciation
•	3S/0x/B1	Soil	↓	↓	↓	Total Hg; Methyl Hg
•	3S/0x/B2	Soil	↓	↓	↓	Total Hg
•	3Aq/0x/A1	H2O	↓	↓	↓	Total Hg; Methyl Hg
•	3Aq/0x/A2	H2O	↓	↓	↓	Total Hg
•	5S/0x/A1	Soil	↓	↓	↓	Hg Speciation
•	5S/0x/A2	Soil	↓	↓	↓	Hg Speciation
•	5S/0x/B1	Soil	↓	↓	↓	Total Hg; Methyl Hg

Frontier Sample Receipt
C.O.C. Seal Intact? ☒ YES ☐ NO
Cooler Temperature: 1.6 °C
Comments:
COC seal opened @ 1030
VTSR: 900

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 1500	Date: Time:	Date: Time:
Received by: Caroline Christy	Received by:	Received by:
Print name: CAROLINE CHRISTY	Print name:	Print name:
Company: FGS	Company:	Company:
Date: 2-8-01 Time: 1245	Date: Time:	Date: Time:

Frontier Geosciences Inc.

Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 6 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201 9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 1Z757 189 01 4243501Y

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
•	5Aq/Ox/A1	H_2O	7/2/01	B. Bolles	None	Total Hg; Methyl Hg
•	5Aq/Ox/A2	H_2O		↓	↓	Total Hg
•	7S/Ox/A1	Soil				Hg Speciation
•	7S/Ox/B1	Soil				Total Hg; Methyl Hg
•	7S/Ox/B2	Soil				Total Hg
•	7Aq/Ox/A1	H_2O				Total Hg, Methyl Hg
•	Column 4	Soil + H_2O	N.A.			Separate Pore Water from
	" "					Sediment - Then:
	" "					Aqueous - 2 Total Hg; 1 Methyl Hg
•	☆ Column 3+4 T-0	Soil	Hg speciation			Sediment - 2 Hg Speciation; 1 THg; 1 MeHg

Sample Receipt	Relinquished / Received	Relinquished / Received	Relinquished / Received
C.O.C. Seal Intact? ☒ YES ☐ NO	Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Cooler Temperature: 1.6 °C	Print name: Bethany Bolles	Print name:	Print name:
Comments: ☆ wasn't written on COC	Company: EERC	Company:	Company:
COC seal opened @ 1030	Date: 2/7/01 Time: 1500	Date: Time:	Date: Time:
	Received by: Caroline Christy	Received by:	Received by:
	Print name: CAROLINE CHRISTY	Print name:	Print name:
	Company: FGS	Company:	Company:
VTSR: 900	Date: 2.8.01 Time: 1245	Date: Time:	Date: Time:

Frontier Geosciences Inc.
Environmental Research & Specialty Analytical Laboratory
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Chain of Custody Record & Laboratory Analysis Request

Client Company: Energy and Environmental Research Center	Frontier Project Manager: Nicolas Bloom
Address: 15 N. 23rd St. Grand Forks, ND 58201	Guaranteed Turnaround Time:
	Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
	Quality Assurance Level: ☐ Standard ☐ High
CONTACT: Bethany Bolles	Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
Phone: 701-777-5050 Fax: 701-777-5181	*All samples are held for at least ~~3 months~~ 60 days JK 10/10/00 after date of receipt. Please note that after this time they are disposed of or returned to the client.
Email: bbolles@undeerc.org	Clients may request a longer holding time by writing to the Frontier Project Manager.
Project Name: Hg Speciation	**Please discuss this with the Frontier Project Manager.
Contract/PO #:	Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking # 1Z X57 189 0142435014

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
		Soil	NA	B. Bolles	None	Separate pore water from sediment.
•	Column 6	AND				Aqueous: 2 total Hg; 1 Methyl Hg
		H_2O				Sediment: 2 Hg Speciation; 1 THg; 1 MeHg
		Soil		B. Bolles	None	Separate pore water from sediment
•	Column 8	AND	NA			Aqueous: 2 total Hg, 1 methyl Hg
		H_2O				Sediment: 2 Hg Speciation; 1 THg; 1 MeHg
★	columns 5+6 T=0	soil	- Hg spec			

Sample Receipt			
C.O.C. Seal Intact? ☒ YES ☐ NO	Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Cooler Temperature: 1.6 °C	Print name: Bethany Bolles	Print name:	Print name:
Comments: ★ not on COC	Company: EERC	Company:	Company:
COC seal opend @ 1030	Date: 2/7/01 Time: 16:00	Date: Time:	Date: Time:
	Received by: Caroline Christy	Received by:	Received by:
	Print name: CAROLINE CHRISTY	Print name:	Print name:
	Company: FGS	Company:	Company:
VTSR: 906	Date: 2.8.01 Time: 1245	Date: Time:	Date: Time:

Frontier Geosciences Inc.

Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 1 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St.
Grand Forks, ND 58201-9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days after date of receipt. JK 10/10/00
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**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	EERC 1	water	2/7 10^55	SOL/BOLLES	NO	TOTAL MERCURY THg
	EERC 2		2/7 11^15			THg
	EERC 3		2/7 11^23			THg
	EERC 4		2/7 11^30			THg
	EERC 5		2/7 11^37			THg
	EERC 6		2/7 11^47			THg
	EERC 7		2/7 11^57			THg
	EERC 8		2/7 12^17			THg
	EERC 9		2/7			THg
	EERC 10	water	2/7 12^40			THg

[redacted]
C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: °C
Comments:

VTSR:

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 15^00	Date: Time:	Date: Time:
Received by:	Received by:	Received by:
Print name:	Print name:	Print name:
Company:	Company:	Company:
Date: Time:	Date: Time:	Date: Time:

Frontier Geosciences Inc.

Environmental Research & Specialty Analytical Laboratory
414 Pontius Avenue North Seattle WA 98109
(206) 622-6960 fax (206) 622-6870 Info@Frontier.WA.Com

Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 2 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201 9018
CONTACT: Bethany Belles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbelles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days after date of receipt. JK 12/12/00
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	EERC 11 1	water	2/7 1300	Pocc/Balles	NO	TOTAL MERCURY THg
	EERC 12		2/7 1405			THg
	EERC 13		2/7 1415			THg
	EERC 14		2/7 1545			THg
	EERC 15		2/7 1645			TOTAL MERCURY THg
	EERC-SOIL 1	SOIL	2/7		—	THg, Fe, Mn, Cr, Ni, Zn, Cu, Pb, Cd
	EERC-SOIL 2		2/7		—	— " —

Sample Receipt Information
C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: °C
Comments:
VTSR:

Relinquished by: Bethany Belles	Relinquished by:	Relinquished by:
Print name: Bethany Belles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 1500	Date: Time:	Date: Time:
Received by:	Received by:	Received by:
Print name:	Print name:	Print name:
Company:	Company:	Company:
Date: Time:	Date: Time:	Date: Time:

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Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 3 of 7

Client Company: Energy and Environmental Research Center	Frontier Project Manager: Nicolas Bloom
Address: 15 N 23rd St Grand Forks, ND 58201 9018	Guaranteed Turnaround Time:
	Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
	Quality Assurance Level: ☒ Standard ☐ High
CONTACT: Bethany Bolles	Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
Phone: (701) 777-5056 Fax: (701) 777-5181	*All samples are held for at least ~~3 months~~ 60 days after date of receipt.
Email: bbolles@undeerc.org	Please note that after this time they are disposed of or returned to the client. Clients may request a longer holding time by writing to the Frontier Project Manager.
Project Name: Hg Speciation	**Please discuss this with the Frontier Project Manager.
Contract/PO #:	Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	EERC - 1M	water	2/7	SOL/BOLLES	NO	Fe, Mn, Cr, Ni, Zn, Cu, Pb, Cd
	EERC - 2M					-"-
	EERC - 3M					-"-
	EERC - 4M					-"-
	EERC - 5M					-"-
	EGRC - 6M					-"-
	EERC - 7M					-"-
	EGRC - 8M					-"-
	EGRC - 9M					-"-
	GERC - 10M		2/7			-"-

Sample Receipt [illegible]	Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
C.O.C. Seal Intact? ☐ YES ☐ NO	Print name: Bethany Bolles	Print name:	Print name:
Cooler Temperature: °C	Company: EERC	Company:	Company:
Comments:	Date: 2/7/01 Time: 15:00	Date: Time:	Date: Time:
	Received by:	Received by:	Received by:
	Print name:	Print name:	Print name:
	Company:	Company:	Company:
VTSR:	Date: Time:	Date: Time:	Date: Time:

Frontier Geosciences Inc.
Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 4 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201-9018

CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK 12/10/01 after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	EERC - 11M	water	2/7	Bolles/Sole	NO	Fe, Mn, Cr, Ni, Zn, Cu, Pb, Cd
	EERC - 12M	}	}	}		— " —
	EERC - 13M	}	}	}		— " —
	EERC - 14M	}	}	}		— " —
	EERC - 15M	water	2/7			— " —

Sample Receipt
C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: °C
Comments:

VTSR:

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 1500	Date: Time:	Date: Time:
Received by:	Received by:	Received by:
Print name:	Print name:	Print name:
Company:	Company:	Company:
Date: Time:	Date: Time:	Date: Time:

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Environmental Research & Specialty Analytical Laboratory
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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 85 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201 9018
CONTACT: Bethany Belles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbelles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.
Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	2S/0x/A	Soil	7/2/01	B. Belles	None	Total Hg
	2Aq/0x/A	H_2O	7/2/01	↓	↓	Total Hg
	3S/0x/A1	Soil	↓	↓	↓	Hg Speciation
	3S/0x/B1	Soil	↓	↓	↓	Total Hg, Methyl Hg
	3S/0x/B2	Soil	↓	↓	↓	Total Hg
	3Aq/0x/A1	H_2O	↓	↓	↓	Total Hg, Methyl Hg
	3Aq/0x/A2	H_2O	↓	↓	↓	Total Hg
	5S/0x/A1	Soil	↓	↓	↓	Hg Speciation
	5S/0x/A2	Soil	↓	↓	↓	Hg Speciation
	5S/0x/B1	Soil	↓	↓	↓	Total Hg, Methyl Hg

Sample Receipt
C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: °C
Comments:
VTSR:

Relinquished by: Bethany Belles	Relinquished by:	Relinquished by:
Print name: Bethany Belles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 15[00]	Date: Time:	Date: Time:
Received by:	Received by:	Received by:
Print name:	Print name:	Print name:
Company:	Company:	Company:
Date: Time:	Date: Time:	Date: Time:

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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 6 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St
Grand Forks, ND 58201 9018
CONTACT: Bethany Bolles
Phone: (701) 777-5050 Fax: (701) 777-5181
Email: bbolles@undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.
Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
	5Aq/Ox/A1	H_2O	7/2/01	B. Bolles	None	Total Hg, Methyl Hg
	5Aq/Ox/A2	H_2O		↓	↓	Total Hg
	7S/Ox/A1	Soil				Hg Speciation
	7S/Ox/B1	Soil				Total Hg, Methyl Hg
	7S/Ox/B2	Soil				Total Hg
	7Aq/Ox/A1	H_2O				Total Hg, Methyl Hg
	Column 4	Soil + H_2O	N.A.			Separate Pore Water from
	" "					Sediment - Then:
	" "					Aqueous - 2 Total Hg, 1 Methyl Hg
						Sediment - 2 Hg Speciation, 1 THg, 1 MeHg

Sample Receipt
C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: °C
Comments:
VTSR:

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 15^00	Date: Time:	Date: Time:
Received by:	Received by:	Received by:
Print name:	Print name:	Print name:
Company:	Company:	Company:
Date: Time:	Date: Time:	Date: Time:

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Chain of Custody Record & Laboratory Analysis Request

Date: 2/7/01 Page: 7 of 7

Client Company: Energy and Environmental Research Center
Address: 15 N 23rd St.
Grand Forks, ND 58201
CONTACT: Bethany Bolles
Phone: 701-777-5050 Fax: 701-777-5181
Email: bbolles @ undeerc.org
Project Name: Hg Speciation
Contract/PO #:

Frontier Project Manager: Nicolas Bloom
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☐ Standard ☐ High
Disposal*: ☒ FGS Dispose ~~_~~ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK 12/16/00 after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☐ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
		Soil	NA	B. Bolles	None	Separate pore water from sediment
	Column 6	AND				Aqueous 2 total Hg, 1 Methyl Hg
		H2O				Sediment 2 Hg Speciation, 1 THg, 1 MeHg
		Soil		B. Bolles	None	Separate pore water from sediment
	Column 8	AND	NA			Aqueous 2 total Hg, 1 methyl Hg
		H2O				Sediment 2 Hg Speciation, 1 THg, 1 MeHg

C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: °C
Comments:

VTSR:

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 2/7/01 Time: 16:00	Date: Time:	Date: Time:
Received by:	Received by:	Received by:
Print name:	Print name:	Print name:
Company:	Company:	Company:
Date: Time:	Date: Time:	Date: Time:

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Environmental Research & Specialty Analytical Laboratory

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Chain of Custody Record & Laboratory Analysis Request

Date: 10/13/00 Page: 1 of 1

Client Company: Energy & Environmental Research Center (EERC)
Address: 15 N 23rd St
Grand Forks, ND 58202
CONTACT: Bethany Bolles
Phone: 701-777-5050 Fax:
Email: bbolles@undeerc.org
Project Name: Mercury Project
Contract/PO #:

Frontier Project Manager:
Guaranteed Turnaround Time:
Confirmation of Sample Arrival at Frontier: ☐ YES ☐ NO
Quality Assurance Level: ☒ Standard ☐ High
Disposal*: ☒ FGS Dispose ☐ Return to Client ☐ Ship to 3rd Party**
*All samples are held for at least ~~3 months~~ 60 days JK 10/16/00 after date of receipt.
Please note that after this time they are disposed of or returned to the client.
Clients may request a longer holding time by writing to the Frontier Project Manager.
**Please discuss this with the Frontier Project Manager.

Carrier Information: FED EX ☐ UPS ☒ Other ☐ Tracking #

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected by	Preservation	Analysis Required/Comments
1	Control #1 - Time = Ø	Sed.	10/13/00	B. Bolles	NONE	Total Hg
2	Control #2 - Time = Ø	Sed.	10/13/00	" "	" "	Total Hg
3	Control #1 - T = Ø	Water	10/13/00	" "	" "	Total Hg
4	Control #2 - T = Ø	Water	10/13/00	" "	" "	Total Hg

[illegible]
C.O.C. Seal Intact? ☐ YES ☐ NO
Cooler Temperature: 9.4°C °C
Comments:
UPS 1Z X57 189 01 4111 840
VTSR: ~~08~~ a.m. f. 10.16.00 0900

Relinquished by: Bethany Bolles	Relinquished by:	Relinquished by:
Print name: Bethany Bolles	Print name:	Print name:
Company: EERC	Company:	Company:
Date: 10/13/00 Time: 4.30 PM	Date: Time:	Date: Time:
Received by: A. Malaske Laffaety	Received by:	Received by:
Print name: A. Malaske Laffaety	Print name:	Print name:
Company: FGS	Company:	Company:
Date: 10.16.00 Time: 1140	Date: Time:	Date: Time:

P.M. Ralph

Frontier Geosciences Inc.
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Seattle, WA 98109
(206) 622-6960 fax (206) 622-6870
info@frontier.wa.com

Chain-of-Custody (COC)

Frontier Client: EERC - UND
Client Contact: JAROSLAV POLC.
Address: 15N 23rd ST. GRAND FORKS ND 58201

Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: Hg Rokogk
Contract/PO #:

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB Y (Y/N) QA Level: Standard ☒ High ☐

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
CENT-891	EERC 29	water	9/22 15:00	Polc/Boiko	—	THg
NIC-109	EERC 30	}	}	}	—	THg
TS-28	EERC 31				—	THg
CENT 600	EERC 32	water			—	THg
	WE ARE RETURNING UNUSED, CROSS-CONT OR INADVERTENTLY EXPOSED BOTTLES THANK YOU.					
						FGS NOTES: Bottles are filled to various capacities. [illegible] is approx. ½ full

Relinquished by (signature): [signature]
Print Name: JAROSLAV POLC
Company: EERC UND
Date: 9/22/99 Time: 16:00

Received by (signature): [signature]
Date: 9-23-99 Time: 1500
Print Name: A. Malaika Lafferty
Cooler Temp.: 2.0 °C
Company: FGS
Blue Ice/Ice pac: [illegible]
Condition of Samples upon arrival: [illegible]

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager)
All samples are held for at least 3 months after date of receipt. After this time they are disposed of or returned to the client.
Clients may request a longer holding time. Requests must be made in writing to the Frontier Project Manager.

