ICP-MS AND XRF SOIL METAL CONCENTRATION SCREENING COMPARISON BY QUANTITATIVE ANALYSIS

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A thesis submitted to the faculty of
San Francisco State University
In partial fulfillment of
the requirements for
the Degree

Master of Science

In

Geosciences

by

Michael Paul Wrigley

San Francisco, California

Spring 2014

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CERTIFICATION OF APPROVAL

I certify that I have read ICP-MS and XRF Soil Metal Concentration Screening Comparison by Quantitative Analysis by Michael Paul Wrigley, and that in my opinion this work meets the criteria for approving a thesis submitted in partial fulfillment of the requirement for the degree Master of Science in Geosciences San Francisco State University.

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ICP-MS AND XRF SOIL METAL CONCENTRATION SCREENING COMPARISON BY QUANTITATIVE ANALYSIS

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San Francisco, California
2014

Urbanization increases human exposure to harmful and potentially deadly levels of toxic metals and their compounds from both natural background and anthropogenic fractions. Quantifying metal levels in soil and bedrock can result in highly variable concentrations. Detections for a suite of 18 metals (As, Ba, Cd, Cr, Co, Cu, Fe, Hg, Mn, Ni, Pb, Rb, Sb, Sn, Sr, Ti, Zn, and Zr) were conducted in my comparative analysis. My goals with this project are twofold: 1) quantify error and accuracy of the X-ray Fluorescence (XRF) against the Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) detection ability, accuracy and reliability, and 2) accurately determine which specific metals of the listed 18 are best screened for by XRF. To accomplish these goals I conducted analytical screening by XRF and compared results from ICP-MS for the 18 metals in a batch of 29 samples of young bay mud (San Antonio Formation). Soil samples were analyzed using the standard EPA method 6020 for bulk content by ICP-MS at a commercial laboratory, and XRF tests were performed in a lab on the SFSU campus. Results varied greatly with best fits to ICP-MS results: Zn, Pb, As, Hg, and Sn, and poorest fits: Cd, Zr, Ba, Co, Sr, and Cr. Highly significant statistical connection to concentration was found correlative (99.9% confidence) to the ability of the XRF to agree with ICP-MS soil metals screening analysis results.

I certify that the Abstract is a correct representation of the content of this Thesis.

Chair, Thesis Committee

5-15-2014

Date

ACKNOWLEDGMENTS

I would like to thank my advisor, Jason Gurdak for all of his help during my learning process and creating this thesis. A special thanks to my committee member, Leonard Sklar for his continued support in statistical review and near-evangelical promotion of competent scientific procedure. This investigation could not have been accomplished without the instruction and feedback from professor Bruce Manning and his depth of understanding with respect to XRF analysis, operation, and purpose. Without the privilege to conduct this work at San Francisco State University from Dr. Peter Palmer, the use of his lab and device, my work would have taken much longer than it did. A big part of my success goes out to the department chair, Karen Grove, for initial opportunity to the preliminary graduate process and her sincere professional enthusiasm along the way. To all those students and colleagues who rallied to meet the demands and rigor of the evolving curriculum of San Francisco State University's graduate requirements, congratulations and thank you for all of the peer advice and encouragement.

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1. Introduction

In response to adverse human health effects and environmental concerns, geoscientists, policy makers, and regulators have identified the critical need to better understand the controls on background levels of trace metals in urbanized soil (Duvergé, 2011; Diamond, 2009; Smith et al., 2012). Metal exposure to humans can cause various illnesses such as heart disease, severe birth defects, cancer, neurological and psychological symptoms, as well as nervous system, respiratory and pulmonary problems (Järup, 2003). Detectable concentrations of metals in soils occur from natural and anthropogenic sources. Without established background concentrations, environmental investigators and regulators face the challenge of determining sources of metal contamination and assigning responsibility for remediation. Use of a single value of background concentration, regardless of the spatial variations could give rise to either overestimation or underestimation of metal contamination and the associated risks. Nevertheless, it is important to estimate the background concentrations because they are essential for risk assessment and regulation of metals in water/soil systems (Shah et al., 2011).

Metals occur naturally in soils across the U.S. (Zhang and Selinus 1998), but are a critically important problem in many parts of California where the complex lithology and varied concentrations can make determination of potential contamination an ongoing and costly challenge for scientists, policy makers, and government regulators (Duvergé 2011, Smith et al., 2012). Research of metal distribution and concentration in soil has increased over the past decade (Shah et al., 2012). Despite abundant research and continued projects in

the fields of cleanup and abatement, potentially dangerous levels of toxic metals are released every year into the environment (Dong et al., 2010).

The California Code of Regulations (CCR), Title 22, section 66261.24 specifies 17 metals that can qualify as hazardous waste (Ag, As, Ba, Be, Cd, Cr, Cu, Co, Hg, Mo, Ni, Pb, Sb, Se, Tl, V, and Zn). 10 of these 17 metals (As, Ba, Cd, Cr, Co, Cu, Hg, Ni, Pb, and Zn) are detectable by both X-ray fluorescence (XRF) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). Eight additional metals (Fe, Mn, Rb, Sb, Sn, Sr, Ti, and Zr) not on the Title 22 list were also included in this study because they are detectable by XRF. Metals such as As, Hg, and Pb can be toxic at relatively low levels and are often screened in soil analyses and health hazard investigations (US EPA 2012). Their concentration concerns are based on risk to human health, toxicological effects on biota, and hazards to the total environment. Both XRF and ICP-MS are typically used in site soil characterization reports as well as bedrock analysis. However, the ICP-MS is definitive in legal disputes, while the XRF's primary function is for rapid field screening and explorative mining investigations (Kabata-Pendias, 1993).

Previous geochemical studies have focused on the association between groups of trace metals and their potential to predict other geochemical properties, the effect of rock type and land use on metal concentrations at the scale of cities, and the advantages and disadvantages of various methods for characterizing geochemical background environments (Facchinelli et al., 2001, Pouyat et al., 2008 Li et al., 2009). My work

builds on Duvergé's 2011 study of background As concentrations in San Francisco regional soils. Duvergé (2011) found that background concentrations of arsenic from Holocene alluvium (5.10 mg/kg) were statistically greater than concentrations within Pleistocene alluvium (3.65 mg/kg) and other Quaternary units (3.30 mg/kg), which indicates a significant geologic control on arsenic concentrations. Prior studies have estimated the mean and range of As for California (3.5, <0.2–11 mg/kg) (UCR, 1996), and localized areas in the Bay Area (11.6, <detection level–42 mg/kg) (Diamond, 2009). However, these and other studies about background concentrations have not focused on the specific abilities and errors associated with the XRF and quantitative challenges of soil metal analyses (Congiu et al., 2013). In this thesis, the functional limits of the XRF were tested against the precision of ICP-MS, where paralleled comparisons of results help determine what metals may be more reliably screened using XRF.

To address these challenges, my primary research goal is to evaluate XRF as a screening tool for the 18 commonly analyzed metals (As, Ba, Cd, Co, Cr, Cu, Fe, Hg, Pb, Mn, Ni, Rb, Sb, Sn, Sr, Ti, Zn, and Zr) in representative San Francisco Bay Area (Bay Area) soil samples (San Antonio bay mud material). I hypothesize that the metals with higher XRF concentrations will have greater agreement with the ICP-MS distributions and fewer non-detects (NDs) when compared to the relatively lower concentration metals. To test the hypotheses, I conducted XRF screening of 29 soil samples that were previously analyzed for 51 elements by ICP-MS at a professional commercial laboratory. The results of metal detection by XRF were compared to the ICP-MS data using

statistical analyses. Findings of this study will help evaluate the performance of the XRF screening and agreement with ICP-MS results in quantifying metal concentrations in soils.

2. Study Region

Natural metal content in soils largely arise from erosion and weathering of parent material (Horv'ath and Harty'ani, 2000). The soils in the Bay Area are complex due to variability in the region's geology, which has been mapped in great detail with key distinctions of bedrock composition and extent (Figure 1). The Bay Area has three distinct geologic provinces: 1) the Salinian block, 2) the Franciscan complex, and 3) the Great Valley sequence (Figure 1). The Salinian block lies west of the San Andreas Fault and is composed primarily of granitic plutonic rocks, which are believed to be rocks of the Sierra Nevada batholith that were displaced northwest along the San Andreas Fault. The Franciscan complex is bounded by the San Andreas Fault on the west and the Hayward Fault on the east. Franciscan rocks are primarily deep marine sandstone and shale from the former oceanic crust that were accreted to North America by subduction and collision. Chert, marble, serpentine, and limestone define the variable Franciscan assemblage. The Great Valley sequence lies east of the Hayward Fault and is composed primarily of Cretaceous and Tertiary marine sedimentary rocks (URS, 2003). Attempts to define a single background concentration for many different metals in the Franciscan units has proven difficult due to the physical heterogeneity of regional geochemistry and

the concept that background concentration represents a range, not a single value (Reimann and Garrett 2005).

3. Methods

This thesis uses data from 29 bulk soil samples of the San Francisco Bay sediments within the San Antonio formation that were collected by a consulting company as part of a professional environmental investigation (Bruce Castle, personal communication, 2013). The location and purpose of the bulk soil samples is confidential (Bruce Castle, personal communication, 2013). The 29 soil samples are used in this thesis as representative samples of the regional geologic material that is commonly screened in local environmental characterization reports. The 29 soil samples are not evaluated here to geochemically characterize the San Antonio formation.

As part of the professional environmental investigation, metal concentrations of the 29 bulk soils samples (reported on a dry-weight basis) were determined by ICP-MS using the EPA Method 6020 (US EPA, 2007). The EPA Method 6020 is recognized by regulatory agencies as the standard analytical method for determination of trace elements when addressing possible soil contamination (Cal-EPA, 1997; DTSC, 1997).

3.1 Sample Preparation

As part of the EPA Method 6020 for ICP-MS analysis, the bulk soil samples were disaggregated at the professional laboratory using an agate mortar and pestle and sieved to <2.0 mm. Prior to ICP-MS analysis, the soil samples were processed using a 2-acid digestion (HNO_3, HCl). The samples analyzed for trace elements were stored in plastic containers.

I processed and analyzed a sub-sample of the 29 bulk soil samples using XRF in Dr. Palmer's laboratory at San Francisco State University. I received the soil sub-samples completely dried, crushed to a fine powder, and separated in re-sealable paper bags. Approximately 100 to 150 grams of powder soil material was transferred from each of the re-sealable paper bags and weighed in plastic sample containers. The plastic sample containers were numbered and placed in the XRF device to determine metal concentrations, using three replicate scans per sample container.

3.2 XRF Analysis

I used the INNOV-X-Systems™ Delta hand-held XRF analyzer to determine metal concentrations in the 29 soil samples. The hand-held XRF is designed for rapid, field-operated readings to give instant results for dry-bulk chemical analysis of soils or bedrock material. XRF analysis is based on the phenomenon of atomic x-ray emission of an element when excited by an external source of radiation (Marcos, et al., 2011). If a gamma of a sufficiently energetic X-ray from an isotope or x-ray tube impinges on an

atom of the sample material, it may eject one of the inner shell electrons of the atom. The vacancy created is almost instantaneously filled by one of the electrons from the higher energy outer shell. The difference between the binding energy of the two shells involved in the process is emitted in the form of x-ray radiation. This emitted radiation is referred to as a "characteristic" x-ray because its energy is specific and unique to the element from which it is emitted (Figure 2). Energy and intensity measurements of the characteristic x-rays derive qualitative and quantitative aspects of x-ray analysis (Potts et al., 2006).

Measurements were transferred from the INNOV-X® software to Microsoft™ Excel® and JMP™ for statistical analysis. A determination of correlation by R^2 value was derived from fit of the multiple XRF measurements to the ICP-MS results by linear regression (Appendix 1

4. Results

4.1 ICP-MS

The results of the ICP-MS analysis are shown in Table 1. The concentrations of the 18 metals vary greatly by soil sample, spanning six orders of magnitude. Summary statistics (Table 2) reveal means range from 0.80 mg/kg (Cd) to nearly 30,000.0 mg/kg (Fe), while all other means are below 100.0 mg/kg with the four exceptions of Ba (195.5

mg/kg), Mn (469.3 mg/kg), Pb (324.6 mg/kg), and Zn (267.7 mg/kg). The highest ICP-MS concentrations are Fe (74,800.0 mg/kg), Ti (6,440.0 mg/kg), Pb (3,820.0 mg/kg), and Zn (1,700.0 mg/kg) and all others hold maximum levels lower than 1,000 mg/kg. Fe and Ti concentrations span 2 orders of magnitude, while Pb and Zn have the greatest range in concentrations, spanning 4 orders of magnitude.

4.2 XRF

The results of the XRF analysis are shown in Table 3 and summarized in Table 4. The concentrations of the 18 metals in the soil samples are highly variable, spanning over six orders of magnitude (Table 3). As expected, Fe has the highest of all metal concentrations with a maximum of 103,445 mg/kg, minimum of 6,530 mg/kg, and a mean of 38,116 mg/kg (Table 4). Similar to Fe, Ti concentrations span three orders of magnitude and are the second highest XRF concentration of any metal, with a maximum of 15,860 mg/kg, minimum of 840 mg/kg, and mean of 3,491 mg/kg (Table 4). Both XRF and ICP-MS Pb concentrations span four orders of magnitude, with an XRF maximum of 1,422 mg/kg, minimum of 6.0 mg/kg, and a mean of 249 mg/kg (Table 2, Table 4).

Toxic metals such as As hold lower comparative XRF concentrations to many of the other metals screened (Ba, Cu, Co, Fe, Mn, Pb, Sb, Sr, Ti, Zn, and Zr) yet with a maximum of 314 mg/kg, minimum of 2.6 mg/kg and mean of 42.3 mg/kg (Table 4) results show high (10 times) concentrations relative to a recent As background level

study based in San Francisco regional soils (mean of 3.3 mg/kg in Quaternary units, Duvergé 2011). Cobalt (Co) also has high relative XRF concentrations with a maximum of 248 mg/kg, minimum of 57 mg/kg, and mean of 162 mg/kg compared to an upper estimated regional background level of 23 mg/kg (LBNL 2002), statistical mean of 14.9 mg/kg found in a California soils survey (UCR 1996), and arithmetic mean of 14 mg/kg in a background study of 17 metals conducted at Berkeley National Labs (LBNL 2009).

Despite a statistically limiting number of 75 total non-detects (see *Non-Detects* below), Hg concentrations from XRF screening were much higher than two regional background metal soils studies (mean of 0.26 mg/kg, UCR 1996, estimated upper limit of 0.2 mg/kg, LBNL 2002) with a maximum of 56.0 mg/kg, minimum of 6.0 mg/kg, and mean of 20.4 mg/kg (Table 4).

4.3 Environmental Screening Levels (ESLs)

Environmental Screening Levels (ESLs) were developed by the local State government regulatory agency, the Regional Water Quality Control Board, San Francisco Bay Area, to address environmental protection goals of the Basin Plan (RWQCBSF 2006). These protection goals include surface water, groundwater, aquatic habitats, buildings from vapor intrusion, and against adverse nuisance conditions.

Several potentially toxic metals from the 29 samples have XRF concentrations that exceed the ESL (Table 5). The ESL for Pb in shallow soil is 80.0 mg/kg (SF Bay RWQCB, 2013). The XRF Pb lower 95% of the mean concentration is 126.0 mg/kg, or

157% of the ESL (Table 3). The median concentration of As (40 mg/kg) exceeds the ESL for shallow soil under residential land use (0.39 mg/kg) by two orders of magnitude (Table 4). The mean concentration of Co (162 mg/kg) is six times the shallow soil ESL (23.0 mg/kg), and the maximum concentration of Co (223.0 mg/kg) is an order of magnitude greater than the ESL. The mean concentration of Hg (13.0 mg/kg) is only 19% of the ESL (67.0 mg/kg). However, the Hg XRF results had many NDs that reduced the statistical significance of the Hg distribution.

4.4 Non-Detects in XRF Analysis

Many of the 18 metals have NDs from the XRF analysis, ranging from 0 to 89% NDs (Table 6). Results from the greatest to least number of NDs (with % of total readings as NDs): Cd, 77 NDs (89 %), Hg, 75 NDs (86 %), Ni, 58 NDs (67 %) Sn, 53 NDs (61 %), Sb, 52 NDs (60 %), Ba, 19 NDs (22 %), As and Co with 9 NDs each (10 %), Cr and Cu with 7 NDs (8 %) each, Pb, 3 NDs (3 %), and the remaining seven metals tied for lowest number of NDs with zero: Fe, Mn, Rb, Sr, Ti, Zn, and Zr (Table 6). The final seven listed, along with As, Co, Cr, Cu, and Pb represent the strongest statistical results based on the highest total of successful recordings from the entire set of 87 screens.

The frequency of NDs reflects the limited sensitivity of the XRF and its higher detection limits relative to ICP-MS (Wolf et al., 2005). While typical detection limits for

most XRF elemental screening record between 1.0 - 10.0 mg/kg, ICP-MS has lower limits of detection for several metals in the 0.01 – 0.10 ug/kg range with heavier elements near limits of 0.001 ug/kg, approximately six orders of magnitude under XRF for similar metals (Figure 3).

In soil investigations it may prove worthwhile for additional testing by ICP-MS for the more toxic metals with frequent NDs by XRF. Conversely, potentially toxic metals with higher XRF detection rate may require further validation by ICP-MS. A key difference between the ICP-MS and XRF methods is the preparation procedures. Where XRF is relatively non-destructive, ICP-MS uses a two or four-acid digestion to dissolve the soil material to solution (a two-acid digestion was performed for the ICP-MS samples in this study). This procedure can produce detection limits by preferential solubility of certain metals, like Pb, when subject to the partial extraction of acid digestion as opposed to total bulk content readings by XRF analysis (Delgado et al., 2011).

4.5 Linear Regressions

A linear regression was run for each metal concentration data set of XRF against the ICP-MS data (Appendix 1). The top-10 best-fitting metals with the greatest XRF screening agreement with the ICP-MS are As, Fe, Hg, Mn, Ni, Pb, Rb, Sb, Sn, and Zn (Table 7). These XRF results indicate a strong statistical agreement (minimum of $R^2 >$ 0.80 against the ICP-MS values) between the XRF and ICP-MS. Ambiguous results of

the poorest-fitting metals (Ba, Cd, Cr, Cu, Sr, and Zr with $R^2 < 0.80$) were consistent with the XRF's behavior to preferentially detect the higher quantities with otherwise poor quantitative determination of the lower-concentration elements (Han, et al., 2003).

