APPENDIX G

BIOACCESSIBILITY

BIOACCESSIBILITY OF MERCURY, ARSENIC, CADMIUM, COPPER, LEAD, SELENIUM, AND ZINC IN SOILS FROM THE FLIN FLON AREA

2008 Final Report

Prepared for

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TABLE OF CONTENTS

I. INTRODUCTION

Intrinsik Environmental Sciences Inc. contracted the Environmental Sciences Group (ESG) of the Royal Military College of Canada (RMC), and the Analytical Services Unit (ASU) of Queen's University, to determine the bioaccessibility of mercury (Hg), arsenic (As), cadmium (Cd), copper (Cu), lead (Pb), selenium (Se), and zinc (Zn) in soils from the Flin Flon, Manitoba, area. Intrinsik provided the soils.

The bioaccessibility of a contaminant is the portion of a contaminant in a sample that is soluble in the gastro-intestinal environment, and can be measured in an *in vitro* laboratory setting. It can be used as a surrogate for bioavailability, which is the portion of a contaminant that is actually absorbed by an organism. The measurement of bioavailability usually requires animal studies, which are more logistically and ethically challenging. The use of bioaccessibility in risk assessment is considered to be valid and applicable at specific sites, provided that some basic data requirements are fulfilled to permit regulatory groups (e.g., Health Canada) to ascertain the validity and defensibility of such measurements (Health Canada 2007). The following parameters have been suggested as relevant for investigation at specific sites:

1. Soil particle size – Other researchers have suggested that the <250 um fraction, typically used in risk assessment, may not be representative of the soil size fraction of greatest relevance to human health risk assessment. Therefore studying a range of soil particle sizes, to ascertain if the particle size affects the bioaccessibility, has been proposed (Health Canada, 2007).
2. Ratio of simulated bioaccessibility fluid volume to soil mass – Other researchers have observed that, for some elements and soils, the ratio of the fluid to the soil mass during the bioaccessibility extraction can affect the bioaccessibility (Health Canada, 2007). To determine if this variable affects the bioaccessibility results, measurements with varying liquid volume to soil mass ratios are recommended (Health Canada, 2007).

The present study was designed in two parts: the first was to investigate the two parameters listed above for three soils and determine the most appropriate method for measuring bioaccessibility, and the second was to measure the bioaccessibility in the remaining soils using this method.

II. METHODS

The ESG laboratory (Analytical Sciences Division) at the Royal Military College of Canada conducted the *in vitro* bioaccessibility extractions to measure the bioaccessible Hg, As, Cd, Cu, Pb, Se, and Zn content of the soil samples. The Analytical Services Unit (ASU) at Queens University in Kingston, Ontario carried out the analysis of the resulting extracts, as well as the analysis of total metals and metalloids in the <45 µm fraction of three soils in Part 1 of the study. The Analytical Sciences Group (ASG) at the Royal Military College, Kingston, Ontario digested several of the bioaccessibility extracts prior to analysis at ASU and analyzed Hg in the <45 µm fraction of two of the three soils used in Part 1. Intrinsik provided the total concentration of the elements of interest in the <250 µm fraction of the soils.

A. *In Vitro* Bioaccessibility Extractions

1. Method Overview

The bioaccessible Hg, As, Cd, Cu, Pb, Se, and Zn content of soils was determined using an *in vitro* bioaccessibility extraction methodology coupled to elemental detection as mentioned previously. The bioaccessibility method is designed to simulate a human receptor and it is a modification of two previously published methods, that of Ruby (1993; 1996) and Rodriguez (1999) incorporating the 100:1 liquid to solid ratio, low molecular weight organic acids, pepsin, bile and pancreatin concentrations used by Ruby and the NaCl and pH conditions of Rodriguez.

For Part 1 of the study, three methods were tested on three soils (FF208B, CS102B, FF276F), in triplicate. The first method (Method 1) used an extraction fluid volume:soil mass of 100:1 and the <45 µm fraction of the soils; the second method (Method 2) used an extraction fluid volume:soil mass of 100:1 and the <250 µm fraction of the soils; and the third method (Method 3) used an extraction fluid volume:soil mass of 2000:1 and the <250 µm fraction of the soils.

Because of the nature of Hg to adhere to adhere to plastic surfaces, Teflon® equipment was used for the extractions, and then the extracts were each split into separate aliquots for As, Cd, Cu, Pb, Se, and Zn analysis, and for Hg analysis. In past studies we have determined that the use of pyrolysis – cold vapour generation – atomic absorption spectrometry (pyrolysis-CVAAS) to measure Hg in bioaccessibility extracts was suitable and convenient, but limited with respect to detection limit (0.2 mg/kg bioaccessible

concentration). Therefore to ensure as many detectable Hg results as possible, the bioaccessibility extracts of 16 samples (reports ASD0809b, c) that had total Hg concentrations less than 7 mg/kg were analyzed by cold vapour generation – atomic fluorescence spectrometry (CV-AFS). This method modification necessitated a larger volume of bioaccessibility extract and this is reflected in the method details.

2. Extraction Procedure

All reagents used in the extraction procedures were of environmental quality or purer. All samples were stored at a low temperature before and after analysis to ensure matrix stability. Samples were clearly and uniquely labelled to ensure traceability.

Two aliquots of dried, sieved (<250 µm for most soils; <45 µm for FF208B, CS102B, FF276F in Part 1, using Method 1) and homogenized soil (0.2 g for samples in reports ASD0809a, d, e, f and samples FF208B, CS102B, FF276F by Methods 1 and 2; 0.6 g for reports ASD0809b, c; 0.02 g for samples FF208B, CS102B, FF276F by Method 3) were accurately weighed into uniquely labelled Teflon® extraction vessels with 20 ml (ASD0809a, d, e, f, and samples FF208B, CS102B, FF276F by Methods 1 and 2), in glass jars with 60 ml (ASD0809b, c), or in Teflon® extraction vessels with 40 ml (samples FF208B, CS102B, FF276F by Method 3) of gastric bioaccessibility solution. Samples were labelled P1 for the simulation of the gastric phase and P2 for simulation of the whole GI process (gastric + intestinal). The gastric bioaccessibility solution, which consisted of 1.25g/L pepsin (P-7000), 0.5 g/L sodium citrate, 0.5 g/L malic acid, 1 ml/L glacial acetic acid, 0.15 M NaCl adjusted to pH 1.8 with 32 % HCl, was added to both the P1 and P2 samples to obtain a liquid to solid ratio of 100:1, except for samples FF208B, CS102B, FF276F by Method 3, where 2000:1 was used. On addition of the gastric solution the pH of both P1 and P2 extraction phases was checked to ensure that it was within 1.8 ± 0.05. If the pH was outside of this tolerance the pH was manually adjusted with 2 or 32 % HCl as required, and the starting pH was recorded. To carry out the stomach phase of the *in vitro* bioaccessibility test, both the P1 and P2 extraction vessels were incubated for 1 hour at 37°C in an incubated shaker at 275 rpm. Thirty minutes into the stomach phase, the pH of the extraction vessels was monitored. If the pH was outside the tolerance of 1.8 ± 0.05, the pH was adjusted as necessary with 2% or 32% HCl and the extraction vessels were returned to the shaking incubator for the remaining 30 minutes. After the 1 hour incubation period, the pH and weight of the P1 samples were recorded. The P1 samples were then prepared for analysis and the P2 samples were further extracted under intestinal conditions. To alter the conditions in the P2 extraction vessels to simulate conditions in the small intestine of the receptor of interest, the pH was

increased to 7 by the addition of saturated sodium carbonate (Na_2CO_3). Once a neutral pH was achieved pre-weighed masses of bile and pancreatin, according to Table 1, were added to each vessel.

Table 1. Experimental conditions of the bioaccessibility extractions.

Experiment or soils	Solution volume (ml)	Soil mass (g)	Soil fraction (μm)	Reagents in P1	Reagents in P2
Part 1: Method 1	20	0.2	<45	1.25g/L pepsin 0.5 g/L sodium citrate 0.5 g/L malic acid 1 ml/L glacial acetic acid 0.15 M NaCl pH 1.8 adjusted with 32 % HCl	35 mg bile extract 10 mg pancreatin
Part 1: Method 2	20	0.2	<250		35 mg bile extract 10 mg pancreatin
Part 1: Method 3	40	0.02	<250		70 mg bile extract 20 mg pancreatin
Part 2: Total Hg>7 mg/kg	20	0.2	<250		35 mg bile extract 10 mg pancreatin
Part 2: Total Hg<7 mg/kg	60	0.6	<250		105 mg bile extract 30 mg pancreatin

Each P2 sample was then further incubated for a total of 4 hours at 37°C. After 2 hours of the intestine phase incubation period, the pH was checked and adjusted to 7.0 with saturated Na_2CO_3 and returned to the incubator for the remaining 2 hours. On completion of the intestinal incubation the final pH and weight of each sample were recorded. The P2 samples were prepared for analysis.

To prepare both the stomach (P1) and intestine (P2) phase samples for analysis, each extract was filtered through a 0.45 μm disposable syringe filter with glass microfibre membrane. Extracts in Part 1 of the study, and extracts of soils with total Hg less than 7 mg/kg were then split into two aliquots: one for analysis of As, Cd, Cu, Pb, Se and Zn (10 ml), and the other for analysis of Hg (4 ml in Part 1, 50 ml otherwise). In Part 1, sample aliquots destined for Hg analysis were preserved with BrCl. Because precipitation of solids was observed in some of these sample aliquots upon preservation, this step was not included in the rest of the study. All filtered extracts were stored refrigerated prior to analysis.

Along with each batch of 8 unknown samples one duplicate sample, one sample of NIST 2710 reference material and two extraction blanks were included. A known amount of Hg was also added to one of the extraction blanks prior to analysis.

B. As, Cd, Cu, Pb, Se, and Zn by (ICP-AES)

ASU determined the inorganic elements As, Cd, Cu, Pb, Se and Zn in the filtered extracts and in the <45 um fraction of three soil samples (FF208B, CS102B, FF276F) used in Part 1 of the present study. ASU is a CAEAL accredited laboratory and the measurement of inorganic elements (including the elements of interest) in soil and water samples by ICP-AES is listed in its scope of accreditation. For soil analysis, the <45 um fractions of the samples (provided by ESG) were ground to a fine powder with a mortar and pestle. Approximately 0.5 g of powdered sample were heated with 2 mL of nitric acid and 6 mL of hydrochloric acid overnight so that the volume was reduced to 1-2 mL. This solution was then made up to 25 mL with distilled deionized water). Samples were analyzed together with one blank, one duplicate and one sample of reference material (SS-2). No further pH adjustment or modifications were made to bioaccessibility extracts prior to analysis. Sample volume was not sufficient to analyze any extracts in duplicate. The elements As, Cd, Cu, Pb, Se, and Zn were analyzed in digests and extracts using a Varian VISTA AX CCD Simultaneous ICP-AES. Calibration control standards (prepared from a separate source from that used for calibration) were used to check the calibration periodically, and instrument blanks were also included with all analyses.

C. Hg in <45 um Fraction of Soils (Part 1)

Analyses were conducted by the ASU and the Analytical Sciences Group (ASG), Royal Military College, Kingston, Ontario. The following methods are included in the scope of accreditation for the two labs. At ASG, the <45 um fraction of two soils (FF208B, FF276F), provided by ESG, were digested using approximately 0.25 to 0.50g soil and 4mL of Trace Metal Grade Nitric Acid, with 1mL each 5% w/v aqueous potassium permanganate and potassium persulfate, in a CEM Microwave Accelerated Reaction System (MARS) microwave digestion system utilizing MARS Xpress 60mL Teflon vessels (ramp to 200^{o}C in 15minute, held at 200^{o}C for 35minutes). After the digestion 0.5mL hydroxylamine sulphate solution (12% w/v) was added on cooling and the samples were made up to 25mL with distilled deionized water. A set of standards, a duplicate, a blank, and a soil reference were digested with each soil run. The soil digests were analyzed using a Perkin-Elmer FIMS-100 Mercury System equipped with a 253.7 ηm source mercury lamp, quartz cell, Perkin-Elmer AS-90 autosampler, and the Perkin Elmer AA WinLab Analyst software. The carrier solution was 3% HCl and the reducing agent was 1.1% tin chloride ($SnCl_2$) in 3% HCl. Ultra high-purity argon was used as the

carrier gas, with the flow rate set between 40 and 70 mL/min. Three 500 μL replicates for each sample were analyzed. A signal was generated in the quartz cell by measuring the amount of light (wavelength 253.7 ηm) absorbed. The mercury concentrations in the samples were determined by comparing sample absorbance responses to a calibration curve generated from standards of known concentration.