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Seattle, WA 98109
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info@frontier.wa.com

PM = RALPH

Chain-of-Custody (COC)

Frontier Client: EERC - UND
Client Contact: JAROS SOLC
Address: 15N 23rd. St.
GRAND FORKS ND
58201

Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: Hg RELEASE
Contract/PO #:

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ✓ (Y/N) QA Level: Standard ☒ High ☐

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
✓	ERCW - 19	water	9/13	SOLC/BOLLES	—	THg
✓	ERCW - 20	}	9/13	|	—	THg
✓	ERCW - 21	}	9/13	|	—	THg
	~~ERCW - 22~~	~~water~~	~~9/13~~	|	—	THg
	ERCA -	trap		|	—	THg
✓	ERCA - 4	|		|	—	THg
✓	ERCA - 5 ✓	|		|	—	THg
✓	ERCA - 6	|	9/13	|	—	THg
✓	ERCA - 7	|		|	—	THg
✓	ERCA - 8	|		|	—	THg
✓	ERCA - 9	|			—	THg
✓	ERCA - 10	trap		SOLC/BOLLES		

Relinquished by (signature): [signature]
Print Name: J. SOLC
Company: EERC - UND
Date: 9/13/99 Time: 14:00

Received by (signature): H Ferguson Date: 9.14.99 Time: 11:38
Print Name: H. FERGUSON Cooler Temp.:
Company: FGS Blue Ice/Ice: ≤5.8°C
Condition of Samples upon arrival:

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager)
All samples are held for at least 3 months after date of receipt. <u>After this time they are disposed of or returned to the client.</u>
Clients may request a longer holding time. Requests <u>must</u> be made in writing to the Frontier Project Manager.

Page ___ of ___

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Seattle, WA 98109
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info@frontier.wa.com

Chain-of-Custody (COC)

Frontier Client: JAROA POLC
Client Contact: EERC - UND
Address: 15 N 23rd St. GRAND FORKS, ND 58201

Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: MERCURY RELEASE
Contract/PO #:

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ____ (Y/N) QA Level: Standard ☒ High ☐

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
✓	ERCA 2	CTRAP	5/21/99	PORTER/BOLLES	—	THg
✓	ERCA 3	CTRAP	5/25/99	BOLLES/POLC	—	THg
✓	ERC 4	SOIL	6/29/99	BOLLES/POLC	—	THg, MeHg, Fe, Mn, LOI
✓	ERCW 10	WATER	6/29/99	BOLLES/POLC	—	THg, MeHg
✓	ERCW 10/1			PORTER	—	TOC
✓	ERCW 10/2				—	Fe, Mn
✓	ERCW 12				—	THg, MeHg
✓	ERCW 12/1				—	TOC
✓	ERCW 12/2				—	Fe, Mn
✓	ERCW 17				—	THg
✓	ERCW - MB				—	THg
✓	ERCW - 23				—	THg
✓	ERCW - 23/1				—	TOC
✓	ERCW - 23/2				—	Fe, Mn
✓	ERCW - 24				—	THg

Relinquished by (signature): [signature]
Print Name: JAROSLAV POLC
Company: EERC - UND
Date: 6/29/99 Time: 17:00

Received by (signature): H Ferguson
Date: 6-30-99 Time: 10:35
Print Name: H Ferguson
Cooler Temp.: 5°C
Company: FGS
Blue Ice / Ice (circled: Ice)
Condition of Samples upon arrival: Gen. Good

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager)
All samples are held for at least 3 months after date of receipt. After this time they are disposed of or returned to the client.
Clients may request a longer holding time. Requests must be made in writing to the Frontier Project Manager.

Frontier Geosciences Inc. COC Form. Version V.3/23/99

Frontier Geosciences Inc.
414 Pontius Avenue North
Seattle, WA 98109
(206) 622-6960 fax (206) 622-6870
info@frontier.wa.com

James

Chain-of-Custody (COC)

Frontier Client: EERC
Client Contact: J. Pocc
Address: 15N 23rd. Street GRAND FORKS, ND 58201

Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: MERCURY RELEAS
Contract/PO #: ______

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ______ (Y/N) QA Level: Standard [X] High [ ]

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
✓	ERCW 24/1	water	6/29/99	FOSTER/	—	TOC
✓	ERCW 24/2			BELLOS/		Fe, Mn
✓	ERCW 25			POCC		THg
✓	ERCW 25/1					TOC
✓	ERCW 25/2					Fe, Mn
✓	ERCW 26					THg
✓	ERCW 26/1					TOC
✓	ERCW 26/2					Fe, Mn
✓	ERCW 27					THg
✓	ERCW 27/1				—	TOC
✓	ERCW 27/2	water	6/29/99			Fe, Mn

Relinquished by (signature): [signature]
Print Name: JAROSLAV POCC
Company: EERC - UND
Date: 6/29/99 Time: 17:00

Received by (signature): H Ferguson Date: 6-30-99 Time: 10 35
Print Name: H. Ferguson Cooler Temp.: 5°C
Company: FGS Blue Ice/(Ice)
Condition of Samples upon arrival: Gen Good

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager)
All samples are held for at least 3 months after date of receipt. After this time they are disposed of or returned to the client.
Clients may request a longer holding time. Requests must be made in writing to the Frontier Project Manager.

P.M. Ralph

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414 Pontius Avenue North
Seattle, WA 98109
(206) 622-6960 fax (206) 622-6870
info@frontier.wa.com

Chain-of-Custody (COC)

Frontier Client: EERC - UND
Client Contact: JAROA SOLC
Address: 15 N 23rd St. GRAND FORKS ND 58203
Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: MERCURY RELEASE
Contract/PO #:

QA Level: Standard ☐ High ☐

Guaranteed TAT ____ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ____ (Y/N)

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
JAR	ERC 5	soil	5/19/99 1500	FOSTER	–	THg, MeHg, LOI, Fe, Mn
I.C. TRAP	ERCA 1	C trap	5/19-5/20/99	VOLC	–	THg
~~VIAL~~	ERCW 9	water	5/20/99 1050	VOLC/FOSTER	–	THg
VIAL	ERCW 9/1	✓	1050		–	TOC
TM-4967	ERCW 9/2	✓	1050		–	Fe, Mn
TS-18	ERCW 11	✓	1120		–	THg, MeHg
VIAL	ERCW 11/1	✓	1120		–	TOC
IM-6589	ERCW 11/2	✓	1120		–	Fe, Mn
CENT 731	ERCW 16	✓	1200		–	THg
FGS-B 12	ERCW 18	✓	1220		–	THg, MeHg
VIAL	ERCW 18/1	✓	1220		–	TOC
IM-5773	ERCW 18/2	✓	1220		–	Fe, Mn
1/S.	~~[illegible]~~	~~[illegible]~~	5/20/99 1520	VOLC/FOSTER	–	~~[illegible]~~

Margin notes: * FGS-B 18C

Relinquished by (signature): [signature]
Print Name: JAROSLAV SOLC
Company: EERC UND
Date: 5/20/99 Time: 16^00

Received by (signature): [signature] Date: 5/21/99 Time: 1015
Print Name: A. Malinka Lollhausky
Cooler Temp.: 1.6°C
Company: FGS
Blue Ice/(Ice): melted
Condition of Samples upon arrival: Generally Good

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager)
All samples are held for at least 3 months after date of receipt. After this time they are disposed of or returned to the client.
Clients may request a longer holding time. Requests must be made in writing to the Frontier Project Manager.

Page 1 of 1

* Samples were labelled only as ERCW - 9 (?) ... which of these were 9/1, ...

Frontier Geosciences Inc.
414 Pontius Avenue North
Seattle, WA 98109
(206) 622-6960 fax (206) 622-6870
info@frontier.wa.com

Chain-of-Custody (COC)

Frontier Client: EERC - UND
Client Contact: JAROSLAV SOLC
Address: 15 N 23 ST. GRAND FORKS ND 58201
Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: MERCURY RELEASE
Contract/PO #:

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ____ (Y/N) QA Level: Standard [X] High [ ]

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
RAIN-153	ERCW 1	H_2O	5/2 / 12:00 PM	FOSTER/SOLC	—	THg, MeHg
RAIN-156	ERCW 2	✓	5/2 12:15		—	THg, MeHg
CR-T-561	ERCW 3	✓	5/2 12:30		—	THg, MeHg
FGS-B-136	ERCW 4	✓	5/2 12:40		—	THg,
RAIN-165	ERCW 5	✓	5/2 13:45		—	THg, MeHg
LB-551	ERCW 6	✓	5/2 10:40		—	THg, MeHg
NIC-025	ERCW 7	✓	6/2 13:55		—	THg,
FGS-B-133	ERCW 8	✓	5/2		—	THg, MeHg
NSB-392	ERCW X	✓	5/2 10:30	FOSTER/SOLC	—	THg,
			5/2			
JAR	ERCS 1	soil	5/2 7:10	SOLC		THg, MeHg, LOI, Fe, Mn
	ERCS 2	soil	5/2 7:20	SOLC		THg, MeHg, LOI, Fe, Mn
	ERCS 3	soil	5/2 7:30	SOLC		THg, MeHg, LOI, Fe, Mn
			~~5/2~~	~~6:20~~		
	FSC 1	soil/core	5/2 6:20	SOLC		pore water extract - ???
JAR	FSC 2	soil/core	5/2 6:50	SOLC		→ THg, MeHg, LOI, Mn, Fe

Relinquished by (signature): [signature]
Print Name: JAROSLAV SOLC
Company: EERC - UND
Date: 5/2 Time:

Received by (signature): [signature] Date: 5-7-?? Time: 1115
Print Name: [illegible]
Cooler Temp.: ≤ 4°C
Company: FGS
Blue Ice/Ice: [illegible]
Condition of Samples upon arrival: [illegible]

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager).
All samples are held for at least 3 months after date of receipt. After this time they are disposed of or returned to the client.
Clients may request a longer holding time. Requests must be made in writing to the Frontier Project Manager.