4.6 Wilcoxon Rank-Sum Test

In order to determine if the results of the XRF screening and the ICP-MS results are statistically different, the Wilcoxon rank-sum test (α=0.1) was conducted on each of the 18 metals (Table 8). Based on the results of the Wilcoxon rank-sum test, the XRF and ICP-MS results differ for most of the 18 metals, including Cd, Co, Cr, Cu, Hg, Ni, Rb, Sb, Sn, Sr, Ti, and Zr (Table 8). The results of the Wilcoxon rank-sum test indicate that the XRF and ICP-MS results are statistically similar for As, Ba, Fe, Mn, Pb, and Zn (Table 8). The results of the Wilcoxon rank-sum tests are shown on box plot distribution of the concentrations of the 18 metals from the XRF and ICP-MS results (Figure 4).

4.7 Bivariate log-transformed analysis

To test the agreement of the XRF with the ICP-MS results, log-transformed concentration plots were generated for each metal (Figure 6, Appendix 2). The absolute difference between the XRF and ICP-MS log-transformed concentrations was plotted as a function of the log-transformed ICP-MS results. In this case, since the ICP-MS has much lower detection limits, higher accuracy, and greater number of detectable elements, the results were chosen as a closer representation of actual metal concentrations (Congiu et al., 2013). Log-transformations were conducted due to the six orders of magnitude ICP-MS concentration variability (Figure 4).

The null hypothesis, that no relation exists between concentration and agreement of the two detection methods, XRF and ICP-MS, was tested by way of these plots (Figure 6). If the null hypothesis were true, a flat line with random scatter should result. After running all 18 plots, many (16 of 18 metals) elements were found in agreement with the influence of concentration on agreement. As expected, low concentration scatter with higher concentration truncation of the plotted functions resulted for most of the metals. 16 of the 18 regressions were statistically significant at the 95% confidence level or greater ($p < 0.05$) with negative coefficients (Figure 7). I accepted the original hypothesis based on high level (99.9%) of confidence in the odds of getting 16 or more of the 18 metals to match the original hypothesis not by random chance.

5. Discussion

The distribution of metal concentrations from XRF and ICP-MS analysis are shown in Figure 3. The box plot distributions help to visualize the XRF results against the ICP-MS for comparative evaluation of the entire dataset. For example, while some metals like As, Ba, Fe, Mn, Ni, Pb, and Zn appear to have relatively similar median concentrations (based on Wilcoxon rank-sum tests (Table 8, Figure 3), the metals Ba and Ni have relatively weak correlation ($R^2 > 0.80$) between the XRF and ICP-MS results.

In order to discuss the XRF and ICP-MS results further, the 18 metals are categorized based on results of the linear regression (R^2) and Wilcoxon rank-sum test (p-value) (Figure 5). The four quadrants of Figure 5 are defined as: 1) high R^2 (>0.80) and high P-value (>0.10), 2) low R^2 (<0.80) and high P-value, 3) low R^2 and low P-value (<0.10), and 4) high R^2 and low P-value. The greatest portion of metals occupy the 3rd quadrant (low R^2 and P-values) with 7 of the 18 (~39%), while the second greatest portion split between the 1st (high on both R^2 and P-values) and 4th (high R^2 and low P-values) with 5 metals in each quadrant (28% of total 18 metals). The last remaining metal, Ba is the lone member in the 2nd quadrant (low R^2 and high P-value) comprising 5% of the total metals. Ba scored a R^2 of a weak 0.05 (17th out of the 18 metals) and yet managed a relatively high P-value of 0.15 (5th out of the 18 metals).

Regarding wellness of fit, Ti is strongly controlled by the three highest end-members (15,966 mg/kg, XRF and 6,440 mg/kg, ICP-MS), which are only a single sample of the 29. With end-members excluded, the Ti R^2 reduced from 0.80 to 0.10. The

Ti results illustrate that the XRF analysis for some metals may provide a more qualitative screening result than a quantitative value comparable to ICP-MS results. The concentrations of As, Fe, Mn, Pb, and Zn all have relatively high correlation ($R^2 > 0.80$) and similar distributions (p-values > 0.10 between the XRF and ICP-MS analysis. Although the XRF and ICP-MS concentrations of Ti were relatively high compared to some SF Bay Area soils (Simpson, 2004), the concentrations were not exceptional as compared to many California soils (Bradford, et al., 1996). Many background metal reports and soil surveys exclude Ti as a contaminant (Brooks, 2004; Diamond, 2009; URS, 2003) since Ti currently has no ESL for soil or groundwater (SF Bay RWQCB, 2013). Despite the lack of an ESL, Ti has relatively high crustal abundance (9^{th} of all known elements, 6^{th} among metals) of approximately 5,650 mg/kg (Lide, 2007).

The relatively high (mean of 38.0 mg/kg) and variable XRF As concentrations (Table 5) could generate legal disputes because the regulatory human-health ESL for As in shallow soils (<3.0 m below ground surface (bgs)) is 0.39 mg/kg (SF Bay RWQCB, 2013). Several of the other 18 metals also have relatively high R^2 values and poor one-to-one relation compared to the ICP-MS results (Figure 5).

Sb had a mean of 128 mg/kg while maintaining a R^2 fit of 0.91 (Figure 4, Appendix 1). Variability of Sb resulted in a maximum concentration of 384 mg/kg and 50 non-detects (ND) out of the total 87 screens (Table 6). Sb has an ESL of 20.0 mg/kg for shallow soils, only 1/6th the mean for the XRF screening of the soil sample. Regulatory

agencies may raise concern over results of toxic metals recording six times the ESL, however in this case with the mode being non-detects (ND < 5.0 mg/kg) the statistical relevance is removed. Resulting analyses of this nature present budget challenges when attempting to characterize contaminated sites and develop remediation action for cleanup efforts (Fonseca et al., 2009).

6. Conclusions

I rejected the null hypothesis due to the agreement of the XRF with the ICP-MS results by the log-transformed concentration plots generated for each metal (Figure 6). This finding represents an opportunity for expansion in the field of comparative analysis for metal soils screening. It will prove worthy of further investigation to increase the variety and sample size of geologic materials screened for in order to determine any consistent relation or signal for the elemental agreement between XRF and ICP-MS.

Five of the elements (Cd, Hg, Ni, Sb, and Sn) screened by XRF have more than 50 NDs (~57% of the 87 total screens, Table 6) rendering a weak and insignificant statistical representation. Despite this performance limitation of the XRF, the log-transformed plots indicate strong relation to concentration even for the higher ND metals.

Despite the inability of the XRF to accurately screen for several human-health risk metals (Cd, Sr, Ba, Co, Cr) it does provide value for instant, rapid on-site field

investigations and a general geochemical tool for two of the more toxic metals considered in this thesis, As and Pb. In addition, many of the non-hazardous metals such as Fe, Mn, and Zn should be considered reliable XRF screening analytes due to their high scoring R^2 and p-values and a minimum of NDs. Therefore, I suggest further expansion of XRF and ICP-MS comparison analyses for soil metals testing, while keeping a close look on key elements such as As, Ba, Cd, Co, Mn, Ni, Pb, Zn, and Zr for both consistent readings of accuracy and precision.

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8. Tables

Table 1. ICP-MS metal concentration results for the 29 soil samples, in mg/kg.

sample	As	Ba	Cd	Co	Cr
1	9.5	220.0	0.6	15.0	47.0
2	82.5	40.0	0.4	12.8	40.0
3	33.7	220.0	0.7	12.3	40.0
4	25.1	150.0	0.6	16.1	52.0
5	8.7	160.0	0.1	4.7	52.0
6	8.6	470.0	1.2	10.5	37.0
7	4.3	60.0	0.1	17.2	48.0
8	71.4	170.0	0.5	13.1	43.0
9	39.1	180.0	7.0	13.2	249.0
10	321.0	80.0	2.6	19.7	112.0
11	122.0	470.0	0.1	3.1	63.0
12	119.5	220.0	0.9	8.9	34.0
13	5.2	170.0	0.2	21.1	51.0
14	4.8	50.0	0.2	23.5	59.0
15	4.8	50.0	0.1	18.0	51.0
16	4.0	70.0	0.1	20.3	71.0
17	11.5	190.0	0.9	17.2	44.0
18	40.6	190.0	0.2	10.4	41.0
19	11.3	280.0	0.9	16.0	83.0
20	8.1	220.0	0.1	5.9	20.0
21	13.3	100.0	0.0	1.4	35.0
22	7.0	200.0	0.3	8.8	44.0
23	11.0	420.0	1.9	16.7	25.0
24	4.7	250.0	0.0	0.7	19.0
25	64.9	200.0	0.5	13.9	27.0
26	8.0	150.0	1.7	14.5	46.0
27	6.3	280.0	0.6	15.4	35.0
28	4.9	280.0	0.4	16.9	31.0
29	3.5	130.0	0.3	12.2	7.0

Table 1 (cont.) ICP-MS metal concentration results for the 29 soil samples, in mg/kg.

sample	Cu	Fe	Hg	Mn	Ni
1	36.9	28300.0	0.3	561.0	64.2
2	83.0	74800.0	0.5	673.0	49.2
3	33.2	22600.0	0.2	427.0	50.7
4	73.5	36700.0	0.9	723.0	41.0
5	22.2	26200.0	0.1	168.0	31.4
6	40.5	26100.0	0.3	459.0	36.4
7	39.3	27300.0	0.0	572.0	27.1
8	31.5	28100.0	0.2	445.0	56.4
9	261.0	27900.0	60.6	178.0	82.4
10	212.0	32600.0	11.8	461.0	79.7
11	102.5	9200.0	7.5	208.0	22.3
12	532.0	37800.0	6.9	174.0	30.0
13	47.5	36300.0	2.3	754.0	36.8
14	60.1	47300.0	2.3	915.0	37.5
15	46.5	45400.0	1.7	893.0	30.7
16	39.5	44300.0	1.6	652.0	74.2
17	22.9	20900.0	0.4	593.0	33.9
18	32.0	23200.0	1.5	371.0	71.6
19	149.5	39800.0	0.8	617.0	86.9
20	22.7	10700.0	4.1	101.0	34.1
21	40.4	27300.0	0.0	36.0	12.0
22	28.0	26100.0	0.0	121.0	39.5
23	52.4	18600.0	0.1	238.0	23.1
24	25.3	4700.0	0.5	31.0	6.1
25	22.9	27400.0	0.1	471.0	27.1
26	139.5	28400.0	0.7	269.0	215.0
27	32.4	19700.0	0.4	804.0	50.4
28	22.0	15200.0	0.3	743.0	30.5
29	17.2	44700.0	0.2	952.0	9.3

Table 1 (cont.) ICP-MS metal concentration results for the 29 soil samples, in mg/kg.

sample	Pb	Rb	Sb	Sn	Sr
1	142.5	29.5	0.9	4.1	55.9
2	748.0	11.2	3.45	228.0	67.7
3	243.0	20.8	1.3	5.9	62.0
4	76.2	5.4	2.16	5.7	76.9
5	15.6	14.7	0.69	2.2	39.6
6	266.0	6.7	5.42	6.9	51.2
7	130.5	3.3	1.38	1.0	23.0
8	53.1	20.1	0.95	13.5	52.0
9	577.0	17.6	6.85	176.0	49.4
10	372.0	11.6	13.8	96.7	51.2
11	479.0	13.0	6.36	54.6	39.6
12	213.0	9.2	286	21.3	31.6
13	7.6	6.8	0.35	0.6	88.9
14	92.7	4.7	0.21	1.3	105.0
15	14.3	4.0	0.16	5.0	119.0
16	3.1	5.7	0.15	0.6	98.5
17	38.2	13.6	82.5	1.8	43.9
18	123.0	17.6	97.8	9.7	101.0
19	3820.0	6.9	22	12.4	74.4
20	64.3	9.1	152	4.8	43.6
21	7.8	25.3	6.87	0.5	29.3
22	5.2	10.2	0.79	0.5	27.2
23	295.0	6.3	2.84	15.2	173.0
24	17.5	9.4	135.5	3.5	38.5
25	188.0	9.2	1.91	209.0	81.0
26	1165.0	3.8	11.15	23.1	104.5
27	203.0	16.0	36.3	8.5	58.1
28	45.7	20.3	117	1.3	38.1
29	6.2	3.8	0.41	0.9	24.2

Table 1 (cont.) ICP-MS metal concentration results for the 29 soil samples, in mg/kg.

sample	Ti	Zn	Zr
1	230	185.0	3.7
2	460	258.0	4.6
3	310	212.0	3.3
4	1100	154.0	7.7
5	200	56.0	5.1
6	2440	521.0	14.7
7	6440	59.0	7.8
8	290	106.0	4.8
9	400	1700.0	8.6
10	670	717.0	13.2
11	560	115.0	12.5
12	490	744.0	18.8
13	1050	54.0	11.1
14	1480	80.0	16.4
15	1230	75.0	14.7
16	1550	59.0	18.3
17	220	239.0	6.0
18	380	90.0	4.9
19	750	371.0	7.9
20	190	35.0	7.8
21	550	21.0	3.8
22	220	51.0	6.2
23	330	577.0	4.5
24	400	6.0	2.1
25	1070	119.0	13.4
26	350	729.0	7.9
27	330	210.0	3.2
28	350	115.0	4.4
29	710	104.0	17.4

Table 2. ICP-MS summary statistics metal concentration results reported by mass (mg/kg) for the set of 29 soil samples.

ICP-MS	As	Ba	Cd	Co	Cr	Cu	Fe	Hg	Pb
mean	36.5	195.5	0.8	13.1	51.9	78.2	29572.4	3.7	324.6
stdev	64.3	113.8	1.3	5.8	43.0	105.3	13849.7	11.3	721.4
max	321.0	470.0	7.0	23.5	249.0	532.0	74800.0	60.6	3820.0
min	3.5	40.0	0.0	0.7	7.0	17.2	4700.0	0.0	3.1

ICP-MS	Mn	Ni	Rb	Sb	Sn	Sr	Ti	Zn	Zr
mean	469.3	47.9	11.6	34.4	31.5	63.7	853.4	267.7	8.8
stdev	277.0	38.7	6.9	65.8	63.3	34.3	1192.0	354.8	5.1
max	952.0	215.0	29.5	286.0	228.0	173.0	6440.0	1700.0	18.8
min	31.0	6.1	3.3	0.2	0.5	23.0	190.0	6.0	2.1

Table 3. Mean XRF metal concentrations, listed alphabetically per metal in mg/kg. Non-detects (ND) inserted where statistics are indeterminable.

sample	As	Ba	Cd	Co	Cr	Cu
1	10.9	196.3	ND	148.0	102.0	84.5
2	117.0	229.0	ND	202.3	89.7	ND
3	32.3	171.3	ND	157.7	74.3	97.0
4	26.0	221.7	ND	135.0	76.3	70.7
5	8.5	106.0	ND	140.7	76.7	71.0
6	13.7	397.7	ND	207.3	103.0	50.7
7	7.0	448.7	ND	198.0	104.0	ND
8	64.6	188.0	7.0	147.7	64.3	104.7
9	45.0	129.7	10.7	197.0	282.7	252.0
10	312.3	190.7	7.0	168.0	157.0	241.0
11	147.3	219.5	ND	ND	79.3	299.0
12	120.3	154.3	ND	186.7	78.0	505.3
13	4.1	156.0	ND	161.7	60.7	79.7
14	5.3	127.0	ND	220.0	53.0	72.0
15	5.3	ND	ND	206.3	56.0	54.3
16	4.3	ND	ND	222.7	173.3	61.0
17	11.1	125.0	ND	144.3	100.0	110.0
18	40.7	139.7	ND	101.0	107.0	132.7
19	ND	220.7	8.0	186.3	152.0	230.7
20	7.6	97.0	ND	73.3	43.5	60.0
21	11.4	165.0	ND	156.7	93.7	115.3
22	5.2	122.3	ND	136.0	70.7	76.3
23	12.3	291.0	11.0	88.3	60.5	109.3
24	4.8	ND	ND	ND	83.0	145.0
25	52.3	258.0	ND	172.0	63.0	81.0
26	26.0	129.0	7.0	209.0	74.3	141.3
27	7.2	194.0	ND	113.0	122.7	94.0
28	5.0	150.3	ND	96.5	73.3	102.0
29	2.9	131.3	8.0	165.7	ND	32.3

Table 3 (cont.) Mean XRF metal concentrations, listed alphabetically per metal in mg/kg. Non-detects (ND) inserted where statistics are indeterminable.

sample	Fe	Hg	Mn	Ni	Pb	Rb
1	57687.0	ND	624.3	39.0	307.3	80.0
2	100165.3	ND	750.7	ND	684.3	44.3
3	27626.0	ND	382.3	21.5	221.0	75.0
4	49219.7	ND	848.3	23.0	74.6	31.9
5	32309.0	ND	145.7	ND	19.6	51.0
6	40616.7	ND	668.3	ND	290.3	41.4
7	76195.0	6.0	1106.7	ND	154.3	13.9
8	30471.3	ND	417.3	34.0	53.5	76.0
9	36337.7	53.3	182.3	47.0	751.3	56.1
10	38665.7	12.2	492.0	54.0	415.0	37.4
11	14738.0	6.0	234.0	122.0	505.7	61.1
12	42953.7	9.8	189.7	ND	254.3	39.1
13	44269.3	6.4	837.7	ND	10.2	30.2
14	57494.3	ND	1003.7	ND	76.8	15.9
15	52171.0	ND	923.7	ND	18.2	14.2
16	52490.7	ND	717.0	41.7	ND	15.1
17	22778.7	ND	510.3	ND	41.4	56.2
18	27955.3	ND	366.3	74.0	131.0	65.3
19	51251.3	ND	780.7	66.3	757.7	38.1
20	13587.3	ND	102.3	ND	69.9	46.3
21	25693.0	ND	55.3	ND	10.8	78.4
22	27612.3	ND	113.7	ND	8.3	40.0
23	19502.0	ND	241.3	ND	311.3	59.1
24	6741.0	ND	35.3	ND	22.2	60.6
25	32817.3	ND	512.0	ND	173.0	51.3
26	35778.3	ND	321.0	322.3	1334.7	16.5
27	21859.7	ND	718.3	32.3	212.0	64.1
28	16584.3	ND	603.3	ND	48.7	70.2
29	49796.0	ND	1031.0	ND	6.4	17.0

Table 3 (cont.) Mean XRF metal concentrations, listed alphabetically per metal in mg/kg. Non-detects (ND) inserted where statistics are indeterminable.