At ASU, the <45 um fraction of sample CS102B, provided by ESG, was detected by CV-AAS, using the Milestone (DMA-80) direct Hg analyser. This instrument allows for direct measurement and has been designed to meet the criteria outlined in US EPA Method 7473. Very small quantities of liquid or solid sample were required with little or no preparation and measurements were made on the total mass of Hg in a test portion. The instrumentation afforded a wide concentration range of measurements to be made within the test samples (0.05 ng to 600 ng) under investigation.

An appropriate mass of soil was weighed into quartz or nickel boats. Within the instrumentation each sample underwent a process of drying followed by thermal decomposition within a continuous flow of oxygen. The combustion products are carried off in the oxygen flow and are further decomposed in a hot catalyst bed. Any Hg vapours are trapped on a gold amalgamator tube and subsequently desorbed for spectrophotometric quantification at 254 nm. Long and short path length cells enable the measurement of low range (0.05 – 35 ng) and high range (35 – 600 ng) Hg concentrations respectively.

The instrument was calibrated from working standards prepared from a 1000 mg/L Hg ICP-AES/ICP MS (PlasmaCAL) stock standard. An aqueous QC (or calibration check sample) was included in all analysis runs and was prepared from an independent Hg source to that used to prepare the calibration standards. The aqueous QC was prepared at a mid-range Hg concentration within each detection cell (i.e. low range or high range). A blank was also included.

D. Hg in Bioaccessibility Extracts (Total Hg in soils >7 mg/kg) pyrolysis-CV-AAS

ASU measured total Hg in the bioaccessibility extracts produced at ESG; the following method for Hg in water was accredited until recently but as described above, analysis of Hg in soil using the same methodology is currently accredited.

As described for Hg in soil, the Hg content of the bioaccessibility extracts was detected by CV-AAS. In order to measure the Hg content of each test sample the maximum boat capacity of 500 μl was volumetrically weighed into quartz or nickel boats.

A blank and an aqueous QC (or calibration check sample) was included in all analysis runs; the latter was prepared from an independent Hg source to that used to prepare the calibration standards.

E. Hg in Bioaccessibility Extracts (Total Hg in soils <7 mg/kg) by CV-AFS

ASG digested P2 bioaccessibility extracts and then ASU measured Hg in all extracts. ASG used the digestion method that is included in the Hg in water analysis method included in its scope of accreditation. The ASU method for Hg in water by CV-AFS was recently accredited.

The extract aliquot was mixed with 4ml of trace metal grade nitric acid in a 60mL Teflon® microwave vessel. Aqueous potassium permanganate (1mL, 5% w/v), and subsequently potassium persulfate (1mL, 5%) are added to the solution prior to microwave digestion. Teflon® vessels were then capped and sealed. Digestion conditions were: ramp to 200^{o}C in 10 minutes and held at 200^{o}C for 30 minutes, and samples were under pressure while microwave digesting. A quantity of 0.5mL hydroxylamine sulphate solution (12% w/v) is added on cooling. The final digest was made up to a volume of 25 mL with distilled deionized water.

For the analysis of Hg in the extracts, a fully automated CV-AFS method combining a Tekran model 2620 autosampler with a 2610 pump unit and 2600 control unit was used. The technique is based on dual stage gold amalgamation with atomic fluorescence. This instrumentation allows for unattended analysis of total mercury in waters at ultra-trace levels (<0.5ppt, limited by reagent purity, trace contamination in preparation areas and sample handling techniques) and can be adapted to analyze other matrices[1]. The method has been designed to meet the criteria outlined in US EPA Method 1631 (with modifications). Small quantities of liquid are required (typically 1-50 ml) with minimal preparation.

All sample preparation and analyses were carried out in a two stage clean room. Samples were digested with bromine monochloride (BrCl) and excess halide was neutralized with hydroxylamine hydrochloride. Mercury was converted from Hg^{2+}

[1] Tekran Instruments Corporation. Automated Ultra-Trace Mercury Analysis of Aqueous Samples, Series 2600, Powerpoint presentation, Rev: 1.30 Oct 07, 2006.

(soluble and stable) to Hg^0 (volatile) by reduction with stannous chloride. A gas liquid separator is used to purge the Hg^0 from solution prior to being concentrated on two gold concentrator columns. Computer controlled event tables were used to accurately manage the timing and operation of the peristaltic pump, cartridge heating systems, mass flow controller and valve switching to direct the trapped mercury vapor to the CVAFS detector where it was measured. Quality control measures included blanks, calibration standards, and calibration check standards.

III. RESULTS AND DISCUSSION

A. Part 1: Determination of the most appropriate bioaccessibility method

Details of the results were given in the cover letter for this part of the study and only a summary is given here. Three samples (FF208B, CS102B, FF276F) were studied in this part of the study:

Hg bioaccessibility was higher in all samples in Phase 2 extracts (mimicking gastric + intestinal conditions) compared with the Phase 1 extracts (mimicking gastric conditions). All other element concentrations were either similar (As, some Cu) or lower in Phase 2 extracts. Se bioaccessibility was mostly undetectable. Hg is likely more soluble in the Phase 2 extraction because of increased concentrations of organic components (bile extract and pancreatin) in this phase, since Hg binds strongly to organic material. Therefore to obtain the most conservative bioaccessibility results, both phases must be included for this suite of elements.

The bioaccessibility results were generally higher using Method 3. However, the higher dilution used in Method 3 (2000:1) resulted in a large number of undetectable sample results (56% of all measurements), in spite of the attempt to use three samples that had relatively high concentrations of metals and metalloids in them. Therefore, to obtain as many detectable results as possible, we did not recommend using Method 3 for the remaining samples, since the detection is compromised with this method and the remainder of the samples have lower concentrations of contaminants than the samples analyzed in Part 1.

Statistical analysis revealed that although the smaller particle size (<45 μm) may have higher contaminant concentrations, the chemical or mineralogical form of these contaminants is less bioaccessible when bioaccessibility is expressed as a percent of the

total concentration. Thus, since the statistics do not support the use of the <45 um fraction, we recommended proceeding with the <250 um fraction, and Method 2 was used for the remainder of the samples.

B. Part 2: Bioaccessibility of Hg, As, Cd, Cu, Pb, Se and Zn

Bioaccessible concentrations (mg/kg) and percent bioaccessibilities were determined for all samples and are summarized in Tables 2 and 3.

Hg bioaccessibilities in Phase 2 (gastric + intestinal) were approximately double those in Phase 1 (gastric only), but did not range higher than 11% overall. Bioaccessibilities for As were generally also slightly higher in Phase 2 and ranged as high as 93%. On the other hand, Cd, Cu, Pb and Zn bioaccessibilities were generally higher in Phase 1. Maximum values for these elements overall were: Cd, 100%, Cu, 69%, Pb, 100%, and Zn, 99%. Bioaccessible Se was detectable in only one sample (FF276F) in Phase 2 (bioaccessible Se was not detectable in Phase 1). A paired t-test comparing percent bioaccessibility values between phases showed significant differences for all elements. This was observed even for As and Cu, which had similar bioaccessibility ranges and averages: average percent bioaccessibility of As in Phase 1 was 28 ± 14, compared with 33 ± 16 in Phase 2, and average percent bioaccessibility of Cu in Phase 1 was 41 ± 11, compared with 33 ± 9 in Phase 2. The likely reason that statistical differences were seen, even when element ranges were similar, was that for most elements the percent bioaccessibilities were remarkably consistent across the range of soil samples. Specifically, relative standard deviations (of overall means) ranged from 21% to 50% for most elements and phases. The results that were outside of this range, corresponding to the highest spread in bioaccessibilities, were observed for Hg (RSDs of 140% in Phase 1 and 65% in Phase 2) and Pb in Phase 2 (91%).

IV. CONCLUSIONS

The project commissioned by Intrinsik Environmental to determine the bioaccessibility in Hg, As, Cd, Cu, Pb, Se and Zn in soils from the Flin Flon area was carried out in two parts.

The first part, using three soils, allowed the selection of the most appropriate bioaccessibility extraction method for this site, which included the use of the <250 µm fraction of soils, and a bioaccessibility extraction fluid volume to soil mass ratio of 100:1.

Bioaccessibility of metals and metalloids in the remaining samples was determined with this method, and generally Hg and As bioaccessibilities were higher in Phase 2 (gastric + intestinal) extracts, whereas bioaccessibilities of Cd, Cu, Pb, and Zn were higher in Phase 1 (gastric) extracts. Bioaccessibility of Se was mostly not detectable.

V. LIMITATIONS AND USE OF REPORT

The present report and all previous reports and accompanying cover letters were prepared for the exclusive use of Intrinsik Environmental. The report and other materials, which specifically include all tables and appendices, is based on data obtained from the analysis of samples sent by Intrinsik.

The content of the present report is based on information collected during our analysis, our present understanding of the methods, and our professional judgement in light of such information available in this report. This report provides a professional opinion and does not provide a legal opinion regarding compliance with applicable laws.

The services performed as described in this report were conducted in a manner consistent with that level of care and skill normally exercised by other members of the science and engineering professions currently practising under similar conditions.

The findings and conclusions of this report are valid only as of the date of this report.

We trust that this report provides you with the information you require at this time. If you have any questions, please do not hesitate to contact us.

Dr. Allison Rutter, Director, ASU

Dr. Ken Reimer, Director, ESG

Dr. Iris Koch, Senior Analytical Manger, ESG

VI. REFERENCES

Health Canada, 2007. Federal contaminated site risk assessment in Canada, Part V: guidance on complex site specific human health risk assessment of chemicals (SSRMChem), Version 1.0 Draft final, Prepared by Contaminated Sites Division, Safe Environments Programme.

APPENDIX A: SUMMARY OF RESULTS FROM PART 1

The summary of results from Part 1 is given Table A-1.

APPENDIX B: QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

ESG follows an internal quality assurance/quality control program that was implemented to allow data quality to be monitored on an ongoing basis. However, total metal and metalloid contents for the <250 um fraction were not included in this program since they were provided by the client, and neither ASD nor ASU reviewed the quality control results for the provided data, nor was it verified by either group.

All samples are given sequential, numerical codes before submission to the analytical firms; these codes mask any information concerning sample type or possible concentration of the sample.

Accuracy is measured and controlled by instrument calibration, the use of control spikes and samples, and the preparation and analysis of blanks.

Control spikes and samples are reference materials with known concentrations. In the present study, the soil standard reference material NIST 2710 was used during the bioaccessibility extraction, in which control ranges have been established for most of the elements of interest (As, Cd, Cu, Pb and Zn). NIST 2710 results for each element that are within the acceptable range indicate that the bioaccessibility extractions for that batch are in control. The instrument calibration is evaluated based on comparison of the results with the target concentration and recoveries within the range of 70 to 130 % are considered to be acceptable.

Analytical blanks are processed through extraction/digestion and analysis procedures. These blanks give a measure of the quantity of any contaminant (analyte) that may be added to the overall result during the analysis. Contamination is assumed to be absent when blank results are less than the detection limits for the method.

Precision is measured and controlled by the analysis of duplicates. Extraction duplicates are replicate preparations and analyses of the same sample. Comparison of the average relative standard deviations (RSD) – also known as coefficients of variation, which are calculated as the standard deviation divided by the mean – are used to evaluate laboratory precision. Acceptable limits are generally considered to be less than 40 percent RSD with 20 percent or less considered good agreement.

QC results were generally in control for all samples batches, and results are summarized in Tables B-1, B-2, and B-3. Exceptions and reasons for accepting the data are given below.

In most batches the standard reference soil NIST 2710 results for all elements were within the control limits; the exceptions were Phase 2 results for Cd, Pb and Zn in report ASD0809d and Pb and Zn in ASD0809f, which were slightly low, the Phase 2 Pb result in report ASD0807a, which was slightly high, and Phase 1 results for As, and Zn in report ASD0807a, and As, Cu, Pb and Zn in report ASD0809f, which were slightly high. Because of the preliminary nature of the control limits it is possible that they could be expanded for future use. The sample analyses are considered to be in control since most of the NIST 2710 non-conformances were no more than 40% from the control means.

Hg spike results are included in the absence of available control limits for Hg in the 2710 control sample. One spike was lost to vial breakage (Hg P2 in ASD0809b). One Phase 1 spike result (ASD0809a) and three Phase 2 Hg spike results (ASD0809c, e, f) were lower than the acceptable range (70-130%). The reason for the lower recoveries (P1: 65%; P2: 35%, 62% and 53%) is unknown and is being investigated; it may be related to adsorption/precipitation reactions, resulting from the hypothesized association of Hg with the increased organic components (bile extract, pancreatin) in this phase. In all cases the higher percent bioaccessibility was obtained in Phase 2 for Hg, where the spike recovery was acceptable.