Frontier Geosciences Inc. COC Form, Version V, 3/23/99 *AA Bag reads 'No Number' but bottle engraved RAIN 153.

PM: Ralph

Frontier Geosciences Inc.
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Chain-of-Custody (COC)

Frontier Client: EERC - UND
Client Contact: JAROB SOLC
Address: 15 N 23 St. GRAND FORKS ND 58201

Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: MERCURY RELEA[illegible]
Contract/PO #:

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ____ (Y/N) QA Level: Standard ☒ High ☐

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
TM-6642	*ERCW 1/2	water	5/2 12⁰⁰ AM	FJV/JOC	–	Fe, Mn
TM-6521	*ERCW 2/2	✓	12[illegible]		–	Fe, Mn
-5810	*ERCW 3/2	✓	12³⁰		–	Fe, Mn
TM-6538	*ERC W4/2	✓	12[illegible]		–	Fe, Mn
TM-5845	*ERCW 5/2	✓	12⁴⁵		–	Fe, Mn
-5865	*ERCW 6/2	✓	10⁰⁰		–	Fe, Mn
-5275	*ERCW 7/2	✓	13[illegible]		–	Fe, Mn
-5812	ERCW 8/2	✓			–	Fe, Mn
TM-6611	*ERCW X/2	✓	10³⁰		–	Fe, Mn

Relinquished by (signature): [signature]
Print Name: JAROSLAV SOLC
Company: EERC - UND
Date: 5/2 Time:

Received by (signature): [signature] Date: 5-4-[illegible] Time: 1115
Print Name: [illegible] Cooler Temp.: [illegible]
Company: FGS
Blue Ice/Ice: [illegible]
Condition of Samples upon arrival: [illegible]

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager)
All samples are held for at least 3 months after date of receipt. <u>After this time they are disposed of or returned to the client.</u>
Clients may request a longer holding time. Requests <u>must</u> be made in writing to the Frontier Project Manager.

* Bag reads 6521, bottle [illegible] 6521.

Dim Ralyk

Frontier Geosciences Inc.
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Seattle, WA 98109
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Chain-of-Custody (COC)

Frontier Client: EERC - UND
Client Contact: JAROA SOCC
Address: 15 N 23 St. GRAND FORKS ND 58201

Phone #: (701) 777 5217
Fax #: (701) 777 5181
Project Name: MERCURY RELEAS
Contract/PO #:

Guaranteed TAT ______ CONFIRMATION OF SAMPLE ARRIVAL AT LAB ___ (Y)/N) QA Level: Standard [X] High [ ]

Bottle #	Sample ID	Matrix	Date/Time Sampled	Collected By	Preservation	Analysis Required/Comments
VIAL	ERCW 1/1	Water	5/2 12^00 PM	PORTER	–	LO1
	✓ERCW 2/1	✓	12^15		–	
	✓ERCW 3/1	✓	12^30		–	
	✓ERCW 4/1	✓	12^40		–	
	✓ERCW 5/1	✓	15^45		–	
	✓ERCW 6/1	✓	10^40		–	
	✓ERCW 7/1	✓	13^55		–	
	ERCW 8/1	✓			–	
VIAL	✓ERCW X/1	Water	10^30	PORTER	–	LO1

Relinquished by (signature): [signature]
Print Name: JAROSLAV SOCC
Company: EERC - UND
Date: 5/2 Time:

Received by (signature): [signature] Date: [illegible] Time: 1115
Print Name: [illegible]
Company: FGS
Cooler Temp.: [illegible]
Blue Ice/Ice: [illegible]
Condition of Samples upon arrival: [illegible]

Disposition of unused sample (circle one) Frontier Dispose/Return to Client/Ship to 3rd party (discuss with Frontier Project Manager).
All samples are held for at least 3 months after date of receipt. <u>After this time they are disposed of or returned to the client.</u>
Clients may request a longer holding time. Requests <u>must</u> be made in writing to the Frontier Project Manager.

APPENDIX B

COLUMN WATER PARAMETERS DURING THE LONG-TERM COLUMN STUDY

NW-1
natural water

Date	pH	EC (mS)	DO (%)	DO (mg/L)	Eh (mV)	T (^oC)
5/11/99	7.00	6.97	13.4	1.12	2.4	23.3
5/12/99	7.07	6.86	15.2	1.38	0.6	18.8
5/13/99	7.08	6.97	16.7	1.58	-1.1	17.6
5/14/99	7.13	6.97	17.6	1.53	-2.5	21.8
5/15/99	7.13	6.87	14.0	1.23	-2.7	20.9
5/16/99	7.12	6.75	14.3	1.25	-3.3	21.1
5/17/99	7.18	6.81	15.2	1.35	-5.4	21.0
5/18/99	7.22	6.8	14.0	1.24	-8.8	20.9
5/19/99	7.34	6.86	14.1	1.23	-15.3	22.4
5/20/99	7.38	6.77	16.1	1.43	-18.1	20.5
5/21/99	7.58	6.86	16.4	1.46	-30.4	21.2
5/24/99	7.60	6.89	17.5	1.54	-31.2	21.5
6/1/99	7.66	6.76	14.9	1.28	-42.0	22.9
6/9/99	7.81	7.07	15.8	1.35	-60.7	22.9
6/14/99	7.79	6.75	20.4	1.80	-55.0	21.4
6/21/99	7.62	6.90	17.2	1.43	-40.2	24.2
6/29/99	7.64	6.98	21.5	1.85	-38.8	20.8

NW-2
natural water

Date	pH	EC (mS)	DO (%)	DO (mg/L)	Eh (mV)	T (^oC)
05/11/99	7.05	7.01	16.0	1.37	1.0	23.3
05/12/99	7.07	7.02	16.4	1.50	1.8	18.8
05/13/99	7.07	6.99	17.0	1.59	2.4	17.6
05/14/99	7.08	7.06	15.7	1.37	1.4	21.7
05/15/99	7.08	7.00	14.6	1.29	0.8	21.0
05/16/99	7.07	7.09	13.3	1.16	1.7	21.2
05/17/99	7.03	6.95	15.4	1.35	3.4	21.2
05/18/99	7.06	6.73	13.5	1.18	2.6	20.9
05/19/99	7.07	6.91	14.0	1.21	1.6	22.4
05/20/99	7.05	6.84	13.7	1.21	4.0	20.5
05/20/99	6.79	6.93	17.0	1.70	0.1	21.4
05/21/99	7.03	6.92	5.4	0.47	3.7	21.3
05/24/99	7.08	6.86	10.4	0.92	1.0	21.5
06/01/99	6.94	7.01	7.3	0.63	2.0	22.9
06/09/99	7.04	6.76	15.4	1.30	-9.8	22.8
06/14/99	7.10	6.73	18.4	1.62	-12.0	21.4
06/21/99	7.03	6.99	15.7	1.33	-4.5	24.2
06/29/99	7.04	6.86	21.1	1.81	-3.1	22.8

NW-3
natural water

Date	pH	EC (?S)	DO (%)	DO (mg/L)	Eh (mV)	T (^oC)
05/11/99	7.05	7.02	22.3	1.83	0.9	23.1
05/12/99	7.11	6.92	15.3	1.41	1.4	18.9
05/13/99	7.27	7.00	15.7	1.49	-7.4	17.7
05/14/99	7.09	6.97	16.5	1.43	-1.8	21.7
05/15/99	7.10	6.81	14.7	1.29	-1.1	21.0
05/16/99	7.10	6.80	15.5	1.38	-1.5	21.2
05/17/99	7.11	6.81	15.4	1.35	-0.6	21.2
05/18/99	7.12	6.81	15.1	1.34	-2.2	21.0
05/19/99	7.14	6.92	17.4	1.49	-2.7	22.4
05/20/99	7.16	6.86	17.9	1.59	-3.5	20.7
05/21/99	7.15	6.71	17.1	1.49	-4.0	21.2
05/24/99	7.12	6.73	17.2	1.50	-2.6	21.5
06/01/99	7.00	6.81	17.3	1.47	-2.8	22.9
06/09/99	7.03	6.97	16.6	1.40	-5.4	22.9
06/14/99	7.03	6.78	18.0	1.59	-8.8	21.7
06/21/99	6.94	6.95	26.7	2.22	-8.4	24.3
06/29/99	7.04	6.73	21.0	1.81	-2.7	22.6
07/06/99	6.97	6.80	23.6	2.12	-6.4	20.5