sample	Sb	Sn	Sr	Ti	Zn	Zr
1	ND	260.0	204.1	3583.0	237.0	224.5
2	ND	257.0	185.1	2641.3	279.3	161.9
3	ND	ND	207.7	3154.7	234.0	273.7
4	ND	ND	196.5	3158.3	153.0	132.7
5	ND	ND	137.6	3210.0	58.3	228.8
6	22.5	17.0	235.3	5238.3	596.0	402.0
7	ND	ND	207.3	15759.3	97.7	320.3
8	ND	14.5	194.3	3521.3	116.3	288.7
9	14.0	138.3	118.4	3501.0	1754.7	197.2
10	23.5	91.7	129.2	2265.3	811.3	296.7
11	26.0	40.0	172.3	3565.3	165.3	353.3
12	371.7	15.0	179.1	1652.3	828.0	635.7
13	ND	ND	205.3	2625.7	59.3	137.3
14	ND	ND	180.3	3127.7	81.7	80.0
15	ND	ND	190.0	2961.3	73.0	98.4
16	ND	ND	174.2	3432.0	62.7	117.2
17	112.3	ND	170.2	3323.0	264.3	408.0
18	149.3	108.3	213.5	3305.3	114.7	341.0
19	43.3	23.7	227.7	3318.0	436.3	265.0
20	283.0	ND	156.6	3244.7	43.7	411.7
21	17.0	ND	167.2	3997.0	21.7	369.7
22	ND	ND	128.1	2376.3	54.7	156.4
23	ND	22.0	278.7	1067.0	636.0	266.7
24	321.0	ND	194.4	3645.0	20.0	424.7
25	ND	213.7	244.4	2372.7	127.3	301.0
26	21.7	15.0	172.8	1014.0	841.0	149.3
27	56.7	ND	194.5	3410.7	234.0	335.7
28	140.0	ND	177.0	3294.3	114.3	377.0
29	ND	ND	93.3	3473.0	104.3	141.5

Table 4. XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation.
ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	As				
	run			median	stdev.
	1	2	3		
1	12.5	ND	9.3	10.9	2.3
2	123.0	119.0	109.0	119.0	7.2
3	36.0	29.0	32.0	32.0	3.5
4	24.5	27.4	26.0	26.0	1.5
5	8.9	7.8	8.8	8.8	0.6
6	12.0	15.0	14.0	14.0	1.5
7	ND	ND	7.0	7.0	Na
8	65.3	64.5	63.9	64.5	0.7
9	29.0	40.0	66.0	40.0	19.0
10	314.0	312.0	311.0	312.0	1.5
11	149.0	150.0	143.0	149.0	3.8
12	121.0	120.0	120.0	120.0	0.6
13	3.2	4.2	4.8	4.2	0.8
14	5.3	ND	ND	5.3	Na
15	5.7	6.0	4.3	5.7	0.9
16	5.1	4.1	3.7	4.1	0.7
17	11.4	11.5	10.5	11.4	0.6
18	40.0	39.9	42.3	40.0	1.4
19	ND	ND	ND	Na	Na
20	8.2	5.9	8.7	8.2	1.5
21	10.7	12.1	11.4	11.4	0.7
22	5.0	6.3	4.3	5.0	1.0
23	12.0	15.0	10.0	12.0	2.5
24	5.4	4.6	4.3	4.6	0.6
25	54.0	54.0	49.0	54.0	2.9
26	14.0	32.0	32.0	32.0	10.4
27	8.2	ND	6.1	7.2	1.5
28	3.9	5.9	5.3	5.3	1.0
29	3.0	3.1	2.6	3.0	0.3

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Ba				
	run			median	stdev.
	1	2	3		
1	271.0	148.0	170.0	170.0	65.6
2	148.0	236.0	303.0	236.0	77.7
3	208.0	122.0	184.0	184.0	44.4
4	206.0	270.0	189.0	206.0	42.7
5	ND	103.0	109.0	106.0	4.2
6	433.0	365.0	395.0	395.0	34.1
7	459.0	441.0	446.0	446.0	9.3
8	176.0	168.0	220.0	176.0	28.0
9	112.0	144.0	133.0	133.0	16.3
10	200.0	189.0	183.0	189.0	8.6
11	330.0	109.0	ND	219.5	156.3
12	175.0	144.0	144.0	144.0	17.9
13	191.0	169.0	108.0	169.0	43.0
14	ND	ND	127.0	127.0	Na
15	ND	ND	ND	Na	Na
16	ND	ND	ND	Na	Na
17	143.0	ND	107.0	125.0	25.5
18	160.0	110.0	149.0	149.0	26.3
19	212.0	264.0	186.0	212.0	39.7
20	97.0	ND	ND	97.0	Na
21	ND	165.0	ND	165.0	Na
22	145.0	95.0	127.0	127.0	25.3
23	237.0	321.0	315.0	315.0	46.9
24	ND	ND	ND	Na	Na
25	248.0	225.0	301.0	248.0	39.0
26	129.0	ND	ND	129.0	Na
27	160.0	252.0	170.0	170.0	50.5
28	146.0	137.0	168.0	146.0	15.9
29	118.0	141.0	135.0	135.0	11.9

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Cd				
	run			median	stdev.
	1	2	3		
1	ND	ND	ND	Na	Na
2	ND	ND	ND	Na	Na
3	ND	ND	ND	Na	Na
4	ND	ND	ND	Na	Na
5	ND	ND	ND	Na	Na
6	ND	ND	ND	Na	Na
7	ND	ND	ND	Na	Na
8	ND	7.0	ND	7.0	Na
9	11.0	11.0	10.0	11.0	Na
10	ND	7.0	ND	7.0	Na
11	ND	ND	ND	Na	Na
12	ND	ND	ND	Na	Na
13	ND	ND	ND	Na	Na
14	ND	ND	ND	Na	Na
15	ND	ND	ND	Na	Na
16	ND	ND	ND	Na	Na
17	ND	ND	ND	Na	Na
18	ND	ND	ND	Na	Na
19	ND	ND	8.0	8.0	Na
20	ND	ND	ND	Na	Na
21	ND	ND	ND	Na	Na
22	ND	ND	ND	Na	Na
23	ND	11.0	ND	11.0	Na
24	ND	ND	ND	Na	Na
25	ND	ND	ND	Na	Na
26	ND	7.0	7.0	7.0	0.0
27	ND	ND	ND	Na	Na
28	ND	ND	ND	Na	Na
29	8.0	ND	ND	8.0	Na

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Co				
	run			median	stdev.
	1	2	3		
1	132.0	159.0	153.0	153.0	14.2
2	159.0	208.0	240.0	208.0	40.8
3	128.0	169.0	176.0	169.0	25.9
4	156.0	130.0	119.0	130.0	19.0
5	160.0	162.0	100.0	160.0	35.2
6	203.0	222.0	197.0	203.0	13.1
7	185.0	202.0	207.0	202.0	11.5
8	150.0	130.0	163.0	150.0	16.6
9	203.0	153.0	235.0	203.0	41.3
10	160.0	162.0	182.0	162.0	12.2
11	ND	ND	ND	Na	Na
12	222.0	169.0	169.0	169.0	30.6
13	158.0	162.0	165.0	162.0	3.5
14	196.0	243.0	221.0	221.0	23.5
15	248.0	169.0	202.0	202.0	39.7
16	241.0	213.0	214.0	214.0	15.9
17	146.0	128.0	159.0	146.0	15.6
18	ND	80.0	122.0	101.0	29.7
19	189.0	165.0	205.0	189.0	20.1
20	99.0	57.0	64.0	64.0	22.5
21	155.0	146.0	169.0	155.0	11.6
22	135.0	123.0	150.0	135.0	13.5
23	82.0	107.0	76.0	82.0	16.4
24	ND	ND	ND	Na	Na
25	185.0	159.0	ND	172.0	18.4
26	225.0	201.0	201.0	201.0	13.9
27	82.0	117.0	140.0	117.0	29.2
28	106.0	87.0	ND	96.5	13.4
29	210.0	143.0	144.0	144.0	38.4

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Cr				
	run			median	stdev.
	1	2	3		
1	83.0	100.0	123.0	100.0	20.1
2	100.0	82.0	87.0	87.0	9.3
3	48.0	109.0	66.0	66.0	31.3
4	78.0	70.0	81.0	78.0	5.7
5	83.0	69.0	78.0	78.0	7.1
6	73.0	117.0	119.0	117.0	26.0
7	115.0	100.0	97.0	100.0	9.6
8	76.0	42.0	75.0	75.0	19.3
9	264.0	305.0	279.0	279.0	20.7
10	163.0	159.0	149.0	159.0	7.2
11	113.0	48.0	77.0	77.0	32.6
12	86.0	74.0	74.0	74.0	6.9
13	67.0	58.0	57.0	58.0	5.5
14	48.0	ND	58.0	53.0	7.1
15	62.0	63.0	43.0	62.0	11.3
16	182.0	181.0	157.0	181.0	14.2
17	92.0	86.0	122.0	92.0	19.3
18	135.0	80.0	106.0	106.0	27.5
19	169.0	135.0	152.0	152.0	17.0
20	36.0	ND	51.0	43.5	10.6
21	106.0	67.0	108.0	106.0	23.1
22	76.0	71.0	65.0	71.0	5.5
23	73.0	ND	48.0	60.5	17.7
24	100.0	75.0	74.0	75.0	14.7
25	ND	63.0	ND	63.0	Na
26	107.0	58.0	58.0	58.0	28.3
27	124.0	123.0	121.0	123.0	1.5
28	69.0	65.0	86.0	69.0	11.2
29	ND	ND	ND	Na	Na

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Cu				
	run			median	stdev.
	1	2	3		
1	77.0	ND	92.0	84.5	10.6
2	ND	ND	ND	Na	Na
3	97.0	96.0	98.0	97.0	1.0
4	56.0	82.0	74.0	74.0	13.3
5	69.0	74.0	70.0	70.0	2.6
6	60.0	43.0	49.0	49.0	8.6
7	ND	ND	ND	Na	Na
8	96.0	122.0	96.0	96.0	15.0
9	236.0	295.0	225.0	236.0	37.6
10	246.0	238.0	239.0	239.0	4.4
11	420.0	257.0	220.0	257.0	106.4
12	492.0	512.0	512.0	512.0	11.5
13	71.0	80.0	88.0	80.0	8.5
14	75.0	69.0	72.0	72.0	3.0
15	70.0	51.0	42.0	51.0	14.3
16	58.0	62.0	63.0	62.0	2.6
17	114.0	108.0	108.0	108.0	3.5
18	189.0	105.0	104.0	105.0	48.8
19	413.0	143.0	136.0	143.0	157.9
20	49.0	73.0	58.0	58.0	12.1
21	101.0	120.0	125.0	120.0	12.7
22	78.0	84.0	67.0	78.0	8.6
23	109.0	99.0	120.0	109.0	10.5
24	170.0	131.0	134.0	134.0	21.7
25	97.0	65.0	ND	81.0	22.6
26	132.0	146.0	146.0	146.0	8.1
27	89.0	105.0	88.0	89.0	9.5
28	102.0	73.0	131.0	102.0	29.0
29	41.0	30.0	26.0	30.0	7.8

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Fe				
	run			median	stdev.
	1	2	3		
1	35204.0	103445.0	34412.0	35204.0	39629.6
2	103445.0	100218.0	96833.0	100218.0	3306.3
3	27499.0	27686.0	27693.0	27686.0	110.0
4	48404.0	49764.0	49491.0	49491.0	719.5
5	32126.0	32202.0	32599.0	32202.0	254.0
6	39374.0	41861.0	40615.0	40615.0	1243.5
7	75937.0	76628.0	76020.0	76020.0	377.3
8	30476.0	30926.0	30012.0	30476.0	457.0
9	35500.0	36661.0	36852.0	36661.0	731.7
10	38823.0	38435.0	38739.0	38739.0	204.1
11	16124.0	14333.0	13757.0	14333.0	1234.4
12	41941.0	43460.0	43460.0	43460.0	877.0
13	43681.0	44699.0	44428.0	44428.0	527.2
14	56848.0	57217.0	58418.0	57217.0	820.9
15	52618.0	50918.0	52977.0	52618.0	1099.9
16	51821.0	52270.0	53381.0	52270.0	803.1
17	22508.0	22823.0	23005.0	22823.0	251.4
18	28979.0	27460.0	27427.0	27460.0	886.7
19	51704.0	50262.0	51788.0	51704.0	857.8
20	13205.0	13989.0	13568.0	13568.0	392.4
21	25052.0	26226.0	25801.0	25801.0	594.4
22	28059.0	27354.0	27424.0	27424.0	388.4
23	19779.0	19245.0	19482.0	19482.0	267.6
24	6944.0	6749.0	6530.0	6749.0	207.1
25	33032.0	32245.0	33175.0	33032.0	500.8
26	35543.0	35896.0	35896.0	35896.0	203.8
27	21810.0	21768.0	22001.0	21810.0	124.2
28	16235.0	16647.0	16871.0	16647.0	322.6
29	50307.0	49497.0	49584.0	49584.0	444.7

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Hg				
	run			median	stdev.
	1	2	3		
1	ND	ND	ND	Na	Na
2	ND	ND	ND	Na	Na
3	ND	ND	ND	Na	Na
4	ND	ND	ND	Na	Na
5	ND	ND	ND	Na	Na
6	ND	ND	ND	Na	Na
7	6.0	ND	ND	6.0	Na
8	ND	ND	ND	Na	Na
9	52.0	52.0	56.0	52.0	2.3
10	15.9	9.6	11.0	11.0	3.3
11	ND	6.0	ND	6.0	Na
12	8.8	10.3	10.3	10.3	0.9
13	ND	6.4	ND	6.4	Na
14	ND	ND	ND	Na	Na
15	ND	ND	ND	Na	Na
16	ND	ND	ND	Na	Na
17	ND	ND	ND	Na	Na
18	ND	ND	ND	Na	Na
19	ND	ND	ND	Na	Na
20	ND	ND	ND	Na	Na
21	ND	ND	ND	Na	Na
22	ND	ND	ND	Na	Na
23	ND	ND	ND	Na	Na
24	ND	ND	ND	Na	Na
25	ND	ND	ND	Na	Na
26	ND	ND	ND	Na	Na
27	ND	ND	ND	Na	Na
28	ND	ND	ND	Na	Na
29	ND	ND	ND	Na	Na

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Mn				
	run			median	stdev.
	1	2	3		
1	544.0	787.0	542.0	544.0	140.9
2	787.0	719.0	746.0	746.0	34.2
3	382.0	387.0	378.0	382.0	4.5
4	849.0	858.0	838.0	849.0	10.0
5	140.0	142.0	155.0	142.0	8.1
6	654.0	684.0	667.0	667.0	15.0
7	1091.0	1113.0	1116.0	1113.0	13.7
8	419.0	403.0	430.0	419.0	13.6
9	176.0	188.0	183.0	183.0	6.0
10	512.0	475.0	489.0	489.0	18.7
11	267.0	215.0	220.0	220.0	28.7
12	201.0	184.0	184.0	184.0	9.8
13	831.0	822.0	860.0	831.0	19.9
14	977.0	1026.0	1008.0	1008.0	24.8
15	935.0	914.0	922.0	922.0	10.6
16	710.0	723.0	718.0	718.0	6.6
17	500.0	508.0	523.0	508.0	11.7
18	383.0	369.0	347.0	369.0	18.1
19	797.0	783.0	762.0	783.0	17.6
20	97.0	109.0	101.0	101.0	6.1
21	56.0	63.0	47.0	56.0	8.0
22	126.0	105.0	110.0	110.0	11.0
23	228.0	245.0	251.0	245.0	11.9
24	34.0	33.0	39.0	34.0	3.2
25	536.0	498.0	502.0	502.0	20.9
26	321.0	321.0	321.0	321.0	0.0
27	712.0	732.0	711.0	712.0	11.8
28	612.0	599.0	599.0	599.0	7.5
29	1057.0	1010.0	1026.0	1026.0	23.9

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Ni				
	run			median	stdev.
	1	2	3		
1	39.0	ND	39.0	39.0	0.0
2	ND	ND	ND	Na	Na
3	22.0	ND	21.0	21.5	0.7
4	ND	ND	23.0	23.0	Na
5	ND	ND	ND	Na	Na
6	ND	ND	ND	Na	Na
7	ND	ND	ND	Na	Na
8	ND	34.0	ND	34.0	Na
9	37.0	61.0	43.0	43.0	12.5
10	48.0	65.0	49.0	49.0	9.5
11	122.0	ND	ND	122.0	Na
12	ND	ND	ND	Na	Na
13	ND	ND	ND	Na	Na
14	ND	ND	ND	Na	Na
15	ND	ND	ND	Na	Na
16	36.0	47.0	42.0	42.0	5.5
17	ND	ND	ND	Na	Na
18	109.0	57.0	56.0	57.0	30.3
19	69.0	71.0	59.0	69.0	6.4
20	ND	ND	ND	Na	Na
21	ND	ND	ND	Na	Na
22	ND	ND	ND	Na	Na
23	ND	ND	ND	Na	Na
24	ND	ND	ND	Na	Na
25	ND	ND	ND	Na	Na
26	339.0	314.0	314.0	314.0	14.4
27	42.0	26.0	29.0	29.0	8.5
28	ND	ND	ND	Na	Na
29	ND	ND	ND	Na	Na

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Pb				
	run			median	stdev.
	1	2	3		
1	116.0	690.0	116.0	116.0	331.4
2	690.0	696.0	667.0	690.0	15.3
3	214.0	224.0	225.0	224.0	6.1
4	74.8	75.5	73.4	74.8	1.1
5	19.4	20.1	19.4	19.4	0.4
6	287.0	300.0	284.0	287.0	8.5
7	156.0	155.0	152.0	155.0	2.1
8	51.8	54.9	53.7	53.7	1.6
9	566.0	560.0	1128.0	566.0	326.2
10	413.0	413.0	419.0	413.0	3.5
11	469.0	526.0	522.0	522.0	31.8
12	249.0	257.0	257.0	257.0	4.6
13	10.5	10.6	9.5	10.5	0.6
14	79.4	81.1	69.8	79.4	6.1
15	18.1	19.2	17.4	18.1	0.9
16	ND	ND	ND	Na	Na
17	41.5	41.3	41.5	41.5	0.1
18	133.0	130.0	130.0	130.0	1.7
19	967.0	488.0	818.0	818.0	245.1
20	68.5	73.2	68.0	68.5	2.9
21	10.6	10.8	11.0	10.8	0.2
22	9.2	7.3	8.4	8.4	1.0
23	294.0	311.0	329.0	311.0	17.5
24	22.8	21.7	22.0	22.0	0.6
25	169.0	170.0	180.0	170.0	6.1
26	1422.0	1291.0	1291.0	1291.0	75.6
27	210.0	213.0	213.0	213.0	1.7
28	48.4	49.7	48.1	48.4	0.9
29	7.1	6.2	6.0	6.2	0.6