A few blank results were detectable (Pb in ASD0809b, Cu and Pb in ASD0809d, Cd, Cu and Pb in ASD0809e, and Cu in ASD0809f) but in most cases the values were at the limit of detection or barely above it. Since values of Pb, Cu and Cd in samples are well above detection we do not consider sample analyses to be contaminated.

Within-lab repeatability (%RSDs for duplicates) was generally good but in some cases %RSDs were greater than limits usually considered acceptable (40%) (Cu and Pb in ASD0809b, Pb in ASD0809c). In all cases this occurred in Phase 2 extracts and is attributable both to sample heterogeneity and the inconsistent nature of precipitation reactions thought to be occurring in Phase 2 for these elements (Cu and Pb). In the preliminary study, all extracts were preserved at the ESG laboratory with BrCl for Hg analysis. This step caused precipitates to form in the Phase 2 (intestinal) extracts, which, for some samples, caused the repeatability to be higher than the 30% relative standard deviation (RSD) considered to be acceptable.

In some cases greater than 100% bioaccessibility was obtained for samples; these values should be considered to be 100% bioaccessible. Sample heterogeneity (differences in element concentrations in subsamples used in bioaccessibility extracts and total element analysis) is the likely cause of such results, and in most cases the results are within the uncertainty associated with laboratory analysis (30%).

Table A-1. Summary of results for Part 1. Three PBET methods were used: Method 1: <45 um fraction, liquid:solid = 100:1; Method 2: <250 um fraction, liquid:solid = 100:1; Method 3: <250 um fraction, liquid:solid = 2000:1. No substitutions for undetectable values were made when calculating averages and standard deviations.

Bioaccessible Concentrations (mg/kg)

		Bioaccessible Hg mg/kg			Bioaccessible As mg/kg			Bioaccessible Cd mg/kg			Bioaccessible Cu mg/kg			Bioaccessible Pb mg/kg			Bioaccessible Se mg/kg			Bioaccessible Zn mg/kg		
Phase 1		<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1
FF208B	average	0.54	0.47	<4.0	126	72	110	40	33	47	1753	1665	2358	641	443	626	<10	<10	<200	9881	7899	10683
	SD	0.03	0.02	no calc	5	12	6	1	5	4	146	47	70	29	73	21	no calc	no calc	no calc	754	223	804
CS102B	average	<0.2	<0.2	<4.0	13	9.4	<100	10	9.4	<40	245	176	238	139	94	126	<10	<10	<200	1362	1148	2277
	SD	no calc	no calc	no calc	1	0.3	no calc	1	0.3	no calc	20	3	19	11	3	16	no calc	no calc	no calc	82	52	96
FF276F	average	0.96	1.03	<4.0	109	63	<100	56	45	47	2326	2047	2776	495	365	555	<10	<10	<200	9812	8863	11425
	SD	0.03	0.05	no calc	5	2	no calc	2	4	9	108	139	269	12	17	54	no calc	no calc	no calc	748	798	1165
Phase 2		<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1
FF208B	average	4.2	3.6	11	99	54	118	22	16.2	<40	1464	1222	1893	132	79	222	<10	8.9	<200	4427	3541	3181
	SD	1.9	0.4	9	2	2	no calc	0.4	0.5	no calc	19	136	68	20	8	20	no calc	0.4	no calc	304	76	289
CS102B	average	0.53	0.36	<4.0	13	8.5	<100	5.6	5.17	<40	226	167	318	15	12	62	<10	<10	<200	601	423	<2000
	SD	0.23	0.11	no calc	1	0.58	no calc	0.6	0.58	no calc	25	6	61	2	1	7	no calc	no calc	no calc	99	27	no calc
FF276F	average	17	11	29	100	65	106	21	21	41	1752	1368	3216	53	46	249	10.4	12	<200	3478	3102	5464
	SD	2	3	5	9	13	no calc	1	4	no calc	45	128	268	3	7	15	0.8	1	no calc	210	243	669

SD = standard deviation; RSD = relative standard deviation = standard deviation/average x 100%

no calc = no calculation (2 or 3 replicates are undetectable.)

% Bioaccessibility

		% Bioaccessible Hg			% Bioaccessible As			% Bioaccessible Cd			% Bioaccessible Cu			% Bioaccessible Pb			% Bioaccessible Se			% Bioaccessible Zn		
Phase 1		<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1
FF208B	average	0.14	0.20	<1.7	39	56	85	78	86	BND	36	58	83	85	85	BND	<8.1	<19	BND	69	82	BND
	SD	0.01	0.01	no calc	1	9	5	3	14	no calc	3	2	2	4	14	no calc	no calc	no calc	no calc	5	2	no calc
CS102B	average	<1.7	<2.6	<50	12.1	17.9	BND	85	65	BND	31	24.7	34	70	55	74	SND	BND	BND	61	54	BND
	SD	no calc	no calc	no calc	0.6	0.5	no calc	4	2	no calc	3	0.4	3	6	2	9	no calc	no calc	no calc	4	2	no calc
FF276F	average	0.060	0.11	<0.4	35	39	<63	79	78	81	35	37	51	59	49	74	<1.6	<3.5	BND	65	60	77
	SD	0.002	0.01	no calc	2	1	no calc	2	7	17	2	3	5	1	2	7	no calc	no calc	no calc	5	5	8
Phase 2		<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1	<45 100:1	<250 100:1	<250 2000:1
FF208B	average	1.1	1.6	4.6	30.4	42	92	42.9	43	BND	29.8	43	66	17	15	43	<8.1	17	BND	31	36.8	33
	SD	0.5	0.2	4.0	0.5	2	no calc	0.7	1	no calc	0.4	5	2	3	1	4	no calc	1	no calc	2	0.8	3
CS102B	average	4.6	4.7	<51	11.8	16	BND	46	36	BND	28	23.6	45	7.5	7.3	36	SND	BND	BND	27	20	<93
	SD	2.0	1.4	no calc	0.5	1	no calc	5	4	no calc	3	0.9	9	0.8	0.7	4	no calc	no calc	no calc	4	1	no calc
FF276F	average	1.0	1.2	2.9	32	41	67	30	36	71	26	25	59	6.3	6	33	1.6	4.1	BND	23	21	37
	SD	0.1	0.3	0.5	3	8	no calc	2	7	no calc	0.7	2	5	0.4	1	2	0.1	0.4	no calc	1	2	5

SD = standard deviation; RSD = relative standard deviation = standard deviation/average x 100%

BND = % bioaccessibility cannot be calculated because the total concentration is less than the bioaccessible concentration.

SND = % bioaccessibility cannot be calculated becase the total concentration is less than the detection limit.

no calc = no calculation (2 or 3 replicates are undetectable.)

ANALYTICAL SCIENCES DIVISION

Environmental Sciences Group

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ANALYSIS REPORT COVER LETTER

Report Number: ASD0809a
Report Date: August 21, 2008
Sample(s) reported: 8
Issue Status: complete
Analysis commenced on: July 21, 2008

Sample and Analysis Details

All samples were received in good condition.

To obtain the bioaccessible metal and metalloid contents for the human receptor, a two phase (stomach and intestine) physiologically based extraction test (PBET) method was employed. The soils were sieved to <250 um fraction and a ratio of 100:1 extraction fluid:soil mass was used. To simulate stomach conditions a portion of each (0.2 g) was extracted at body temperature (37°C) in Teflon® extraction vessels with 20 ml of simulated gastric solution at pH 1.8 for 1 hour. To simulate gastric+intestinal conditions a portion of each sample was subjected to the stomach extraction described above, and then the conditions were altered to simulate those approximating the small intestine. Intestinal extraction conditions were applied for 4 hours. The filtered (0.45 um) resulting extracts were analyzed by ICP-OES (As, Cd, Cu, Pb, Se, Zn) and by pyrolysis-cold vapour generation-AAS (Hg).

Table 1. Summary of methods and instrumentation used.

Analytes	**Test Method**	**Sample Matrix**
As, Cd, Cu, Pb, Se, Zn	ICP-OES	Bioaccessibility (PBET)
Hg	Pyrolysis-CV-AAS	Bioaccessibility (PBET)

To calculate % bioaccessibility, the following equation was used for each element:

%Bioaccessibility = Bioaccessible Concentration (mg/kg)/Total Concentration (mg/kg) x 100%

Total metal and metalloid contents for the <250 um fraction were provided by the client. Neither ASD nor ASU reviewed the quality control results for the provided data, nor was it verified by either group.

Two spike results were slightly lower than the acceptable range (70-130%) (Hg Phase 1 and Pb Phase 2). The reason for the slightly lower Hg recovery (65%) is unknown and is being investigated. In all cases the higher % bioaccessibility was obtained in Phase 2 for Hg, where the spike recovery was acceptable. A slightly lower Pb spike recovery is not uncommon, since the Phase 2 conditions (pH 7) are not favourable to keeping dissolved Pb in solution.

Because of limitations with the current software used for reporting data, the number of significant figures quoted in the attached tables may not be representative of the actual uncertainty. Data should be considered accurate to no more than two significant figures.

This report is issued under complete status. All analyses requested for the first phase of the study have been completed and results are issued with full compliance of data verification.

Report authorised by: ……Iris Koch…… Date: …Aug 21, 2008

ESG Bioaccessibility Report ASD0809a

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 21 2008
Analysis Date: August 14 -15 2008
Report Date: August 21 2008
Method: PBET method <250 µm soil particle size 100:1

Results

Sample Name	Bioaccessible Hg (mg/kg)	Total Hg (mg/kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg/kg)	Total As (mg/kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg/kg)	Total Cd (mg/kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg/kg)	Total Cu (mg/kg)	% Bioaccessible Cu (%)
PHASE 1												
FF202F P1	0.634	168	0.38	15.1	51	30	22.7	26	86	1221	3290	37
FF206F P1	<0.2	184	<0.11	16.3	42	39	15.4	16	99	914	1880	49
FF209B P1*	<0.2	132	<0.15	33.5	97	34	17.8	20	90	1084	1790	61
FF229F P1	<0.2	320	<0.06	21.2	67	32	26.0	31	84	788	2530	31
FF231F P1	0.366	535	0.068	76.2	124	61	67.5	51	132	2222	3970	56
FF235B P1	<0.2	370	<0.05	85.9	169	51	38.3	50	77	1625	3850	42
FF271F P1	0.191	330	0.058	22.2	99	22	46.3	50	93	2523	5530	46
FF277F P1	<0.2	183	<0.11	127	222	57	30.6	37	84	1458	2940	50
PHASE 2												
FF202F P2	3.60	168	2.1	14.2	51	28	11.2	26	42	868	3290	26
FF206F P2	4.98	184	2.7	14.7	41.7	35	8.80	16	56	615	1880	33
FF209B P2*	6.11	132	4.6	31.9	97.3	33	10.2	20	52	777	1790	43
FF229F P2	4.69	320	1.5	19.7	67	29	10.0	31	32	444	2530	18
FF231F P2	8.93	535	1.7	62.9	124	51	23.6	51	46	1693	3970	43
FF235B P2	2.85	370	0.77	50.3	169	30	16.4	50	33	1338	3850	35
FF271F P2	7.58	330	2.3	22.9	98.9	23	22.4	50	45	1656	5530	30
FF277F P2	5.44	183	3.0	110	222	50	12.0	37	33	1022	2940	35

Sample Name	Bioaccessible Pb (mg/kg)	Total Pb (mg/kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg/kg)	Total Se (mg/kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg/kg)	Total Zn (mg/kg)	% Bioaccessible Zn (%)
PHASE 1									
FF202F P1	238	335	71	<10	42	<24	2395	4010	60
FF206F P1	153	199	77	<10	41	<24	2260	3270	69
FF209B P1*	223	281	80	<10	54	<19	3189	4350	73
FF229F P1	142	353	40	<10	76	<13	2699	4550	59
FF231F P1	481	599	80	<10	124	<8.1	13522	14900	91
FF235B P1	635	746	85	<10	98	<10	10114	14800	68
FF271F P1	468	726	64	<10	95	<11	5270	9220	57
FF277F P1	494	640	77	<10	66	<15	8610	14200	61
PHASE 2									
FF202F P2	33	335	10	<10	42	<24	952	4010	24
FF206F P2	22	199	11	<10	41	<24	1079	3270	33
FF209B P2*	36	281	13	<10	54	<19	1429	4350	33
FF229F P2	18	353	5.0	<10	76	<13	794	4550	17
FF231F P2	130	599	22	<10	124	<8.1	4855	14900	33
FF235B P2	95	746	13	<10	98	<10	2711	14800	18
FF271F P2	53	726	7.2	<10	95	<11	2104	9220	23
FF277F P2	107	640	17	<10	66	<15	3371	14200	24