DI-1
DI Water

Date	pH	EC???S?	DO (%)	DO (mg/L)	Eh (mV)	T (^oC)
5/11/99	6.44	287	31.4	2.70	36.6	22.8
5/12/99	6.57	329	27.6	2.56	31.4	19.0
5/13/99	6.62	363	26.3	2.49	26.9	17.7
5/14/99	6.66	380	29.2	2.57	25.8	17.6
5/15/99	6.74	374	27.6	2.45	20.7	21.0
5/16/99	6.73	402	30.2	2.67	19.8	21.2
5/17/99	6.77	413	29.0	2.57	19.2	21.2
5/18/99	6.78	414	32.0	2.84	20.6	21.0
5/19/99	6.84	438	32.6	2.84	15.8	22.4
5/20/99	6.86	456	32.8	2.93	14.0	20.7
5/21/99	6.92	554	32.9	2.91	11.5	21.2
5/24/99	6.94	495	33.1	2.91	8.9	21.6
6/1/99	6.33	581	32.3	2.78	38.4	22.5
6/9/99	6.50	650	26.3	2.26	57.3	22.8
6/14/99	6.75	705	26.5	2.31	11.5	21.7
6/21/99	6.82	790	31.7	2.66	8.2	24.0
6/29/99	6.81	860	29.4	2.57	11.2	22.6
7/6/99	6.86	972	34.5	3.10	1.3	20.6

DI-2
DI Water

Date	pH	EC???S?	DO (%)	DO (mg/L)	Eh (mV)	T (^oC)
5/11/99	6.29	172	31.6	2.82	46.0	23.1
5/12/99	6.51	184	26.6	2.46	33.4	19.0
5/13/99	6.52	170	30.6	2.91	33.4	17.7
5/14/99	6.58	182	32.0	2.81	28.8	21.7
5/15/99	6.61	185	32.5	2.89	28.6	21.0
5/16/99	6.63	217	29.4	2.60	27.1	21.2
5/17/99	6.63	214	30.0	2.65	29.1	21.3
5/18/99	6.71	273	29.0	2.57	22.4	21.0
5/19/99	6.74	239	32.0	2.78	21.8	22.4
5/20/99	6.80	258	32.6	2.92	18.4	20.7
5/20/99	7.20	940	0.1	0.01	-5.4	21.5
5/21/99	7.26	1119	3.9	0.35	-10.1	21.2
5/24/99	7.16	1033	8.6	0.75	-3.3	21.6
6/1/99	7.02	1056	13.4	1.16	-3.0	22.4
6/9/99	6.95	1178	14.8	1.27	-8.4	22.8
6/14/99	7.05	1227	14.2	1.25	-9.4	21.5
6/21/99	6.94	1303	17.1	1.44	0.7	24.0
6/29/99	6.83	1381	20.0	1.73	9.4	22.7

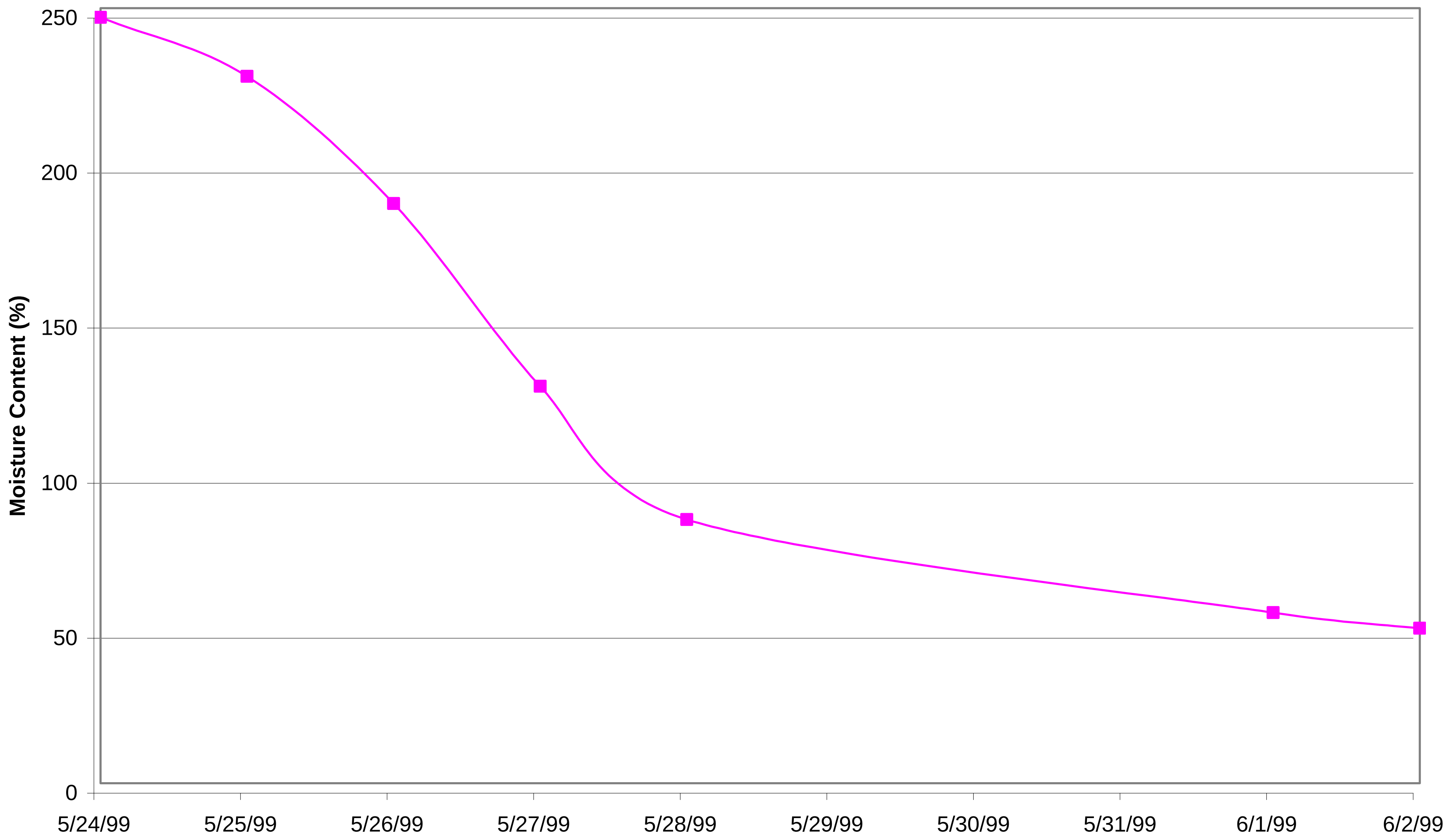
RS-1
Coulee Water

Date	pH	EC (?S)	DO (%)	DO (mg/L)	Eh (mV)	T (^oC)
5/19/99	8.43	1521.0	70.0	6.14	-81.1	21.9
5/20/99	8.42	1528.0	60.8	5.38	-80.1	20.8
5/20/99	7.27	1728.0	0.7	0.07	-12.4	21.4
5/21/99	7.45	1688.0	14.2	1.25	-20.6	21.5
5/24/99	7.56	1682.0	18.4	1.62	-29.2	21.7
6/1/99	7.66	1713	26.4	2.27	-40.4	22.9
6/9/99	7.62	1675	22.8	1.95	-27.3	22.9
6/14/99	7.74	1685	21.9	1.90	-54.6	21.9
6/21/99	6.82	1772	25.7	2.16	8.0	24.0
6/29/99	7.79	1652	31.5	2.76	-46.9	22.4
7/6/99	7.54	1700	23.0	2.06	-41.4	20.7

APPENDIX C

SEDIMENT WATER CONTENT

Average Moisture Content During Drying (5/24/99 - 6/2/99); New Jersey Sediment



Moisture Analyses of the New Jersey Sediment Exposed to Air (All weights are in grams.)