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Rb				
	run			median	stdev.
	1	2	3		
1	98.0	44.7	97.2	97.2	30.5
2	44.7	43.6	44.7	44.7	0.6
3	74.0	75.6	75.3	75.3	0.9
4	31.6	31.8	32.4	31.8	0.4
5	51.8	51.4	49.8	51.4	1.1
6	41.2	41.7	41.4	41.4	0.3
7	14.1	14.0	13.6	14.0	0.3
8	75.9	76.2	75.9	75.9	0.2
9	55.0	57.8	55.5	55.5	1.5
10	37.3	37.0	37.9	37.3	0.5
11	55.6	64.3	63.3	63.3	4.8
12	39.8	38.7	38.7	38.7	0.6
13	29.6	30.7	30.2	30.2	0.6
14	16.8	15.6	15.2	15.6	0.8
15	14.9	13.4	14.2	14.2	0.8
16	14.9	15.6	14.9	14.9	0.4
17	56.9	55.5	56.3	56.3	0.7
18	66.7	64.3	65.0	65.0	1.2
19	38.9	37.1	38.4	38.4	0.9
20	46.8	46.8	45.3	46.8	0.9
21	77.6	79.0	78.6	78.6	0.7
22	40.0	40.5	39.5	40.0	0.5
23	60.1	58.7	58.5	58.7	0.9
24	60.8	60.7	60.4	60.7	0.2
25	51.4	51.6	51.0	51.4	0.3
26	16.2	16.6	16.6	16.6	0.2
27	63.7	64.5	64.0	64.0	0.4
28	70.1	69.1	71.4	70.1	1.2
29	17.3	16.3	17.4	17.3	0.6

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Sb				
	run			median	stdev.
	1	2	3		
1	ND	ND	ND	Na	Na
2	ND	ND	ND	Na	Na
3	ND	ND	ND	Na	Na
4	ND	ND	ND	Na	Na
5	ND	ND	ND	Na	Na
6	20.0	ND	25.0	22.5	3.5
7	ND	ND	ND	Na	Na
8	ND	ND	ND	Na	Na
9	ND	14.0	ND	14.0	Na
10	23.0	24.0	ND	23.5	0.7
11	26.0	ND	ND	26.0	Na
12	381.0	367.0	367.0	367.0	8.1
13	ND	ND	ND	Na	Na
14	ND	ND	ND	Na	Na
15	ND	ND	ND	Na	Na
16	ND	ND	ND	Na	Na
17	117.0	111.0	109.0	111.0	4.2
18	151.0	142.0	155.0	151.0	6.7
19	45.0	44.0	41.0	44.0	2.1
20	301.0	268.0	280.0	280.0	16.7
21	17.0	ND	ND	17.0	Na
22	ND	ND	ND	Na	Na
23	ND	ND	ND	Na	Na
24	327.0	313.0	323.0	323.0	7.2
25	ND	ND	ND	Na	Na
26	19.0	23.0	23.0	23.0	2.3
27	62.0	53.0	55.0	55.0	4.7
28	140.0	141.0	139.0	140.0	1.0
29	ND	ND	ND	Na	Na

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Sn				
	run			median	stdev.
	1	2	3		
1	ND	260.0	ND	260.0	Na
2	260.0	253.0	258.0	258.0	3.6
3	ND	ND	ND	Na	Na
4	ND	ND	ND	Na	Na
5	ND	ND	ND	Na	Na
6	17.0	ND	17.0	17.0	0.0
7	ND	ND	ND	Na	Na
8	ND	14.0	15.0	14.5	0.7
9	137.0	140.0	138.0	138.0	1.5
10	92.0	93.0	90.0	92.0	1.5
11	41.0	40.0	39.0	40.0	1.0
12	17.0	14.0	14.0	14.0	1.7
13	ND	ND	ND	Na	Na
14	ND	ND	ND	Na	Na
15	ND	ND	ND	Na	Na
16	ND	ND	ND	Na	Na
17	ND	ND	ND	Na	Na
18	110.0	104.0	111.0	110.0	3.8
19	26.0	22.0	23.0	23.0	2.1
20	ND	ND	ND	Na	Na
21	ND	ND	ND	Na	Na
22	ND	ND	ND	Na	Na
23	24.0	23.0	19.0	23.0	2.6
24	ND	ND	ND	Na	Na
25	222.0	209.0	210.0	210.0	7.2
26	15.0	15.0	15.0	15.0	0.0
27	ND	ND	ND	Na	Na
28	ND	ND	ND	Na	Na
29	ND	ND	ND	Na	Na

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Sr				
	run			median	stdev.
	1	2	3		
1	215.9	187.8	208.7	208.7	14.6
2	187.8	183.8	183.8	183.8	2.3
3	206.1	207.4	209.6	207.4	1.8
4	189.5	203.2	196.8	196.8	6.9
5	138.2	136.9	137.8	137.8	0.7
6	232.6	235.9	237.5	235.9	2.5
7	206.7	208.2	207.0	207.0	0.8
8	196.0	195.7	191.1	195.7	2.7
9	117.4	119.9	117.8	117.8	1.3
10	129.3	128.6	129.6	129.3	0.5
11	155.0	179.7	182.2	179.7	15.0
12	177.4	179.9	179.9	179.9	1.4
13	203.5	207.0	205.4	205.4	1.8
14	179.7	179.1	182.0	179.7	1.5
15	192.2	187.2	190.7	190.7	2.6
16	172.8	174.9	175.0	174.9	1.2
17	173.3	167.4	170.0	170.0	3.0
18	213.1	215.4	212.1	213.1	1.7
19	226.4	226.9	229.8	226.9	1.8
20	157.1	157.4	155.4	157.1	1.1
21	165.9	168.5	167.2	167.2	1.3
22	126.9	127.4	130.1	127.4	1.7
23	279.0	278.0	279.0	279.0	0.6
24	196.5	192.9	193.9	193.9	1.9
25	244.2	243.9	245.0	244.2	0.6
26	171.1	173.6	173.6	173.6	1.4
27	194.7	195.8	193.0	194.7	1.4
28	176.5	176.8	177.6	176.8	0.6
29	93.4	94.7	91.8	93.4	1.5

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Ti				
	run			median	stdev.
	1	2	3		
1	3893.0	2907.0	3949.0	3893.0	586.1
2	2907.0	2640.0	2377.0	2640.0	265.0
3	3074.0	3189.0	3201.0	3189.0	70.1
4	3155.0	3105.0	3215.0	3155.0	55.1
5	3247.0	3242.0	3141.0	3242.0	59.8
6	4863.0	5549.0	5303.0	5303.0	347.5
7	15599.0	15819.0	15860.0	15819.0	140.4
8	3438.0	3691.0	3435.0	3438.0	146.9
9	3452.0	3412.0	3639.0	3452.0	121.2
10	2198.0	2256.0	2342.0	2256.0	72.5
11	4258.0	3204.0	3234.0	3234.0	600.1
12	1637.0	1660.0	1660.0	1660.0	13.3
13	2555.0	2735.0	2587.0	2587.0	96.0
14	3192.0	3172.0	3019.0	3172.0	94.6
15	2993.0	2892.0	2999.0	2993.0	60.1
16	3488.0	3341.0	3467.0	3467.0	79.5
17	3201.0	3420.0	3348.0	3348.0	111.6
18	3595.0	3054.0	3267.0	3267.0	272.5
19	3423.0	3140.0	3391.0	3391.0	155.0
20	3196.0	3280.0	3258.0	3258.0	43.6
21	4158.0	3776.0	4057.0	4057.0	197.9
22	2479.0	2372.0	2278.0	2372.0	100.6
23	1357.0	1005.0	839.0	1005.0	264.5
24	3827.0	3628.0	3480.0	3628.0	174.1
25	2631.0	2269.0	2218.0	2269.0	225.2
26	976.0	1033.0	1033.0	1033.0	32.9
27	3486.0	3150.0	3596.0	3486.0	232.3
28	3365.0	3255.0	3263.0	3263.0	61.3
29	3658.0	3458.0	3303.0	3458.0	178.0

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Zn				
	run			median	stdev.
	1	2	3		
1	215.0	287.0	209.0	215.0	43.4
2	287.0	287.0	264.0	287.0	13.3
3	230.0	241.0	231.0	231.0	6.1
4	152.0	152.0	155.0	152.0	1.7
5	64.0	54.0	57.0	57.0	5.1
6	582.0	613.0	593.0	593.0	15.7
7	94.0	100.0	99.0	99.0	3.2
8	112.0	123.0	114.0	114.0	5.9
9	1481.0	1668.0	2115.0	1668.0	325.8
10	812.0	803.0	819.0	812.0	8.0
11	187.0	160.0	149.0	160.0	19.6
12	810.0	837.0	837.0	837.0	15.6
13	54.0	61.0	63.0	61.0	4.7
14	77.0	83.0	85.0	83.0	4.2
15	72.0	69.0	78.0	72.0	4.6
16	57.0	64.0	67.0	64.0	5.1
17	263.0	263.0	267.0	263.0	2.3
18	121.0	111.0	112.0	112.0	5.5
19	536.0	375.0	398.0	398.0	87.1
20	46.0	43.0	42.0	43.0	2.1
21	17.0	26.0	22.0	22.0	4.5
22	50.0	58.0	56.0	56.0	4.2
23	637.0	656.0	615.0	637.0	20.5
24	26.0	22.0	12.0	22.0	7.2
25	143.0	134.0	105.0	134.0	19.9
26	831.0	846.0	846.0	846.0	8.7
27	237.0	235.0	230.0	235.0	3.6
28	116.0	109.0	118.0	116.0	4.7
29	102.0	109.0	102.0	102.0	4.0

Table 4 (cont.) XRF summary statistics for each of the 18 metals, listed by samples 1-29, alphabetical series of tables with all concentrations in mg/kg, including each replicant screen (3 total), median, and standard deviation. ND = non-detect, Na = statistic indeterminable, all units in mg/kg.

sample	Zr				
	run			median	stdev.
	1	2	3		
1	243.9	172.5	257.0	243.9	45.5
2	172.5	157.7	155.6	157.7	9.2
3	272.0	271.0	278.0	272.0	3.8
4	126.5	137.8	133.8	133.8	5.7
5	220.1	224.8	241.6	224.8	11.3
6	403.0	400.0	403.0	403.0	1.7
7	317.0	326.0	318.0	318.0	4.9
8	296.0	296.0	274.0	296.0	12.7
9	193.4	202.7	195.5	195.5	4.9
10	291.0	302.0	297.0	297.0	5.5
11	319.0	365.0	376.0	365.0	30.2
12	633.0	637.0	637.0	637.0	2.3
13	134.6	144.9	132.4	134.6	6.7
14	80.0	79.7	80.4	80.0	0.4
15	98.3	98.2	98.8	98.3	0.3
16	112.5	120.5	118.5	118.5	4.2
17	405.0	405.0	414.0	405.0	5.2
18	343.0	347.0	333.0	343.0	7.2
19	252.0	269.0	274.0	269.0	11.5
20	410.0	405.0	420.0	410.0	7.6
21	371.0	378.0	360.0	371.0	9.1
22	153.6	158.2	157.5	157.5	2.5
23	260.0	271.0	269.0	269.0	5.9
24	429.0	422.0	423.0	423.0	3.8
25	303.0	297.0	303.0	303.0	3.5
26	157.8	145.1	145.1	145.1	7.3
27	342.0	332.0	333.0	333.0	5.5
28	381.0	373.0	377.0	377.0	4.0
29	142.1	140.6	141.8	141.8	0.8

Table 5. List of ESLs (Environmental Screening Levels) set by RWQCB (Regional Water Quality Control Board) for the selected 18 metals within residential land use limited to shallow soils (< 3.0 m bgs). Mean XRF resulting concentration (mg/kg), and relation to ESL (as %).

metal	ESL	XRF mean	% of ESL
	mg/kg		%
As	**0.4**	**42.2**	**10821**
Ba	750.0	199.9	27
Cd	12.0	8.7	73
Co	**23.0**	**162.2**	**705**
Cr	750.0	98.0	13
Cu	230.0	129.8	56
Fe	x	38116.0	x
Hg	**6.7**	**20.4**	**304**
Mn	x	514.3	x
Ni	150.0	79.0	53
Pb	**80.0**	**248.7**	**311**
Rb	x	46.4	x
Sb	**20.0**	**136.7**	**684**
Sn	x	88.5	x
Sr	x	184.0	x
Ti	x	3491.0	x
Zn	600.0	297.2	50
Zr	x	272.3	x

x = no listed ESL value

Bold inicates the top-5 XRF mean metal concentrations exceeding their ESL (As, Co, Hg,Pb, and Sb)

Table 6. Total number of XRF non-detects (NDs) for each metal, as percentage of the total 87 screens, with ranking of fewest number of NDs ranking highest.

metal	NDs	ND % of total	Rank*
As	9	10	11
Ba	19	22	13
Cd	**77**	**89**	**18**
Co	9	10	11
Cr	8	9	10
Cu	7	8	9
Fe	0	0	1
Hg	**75**	**86**	**17**
Mn	0	0	1
Ni	**58**	**67**	**16**
Pb	3	3	8
Rb	0	0	1
Sb	**52**	**60**	**14**
Sn	**53**	**61**	**15**
Sr	0	0	1
Ti	0	0	1
Zn	0	0	1
Zr	0	0	1

* =Fe, Mn, Rb, Sr,Ti, Zn, and Zr all tie for highest rank (1st), or lowest number of NDs with zero.
Bold indicates poorest five scores, or highest count of NDs.

Table 7. Linear regression R^2 values of XRF against ICP-MS metal concentration results. Rating categorized as strong ($R^2 > 0.80$), or weak fit ($R^2 < 0.80$), with ranking by R^2 value of all 18 metals.

metal	R^2 value	rating of fit	Rank
As	**0.970**	**strong**	**3**
Ba	0.050	weak	17
Cd	0.200	weak	14
Co	0.080	weak	16
Cr	0.690	weak	12
Cu	0.750	weak	11
Fe	0.800	strong	10
Hg	**0.990**	**strong**	**1**
Mn	**0.860**	**strong**	**6**
Ni	**0.850**	**strong**	**7**
Pb	**0.950**	**strong**	**4**
Rb	0.820	strong	9
Sb	**0.880**	**strong**	**5**
Sn	0.850	strong	7
Sr	0.310	weak	13
Ti	0.100	weak	15
Zn	**0.980**	**strong**	**2**
Zr	0.001	weak	18

Bold indicates top seven scoring R^2 values (< 0.90). Here in alphabetical order: As, Hg, Mn, Ni, Pb, Sb, and Zn.

Table 8. Alphabetical list of the 18 metals with p-values from Wilcoxon tests on XRF and ICP-MS results. Strong ratings (P > 0.100) and weak (P < 0.100) are listed, where strong indicates similar distributions.

metal	p-value	rating
As	**0.744**	**strong**
Ba	**0.152**	**strong**
Cd	0.001	weak
Co	0.000	weak
Cr	0.000	weak
Cu	0.002	weak
Fe	0.103	strong
Hg	0.007	weak
Mn	**0.652**	**strong**
Ni	**0.800**	**strong**
Pb	**0.626**	**strong**
Rb	0.000	weak
Sb	0.002	weak
Sn	0.002	weak
Sr	0.000	weak
Ti	0.000	weak
Zn	**0.630**	**strong**
Zr	0.000	weak

*** bold indicates strongest or most similar metal distributions with the seven highest p-values.**

9. Figures

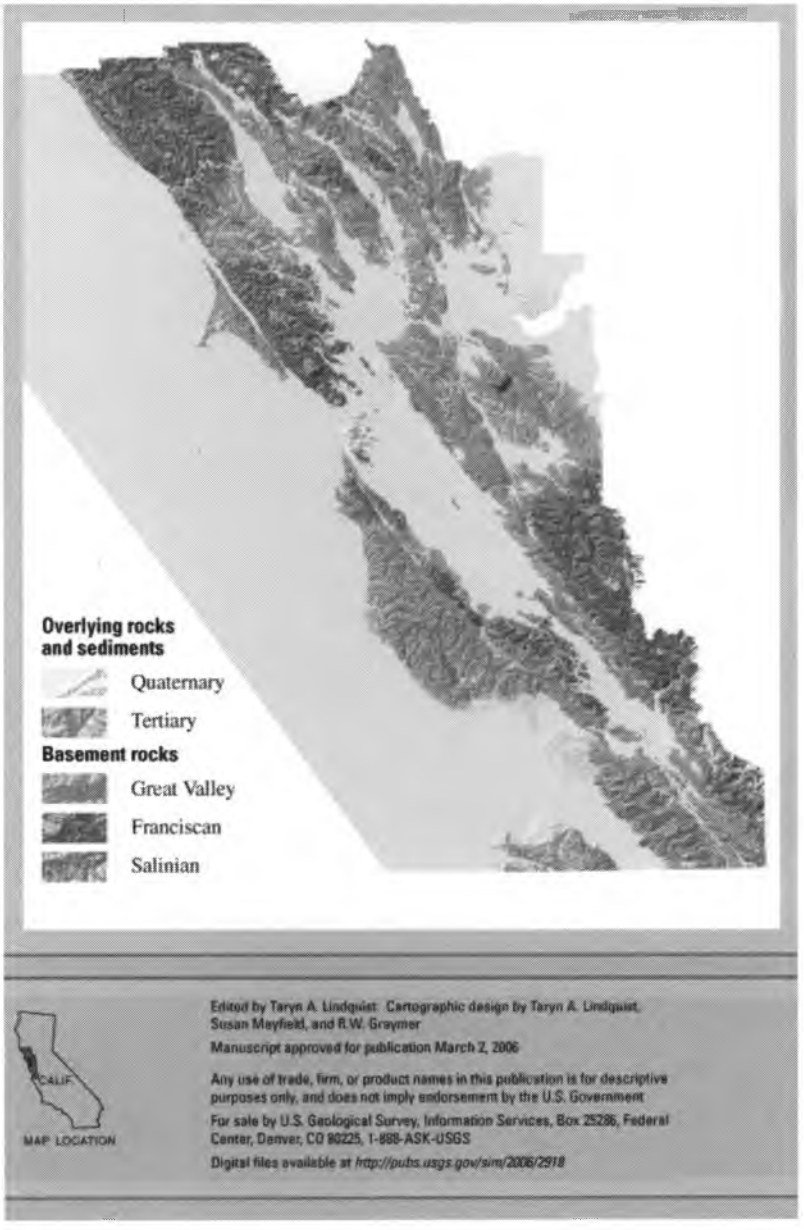


Figure 1. Simplified geologic map of the San Francisco Bay Region, depicting basement rocks, overlying Tertiary rocks, and Quaternary surficial sediments. Modified from Graymer et al., 2006.