* Average of the duplicate is reported

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.20	<5.0	<2.0	<1.0	<0.5	<10	<100
Blank P2	<0.20	<5.0	<2.0	<1.0	<0.5	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF209B P1	no calc	8.4	8.9	7.0	3.5	no calc	0.6
FF209B P2	0.5	8.3	7.3	1.6	41	no calc	2.4

no calc = at least value in the duplicate pair is below detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results (%)	no result	48	73	58	71	not certified	27
Accepted range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	yes	yes	yes	yes	NA	yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results (%)	no result	43	45	51	14	not certified	12
Accepted range (%)	not available	22-47	40-53	45-60	9.6-17	NA	10.0-17
Acceptable	NA	yes	yes	yes	yes	NA	yes
ESG control limits for NIST 2710 100:1							

NA = not applicable

Matrix Spikes

Hg P1 expected conc.		Hg P2 expected conc.	
0.044	ppm	0.050	ppm
Actual Hg concentration		Actual Hg conc.	
0.0287	ppm	0.0415	ppm
Recovery		Recovery	
65	%	83	%

QA/QC from ASU

	Hg	As	Cd	Cu	Pb	Se	Zn
Blank (m/L)	<2.0 ; <2.0 ; <2.0 ; <2.0	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	2.0 ; 2.0	4.3	4.2	8.2	42.3	3.2	15.6
Control Target (mg/L)	2.0	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	100; 100	108	106	102	106	105	104
Control (mg/L)	20.0 ; 20.4	NA	NA	NA	NA	NA	NA
Control Target (mg/L)	20	NA	NA	NA	NA	NA	NA
% Recovery	100; 102	NA	NA	NA	NA	NA	NA
Duplicates	**% RSD Hg**	**% RSD As**	**% RSD Cd**	**% RSD Cu**	**% RSD Pb**	**% RSD Se**	**% RSD Zn**
FF231F P1	3.7	NA	NA	NA	NA	NA	NA
FF229F P2	5.9	NA	NA	NA	NA	NA	NA
FF235B P2	3.1	NA	NA	NA	NA	NA	NA
FF277F P2	0.1	NA	NA	NA	NA	NA	NA

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ANALYTICAL SCIENCES DIVISION

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ANALYSIS REPORT COVER LETTER

Report Number: ASD0809b, c, d, e, f
Report Date: Sept 3, 2008
Sample(s) reported: 47
Issue Status: complete
Analysis commenced on: July 23, 2008

Sample and Analysis Details

All samples were received in good condition.

To obtain the bioaccessible metal and metalloid contents for the human receptor, a two phase (stomach and intestine) physiologically based extraction test (PBET) method was employed. The soils were sieved to <250 um fraction and a ratio of 100:1 extraction fluid:soil mass was used. To simulate stomach conditions a portion of each (0.2 g for samples in reports ASD0809d, e, f; 0.6 g for reports ASD0809b, c) was extracted at body temperature (37°C) in Teflon® extraction vessels with 20 ml (ASD0809d, e, f) or in glass jars with 60 ml (ASD0809b, c) of simulated gastric solution at pH 1.8 for 1 hour. To simulate gastric+intestinal conditions a portion of each sample was subjected to the stomach extraction described above, and then the conditions were altered to simulate those approximating the small intestine. Intestinal extraction conditions were applied for 4 hours. The filtered (0.45 um) resulting extracts were analyzed by ICP-OES (As, Cd, Cu, Pb, Se, Zn) and by pyrolysis-cold vapour generation-AAS (Hg) (reports d, e, f). For samples in reports ASD0809b and ASD0809c, Phase 2 (gastric+intestinal) extracts were digested and then analyzed for Hg by cold vapour generation AFS.

Table 1. Summary of methods and instrumentation used.

Analytes	**Test Method**	**Sample Matrix**
As, Cd, Cu, Pb, Se, Zn	ICP-OES	Bioaccessibility (PBET)
Hg (total Hg>7 ppm)	Pyrolysis-CV-AAS	Bioaccessibility (PBET)
Hg (total Hg<7 ppm)	CV-AFS	Bioaccessibility (PBET), phase 2 extracts digested

To calculate % bioaccessibility, the following equation was used for each element:

%Bioaccessibility = Bioaccessible Concentration (mg/kg)/Total Concentration (mg/kg) x 100%

Total metal and metalloid contents for the <250 um fraction were provided by the client. Neither ASD nor ASU reviewed the quality control results for the provided data, nor was it verified by either group.

Several blank results were detectable (Pb in ASD0809b, Cu and Pb in ASD0809d, Cd, Cu and Pb in ASD0809e, and Cu in ASD0809f) but in most cases the values were at the limit of detection or barely above it. Since values of Pb, Cu and Cd in samples are well above detection we do not consider sample analyses to be contaminated.

Within-lab repeatability (%RSDs for duplicates) was generally good but in some cases %RSDs were greater than limits usually considered acceptable (40%) (Cu and Pb in ASD0809b, Pb in ASD0809c). In all cases this occurred in Phase 2 extracts and is attributable both to sample heterogeneity and the inconsistent nature of precipitation reactions thought to be occurring in Phase 2 for these elements (Cu and Pb).

A standard reference soil was included in each batch, for which control limits have been established in our lab previously. In most batches results for all elements were within the control limits; the exceptions were Phase 2 results for Cd, Pb and Zn in report ASD0809d and Pb and Zn in ASD0809f, which were slightly low, and Phase 1 results for As, Cu, Pb and Zn in report ASD0809f, which were slightly high. Because of the preliminary nature of the control limits it is possible that they could be expanded for future use. The sample analyses are considered to be in control since most of the non-conformances were no more than 40% from the control means.

Hg spike results are included in the absence of available control limits for Hg in the 2710 control sample. One spike was lost to vial breakage (Hg P2 in ASD0809b). Three Phase 2 Hg spike results were lower than the acceptable range (70-130%) (ASD0809c, e, f). The reason for the lower recoveries (35%, 62% and 53%) is unknown and is being investigated; it may be related to adsorption/precipitation reactions, resulting from the hypothesized association of Hg with the increased organic components (bile extract, pancreatin) in this phase.

In some cases greater than 100% bioaccessibility was obtained for samples; these values should be considered to be 100% bioaccessible. Sample heterogeneity (differences in element concentrations in subsamples used in

bioaccessibility extracts and total element analysis) is the likely cause of such results, and in most cases the results are within the uncertainty associated with laboratory analysis (30%).

Because of limitations with the current software used for reporting data, the number of significant figures quoted in the attached tables may not be representative of the actual uncertainty. Data should be considered accurate to no more than two significant figures.

This report is issued under complete status. All analyses requested for the first phase of the study have been completed and results are issued with full compliance of data verification.

Report authorised by: ……Iris Koch……. Date: …Sept 3, 2008

ESG Bioaccessibility Report ASD0809b

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 23 3008
Analysis Date: August 12, 22 2008
Report Date: August 26 2008
Method: PBET method, liquid to solid ratio 100:1 and <250 µm soil particle size

Results

Sample Name	Bioaccessible Hg (mg/kg)	Total Hg (mg/kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg/kg)	Total As (mg/kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg/kg)	Total Cd (mg/kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg/kg)	Total Cu (mg/kg)	% Bioaccessible Cu (%)
PHASE 1												
FF402F P1	0.024	0.31	7.6	<5.0	8.9	<56	2.0	2.9	70	51	118	43
FF408G P1*	0.0081	0.46	1.8	<5.0	7.6	<66	2.0	2.3	87	21	95	22
FF402G P1	0.013	0.55	2.4	6.5	16	42	5.0	5.3	96	59	195	30
CS112G P1	0.036	0.60	6.1	5.1	19	27	5.1	3.8	132	47	141	33
FF324B P1	0.011	1.2	0.94	<5.0	13	<38	7.2	7.9	91	141	389	36
FF401B P1	0.0094	1.2	0.77	<5.0	12	<42	3.1	3.9	78	69	165	42
FF402B P1	0.014	2.4	0.60	5.1	25	20	12	13	96	169	468	36
CS114F P1	0.030	2.6	1.2	5.6	34	16	23	28	84	198	1090	18
PHASE 2												
FF402F P2	0.018	0.31	5.7	<5.0	8.9	<56	<2.0	2.9	<69	48	118	41
FF408G P2*	0.013	0.46	2.9	7.1	7.6	93	<2.0	2.3	<87	66	95	69
FF402G P2	0.020	0.55	3.7	7.4	16	48	2.1	5.3	40	71	195	37
CS112G P2	0.065	0.60	11	6.5	19	35	<2.0	3.8	<53	55	141	39
FF324B P2	0.068	1.2	5.6	<5.0	13	<38	2.0	7.9	25	124	389	32
FF401B P2	0.061	1.2	5.0	<5.0	12	<42	2.1	3.9	54	70	165	43
FF402B P2	0.10	2.4	4.1	7.9	25	31	6.4	13	50	155	468	33
CS114F P2	0.076	2.6	2.9	6.1	34	18	6.1	28	22	194	1090	18

Sample Name	Bioaccessible Pb (mg/kg)	Total Pb (mg/kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg/kg)	Total Se (mg/kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg/kg)	Total Zn (mg/kg)	% Bioaccessible Zn (%)
PHASE 1									
FF402F P1	48	56	85	<10	0.3	BND	245	515	48
FF408G P1*	21	31	68	<10	0.7	BND	233	438	53
FF402G P1	61	107	57	<10	1.1	BND	698	1020	68
CS112G P1	1048	1490	70	<10	5.8	BND	346	350	99
FF324B P1	51	56	92	<10	1.4	BND	371	567	65
FF401B P1	63	74	85	<10	0.8	BND	309	655	47
FF402B P1	122	226	54	<10	2.3	BND	2073	2960	70
CS114F P1	77	200	38	<10	5.6	BND	1019	1270	80
PHASE 2									
FF402F P2	7.3	56	13	<10	0.3	BND	99	515	19
FF408G P2*	23	31	72	<10	0.7	BND	<100	438	<23
FF402G P2	6.4	107	6.0	<10	1.1	BND	254	1020	25
CS112G P2	131	1490	8.8	<10	5.8	BND	<100	350	<29
FF324B P2	5.9	56	11	<10	1.4	BND	<100	567	<18
FF401B P2	9.5	74	13	<10	0.8	BND	112	655	17
FF402B P2	20	226	8.9	<10	2.3	BND	844	2960	29
CS114F P2	7.1	200	3.6	<10	5.6	BND	173	1270	14

*average of duplicate pair reported

BND = total initial concentration is less than the detection limit of bioaccessible fraction, therefore % bioaccessibility cannot be calculated

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.005	<5.0	<2.0	<1.0	<0.5	<10	<100
Blank P2	<0.008	<5.0	<2.0	<1.0	0.5	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF408G P1	31	no calc	0	7	7	no calc	3
FF408G P2	17	no calc	no calc	74	135	no calc	no calc

bioaccessible concentrations used for the RSD calculations

no calc = one or both duplicates are less than detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results	no result	42%	70%	61%	66%	not certified	26%
Accepted range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	yes	yes	yes	yes	NA	yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results	no result	36%	43%	57%	10%	not certified	10%
Accepted range (%)	not available	22-47	40-53	45-60	9.6-17	NA	10.0-17
Acceptable	NA	yes	yes	yes	yes	NA	yes

ESG control limits for NIST 2710 100:1

Matrix Spikes

Hg P1 expected conc.	Hg P2 expected conc.
0.054 ppm	vial lost
Actual Hg concentration	Actual Hg concentration
0.041 ppm	vial lost
Recovery	Recovery
76 %	vial lost

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<0.01;<0.01; <0.01; <0.01	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	0.006; 0.006; 0.005; 0.005; 0.005; 0.006	4.2	4.0	7.7	40.1	3.0	15.4
Control Target (mg/L)	0.005	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	100 -110	105	100	96	100	100	103
Control (mg/L)	0.061; 0.064; 0.082;0.077; 0.072; 0.072	4.0	3.8	7.4	38	2.9	14
Control Target (mg/L)	0.075	4.0	4.0	8.0	40	3.0	15
% Recovery	81 - 109	100	95	93	95	97	94
Duplicates	**% RSD Hg**	**% RSD As**	**% RSD Cd**	**% RSD Cu**	**% RSD Pb**	**% RSD Se**	**% RSD Zn**
FF312B P1	0.0	NA	NA	NA	NA	NA	NA
FF408B DUP P2	3.2	NA	NA	NA	NA	NA	NA

ESG Bioaccessibility Report ASD0809c

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 25 3008
Analysis Date: August 12, 22 2008
Report Date: September 03 2008
Method: PBET method, liquid to solid ratio 100:1 and <250 µm soil particle size