Date: 5/24/99

	Sample #1	Sample #2
Weight of Container:	121.4	121.3
Weight of Container + Soil:	166.1	170.4
Weight of Soil:	44.7	49.1
Weight of Container + Dry Soil:	134.4	135.3
Weight of Dry Soil:	13.0	14.1
Moisture Content (%):	243.9	249.7

Average Moisture Content: **247%**

Date: 5/25/99

	Sample #1	Sample #2
Weight of Container:	124.4	127.0
Weight of Container + Soil:	170.2	176.9
Weight of Soil:	45.8	49.8
Weight of Container + Dry Soil:	138.1	142.5
Weight of Dry Soil:	13.7	15.5
Moisture Content (%):	233.7	221.8

Average Moisture Content: **228%**

Date: 5/26/99

	Sample #1	Sample #2
Weight of Container:	121.4	121.4
Weight of Container + Soil:	158.7	162.4
Weight of Soil:	37.3	41.0
Weight of Container + Dry Soil:	134.4	135.7
Weight of Dry Soil:	13.0	14.3
Moisture Content (%):	187.2	186.4

Average Moisture Content: **187%**

Date: 5/27/99

	Sample #1	Sample #2
Weight of Container:	124.9	122.3
Weight of Container + Soil:	169.2	172
Weight of Soil:	44.3	49.7
Weight of Container + Dry Soil:	144.2	144.2
Weight of Dry Soil:	19.4	21.9
Moisture Content (%):	129.0	127.2

Average Moisture Content: **128%**

Date: 5/28/99

	Sample #1	Sample #2
Weight of Container:	119.3	119.4
Weight of Container + Soil:	155.0	167.3
Weight of Soil:	35.7	47.9
Weight of Container + Dry Soil:	138.3	145.8
Weight of Dry Soil:	19.0	26.4
Moisture Content (%):	87.8	81.3

Average Moisture Content: **85%**

Date: 6/1/99

	Sample #1	Sample #2
Weight of Container:	124.9	122.3
Weight of Container + Soil:	173.4	162.0
Weight of Soil:	48.6	39.7
Weight of Container + Dry Soil:	155.4	148.7
Weight of Dry Soil:	30.5	26.4
Moisture Content (%):	59.0	50.4

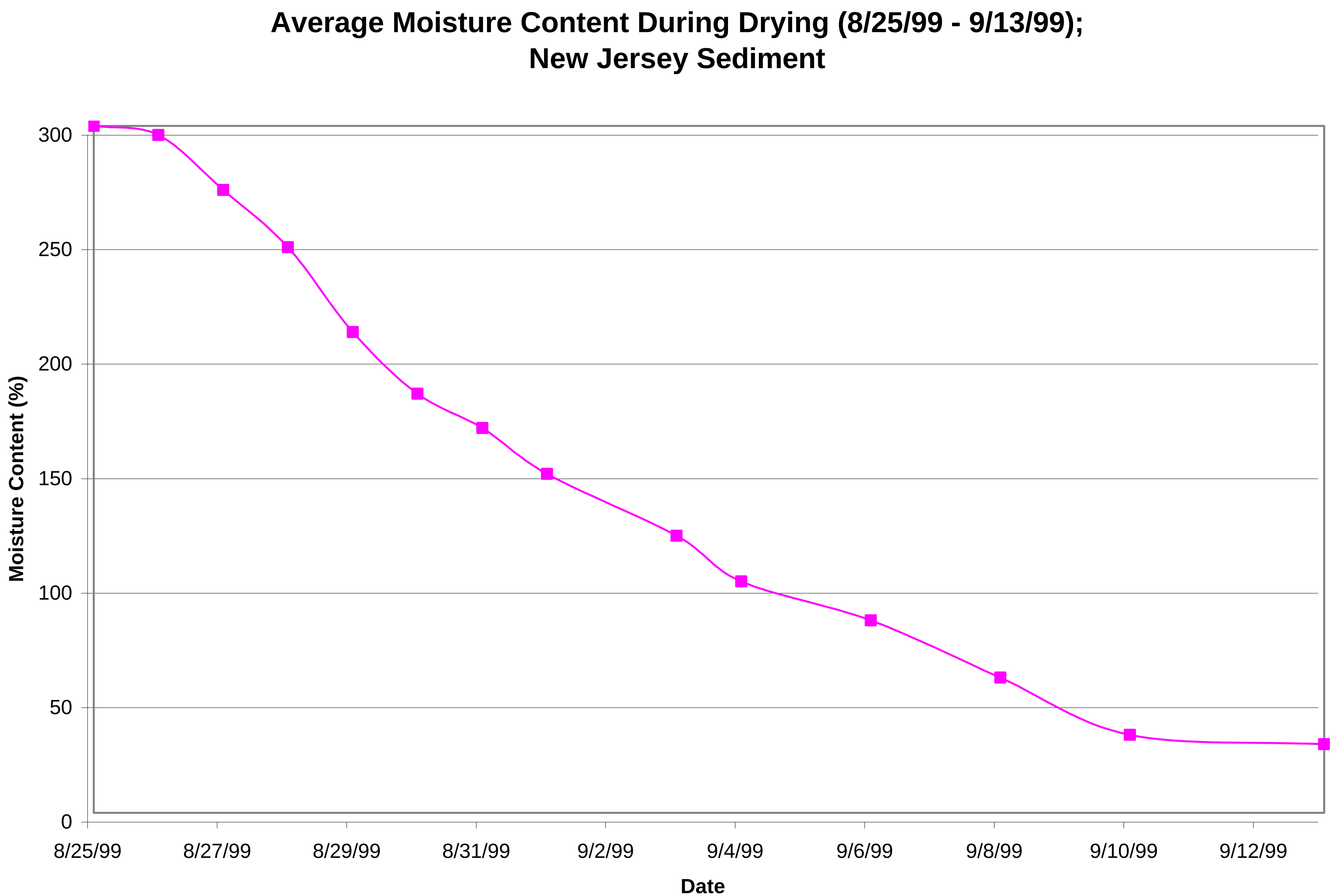
Average Moisture Content: **55%**

Date: 6/2/99

	Sample #1	Sample #2
Weight of Container:	119.2	119.3
Weight of Container + Soil:	159.9	167.1
Weight of Soil:	40.7	47.7
Weight of Container + Dry Soil:	146.5	151.0
Weight of Dry Soil:	27.3	31.7
Moisture Content (%):	49.0	50.5

Average Moisture Content: **50%**

Date	Average Moisture Content (%)
5/24/99	247
5/25/99	228
5/26/99	187
5/27/99	128
5/28/99	85
6/1/99	55
6/2/99	50



Average Moisture Content During Drying (8/25/99 - 9/13/99);
New Jersey Sediment
Moisture Content (%)
300
250
200
150
100
50
0
8/25/99
8/27/99
8/29/99
8/31/99
9/2/99
9/4/99
9/6/99
9/8/99
9/10/99
9/12/99
Date

Moisture Analyses of the New Jersey Sediment Exposed to Air (All weights are in grams.)

Date: 8/25/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.5
Weight of Container + Soil:	54.9	77.9
Weight of Soil:	52.4	75.4
Weight of Container + Dry Soil:	15.5	21.5
Weight of Dry Soil:	13.0	19.0
Moisture Content (%):	303.1	296.8

Average Moisture Content: **300%**

Date: 8/26/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.5
Weight of Container + Soil:	48.7	64.3
Weight of Soil:	46.2	61.8
Weight of Container + Dry Soil:	14.1	18.2
Weight of Dry Soil:	11.6	15.7
Moisture Content (%):	298.3	293.6

Average Moisture Content: **296%**

Date: 8/27/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.5
Weight of Container + Soil:	44.6	51.7
Weight of Soil:	42.1	49.2
Weight of Container + Dry Soil:	14.5	15
Weight of Dry Soil:	12.0	12.5
Moisture Content (%):	250.8	293.6

Average Moisture Content: **272%**

Date: 8/28/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.5
Weight of Container + Soil:	40.6	51
Weight of Soil:	38.1	48.5
Weight of Container + Dry Soil:	13.4	16.6
Weight of Dry Soil:	10.9	14.1
Moisture Content (%):	249.5	244.0

Average Moisture Content: **247%**

Moisture Analyses of the New Jersey Sediment Exposed to Air (All weights are in grams.)

Date: 8/29/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.5
Weight of Container + Soil:	45.8	55.4
Weight of Soil:	43.4	52.9
Weight of Container + Dry Soil:	16.5	19.5
Weight of Dry Soil:	14.1	17.0
Moisture Content (%):	207.8	211.2

Average Moisture Content: **210%**

Date: 8/30/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.5
Weight of Container + Soil:	36.8	48.4
Weight of Soil:	34.3	45.9
Weight of Container + Dry Soil:	14.6	18.8
Weight of Dry Soil:	12.1	16.3
Moisture Content (%):	183.5	181.6

Average Moisture Content: **183%**

Date: 8/31/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.5
Weight of Container + Soil:	36.6	35.9
Weight of Soil:	34.2	33.4
Weight of Container + Dry Soil:	15.6	14.6
Weight of Dry Soil:	13.2	12.1
Moisture Content (%):	159.1	176.0

Average Moisture Content: **168%**

Date: 9/1/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.5
Weight of Container + Soil:	34.7	29.3
Weight of Soil:	32.3	26.8
Weight of Container + Dry Soil:	15.6	13.2
Weight of Dry Soil:	13.2	10.7
Moisture Content (%):	144.7	150.5

Average Moisture Content: **148%**

Moisture Analyses of the New Jersey Sediment Exposed to Air (All weights are in grams.)