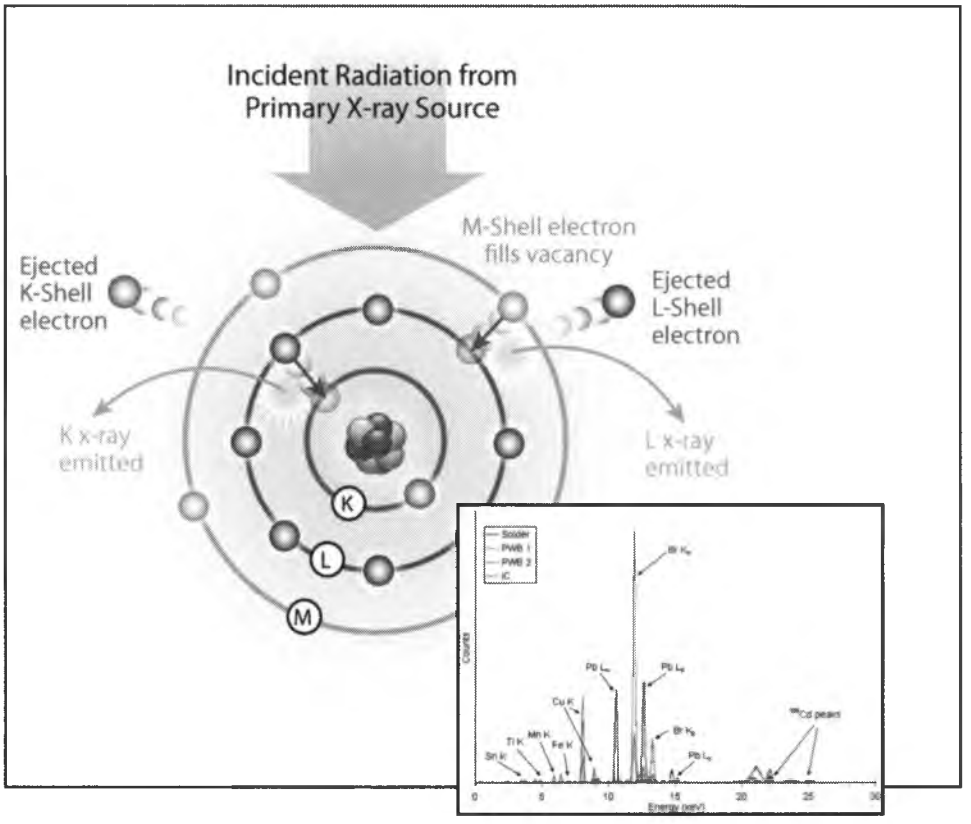


Figure 2. Simplified illustration of X-ray fluorescence (XRF) process on a single atom, where incident x-ray emits inner shell electron (K) and subsequent replacement by the outer shell electrons (L, M) and the characteristic x-ray emission (L shell) from the difference in energy between inner and outer shell electrons. The inset graph shows typical results from an XRF device with variable energy emission readings characteristic to specific elemental signatures. Image modified from Portable Analytical Solutions, 2013.

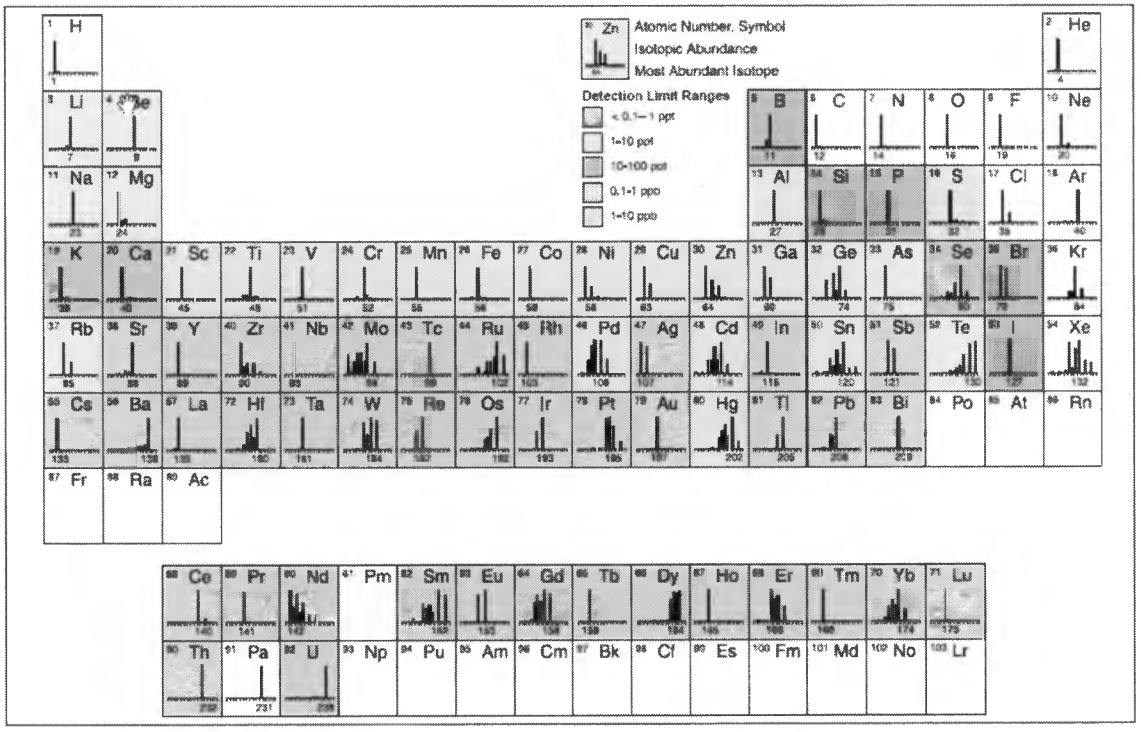


Figure 3. Approximate ICP-MS detection limits for known elements including the 18 metals considered in this study. (Courtesy of PerkinElmer, Inc.) (Wolf et al., 2005).

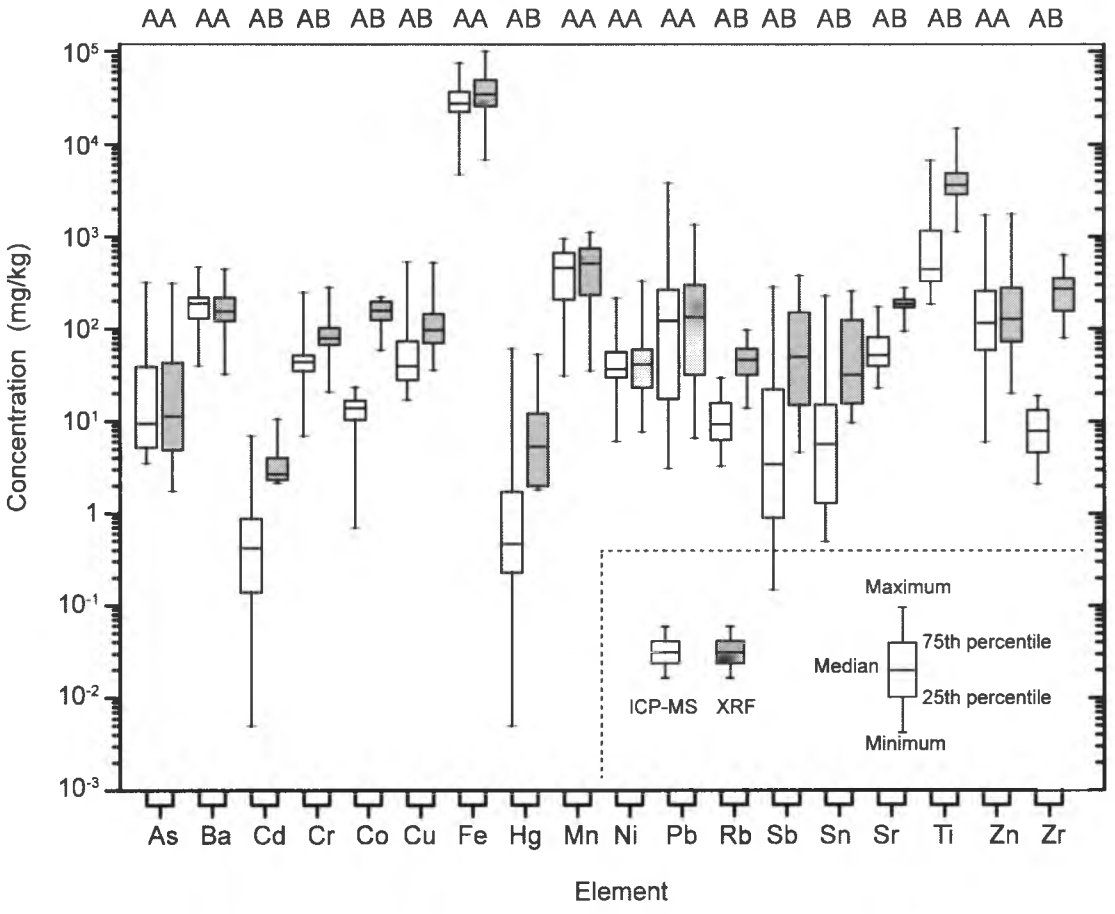


Figure 4. Distribution of the concentrations of 18 metals from ICP-MS and XRF analysis. Metal concentrations with different letters (A B) have significantly different concentrations at $\alpha = 0.1$ (Wilxocon rank-sum test).

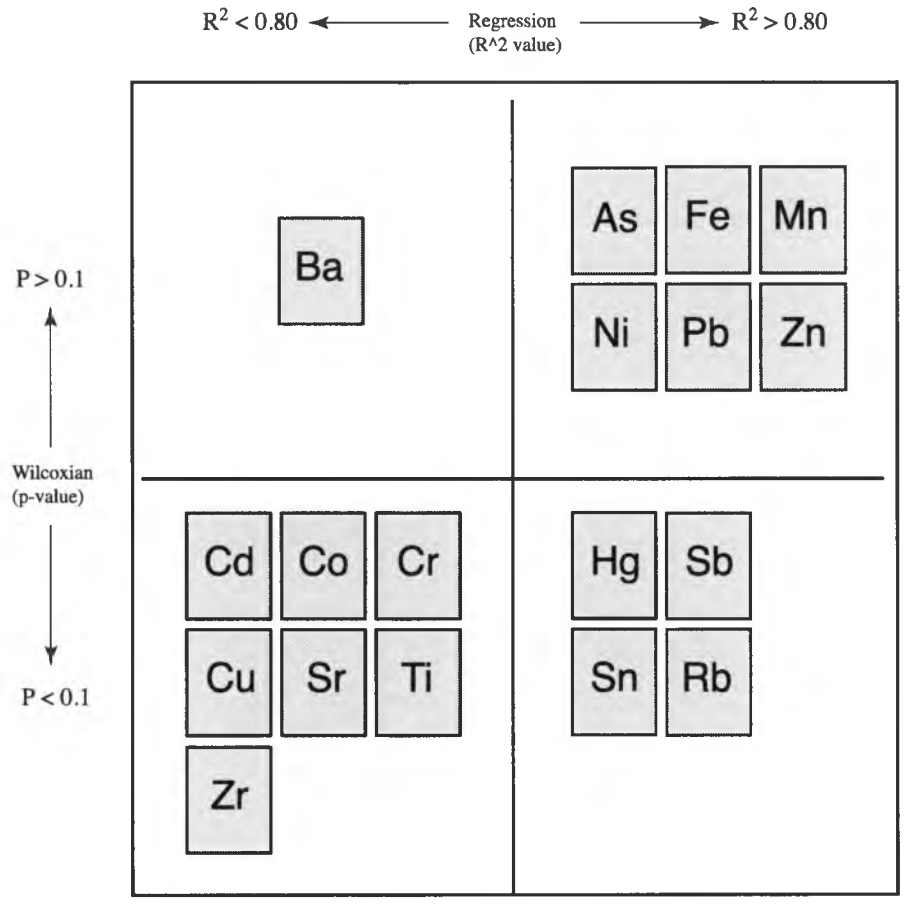


Figure 5. Diagram with four quadrants that categorizes metals according to their respective R^2 and P-values. The upper-right quadrant is defined by higher R^2 values (>0.80) and high Wilcoxon P-values (>0.10), the upper left is defined by low R^2 (<0.80) and high P-values, the lower left is defined by both lower R^2 and P-values, and the final quadrant, the lower right is defined by high R^2 and low P-values.

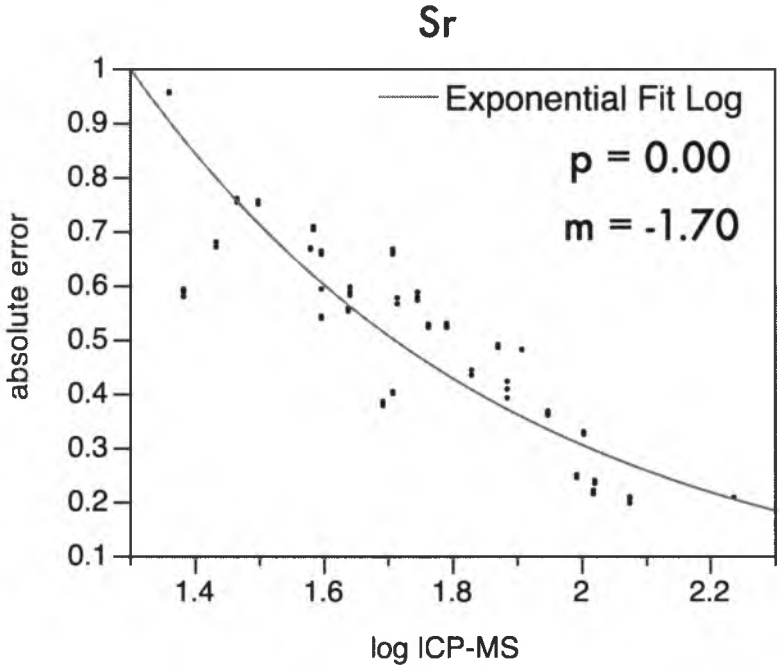


Figure 6. An example of the exponential regression plots where log-transformed concentrations of XRF and ICP-MS differences (absolute error) are plotted as a function of log-transformed ICP-MS concentration. Excellent agreement ($p < 0.05$) was found for 16 of the 18 metals (Appendix 2). Slope (m) is stated along with the p-value for each metal.

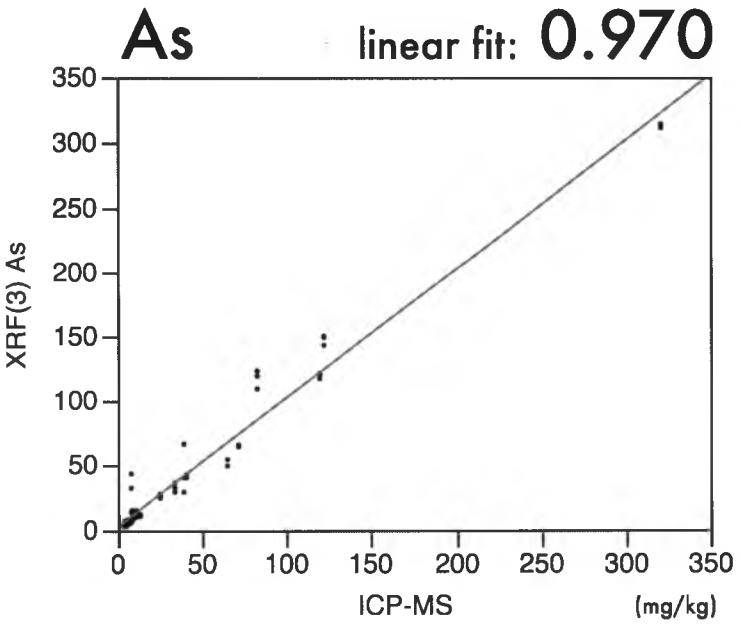
metal	P-value	m
As	0.02	-0.06
Ba	0.00	-1.58
Cd	0.00	-0.83
Co	0.00	-0.66
Cr	0.00	-2.06
Cu	0.00	-2.35
Fe	**0.41**	**-0.26**
Hg	0.00	-1.05
Mn	**0.66**	**-0.11**
Ni	0.04	-0.96
Pb	0.00	-0.56
Rb	0.00	-0.24
Sb	0.00	-0.63
Sn	0.00	-1.61
Sr	0.00	-1.70
Ti	0.00	-1.10
Zn	0.04	-0.46
Zr	0.01	-0.53

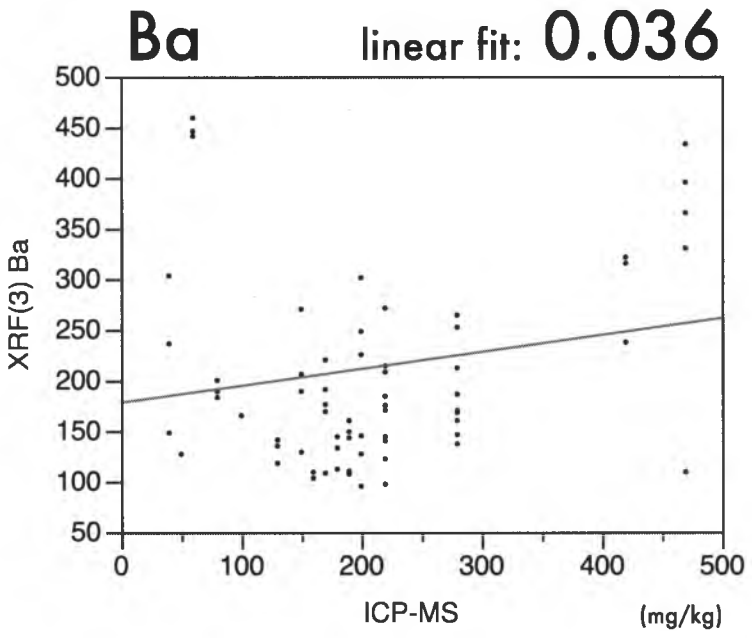
*** bold indicates the two metals with rgsultin p -values notewithin the 95% confidnce le vel (Fe, Mn).**

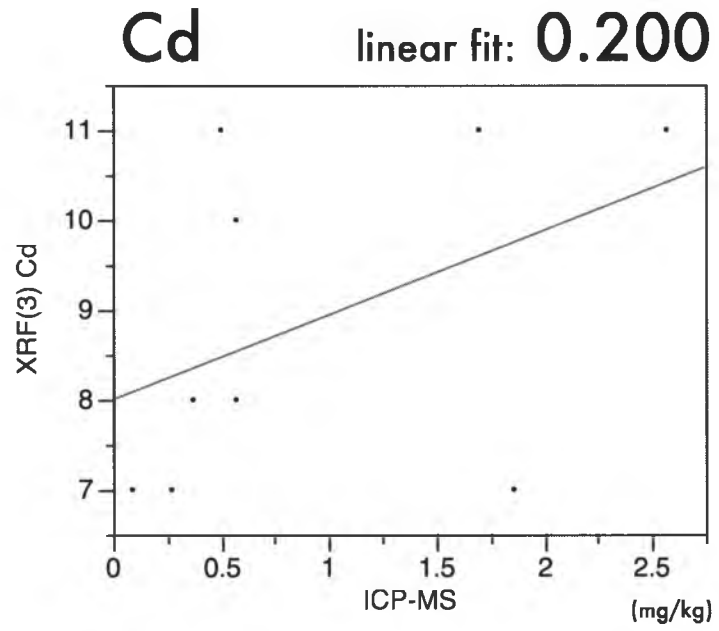
Figure 7. Alphabetical list of the 18 metals with resulting p-values and slope (m) from log-transformed plots (Fig.6). Statistical significance of the exponential regression is determined at the 95% confidence level ($\alpha = 0.05$). 16 of the 18 metals scored within the 95% confidence level, leaving two metals (Fe, Mn) resulting outside of the confidence level.