Results

Sample Name	Bioaccessible Hg (mg/kg)	Total Hg (mg/kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg/kg)	Total As (mg/kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg/kg)	Total Cd (mg/kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg/kg)	Total Cu (mg/kg)	% Bioaccessible Cu (%)
PHASE 1												
FF410 P1	0.037	3.4	1.1	4.4	36	12	8.9	14	62	258	630	41
FF408B P1*	0.028	4.0	0.70	<5.0	14	<36	7.4	9.3	80	112	284	39
CS111F P1	0.038	4.8	0.78	6.4	36	18	6.9	13	54	209	720	29
FF408F P1	0.019	4.8	0.40	<5.0	17	<29	7.8	15	50	94	540	17
FF221F P1	0.040	5.0	0.80	4.9	10	48	4.9	4.0	124	182	335	54
FF404F P1	0.036	5.5	0.66	<5.0	17	<29	14	21	68	108	660	16
FF310F P1	0.031	6.1	0.50	4.3	17	25	11	11	103	211	620	34
FF312B P1	0.020	6.6	0.30	<5.0	14	<36	20	19	104	404	989	41
PHASE 2												
FF410 P2	0.18	3.4	5.4	7.1	36	20	6.1	14	43	206	630	33
FF408B P2*	0.052	4.0	1.3	<5.0	14	<36	5.1	9.3	55	118	284	41
CS111F P2	0.25	4.8	5.3	11	36	31	5.1	13	40	184	720	25
FF408F P2	0.038	4.8	0.79	<5.0	17	<29	5.0	15	33	110	540	20
FF221F P2	0.21	5.0	4.2	7.2	10	70	2.9	4.0	73	171	335	51
FF404F P2	0.046	5.5	0.83	5.1	17	30	4.0	21	20	119	660	18
FF310F P2	0.059	6.1	1.0	5.4	17	32	5.9	11	53	185	620	30
FF312B P2	0.078	6.6	1.2	<5.0	14	<36	8.0	19	42	288	989	29

Sample Name	Bioaccessible Pb (mg/kg)	Total Pb (mg/kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg/kg)	Total Se (mg/kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg/kg)	Total Zn (mg/kg)	% Bioaccessible Zn (%)
PHASE 1									
FF410 P1	100	183	55	<10	3.0	BND	1355	3350	40
FF408B P1*	73	90	81	<10	1.9	BND	906	1310	69
CS111F P1	64	154	42	<10	4.2	BND	243	476	51
FF408F P1	67	163	41	<10	3.1	BND	1324	3000	44
FF221F P1	31	42	75	<10	1.8	BND	331	484	68
FF404F P1	53	136	39	<10	3.0	BND	725	1190	61
FF310F P1	196	111	177	<10	3.6	BND	2228	3350	67
FF312B P1	118	164	72	<10	3.6	BND	1367	1640	83
PHASE 2									
FF410 P2	11	183	6.1	<10	3.0	BND	544	3350	16
FF408B P2*	24	90	27	<10	1.9	BND	410	1310	31
CS111F P2	15	154	10	<10	4.2	BND	96	476	20
FF408F P2	16	163	9.8	<10	3.1	BND	622	3000	21
FF221F P2	4.8	42	11	<10	1.8	BND	127	484	26
FF404F P2	5.1	136	3.7	<10	3.0	BND	116	1190	10
FF310F P2	22	111	19	<10	3.6	BND	576	3350	17
FF312B P2	16	164	9.8	<10	3.6	BND	405	1640	25

* average of duplicate pair

BND = total initial concentration is less than the detection limit of bioaccessible fraction, therefore % bioaccessibility cannot be calculated

QA/QC Experimental

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.005	<5.0	<2.0	<1.0	<0.5	<10	<100
Blank P2	<0.008	<5.0	<2.0	<1.0	<0.5	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF408B P1	2.0	no calc	8.7	4.8	4.8	no calc	8.9
FF408B P2	17	no calc	2.6	0.2	53	no calc	1.0

no calc = RSD % could not be calculated since at least one value was below the detection limit

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results	no result	37	63	55	60	not certified	23
Accepted range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	yes	yes	yes	yes	NA	yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results	no result	40	46	49	12	not certified	12
Accepted range (%)	not available	22-47	40-53	45-60	9.6-17	NA	10.0-17
Acceptable	NA	yes	yes	yes	yes	NA	yes

ESG control limits for NIST 2710 100:1

Matrix Spikes

Hg P1 expected conc.	Hg P2 expected conc.
0.054 ppm	0.054 ppm
Actual Hg concentration	Actual Hg concentration
0.0543 ppm	0.019 ppm
Recovery	Recovery
101 %	35 %

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<0.01; <0.01; <0.01;<0.01; <0.01; <0.01	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	0.006; 0.006; 0.005; 0.005; 0.005; 0.006	4.2	4.0	7.7	40	3.0	15
Control Target (mg/L)	0.005	4.0	4.0	8.0	40	3.0	15
% Recovery	100 - 110	105	100	96	100	100	103
Control (mg/L)	0.061; 0.064; 0.082;0.077; 0.072; 0.072	4.0	3.8	7.4	38	2.9	14
Control Target (mg/L)	0.075	4.0	4.0	8.0	40	3.0	15
% Recovery	81 - 109	100	95	93	95	97	94
Duplicates	**% RSD Hg**	**% RSD As**	**% RSD Cd**	**% RSD Cu**	**% RSD Pb**	**% RSD Se**	**% RSD Zn**
FF312B P1	0.0	NA	NA	NA	NA	NA	NA
FF408B DUP P2	3.2	NA	NA	NA	NA	NA	NA

ESG Bioaccessibility Report ASD0809d

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 28 2008
Analysis Date: August 18, Sept 02, 2008
Report Date: September 03 2008
Method: PBET method <250 µm soil particle size 100:1

Results

Sample Name	Bioaccessible Hg (mg -kg)	Total Hg (mg -kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg -kg)	Total As (mg -kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg -kg)	Total Cd (mg -kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg -kg)	Total Cu (mg -kg)	% Bioaccessible Cu (%)
PHASE 1												
CS104F P1	<0.2	38.8	<0.5	27	314	8.6	16	20.5	76	546	1270	43
FF201F P1	<0.2	45	<0.4	<5.0	18.9	<26	5.8	7.22	81	232	640	36
FF206B P1	<0.2	100	<0.2	23	49.2	46	20	21	97	863	1610	54
FF210B P1	<0.2	101	<0.2	19	88.5	21	20	26.8	76	905	2120	43
FF225B P1 *	<0.2	90.0	<0.2	28	77.9	36	21	24	89	752	1830	41
FF239F P1	<0.2	127	<0.2	27	104	26	49	57.9	85	1614	3770	43
FF258F P1	<0.2	42	<0.5	27	139	19	26	35.1	73	936	2310	41
FF278F P1	<0.2	71	<0.3	16	138	12	16	25.4	64	865	2920	30
PHASE 2												
CS104F P2	1.8	38.8	4.7	48	314	15	3.9	20.5	19	482	1270	38
FF201F P2	0.71	45	1.6	<5.0	18.9	<26	2.9	7.22	41	186	640	29
FF206B P2	4.2	100	4.2	24	49.2	48	11	21	52	645	1610	40
FF210B P2	2.9	101	2.8	35	88.5	40	16	26.8	59	700	2120	33
FF225B P2 *	2.0	90	2.2	26	77.9	34	14	24	57	704	1830	38
FF239F P2	3.1	127	2.4	28	104	27	23	57.9	40	1135	3770	30
FF258F P2	1.3	42	3.0	29	139	21	15	35.1	41	693	2310	30
FF278F P2	3.0	71	4.2	23	138	17	11	25.4	42	767	2920	26

Sample Name	Bioaccessible Pb (mg -kg)	Total Pb (mg -kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg -kg)	Total Se (mg -kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg -kg)	Total Zn (mg -kg)	% Bioaccessible Zn (%)
PHASE 1									
CS104F P1	101	394	26	<10	20.4	<49	743	1690	44
FF201F P1	47	81.5	57	<10	10.2	<98	995	1280	78
FF206B P1	135	209	65	<10	32.4	<31	3258	4410	74
FF210B P1	266	420	63	<10	26.5	<38	2491	4260	58
FF225B P1 *	211	274	77	<10	28.4	<35	3053	4520	68
FF239F P1	407	638	64	<10	43.3	<23	6035	8780	69
FF258F P1	269	476	56	<10	30.5	<33	3977	7210	55
FF278F P1	219	461	47	<10	25.2	<40	2328	5670	41
PHASE 2									
CS104F P2	31	394	7.9	<10	20.4	<49	160	1690	9.5
FF201F P2	4.9	81.5	6.0	<10	10.2	<98	315	1280	25
FF206B P2	18	209	8.5	<10	32.4	<31	1478	4410	34
FF210B P2	31	420	7.5	<10	26.5	<38	1693	4260	40
FF225B P2 *	36	274	13	<10	28.4	<35	1215	4520	27
FF239F P2	41	638	6.4	<10	43.3	<23	2228	8780	25
FF258F P2	22	476	4.6	<10	30.5	<33	1739	7210	24
FF278F P2	28	461	6.1	<10	25.2	<40	1194	5670	21

* Average of the duplicate is reported

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.20	<5.0	<2.0	<1.0	1.0	<10	<100
Blank P2	<0.20	<5.0	<2.0	2.0	5.0	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF209B P1	no calc	0.8	0.8	2.7	0.2	no calc	0.3
FF209B P2	11	8.6	15	27	36	no calc	13

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results (%)	no result	46	75	65	71	not certified	28
Accepted Range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	Yes	Yes	Yes	Yes	NA	Yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results (%)	12	36	36	54	9.2	not certified	7.5
Average	not available	22-47	40-53	45-60	9.6-17	NA	10-16
Acceptable	NA	Yes	No	Yes	No	NA	No

ESG control limits for NIST 2710 100:1

NA = not applicable

Matrix Spikes

Hg P1 expected conc. 0.047 ppm	Hg P2 expected conc. 0.042 ppm
Actual Hg concentration 0.0378 ppm	Actual Hg concentration 0.0327 ppm
Recovery 80 %	Recovery 78 %

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<2.0 , <2.0	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	2.0 ; 2.0	4.3	3.9	7.8	40.0	3.2	14.8
Control Target (mg/L)	2.0	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	100, 100	107	99	98	100	107	99
Control (mg/L)	18.8 ; 19.3	n/a	n/a	n/a	n/a	n/a	n/a
Control Target (mg/L)	20	n/a	n/a	n/a	n/a	n/a	n/a
% Recovery	94, 96.5	n/a	n/a	n/a	n/a	n/a	n/a
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
CS104F P1	no calc	n/a	n/a	n/a	n/a	n/a	n/a
FF278F P1	no calc	n/a	n/a	n/a	n/a	n/a	n/a
FF201F P2	2.0	n/a	n/a	n/a	n/a	n/a	n/a
FF278F P2	0.0	n/a	n/a	n/a	n/a	n/a	n/a

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

ESG Bioaccessibility Report ASD0809d

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 28 2008
Analysis Date: August 18, Sept 02, 2008
Report Date: September 03 2008 **AMENDED REPORT**
Method: PBET method <250 µm soil particle size 100:1

Results

Sample Name	Bioaccessible Hg (mg -kg)	Total Hg (mg -kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg -kg)	Total As (mg -kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg -kg)	Total Cd (mg -kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg -kg)	Total Cu (mg -kg)	% Bioaccessible Cu (%)
PHASE 1												
CS104F P1	<0.2	38.8	<0.5	27	314	8.6	16	20.5	76	546	1270	43
FF201F P1	<0.2	45	<0.4	<5.0	18.9	<26	5.8	7.22	81	232	640	36
FF206B P1	<0.2	100	<0.2	23	49.2	46	20	21	97	863	1610	54
FF210B P1	<0.2	101	<0.2	19	88.5	21	20	26.8	76	905	2120	43
FF225B P1 *	<0.2	90.0	<0.2	28	77.9	36	21	24	89	752	1830	41
FF239F P1	<0.2	127	<0.2	27	104	26	49	57.9	85	1614	3770	43
FF258F P1	<0.2	42	<0.5	27	139	19	26	35.1	73	936	2310	41
FF278F P1	<0.2	71	<0.3	16	138	12	16	25.4	64	865	2920	30
PHASE 2												
CS104F P2	1.8	38.8	4.7	48	314	15	3.9	20.5	19	482	1270	38
FF201F P2	0.71	45	1.6	<5.0	18.9	<26	2.9	7.22	41	186	640	29
FF206B P2	4.2	100	4.2	24	49.2	48	11	21	52	645	1610	40
FF210B P2	2.9	101	2.8	35	88.5	40	16	26.8	59	700	2120	33
FF225B P2 *	2.0	90	2.2	26	77.9	34	14	24	57	704	1830	38
FF239F P2	3.1	127	2.4	28	104	27	23	57.9	40	1135	3770	30
FF258F P2	1.3	42	3.0	29	139	21	15	35.1	41	693	2310	30
FF278F P2	3.0	71	4.2	23	138	17	11	25.4	42	767	2920	26