Date: 9/2/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.5
Weight of Container + Soil:	34.9	35.7
Weight of Soil:	32.5	33.2
Weight of Container + Dry Soil:	18.3	16.8
Weight of Dry Soil:	15.9	14.3
Moisture Content (%):	104.4	132.2

Average Moisture Content: **118%**

Date: 9/3/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.4
Weight of Container + Soil:	32	26.4
Weight of Soil:	29.6	24.0
Weight of Container + Dry Soil:	15.9	13.2
Weight of Dry Soil:	13.5	10.8
Moisture Content (%):	119.3	122.2

Average Moisture Content: **121%**

Date: 9/4/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.4
Weight of Container + Soil:	29.8	32.1
Weight of Soil:	27.3	29.7
Weight of Container + Dry Soil:	16.4	16.8
Weight of Dry Soil:	13.9	14.4
Moisture Content (%):	96.4	106.3

Average Moisture Content: **101%**

Date: 9/6/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.5
Weight of Container + Soil:	31	34.1
Weight of Soil:	28.6	31.6
Weight of Container + Dry Soil:	17.9	19.8
Weight of Dry Soil:	15.5	17.3
Moisture Content (%):	84.5	82.7

Average Moisture Content: **84%**

Moisture Analyses of the New Jersey Sediment Exposed to Air (All weights are in grams.)

Date: 9/8/99

	Sample #1	Sample #2
Weight of Container:	2.4	2.4
Weight of Container + Soil:	29.9	25.7
Weight of Soil:	27.5	23.3
Weight of Container + Dry Soil:	19.4	17.3
Weight of Dry Soil:	17.0	14.9
Moisture Content (%):	61.8	56.4

Average Moisture Content: **59%**

Date: 9/10/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.5
Weight of Container + Soil:	25.2	25.3
Weight of Soil:	22.7	22.8
Weight of Container + Dry Soil:	19.2	19.8
Weight of Dry Soil:	16.7	17.3
Moisture Content (%):	35.9	31.8

Average Moisture Content: **34%**

Date: 9/13/99

	Sample #1	Sample #2
Weight of Container:	2.5	2.4
Weight of Container + Soil:	25.1	29.0
Weight of Soil:	22.6	26.6
Weight of Container + Dry Soil:	20.3	22.3
Weight of Dry Soil:	17.8	19.9
Moisture Content (%):	27.0	33.7

Average Moisture Content: **30%**

Moisture Analyses of the New Jersey Sediment Exposed to Air

Date	Average Moisture Content (%)
8/25/99	300
8/26/99	296
8/27/99	272
8/28/99	247
8/29/99	210
8/30/99	183
8/31/99	168
9/1/99	148
9/2/99	
9/3/99	121
9/4/99	101
9/6/99	84
9/8/99	59
9/10/99	34
9/13/99	30

APPENDIX D

CASE NARRATIVE BY NICOLAS BLOOM (FRONTIER GEOSCIENCES, INC.)

Mercury Speciation and Trace Metals in Core Incubation Sediments and Pore Water

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March 22, 2001

I. Objectives

Sediment, soil, and water samples, as well as whole sediment core tubes from two different experiments were sent for mercury speciation and trace metal analysis, as described on the accompanying chains of custody. Extraction of pore water and mercury speciation was required on a project which looked at the long-term incubation of Hg spiked sediments, while trace metals analyses were performed on samples from a project which investigated the leachability of metals from contaminated soils. Because of Frontier's own interest in these projects, extra samples were analyzed at no additional cost to fill in some details. Data were collected using Frontier's research level protocols, which call for summary data and QC to be supplied, without a complete raw data validation package.

II. Sample Receipt

Samples were received on February 8, 2001, as shown on the accompanying chain of custody forms. All samples were accounted for, and in excellent condition upon arrival. Core samples were placed in a cold room (0-5°C) for two days until they could be processed fore pore water extraction. Sediment and soil samples for total metals analysis only were kept in the laboratory until digestion, while those requiring solid phase Hg speciation were refrigerated. Subsamples were taken immediately from all sediments requiring methyl mercury analysis, and frozen (-18°C) until extraction. Water samples were acidified (1% HCl containing 0.2N BrCl for total Hg samples, 1% HNO_3 for trace metals samples) in the original containers, shaken, and then transferred to ultra-clean Frontier Geosciences containers for digestion and storage. The HNO_3 preserved samples were transferred to HF/HNO_3 cleaned polyethylene containers, while the total

Hg water samples were transferred to glass containers to avoid sample contamination by diffusion of gas phase Hg° through the polyethylene bottle walls.

III. Analytical Procedures

<u>General Procedures.</u> All handling, processing, and analysis of the samples was conducted using clean sample handling protocols, both to avoid contamination of the samples by the laboratory, and to avoid contamination of the laboratory by the samples (Bloom, 1995). These protocols include the use of lab spaces where the air, reagent water, wash water, and reagents are monitored and controlled for Hg levels suitably low for trace mercury analysis. Lab personnel wear disposable cleanroom gloves, which are changed frequently during processing and analysis. All pertinent digestion and analytical data is recorded at the time of acquisition in either bound paper log books, or computer notebooks (backed up daily).

Data from the atomic fluorescence (CVAFS) Hg analyzers was accessed as peak height by chart recorders with two pens offset by a factor of 20, to allow access to a wide linear range (10^3). Calculations were made by Excel spread sheets, starting with the calculation of the mass (ng) of Hg in the aliquot analyzed, and then calculating the final concentration in ng/g (parts per billion) or ng/L (parts per trillion). For direct comparison of Hg species, all concentrations are expressed as the element, not as the individual compound. Trace metals data were directly calculated as dilution-corrected concentrations by the ICP/MS software (Perkin-Elmer Elan-6000), and then manually corrected for blanks and matrix spike additions (MSA) slope correction. Data are method blank corrected, and all have been reviewed and, if necessary, revised, by the quality assurance department. Although more detailed descriptions are given below, the following table summarizes the analytical methods used in this study.

analyte	matrix	method no.	description
porewater extr.	sediment	Mason, et. al 1999	centrifugation
Hg	water	EPA-1631	BrCl, Dual Au, CVAFS
Hg	sediment	EPA-1631 appdx	aqua regia + M-1631
CH_3Hg	water	EPA-1630 draft	ethylation, GC-CVAFS
CH_3Hg	sediments	Bloom, et. al, 1997	HBr extract + M-1630
trace metals	water	EPA-1638 draft	ICP/MS
trace metals	sediments	EPA-1638 draft	HF/HNO_3 digest; ICP/MS
water content	solids	*na*	dry @ 105°C

Lab Extraction of Sediment Pore Water. Incubation cores were opened, handled, and subsampled in an ultra-clean N_2 purged glove box, to avoid changes in Hg speciation and distribution coefficients that can accompany the exposure of anoxic sediments to air. In the glove box, both ends of the core were opened, and approximately 20 cm of mud from each side was scooped out into a glass bowl, and quickly homogenized with a stainless steel spoon. The mud was then packed into four 250 mL centrifuge tubes to contain about 300 grams per tube, such that pairs of tubes weighing within one gram of each other were prepared. Additionally, two each of approximately 20 gram aliquots of the homogenized sediment were sub sampled to 20 mL glass vials. One sediment vial was then sealed with its Teflon-lined cap, and frozen until extraction for methyl mercury analysis, while the other was sealed and refrigerated until it could be extracted and analyzed for total Hg and sequential selective extractions.

Once four centrifuge tubes, were collected and their masses balanced in pairs, they were removed from the glove box and placed in the centrifuge, where they were spun for 30 minutes at 3000 RPM to separate the solids from the pore water. After centrifugation, the centrifuge tubes were returned, unopened, to the glove box for further processing. In the glove box, the supernatant pore water from each jar was then vacuum filtered in the glove box, using disposable acid-cleaned 0.2 μ nitrocellulose membrane filter units. Some of the samples could be rapidly filtered through a single filtration unit, but others required as many as three filtration units to filter 20-40 mL of sample, due to clogging by fine particulate matter that was not settled by centrifugation. The filtered pore water from a single section was poured into 40 mL glass vials with Teflon lined caps, still within the N_2 glove box, and the vials capped and sent to the lab for preservation. The samples were preserved by the addition of 0.4% (v/v) HCl, and stored refrigerated until analysis for Hg, CH_3Hg, and metals.