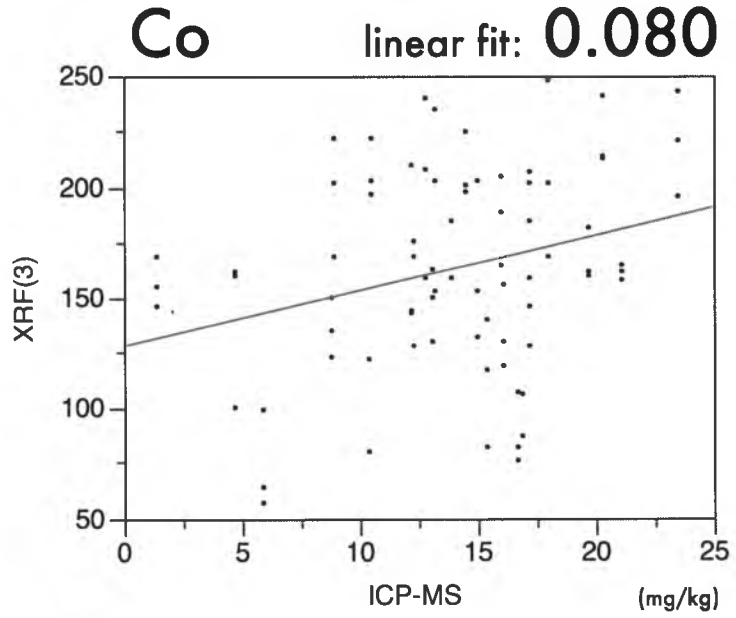
10. Appendices

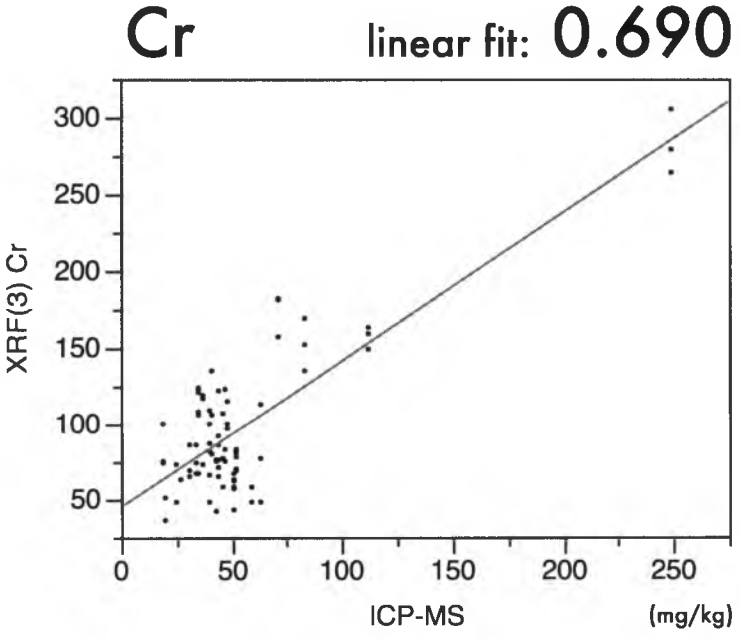
Appendix 1. Linear regressions of the 18 metals XRF screening results against the ICP-MS concentrations, listed in alphabetical order with R^2 best-fit line and value. All concentrations listed in mg/kg, axis scales vary greatly by each metal. Both Pb and Ti have two regressions each, one with higher end members excluded (lower R^2), and the other included (higher R^2).

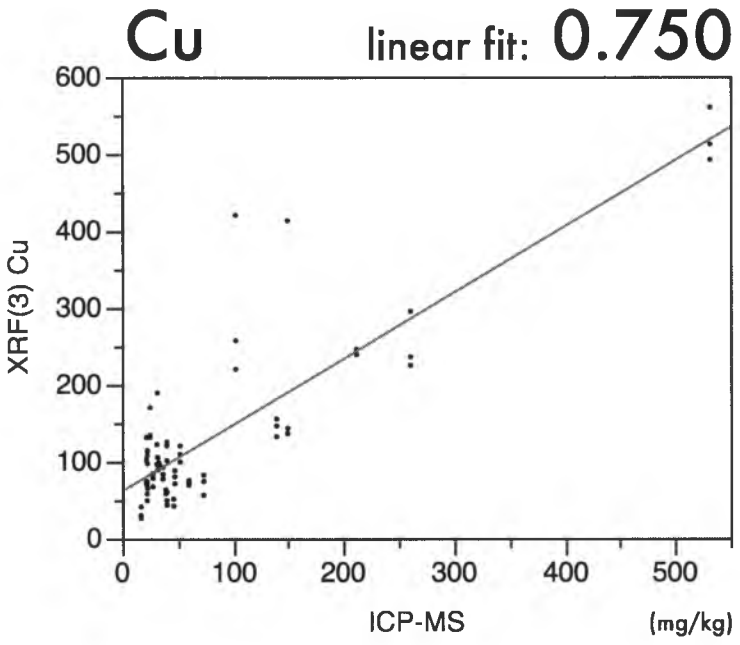


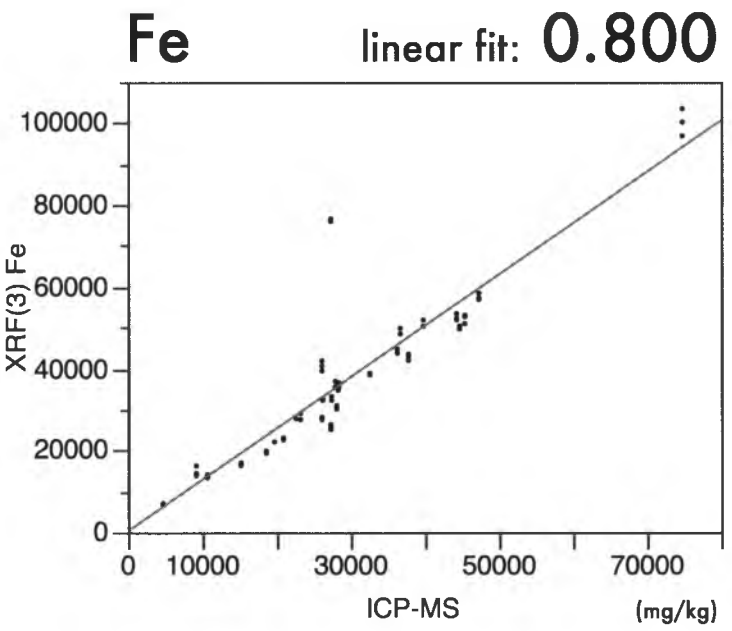

Ba
linear fit: 0.036
XRF(3) Ba
500
450
400
350
300
250
200
150
100
50
0
100
200
300
400
500
ICP-MS
(mg/kg)

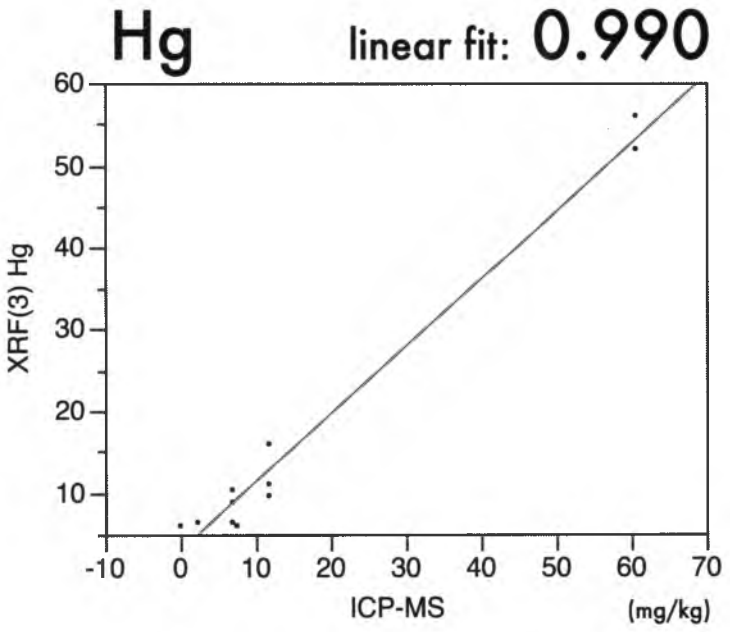

Cd
linear fit: 0.200
XRF(3) Cd
11
10
9
8
7
0
0.5
1
1.5
2
2.5
ICP-MS
(mg/kg)

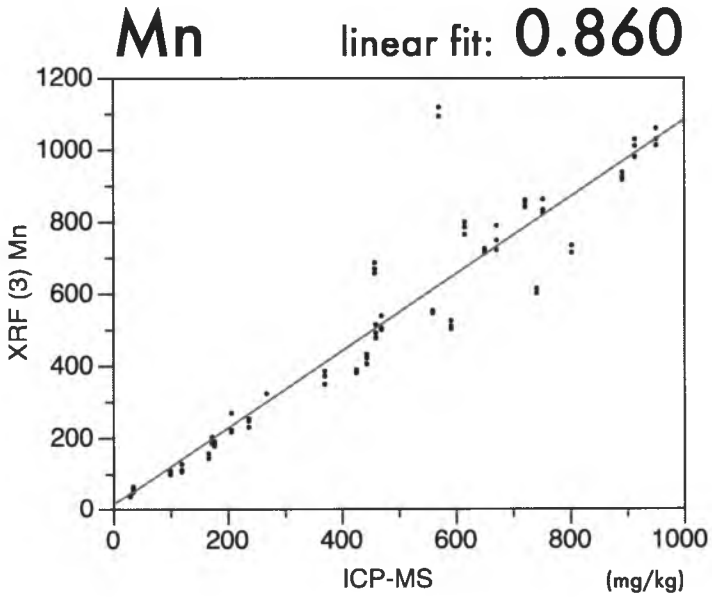

Co
linear fit: 0.080
XRF(3)
250
200
150
100
50
0
5
10
15
20
25
ICP-MS
(mg/kg)

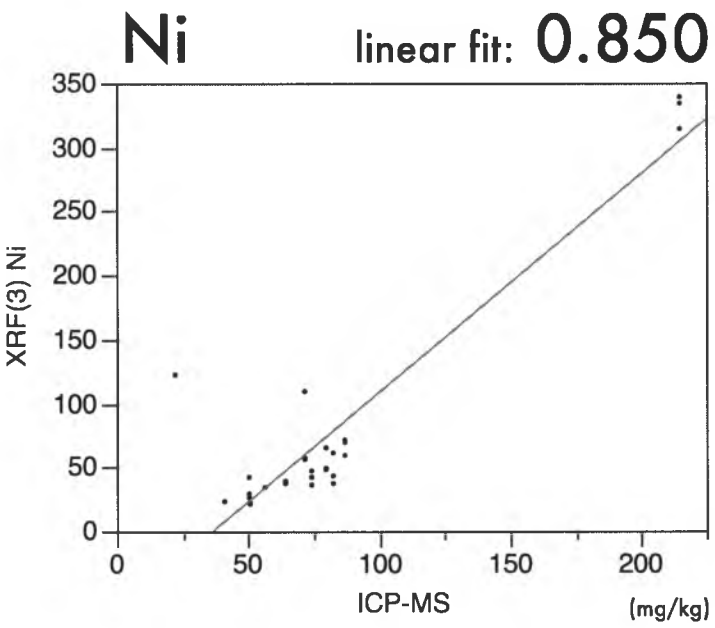

Cr
linear fit: 0.690
XRF(3) Cr
300
250
200
150
100
50
0
50
100
150
200
250
ICP-MS
(mg/kg)

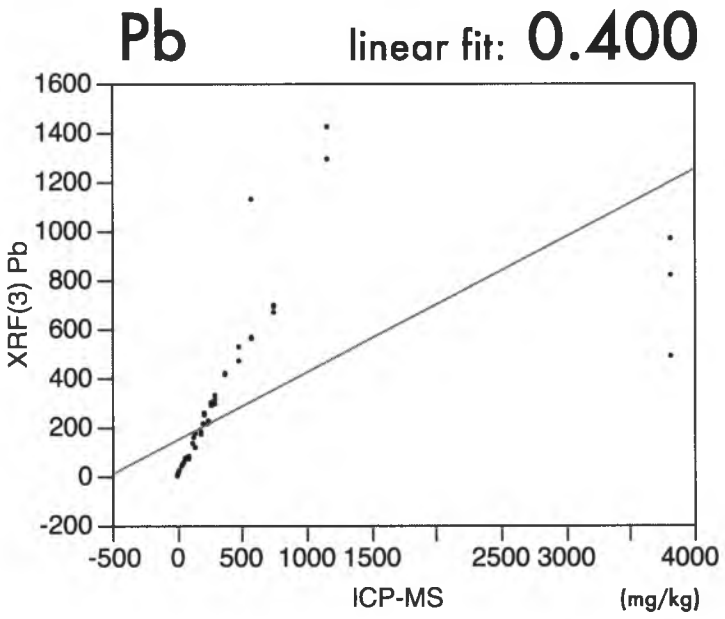

Cu
linear fit: 0.750
XRF(3) Cu
600
500
400
300
200
100
0
0
100
200
300
400
500
ICP-MS
(mg/kg)

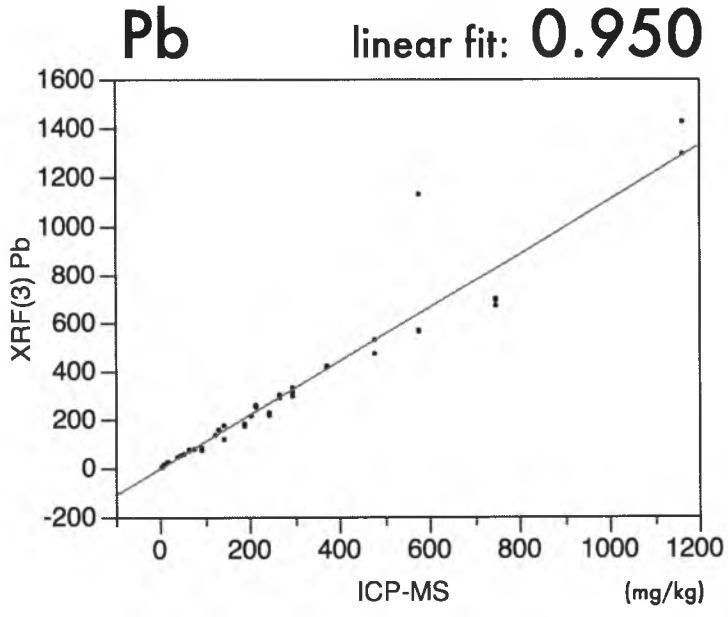

Fe
linear fit: 0.800
100000
80000
60000
40000
20000
0
XRF(3) Fe
0
10000
30000
50000
70000
ICP-MS
(mg/kg)

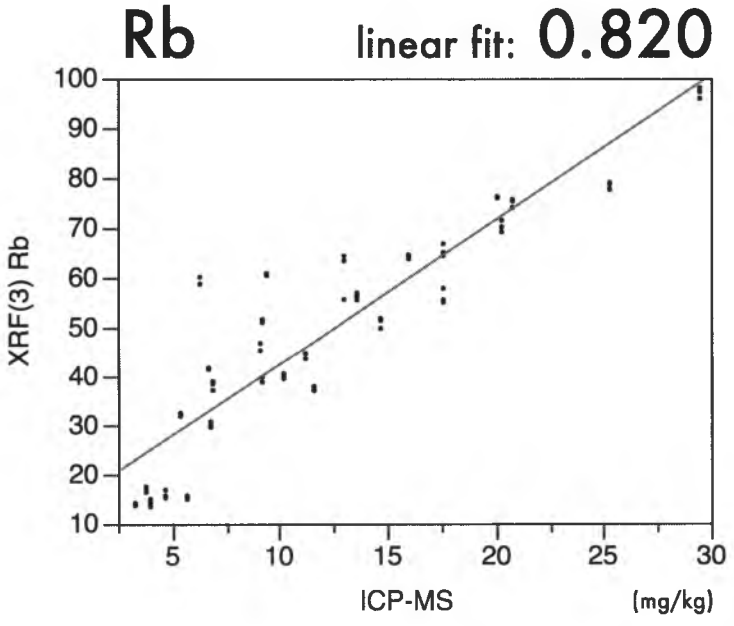

Hg
linear fit: 0.990
60
50
40
30
20
10
XRF(3) Hg
-10
0
10
20
30
40
50
60
70
ICP-MS
(mg/kg)

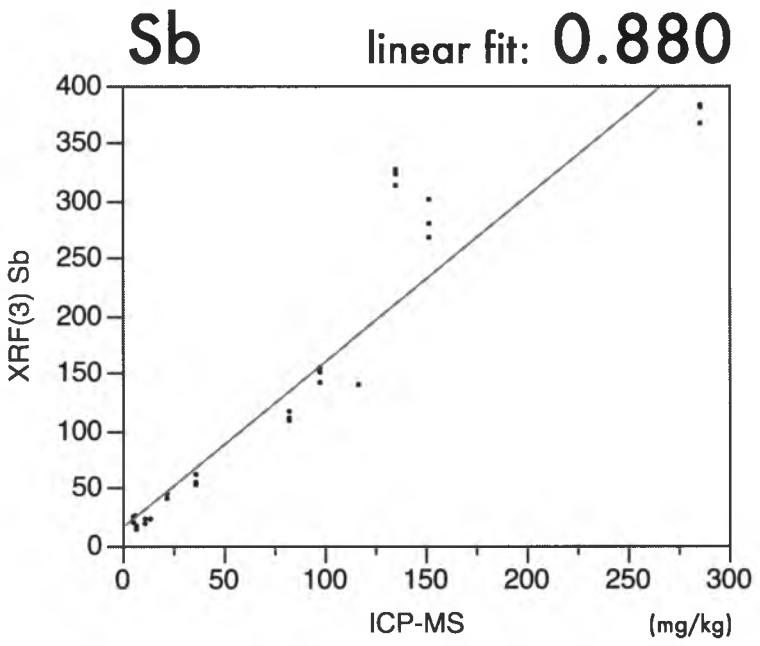

Mn
linear fit: 0.860
XRF (3) Mn
1200
1000
800
600
400
200
0
0
200
400
600
800
1000
ICP-MS
(mg/kg)