Sample Name	Bioaccessible Pb (mg -kg)	Total Pb (mg -kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg -kg)	Total Se (mg -kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg -kg)	Total Zn (mg -kg)	% Bioaccessible Zn (%)
PHASE 1									
CS104F P1	101	394	26	<10	20.4	<49	743	1690	44
FF201F P1	47	81.5	57	<10	10.2	<98	995	1280	78
FF206B P1	135	209	65	<10	32.4	<31	3258	4410	74
FF210B P1	266	420	63	<10	26.5	<38	2491	4260	58
FF225B P1 *	211	274	77	<10	28.4	<35	3053	4520	68
FF239F P1	407	638	64	<10	43.3	<23	6035	8780	69
FF258F P1	269	476	56	<10	30.5	<33	3977	7210	55
FF278F P1	219	461	47	<10	25.2	<40	2328	5670	41
PHASE 2									
CS104F P2	31	394	7.9	<10	20.4	<49	160	1690	9.5
FF201F P2	4.9	81.5	6.0	<10	10.2	<98	315	1280	25
FF206B P2	18	209	8.5	<10	32.4	<31	1478	4410	34
FF210B P2	31	420	7.5	<10	26.5	<38	1693	4260	40
FF225B P2 *	36	274	13	<10	28.4	<35	1215	4520	27
FF239F P2	41	638	6.4	<10	43.3	<23	2228	8780	25
FF258F P2	22	476	4.6	<10	30.5	<33	1739	7210	24
FF278F P2	28	461	6.1	<10	25.2	<40	1194	5670	21

* Average of the duplicate is reported

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.20	<5.0	<2.0	<1.0	1.0	<10	<100
Blank P2	<0.20	<5.0	<2.0	2.0	5.0	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF225B P1	no calc	0.8	0.8	2.7	0.2	no calc	0.3
FF225B P2	11	8.6	15	27	36	no calc	13

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results (%)	no result	46	75	65	71	not certified	28
Accepted Range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	Yes	Yes	Yes	Yes	NA	Yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results (%)	12	36	36	54	9.2	not certified	7.5
Average	not available	22-47	40-53	45-60	9.6-17	NA	10-16
Acceptable	NA	Yes	No	Yes	No	NA	No

ESG control limits for NIST 2710 100:1

NA = not applicable

Matrix Spikes

Hg P1 expected conc. 0.047 ppm	Hg P2 expected conc. 0.042 ppm
Actual Hg concentration 0.0378 ppm	Actual Hg concentration 0.0327 ppm
Recovery 80 %	Recovery 78 %

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<2.0 , <2.0	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	2.0 ; 2.0	4.3	3.9	7.8	40.0	3.2	14.8
Control Target (mg/L)	2.0	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	100, 100	107	99	98	100	107	99
Control (mg/L)	18.8 ; 19.3	n/a	n/a	n/a	n/a	n/a	n/a
Control Target (mg/L)	20	n/a	n/a	n/a	n/a	n/a	n/a
% Recovery	94, 96.5	n/a	n/a	n/a	n/a	n/a	n/a
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
CS104F P1	no calc	n/a	n/a	n/a	n/a	n/a	n/a
FF278F P1	no calc	n/a	n/a	n/a	n/a	n/a	n/a
FF201F P2	2.0	n/a	n/a	n/a	n/a	n/a	n/a
FF278F P2	0.0	n/a	n/a	n/a	n/a	n/a	n/a

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

ESG Bioaccessibility Report ASD0809e

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 30 2008
Analysis Date: August 14 -15, Sept 02, 2008
Report Date: September 03 2008
Method: PBET method <250 µm soil particle size 100:1

Results

Sample Name	Bioaccessible Hg (mg/kg)	Total Hg (mg/kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg/kg)	Total As (mg/kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg/kg)	Total Cd (mg/kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg/kg)	Total Cu (mg/kg)	% Bioaccessible Cu (%)
PHASE 1												
CS108F P1	<0.2	30.5	<0.66	12	97.3	13	14	20.1	68	560	1200	47
CS125F P1	<0.2	20.7	<0.97	19	152	13	20	31.8	63	644	1800	36
CS129F P1	<0.2	19.8	<1.0	29	220	13	6.8	12.2	56	635	1320	48
FF232F P1	0.74	25	3.0	13	33.9	37	14	12.2	111	840	1290	65
FF254F P1	<0.2	22	<0.91	48	142	34	32	46.7	68	964	2190	44
FF341F P1	<0.2	32	<0.63	7.3	34.2	21	24	30.5	80	622	1770	35
FF343F P1 *	<0.2	21.4	<0.93	16	49.7	33	23	30.4	77	739	1640	45
FF344F P1	<0.2	17.9	<1.1	6.2	29	21	25	30.9	81	905	1950	46
PHASE 2												
CS108F P2	0.96	30.5	3.1	14	97.3	14	8.1	20.1	40	328	1200	27
CS125F P2	0.50	20.7	2.4	26	152	17	12	31.8	37	391	1800	22
CS129F P2	1.8	19.8	9.0	50	220	23	3.1	12.2	26	432	1320	33
FF232F P2	0.74	25	3.0	14	33.9	41	7.2	12.2	59	510	1290	40
FF254F P2	0.76	22	3.5	68	142	48	21	46.7	45	679	2190	31
FF341F P2	0.26	32	0.82	9.5	34.2	28	12	30.5	38	449	1770	25
FF343F P2 *	0.32	21.4	1.5	17	49.7	35	14	30.4	45	474	1640	29
FF344F P2	<0.2	17.9	<1.1	7.1	29	24	12	30.9	39	501	1950	26

Sample Name	Bioaccessible Pb (mg/kg)	Total Pb (mg/kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg/kg)	Total Se (mg/kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg/kg)	Total Zn (mg/kg)	% Bioaccessible Zn (%)
PHASE 1									
CS108F P1	165	287	57	<10	13	<77	1514	2810	54
CS125F P1	219	456	48	<10	15	<67	2103	4660	45
CS129F P1	201	392	51	<10	16.3	<61	389	1600	24
FF232F P1	112	121	93	<10	7.6	BND	1321	1520	87
FF254F P1	558	820	68	<10	18.1	<55	5683	9310	61
FF341F P1	202	357	56	<10	11.6	<86	4491	5650	79
FF343F P1 *	350	552	63	<10	10.4	<96	6355	8240	77
FF344F P1	275	380	72	<10	11	<91	3532	5400	65
PHASE 2									
CS108F P2	33	287	12	<10	13	<77	779	2810	28
CS125F P2	25	456	5.6	<10	15	<67	987	4660	21
CS129F P2	32	392	8.3	<10	16.3	<61	132	1600	8.2
FF232F P2	19	121	15	<10	7.6	BND	584	1520	38
FF254F P2	67	820	8.1	<10	18.1	<55	2274	9310	24
FF341F P2	29	357	8.3	<10	11.6	<86	1346	5650	24
FF343F P2 *	58	552	10	<10	10.4	<96	2668	8240	32
FF344F P2	21	380	5.6	<10	11	<91	1200	5400	22

* Average of the duplicate is reported

BND = total initial concentration is less than the detection limit of bioaccessible fraction, therefore % bioaccessibility cannot be calculated

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.2	<5.0	<2.0	<1.0	<0.5	<10	<100
Blank P2	<0.2	<5.0	2	1	1	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF209B P1	no calc	0.5	6.1	6.8	3.1	no calc	2.0
FF209B P2	10	0.6	2.7	2.1	8.8	no calc	20

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results (%)	no result	48	70	67	71	not certified	29
Accepted range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	yes	yes	yes	yes	NA	yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results (%)	no result	46	46	60	14	not certified	12
Accepted range (%)	not available	22-47	40-53	45-60	9.6-17	NA	10.0-17
Acceptable	NA	yes	yes	yes	yes	NA	yes

ESG control limits for NIST 2710 100:1

NA = not applicable

Matrix Spikes

Hg P1 expected conc. 0.043 ppm	Hg P2 expected conc. 0.037 ppm
Actual Hg concentration 0.031 ppm	Actual Hg concentration 0.023 ppm
% Recovery 72 %	% Recovery 62 %

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<2.0 , <2.0	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	1.8 , 1.9	4.3	3.9	7.8	40.0	3.2	14.8
Control Target (mg/L)	2.0	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	90, 95	107	99	98	100	107	99
Control (mg/L)	18.1, 17.1	4.5	4.09	8.14	41.95	3.29	15.65
Control Target (mg/L)	20	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	91, 86	112	102	102	105	110	104
Duplicates	**% RSD Hg**	**% RSD As**	**% RSD Cd**	**% RSD Cu**	**% RSD Pb**	**% RSD Se**	**% RSD Zn**
CS129F P2	no calc	NA	NA	NA	NA	NA	NA

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

ESG Bioaccessibility Report ASD0809e

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 30 2008
Analysis Date: August 14 -15, Sept 02, 2008
Report Date: September 03 2008 **AMENDED REPORT**
Method: PBET method <250 µm soil particle size 100:1

Results

Sample Name	Bioaccessible Hg (mg/kg)	Total Hg (mg/kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg/kg)	Total As (mg/kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg/kg)	Total Cd (mg/kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg/kg)	Total Cu (mg/kg)	% Bioaccessible Cu (%)
PHASE 1												
CS108F P1	<0.2	30.5	<0.66	12	97.3	13	14	20.1	68	560	1200	47
CS125F P1	<0.2	20.7	<0.97	19	152	13	20	31.8	63	644	1800	36
CS129F P1	<0.2	19.8	<1.0	29	220	13	6.8	12.2	56	635	1320	48
FF232F P1	0.74	25	3.0	13	33.9	37	14	12.2	111	840	1290	65
FF254F P1	<0.2	22	<0.91	48	142	34	32	46.7	68	964	2190	44
FF341F P1	<0.2	32	<0.63	7.3	34.2	21	24	30.5	80	622	1770	35
FF343F P1 *	<0.2	21.4	<0.93	16	49.7	33	23	30.4	77	739	1640	45
FF344F P1	<0.2	17.9	<1.1	6.2	29	21	25	30.9	81	905	1950	46
PHASE 2												
CS108F P2	0.96	30.5	3.1	14	97.3	14	8.1	20.1	40	328	1200	27
CS125F P2	0.50	20.7	2.4	26	152	17	12	31.8	37	391	1800	22
CS129F P2	1.8	19.8	9.0	50	220	23	3.1	12.2	26	432	1320	33
FF232F P2	0.74	25	3.0	14	33.9	41	7.2	12.2	59	510	1290	40
FF254F P2	0.76	22	3.5	68	142	48	21	46.7	45	679	2190	31
FF341F P2	0.26	32	0.82	9.5	34.2	28	12	30.5	38	449	1770	25
FF343F P2 *	0.32	21.4	1.5	17	49.7	35	14	30.4	45	474	1640	29
FF344F P2	<0.2	17.9	<1.1	7.1	29	24	12	30.9	39	501	1950	26

Sample Name	Bioaccessible Pb (mg/kg)	Total Pb (mg/kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg/kg)	Total Se (mg/kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg/kg)	Total Zn (mg/kg)	% Bioaccessible Zn (%)
PHASE 1									
CS108F P1	165	287	57	<10	13	<77	1514	2810	54
CS125F P1	219	456	48	<10	15	<67	2103	4660	45
CS129F P1	201	392	51	<10	16.3	<61	389	1600	24
FF232F P1	112	121	93	<10	7.6	BND	1321	1520	87
FF254F P1	558	820	68	<10	18.1	<55	5683	9310	61
FF341F P1	202	357	56	<10	11.6	<86	4491	5650	79
FF343F P1 *	350	552	63	<10	10.4	<96	6355	8240	77
FF344F P1	275	380	72	<10	11	<91	3532	5400	65
PHASE 2									
CS108F P2	33	287	12	<10	13	<77	779	2810	28
CS125F P2	25	456	5.6	<10	15	<67	987	4660	21
CS129F P2	32	392	8.3	<10	16.3	<61	132	1600	8.2
FF232F P2	19	121	15	<10	7.6	BND	584	1520	38
FF254F P2	67	820	8.1	<10	18.1	<55	2274	9310	24
FF341F P2	29	357	8.3	<10	11.6	<86	1346	5650	24
FF343F P2 *	58	552	10	<10	10.4	<96	2668	8240	32
FF344F P2	21	380	5.6	<10	11	<91	1200	5400	22

* Average of the duplicate is reported

BND = total initial concentration is less than the detection limit of bioaccessible fraction, therefore % bioaccessibility cannot be calculated

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.2	<5.0	<2.0	<1.0	<0.5	<10	<100
Blank P2	<0.2	<5.0	2	1	1	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF343B P1	no calc	0.5	6.1	6.8	3.1	no calc	2.0
FF343B P2	10	0.6	2.7	2.1	8.8	no calc	20