Total Hg in pore water and water samples. After methyl Hg was determined and verified on all specified water samples, the remainder contained in the original sample bottle was oxidized by the addition of 0.2N bromine monochloride in 12N HCl (1% by volume for overlying waters, 5% by volume for pore waters). Water sample #13, which contained large amounts of suspended sediment was digested with by mixing 1:1 with a 0.1N BrCl in 6N HCl mixture, and heating for 4 hours at 85°C in a sealed glass bottle. After BrCl oxidation, the samples were analyzed by US EPA Method 1631-modified. Aliquots of the oxidized samples ranging from 0.005 to 100 mL were pre-reduced with $NH_2OH{\cdot}HCl$ (to remove free halogens), and then placed into the purge vessel, and the Hg(II) reduced to

Hg^{o} by the addition of $SnCl_2$. The Hg^{o} formed was purged with nitrogen onto gold coated sand traps, and the traps analyzed by dual amalgamation/CVAFS. The final results are corrected for the volume dilution caused by the addition of the BrCl oxidant.

Methyl Hg in water. The preserved aqueous samples (5 – 45 mL, diluted to 45 mL with 0.4% HCl) were distilled with the addition of APDC, to separate the methyl mercury from the matrix (Bloom and Von der Geest, 1995; Horvat, et. al, 1993). At the end of the distillation, the methyl mercury content of the samples is collected in pure distilled water, and the interfering matrix components (DOC, HCl) remain in the residue. The distillation procedure results in recovery of 78% of the aqueous volume of the original sample, and this has been found to empirically carry over 90.6% of the methyl mercury. Distilled samples are corrected by this empirical recovery factor. The distilled samples or smaller aliquots thereof were then analyzed using aqueous phase ethylation at pH 4.9, purge and trap onto Carbotrap™, isothermal GC separation at 100 °C, and CVAFS detection, as is described in EPA Draft Method 1630 (Bloom and Fitzgerald, 1989; Liang, et. al, 1994).

Trace Metals in Water. Trace metals (Cr, Fe, Mn, Ni, Cu, Zn, Cd, Pb) were determined using direct ICP/MS analysis (EPA Method 1638), after digestion of the samples by heating with 1% HNO_3 at 85°C for 4 hours in closed polyethylene bottles. Sample #13, which contained a very large amount of sediment, was further acidified with 5% of a 3:1 mixture of HNO_3 + HF prior to digestion. Internal standardization was accomplished using ^{45}Sc, ^{115}In, and ^{194}Pt as an internal standards for the low, medium, and high mass ranges respectively. Because of high dissolved solids, all water samples (and corresponding blanks) were analyzed with 5x dilution, except #13, which was diluted 1,000x before analysis.

Dry fraction and Loss on Ignition. Soil samples were analyzed for Hg on a wet (as received) basis, and then corrected to a dry weight basis by the use of an independently measured solids content for each sample. The solids content was determined gravimetrically, by drying an aliquot of the sediment overnight at 105 °C, and calculating the fraction of the mass remaining after drying. Loss on ignition, a measure of the sediment organic matter content, was determined by further heating the samples over night at 525°C, and weighing the residual ash.

Total Hg in Sediments. An aliquot (0.5-1 gram) of each sample was digested over night at room temperature using aqua regia (4:1 HCl + HNO_3), and then diluted to 40.0 mL with 0.01N BrCl solution. After digestion, the samples were analyzed by US EPA Method 1631-modified for sediment extracts. Aliquots of the digested samples ranging from 0.01 to 5 mL were placed into the purge vessel, and the Hg(II) reduced to Hg° by the addition of $SnCl_2$. The Hg° formed was purged with nitrogen onto gold coated sand traps, and the traps analyzed by dual amalgamation/CVAFS.

Methyl Hg in Sediments. Sediment sub-samples for methyl mercury were frozen in 20 mL glass vials until the day of extraction. Because of the observation of significant positive artifact formation for methyl mercury when sediments are distilled, sediments were instead extracted with acidic KBr into CH_2Cl_2 prior to analysis (Bloom et. al, 1997). Sediment aliquots of approximately 0.5 grams were digested at room temperature with a mixture of KBr + H_2SO_4 + $CuSO_4$, and then extracted by shaking with 10 mL of CH_2Cl_2 in Teflon centrifuge tubes. After extraction and centrifugation to separate the layers, 20% of the CH_2Cl_2 was transferred to a Teflon purge vessel containing deionized water, and the samples heated to 45°C with nitrogen purge to remove the solvent. This procedure transfers the methyl mercuric bromide to a pure water matrix for analysis. Samples were diluted to a mark on the vials (57.6 mL), and then aliquots of the extract analyzed by aqueous phase ethylation at pH 4.9, purge and trap onto Carbotrap™, isothermal GC separation at 100°C, and CVAFS detection. This extraction procedure is quantitative, so that no recovery correction is made to the results.

Sequential Selective Extractions (Solid Phase Hg Speciation). The extraction scheme is performed using a 100:1 liquid to solids ratio in 40 mL vials, each extraction step is conducted for 18 ± 3 hours with constant agitation, at room temperature. At the end of an extraction step, the samples are centrifuged, and then the supernatant liquid filtered through a 0.2 μ filter. Each sample is then re-suspended in the same extractant as a rinse step, re-centrifuged, and re-filtered. The two filtrates are combined in a 125 mL bottle, oxidized by the addition of BrCl, and diluted to 125 mL prior to analysis for total Hg by EPA Method 1631. After the rinse step, the sample pellet in the centrifuge tube is resuspended in the next extractant, and the entire process repeated. For the 12N HNO_3 step, the samples are centrifuged, but not filtered, as this strength of acid dissolved the cellulose nitrate filters employed. For the final, aqua regia step, the extraction is carried out once, and then diluted to 40.0 mL directly in the centrifuge tube (same extraction as the total Hg extraction described earlier). In the table below are listed the extraction steps and examples of compounds extracted by each.

step	extractant	description	typical compounds
F1	DI water	water soluble	$HgCl_2$, $HgSO_4$
F2	pH 2 HCl/HOAc	"stomach acid"	HgO
F3	1N KOH	organo complexed	Hg-humics, Hg_2Cl_2
F4	12N HNO_3	strong complexed	mineral lattice, Hg_2Cl_2, Hg^o
F5	aqua regia	cinnabar	HgS, m-HgS, HgSe, HgAu
sum			total Hg

As QA measures, a matrix duplicate and three method blanks were co-extracted. In addition, three solid phase samples were extracted as a gauge of methodological accuracy. These samples were a suspension of pure cinnabar (red HgS) powder in kaolin clay, a suspension of pure $HgCl_2$ in kaolin clay, and the certified reference material NIST-2710. The HgS sample is an end-member which should only be extracted by the last (aqua regia) step, while the $HgCl_2$ sample is an end member which should be almost fully extracted by the first (deionized water) step. These two samples have been repeatedly analyzed for total Hg by the aqua regia digestion method, so that in addition to providing a check on the speciation, they can be used to assess overall accuracy (however, the HgS sample typically comes up a bit low by the selective extraction method, owing to losses of some of the highly hydrophobic HgS powder due to flotation during the F1 and F2 extraction steps). The NIST-2710 is a mine-contaminated natural soil which is certified for total Hg, but not Hg speciation. However, this sample has been repeatedly analyzed (n=15) as a selective extractions laboratory control sample for the past two years, and so comparative consistency can be provided.

Selective extractions data must be viewed with considerable caution when making interpretations about the actual Hg species present. While the method quite accurately reflects how much Hg from a sample is, for example, water soluble (F1), or would be stomach acid soluble (F1 + F2), what species actually give rise to the leaching pattern observed are due to a complex interaction of the *in situ* speciation, Hg concentration, and the adsorptive capacity of the matrix. Generally, more accurate inferences about the species present can be made in higher Hg concentration samples (> 100 µg/g), because at lower concentrations re-adsorption of Hg on the matrix particles becomes more severe.

IV. Analytical Issues

Overall, the QA/QC on this project was excellent, and almost all of the results are of extremely high quality. Except for the case of Fe and Cr in soils, all method blanks, duplicate analyses, matrix spikes, and CRMs were well within acceptance criteria (except in a few cases where the spiking level was too low compared to the ambient analyte concentration). We did observe low recoveries for Fe and Cr in the sediment CRM, which, because the matrix spikes were too low, could not be verified with respect to the matrix spike recoveries. These metals are often difficult to fully recover from sediments and soils by the HF/HNO3 digestion, due to incomplete dissolution of minerals such as magnetite, hematite, and chromite (minerals such as these require a different approach—such as lithium metaborate fusion) to fully solubilize the matrix. We should note that good recoveries for these metals were seen for the water CRM, as well as on a different co-analyzed reference material (NIST-1632b, trace metals in coal) which was run for a different client. While it is likely that some recalcitrant mineral phases of Fe and Cr may not have been fully solubilized by this digestion, it is reasonably to assume that any biogeochemically active forms certainly were dissolved.

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