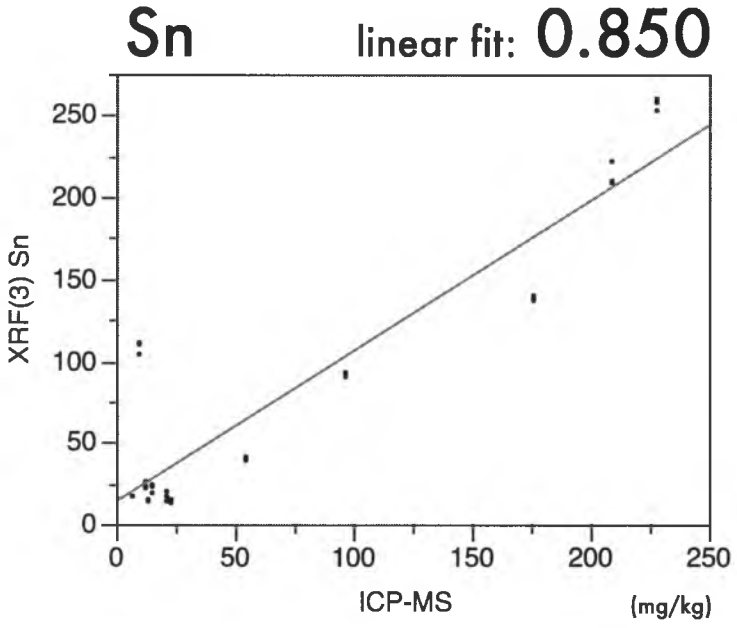

Ni
linear fit: 0.850
350
300
250
200
150
100
50
0
XRF(3) Ni
0
50
100
150
200
ICP-MS
(mg/kg)

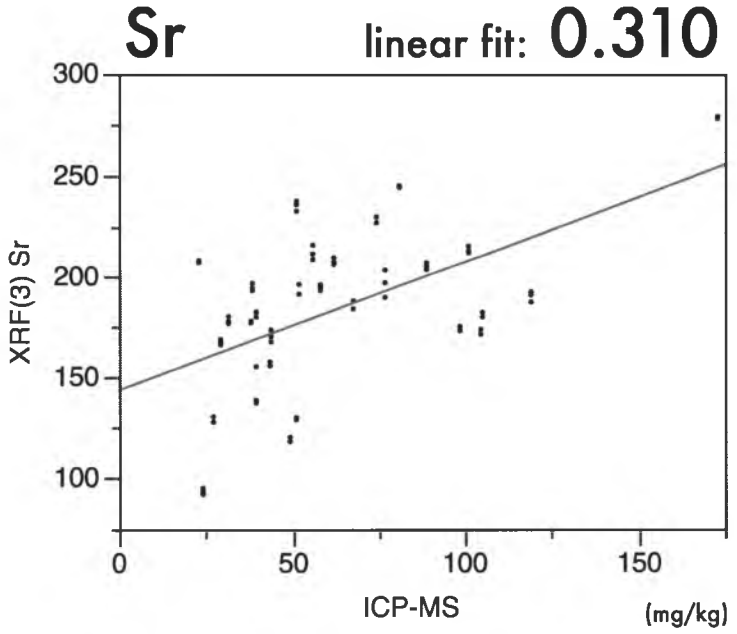

Pb
linear fit: 0.400
XRF(3) Pb
1600
1400
1200
1000
800
600
400
200
0
-200
-500
0
500
1000
1500
2500
3000
4000
ICP-MS
(mg/kg)

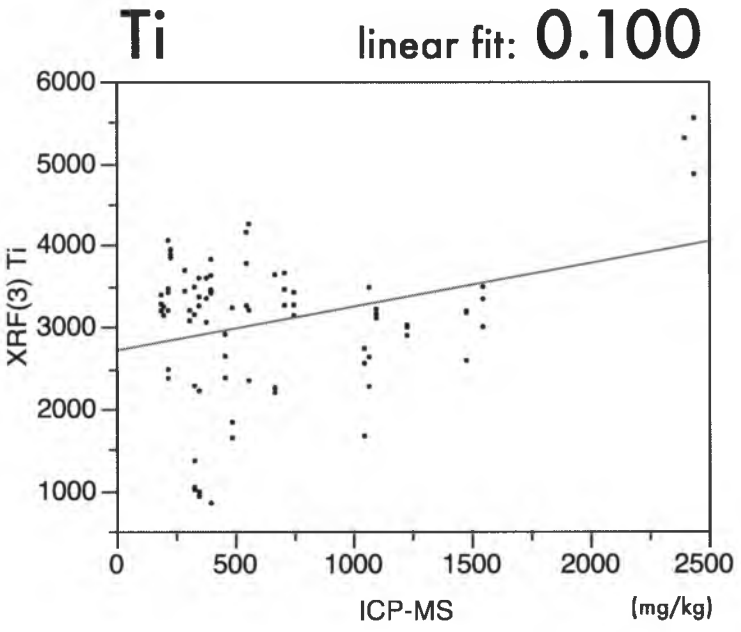

Pb
linear fit: 0.950
XRF(3) Pb
1600
1400
1200
1000
800
600
400
200
0
-200
0
200
400
600
800
1000
1200
ICP-MS
(mg/kg)

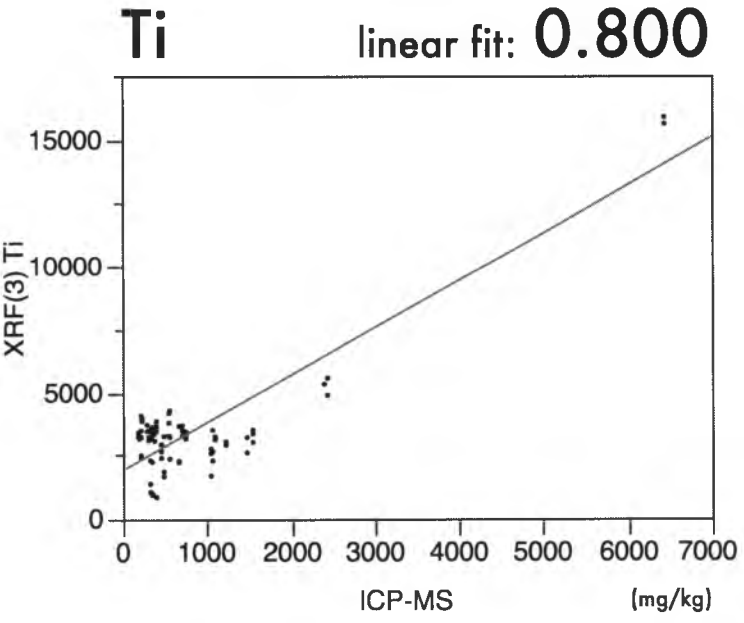

Rb
linear fit: 0.820
XRF(3) Rb
100
90
80
70
60
50
40
30
20
10
5
10
15
20
25
30
ICP-MS
(mg/kg)

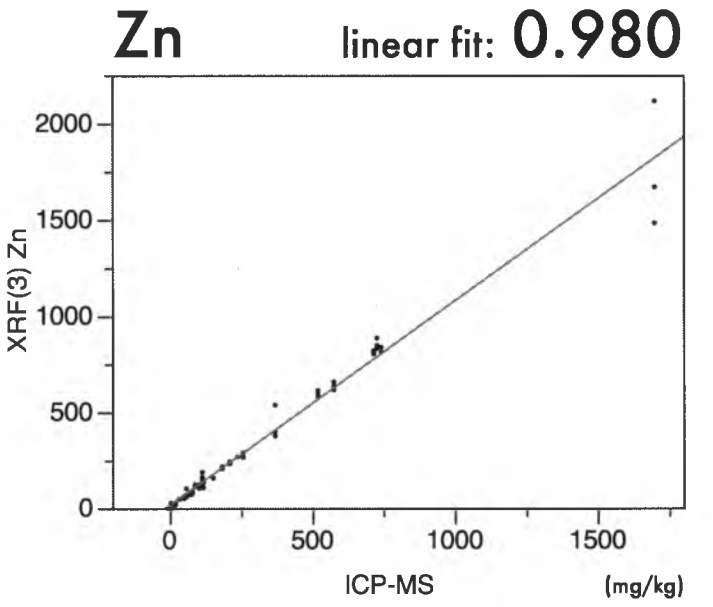

Sb
linear fit: 0.880
XRF(3) Sb
400
350
300
250
200
150
100
50
0
0
50
100
150
200
250
300
ICP-MS
(mg/kg)

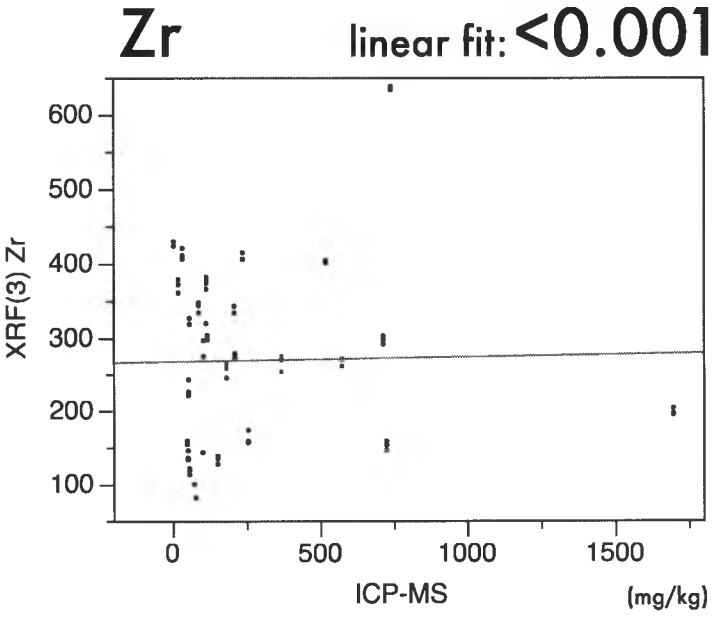

Sn
linear fit: 0.850
XRF(3) Sn
ICP-MS
(mg/kg)
0
50
100
150
200
250


Sr
linear fit: 0.310
XRF(3) Sr
ICP-MS
(mg/kg)
100
150
200
250
300
0
50
100
150

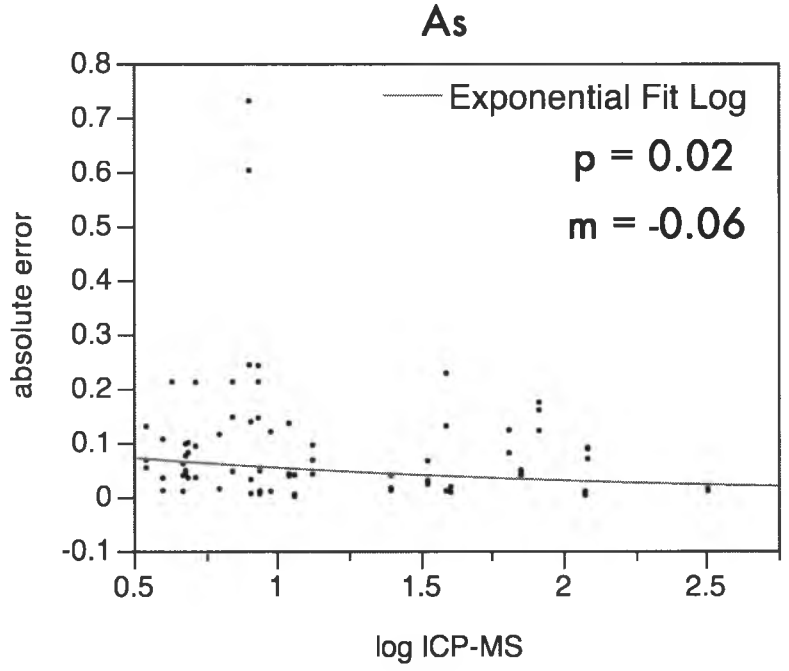

Ti
linear fit: 0.100
6000
5000
4000
3000
2000
1000
XRF(3) Ti
0
500
1000
1500
2000
2500
ICP-MS
(mg/kg)

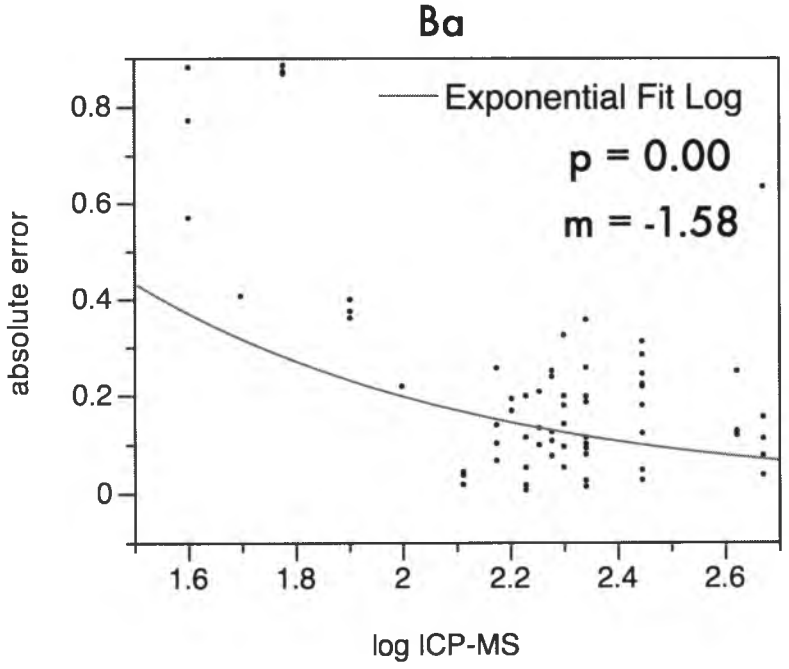

Ti
linear fit: 0.800
15000
10000
5000
0
XRF(3) Ti
0
1000
2000
3000
4000
5000
6000
7000
ICP-MS
(mg/kg)

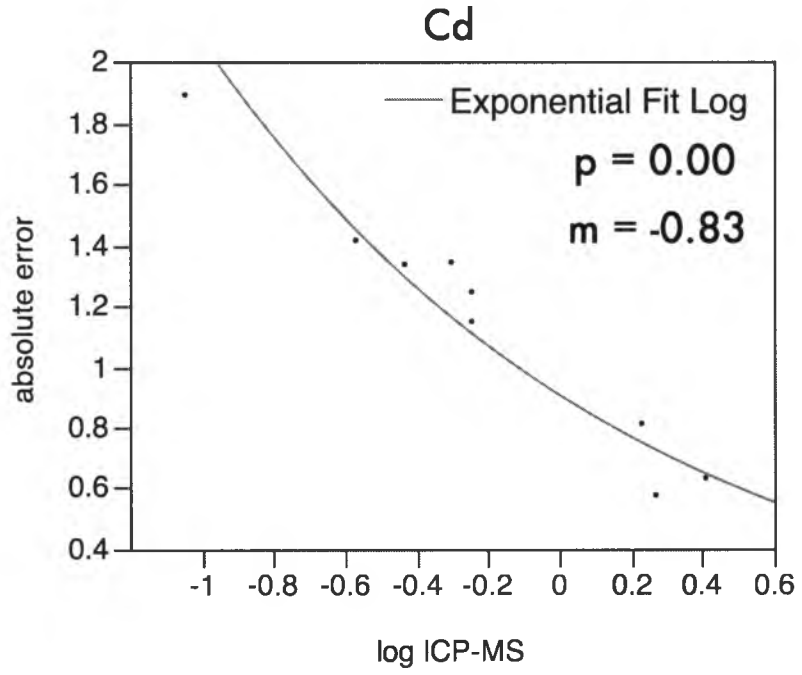

Zn
linear fit: 0.980
2000
1500
1000
500
0
XRF(3) Zn
0
500
1000
1500
ICP-MS
(mg/kg)

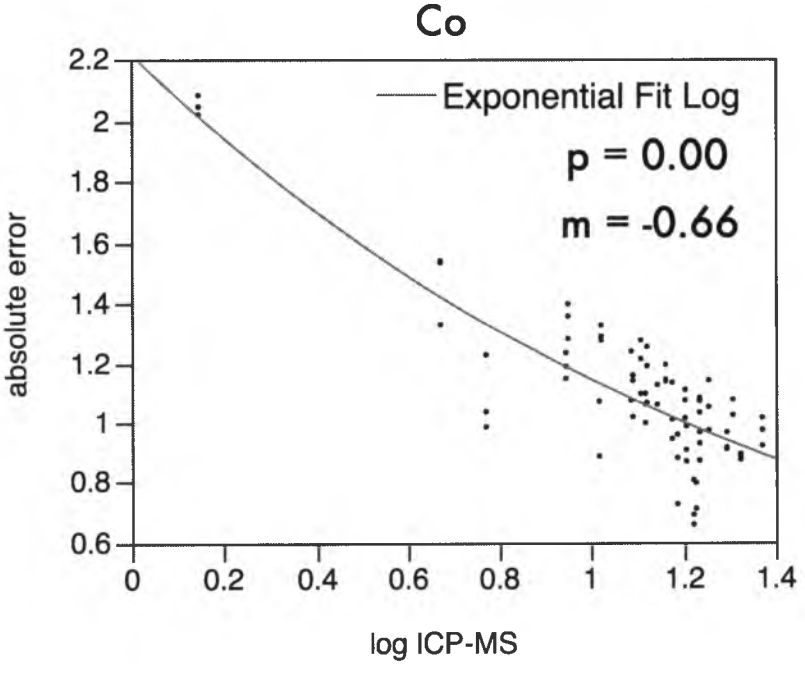

Zr
linear fit: <0.001
600
500
400
300
200
100
XRF(3) Zr
0
500
1000
1500
ICP-MS
(mg/kg)

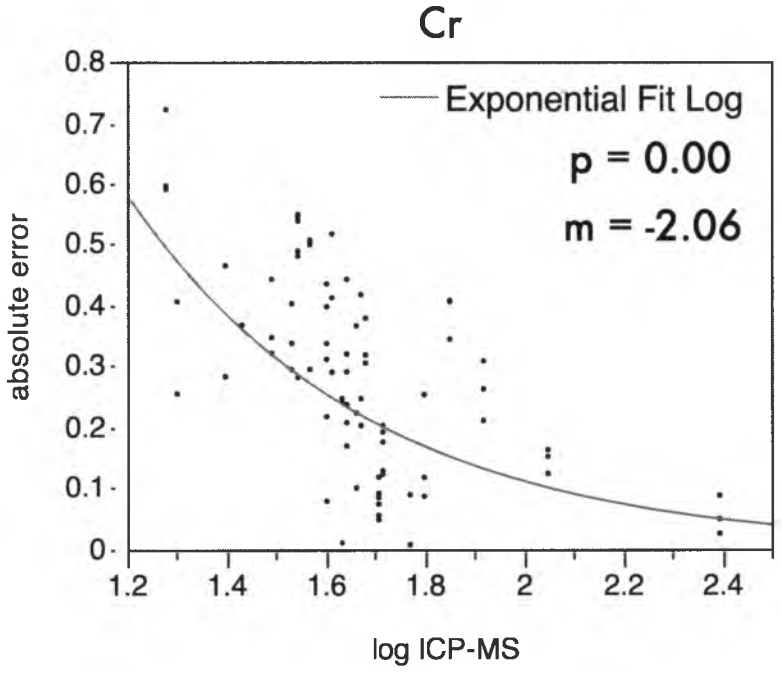
Appendix 2. Log-transformed exponential regression plots for all 18 metals with p-value and slope (m) indicated for each regression.