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results (%)	no result	48	70	67	71	not certified	29
Accepted range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	yes	yes	yes	yes	NA	yes
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results (%)	no result	46	46	60	14	not certified	12
Accepted range (%)	not available	22-47	40-53	45-60	9.6-17	NA	10.0-17
Acceptable	NA	yes	yes	yes	yes	NA	yes

ESG control limits for NIST 2710 100:1

NA = not applicable

Matrix Spikes

Hg P1 expected conc. 0.043 ppm	Hg P2 expected conc. 0.037 ppm
Actual Hg concentration 0.031 ppm	Actual Hg concentration 0.023 ppm
% Recovery 72 %	% Recovery 62 %

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<2.0 , <2.0	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	1.8 , 1.9	4.3	3.9	7.8	40.0	3.2	14.8
Control Target (mg/L)	2.0	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	90, 95	107	99	98	100	107	99
Control (mg/L)	18.1, 17.1	4.5	4.09	8.14	41.95	3.29	15.65
Control Target (mg/L)	20	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	91, 86	112	102	102	105	110	104
Duplicates	**% RSD Hg**	**% RSD As**	**% RSD Cd**	**% RSD Cu**	**% RSD Pb**	**% RSD Se**	**% RSD Zn**
CS129F P2	no calc	NA	NA	NA	NA	NA	NA

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

ESG Bioaccessibility Report ASD0809f

Site: Flin Flon
Analyst: Jessica Harris, Kim House (ESG)
Extraction Date: July 31 2008
Analysis Date: August 21, September 02 2008
Report Date: September 03 2008
Method: PBET method <250 µm soil particle size 100:1

Results

Sample Name	Bioaccessible Hg (mg/kg)	Total Hg (mg/kg)	% Bioaccessible Hg (%)	Bioaccessible As (mg/kg)	Total As (mg/kg)	% Bioaccessible As (%)	Bioaccessible Cd (mg/kg)	Total Cd (mg/kg)	% Bioaccessible Cd (%)	Bioaccessible Cu (mg/kg)	Total Cu (mg/kg)	% Bioaccessible Cu (%)
PHASE 1												
CS104B P1	<0.2	8.9	<2.2	35	285	12	21	22	96	668	1380	48
CS112B P1	<0.2	10.5	<1.9	8.5	60.4	14	18	20	91	434	960	45
FF302F P1	<0.2	7.4	<2.7	<5.0	17.4	<29	22	21	105	562	1100	51
FF305B P1*	<0.2	7.2	<2.8	<5.0	18	<28	13	15	88	408	1020	40
FF307F P1	<0.2	10.2	<2.0	<5.0	20	<25	27	27	97	547	1360	40
FF323F P1	<0.2	9.93	<2.0	6.5	27	24	17	20	85	501	970	52
FF406F P1	<0.2	7	<2.9	6.9	31	22	16	21	76	255	700	36
PHASE 2												
CS104B P2	0.19	8.9	2.2	39	285	14	6.8	22	31	514	1380	37
CS112B P2	<0.2	10.5	<1.9	17	60.4	27	8.3	20	42	376	960	39
FF302F P2	<0.2	7.4	<2.7	<5.0	17.4	<29	6.6	21	32	368	1100	33
FF305B P2*	0.21	7.2	2.9	<5.0	18	<28	3.7	15	25	292	1020	29
FF307F P2	<0.2	10.2	<2.0	<5.0	20	<25	9.1	27	33	393	1360	29
FF323F P2	<0.2	9.93	<2.0	5.9	27	22	6.9	20	35	386	970	40
FF406F P2	<0.2	7	<2.9	8.1	31.4	26	7.6	21	37	244	700	35

Sample Name	Bioaccessible Pb (mg/kg)	Total Pb (mg/kg)	% Bioaccessible Pb (%)	Bioaccessible Se (mg/kg)	Total Se (mg/kg)	% Bioaccessible Se (%)	Bioaccessible Zn (mg/kg)	Total Zn (mg/kg)	% Bioaccessible Zn (%)
PHASE 1									
CS104B P1	153	365	42	<10	15	<67	1290	1900	68
CS112B P1	165	249	66	<10	5.5	BND	550	707	78
FF302F P1	389	304	128	<10	5.1	BND	3525	4160	85
FF305B P1*	207	221	94	<10	6.0	BND	2501	3790	66
FF307F P1	250	275	91	<10	8.6	BND	4473	5490	81
FF323F P1	293	289	102	<10	6.3	BND	2208	3290	67
FF406F P1	203	266	76	<10	4.0	BND	3358	5680	59
PHASE 2									
CS104B P2	19	365	5.1	<10	15	<67	186	1900	10
CS112B P2	31	249	13	<10	5.5	BND	177	707	25
FF302F P2	17	304	5.6	<10	5.1	BND	569	4160	14
FF305B P2*	17	221	7.7	<10	6.0	BND	371	3790	9.8
FF307F P2	17	275	6.2	<10	8.6	BND	1025	5490	19
FF323F P2	31	289	11	<10	6.3	BND	502	3290	15
FF406F P2	18	266	6.8	<10	4.0	BND	1214	5680	21

*average of duplicate pair
BND = total initial concentration is less than the detection limit of bioaccessible fraction, therefore % bioaccessibility cannot be calculated

QA/QC

QC Samples	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg	Bioaccessible mg/kg
Blank	**[Hg]**	**[As]**	**[Cd]**	**[Cu]**	**[Pb]**	**[Se]**	**[Zn]**
Blank P1	<0.20	<5.0	<2.0	<1.0	<0.5	<10	<100
Blank P2	<0.20	<5.0	<2.0	1.0	<0.5	<10	<100
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
FF305B P1	no calc	no calc	5.3	4.3	2.1	no calc	8.8
FF305B P2	3.0	no calc	3.0	2.3	34	no calc	6.0

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

Control

	% BA Hg	% BA As	% BA Cd	% BA Cu	% BA Pb	% BA Se	% BA Zn
2710 P1 results	no result	60%	81%	77%	84%	not certified	33%
Accepted range (%)	not available	19-55	53-85	53-75	58-81	NA	23-30
Acceptable	NA	no	yes	no	no	NA	no
	% BA Hg	**% BA As**	**% BA Cd**	**% BA Cu**	**% BA Pb**	**% BA Se**	**% BA Zn**
2710 P2 results	8.1	47%	40%	59%	8.6%	not certified	8.3%
Accepted range (%)	not available	22-47	40-53	45-60	9.6-17	NA	10.0-17
Acceptable	NA	yes	yes	yes	no	NA	no

ESG control limits for NIST 2710 100:1

NA = not applicable

Matrix Spikes

Hg P1 expected conc. 0.0379 ppm	Hg P2 expected conc. 0.0453 ppm
Actual Hg concentration 0.0285 ppm	Actual Hg concentration 0.0238 ppm
Recovery 75 %	Recovery 53 %

QA/QC from ASU

	Hg (ug/L)	As	Cd	Cu	Pb	Se	Zn
Blank (mg/L)	<2.0, <2.0	<0.05	<0.02	<0.01	<0.005	<0.1	<1.0
Control (mg/L)	1.9 , 1.9	4.5	4.1	8.1	42.0	3.3	15.7
Control Target (mg/L)	2.0	4.0	4.0	8.0	40.0	3.0	15.0
% Recovery	95, 95	112	102	102	105	110	104
Control (mg/L)	18.0 , 17.7	8.1	4.1	42	16	4.5	3.3
Control Target (mg/L)	20	8.0	4.0	40	15	4.0	3.0
% Recovery	90, 89	102	102	105	104	112	110
Duplicates	**RSD % Hg**	**RSD % As**	**RSD % Cd**	**RSD % Cu**	**RSD % Pb**	**RSD % Se**	**RSD % Zn**
CS112B P1	no calc	n/a	n/a	n/a	n/a	n/a	n/a
FF406F P2	no calc	n/a	n/a	n/a	n/a	n/a	n/a

no calc = %RSD not calculated because at least one value in the duplicate pair is below detection

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ANALYTICAL SCIENCES DIVISION

Environmental Sciences Group

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Kingston, Ontario

K7K 7B4

ANALYSIS REPORT COVER LETTER

Report Number: ASD0819
Report Date: November 4, 2008
Sample(s) reported: 2 (reruns)
Issue Status: complete
Analysis commenced on: September 20, 2008

Sample and Analysis Details

Samples were obtained from leftover (previously sieved) samples from the original analysis.

To obtain the bioaccessible metal and metalloid contents for the human receptor, a two phase (stomach and intestine) physiologically based extraction test (PBET) method was employed. The soils previously sieved to <250 um fraction were subjected to a ratio of 100:1 extraction fluid:soil mass. To simulate stomach conditions a portion of each (0.2 g) was extracted at body temperature (37°C) in Teflon® extraction vessels with 20 ml of simulated gastric solution at pH 1.8 for 1 hour. To simulate gastric+intestinal conditions a portion of each sample was subjected to the stomach extraction described above, and then the conditions were altered to simulate those approximating the small intestine. Intestinal extraction conditions were applied for 4 hours. The filtered (0.45 um) resulting extracts were analyzed by ICP-OES (As, Cd, Cu, Pb, Se, Zn).

Table 1. Summary of methods and instrumentation used.

Analytes	**Test Method**	**Sample Matrix**
As, Cd, Cu, Pb, Se, Zn	ICP-OES	Bioaccessibility (PBET)
As, Cd, Cu, Pb, Se, Zn	Aqua regia digestion – ICP-OES	Soil

To calculate % bioaccessibility, the following equation was used for each element:

%Bioaccessibility = Bioaccessible Concentration (mg/kg)/Total Concentration (mg/kg) x 100%

Total metal and metalloid contents for the <250 um fraction were analyzed by ASU. Differences were observed for most elements, with an average relative percent difference (RPD[1]) of 22% for As, Cu, Cd, and Zn between the ASU results and the results provided by Intrinsik. These differences are within reasonable estimates of sample heterogeneity. The differences noted were larger for Pb: 37 % RPD for FF302F and 108% for FF310F (i.e. the ASU results were 3x higher than the results provided by Intrinsik for this sample). The resulting percent bioaccessibilities were therefore lower than in the original report (Table 2).

Table 2. Percent bioaccessibility results for Pb in the two samples.

Sample	**This report**	**Previous report**
FF302F	56% (Phase 1) 6.4% (Phase 2)	128% (Phase 1) 5.6% (Phase 2)
FF310F	54% (Phase 1) 7.1% (Phase 2)	177% (Phase 1) 19% (Phase 2)

The phase 2 blanks have Cu, Cd and Pb contamination and indicate that the phase 2 values are questionable for these elements. The blank contamination may not have been systematic, however, since the standard reference material results were within control limits.

Because of limitations with the current software used for reporting data, the number of significant figures quoted in the attached tables may not be representative of the actual uncertainty. Data should be considered accurate to no more than two significant figures.

This report is issued under complete status. All analyses requested for the first phase of the study have been completed and results are issued with full compliance of data verification.

Report authorised by: ……Iris Koch……. Date: …Nov 4, 2008

[1] RPD = (absolute value of (value 1 – value 2)/average of value 1 and 2) x 100%

ESG Bioaccessibility Report ASD0819

Site: Flin Flon
Analyst: Jessica Harris
Extraction Date: 30-Sep-08
Method: PBET
Ratio: 100 to 1

 # Samples: 2 reruns
Reference Reports: ASU 11571 (2 reports)
ESG Request #: 08-906
Report Date: 04-Nov-08

RESULTS

SAMPLE ID	Bioaccessible conc (mg/kg)	Initial conc in soil (mg/kg)	% Bioaccessibility (%)	Bioaccessible conc (mg/kg)	Initial conc in soil (mg/kg)	% Bioaccessibility (%)	Bioaccessible conc (mg/kg)	Initial conc in soil (mg/kg)	% Bioaccessibility (%)
	As	**As**	**As**	**Cd**	**Cd**	**Cd**	**Cu**	**Cu**	**Cu**
PHASE 1									
FF302F	6.5	25	26	20	30	65	372	1140	33
FF310F	7.6	22	35	10	18	58	180	747	24
PHASE 2									
FF302F	6.7	25	27	11	30	35	291	1140	26
FF310F	8.1	22	37	6.9	18	38	197	747	26

BND = total initial concentration and the bioaccessible fraction are less than the detection limit, therefore % bioaccessibility cannot be calculated

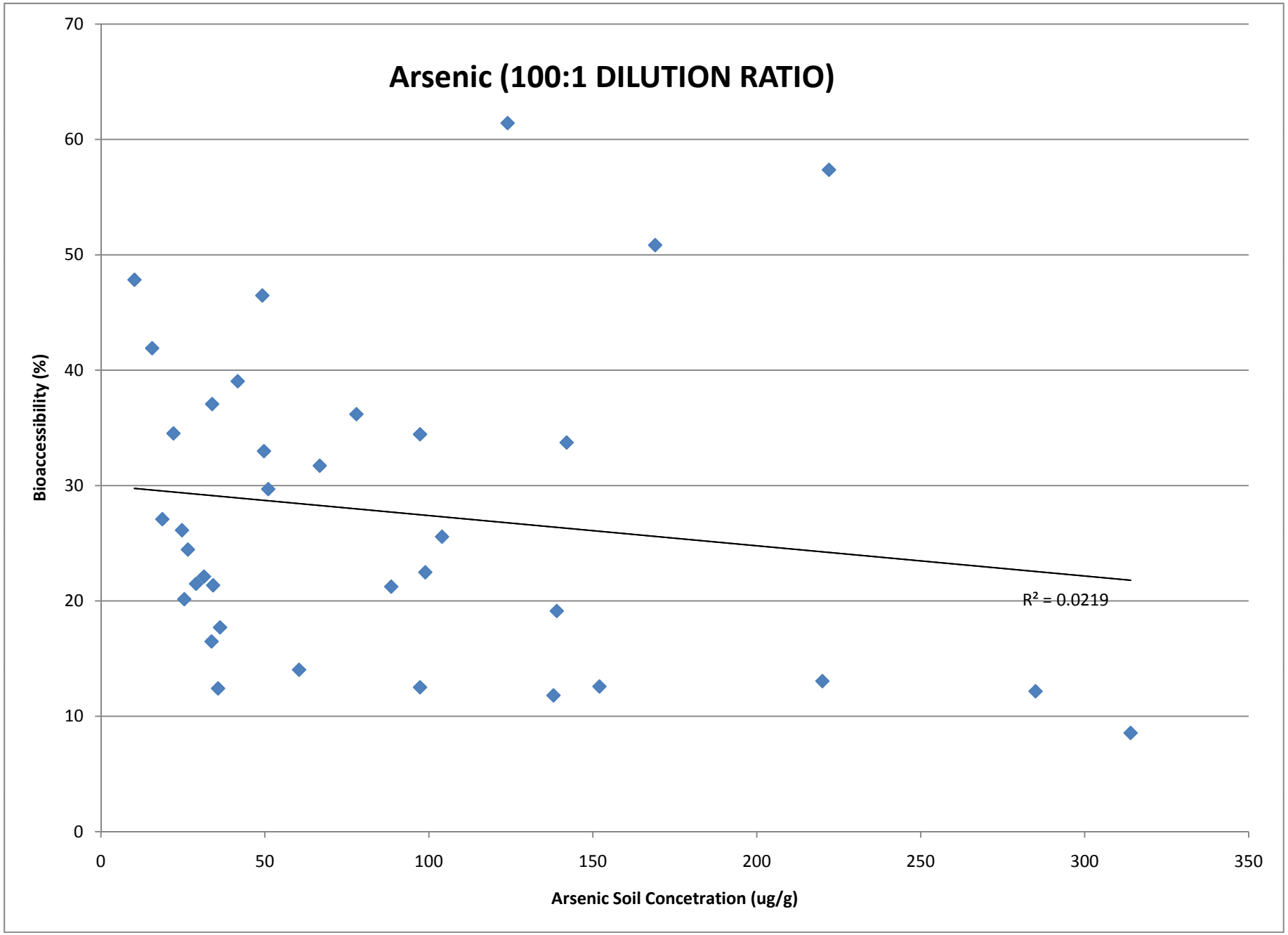
ESG Bioaccessib

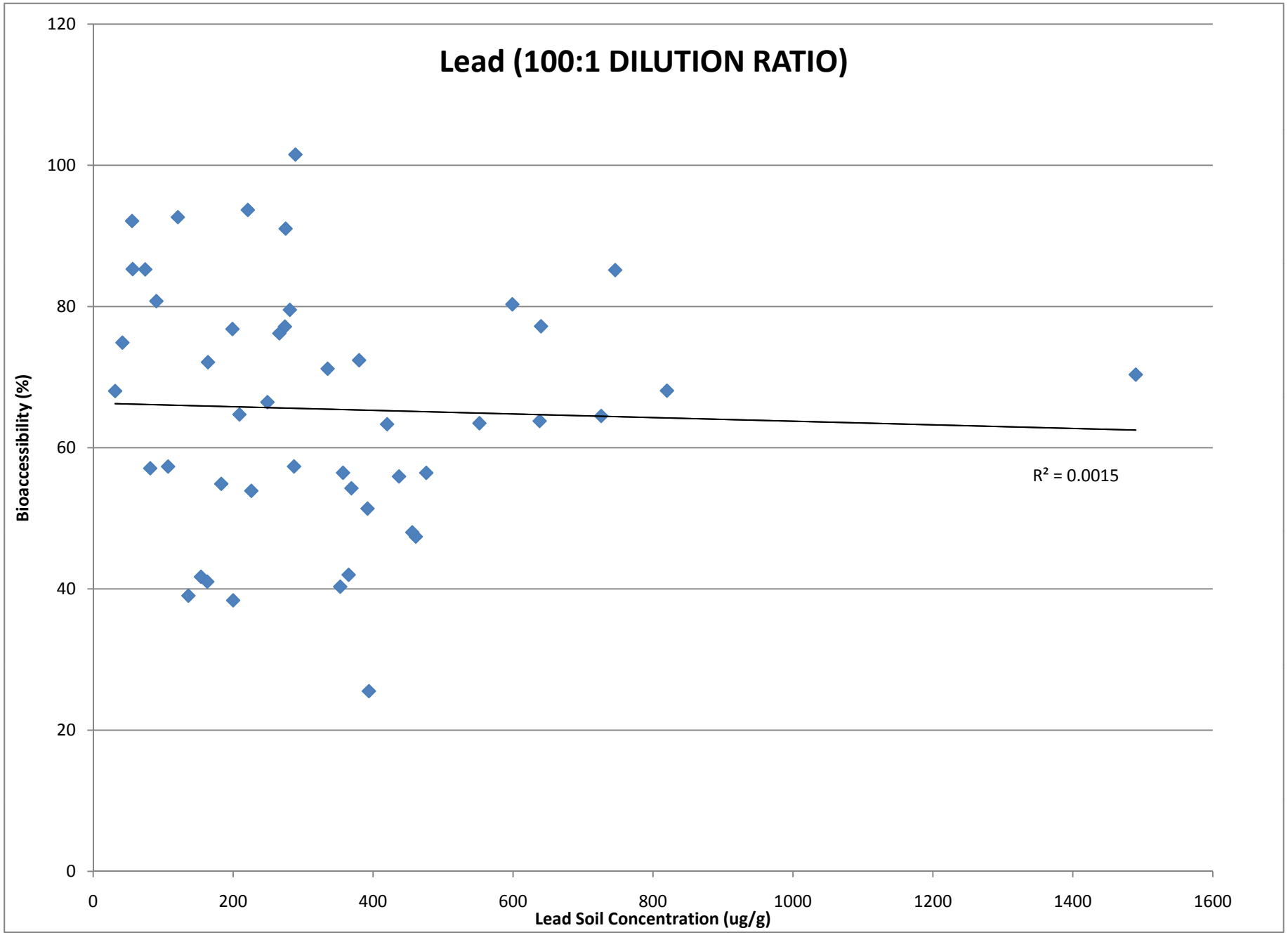
Site:
Analyst:
Extraction Date:
Method:
Ratio:

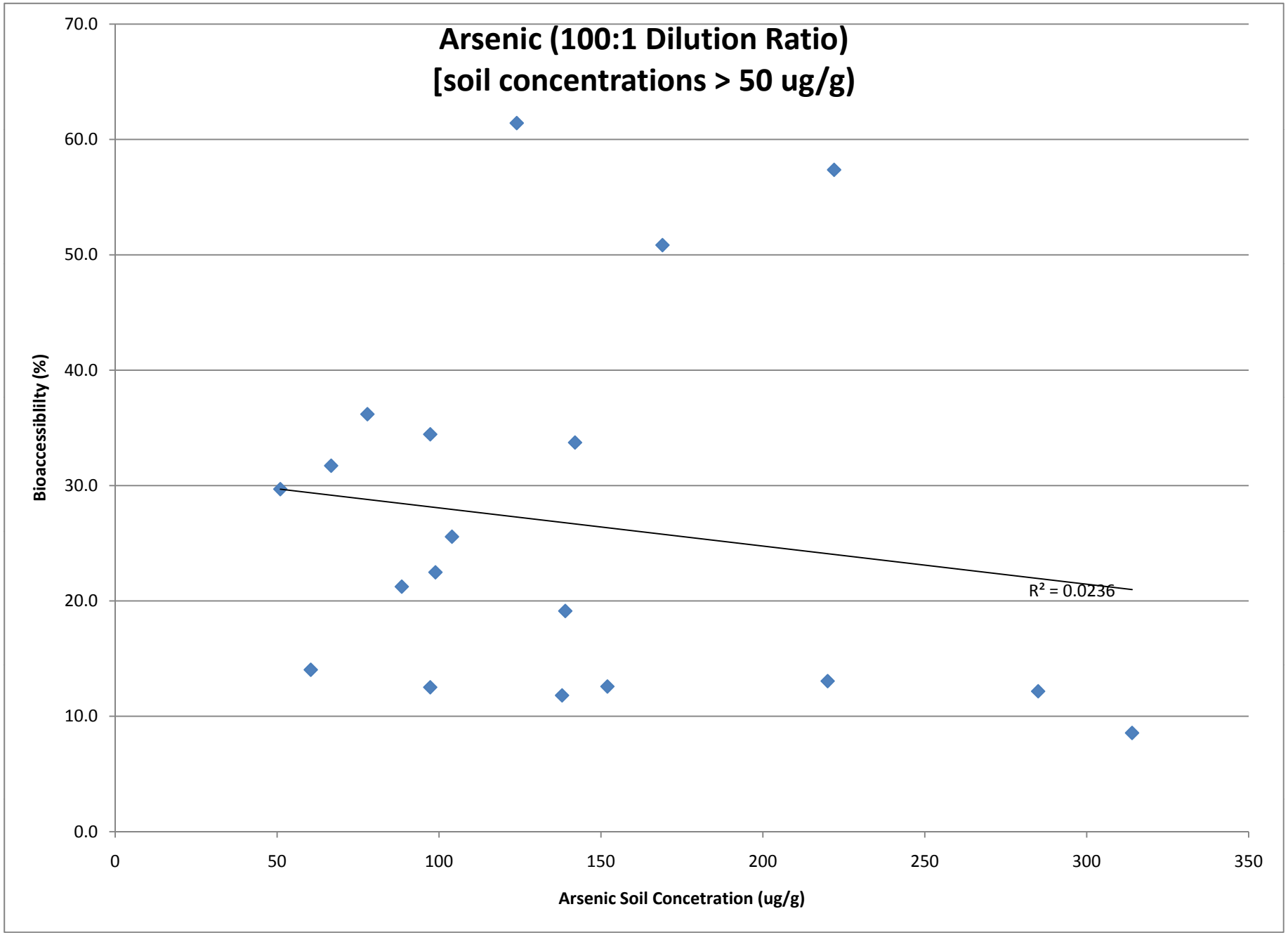
RESULTS

SAMPLE ID	Bioaccessible conc (mg/kg)	Initial conc in soil (mg/kg)	% Bioaccessibility (%)	Bioaccessible conc (mg/kg)	Initial conc in soil (mg/kg)	% Bioaccessibility (%)	Bioaccessible conc (mg/kg)	Initial conc in soil (mg/kg)	% Bioaccessibility (%)
	Pb	**Pb**	**Pb**	**Se**	**Se**	**Se**	**Zn**	**Zn**	**Zn**
PHASE 1									
FF302F	244	437	56	<10	<10	BND	3207	4220	76
FF310F	200	369	54	<10	<10	BND	2174	3520	62
PHASE 2									
FF302F	28	437	6.4	<10	<10	BND	1039	4220	25
FF310F	26	369	7.1	<10	<10	BND	780	3520	22

BND = total initial con


Arsenic (100:1 DILUTION RATIO)
Bioaccessibility (%)
Arsenic Soil Concetration (ug/g)
R² = 0.0219
0
10
20
30
40
50
60
70
0
50
100
150
200
250
300
350


Lead (100:1 DILUTION RATIO)
Bioaccessibility (%)
120
100
80
60
40
20
0
0
200
400
600
800
1000
1200
1400
1600
Lead Soil Concentration (ug/g)
R² = 0.0015


Arsenic (100:1 Dilution Ratio)
[soil concentrations > 50 ug/g)
Bioaccessibility (%)
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
50
100
150
200
250
300
350
Arsenic Soil Concetration (ug/g)
R² = 0.0236