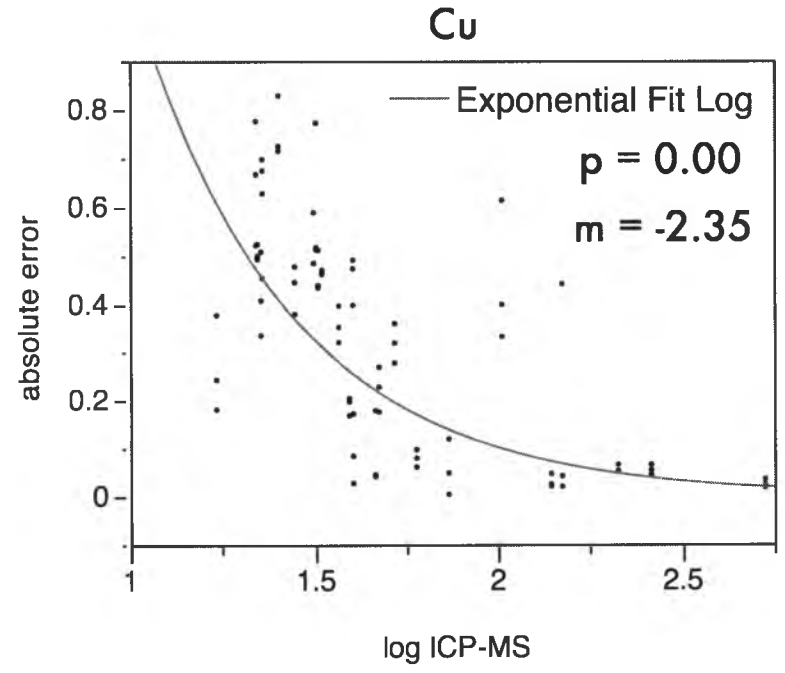


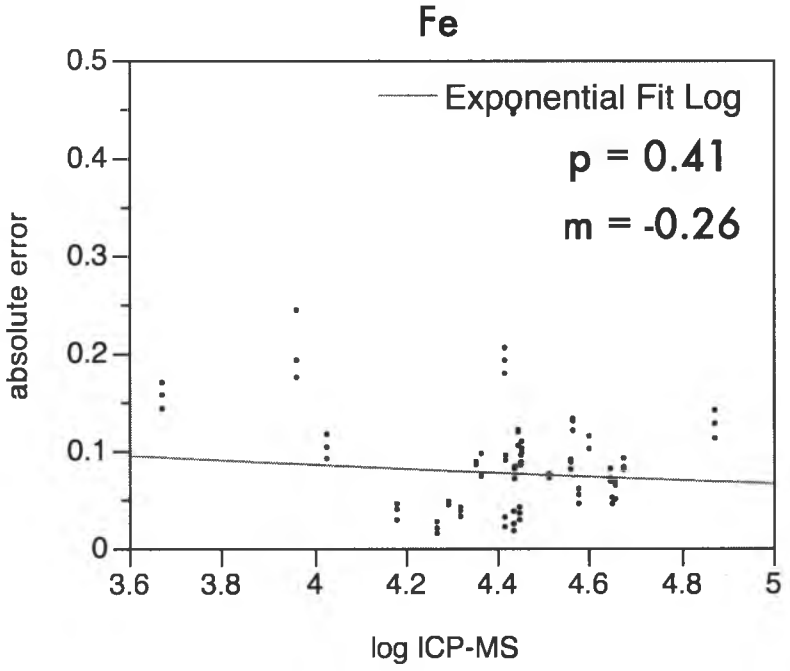


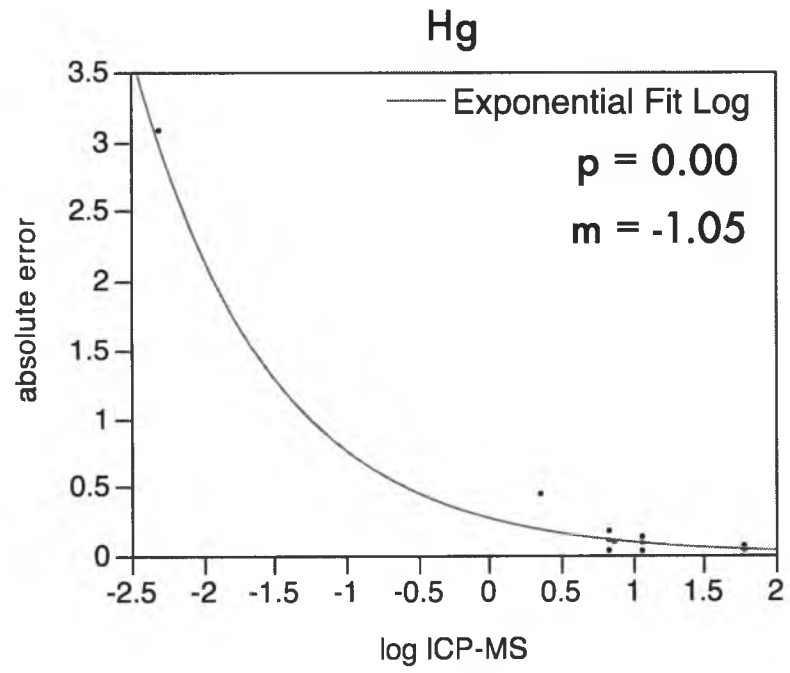

Cd
Exponential Fit Log
p = 0.00
m = -0.83
absolute error
log ICP-MS
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
-1
-0.8
-0.6
-0.4
-0.2
0
0.2
0.4
0.6

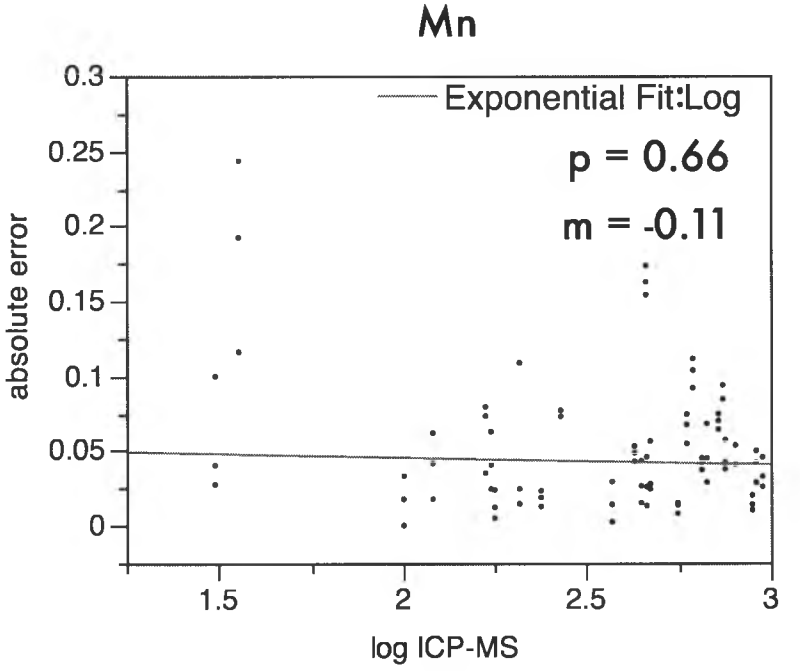

Co
Exponential Fit Log
p = 0.00
m = -0.66
absolute error
log ICP-MS
2.2
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0
0.2
0.4
0.6
0.8
1
1.2
1.4

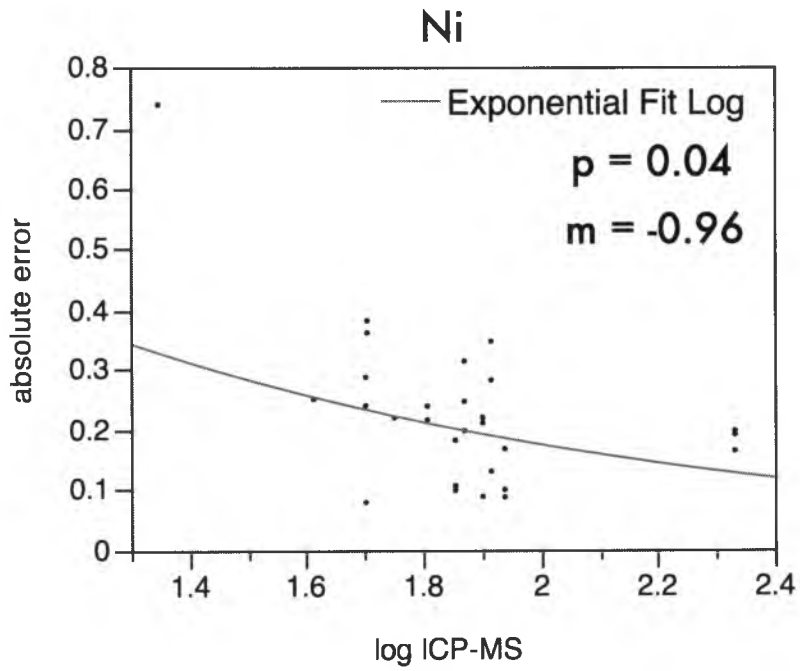

Cr
Exponential Fit Log
p = 0.00
m = -2.06
absolute error
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
1.2
1.4
1.6
1.8
2
2.2
2.4
log ICP-MS

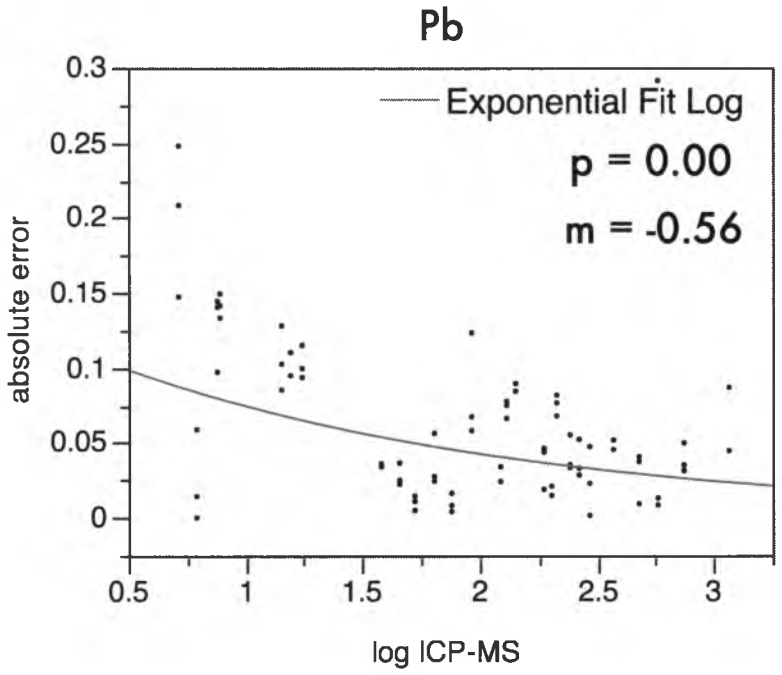

Cu
Exponential Fit Log
p = 0.00
m = -2.35
absolute error
0.8
0.6
0.4
0.2
0
1
1.5
2
2.5
log ICP-MS

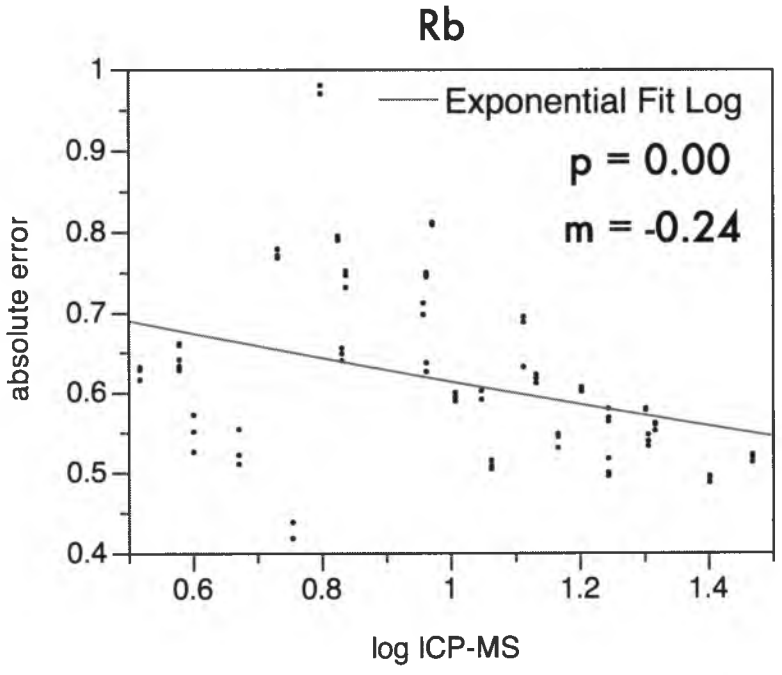

Fe
Exponential Fit Log
p = 0.41
m = -0.26
absolute error
0.5
0.4
0.3
0.2
0.1
0
3.6
3.8
4
4.2
4.4
4.6
4.8
5
log ICP-MS

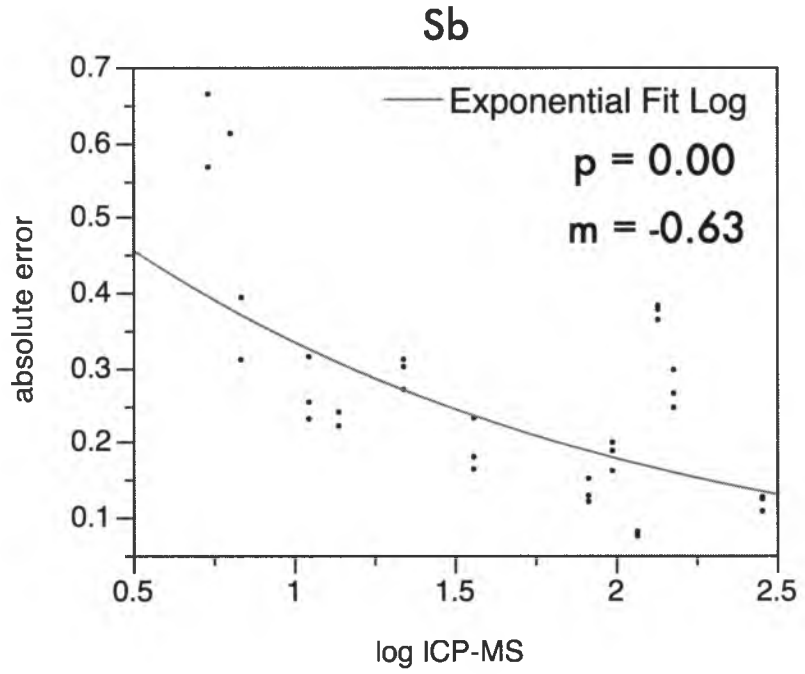

Hg
Exponential Fit Log
p = 0.00
m = -1.05
absolute error
3.5
3
2.5
2
1.5
1
0.5
0
-2.5
-2
-1.5
-1
-0.5
0
0.5
1
1.5
2
log ICP-MS

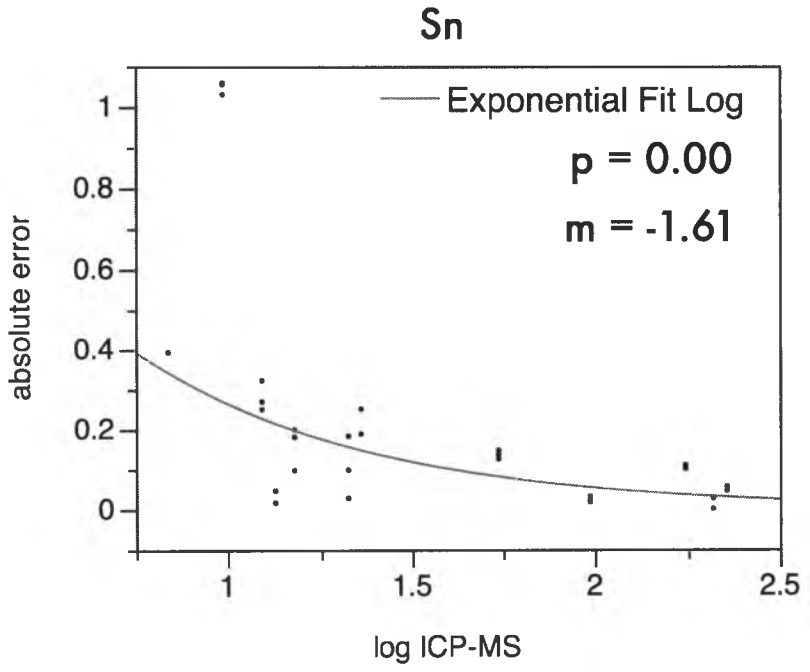

Mn
Exponential Fit:Log
p = 0.66
m = -0.11
absolute error
0.3
0.25
0.2
0.15
0.1
0.05
0
1.5
2
2.5
3
log ICP-MS

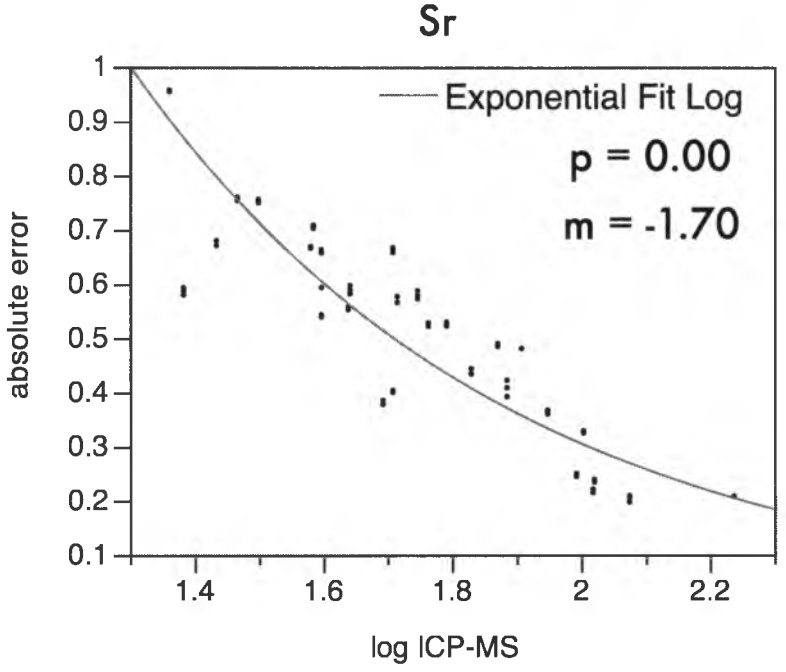

Ni
Exponential Fit Log
p = 0.04
m = -0.96
absolute error
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
1.4
1.6
1.8
2
2.2
2.4
log ICP-MS

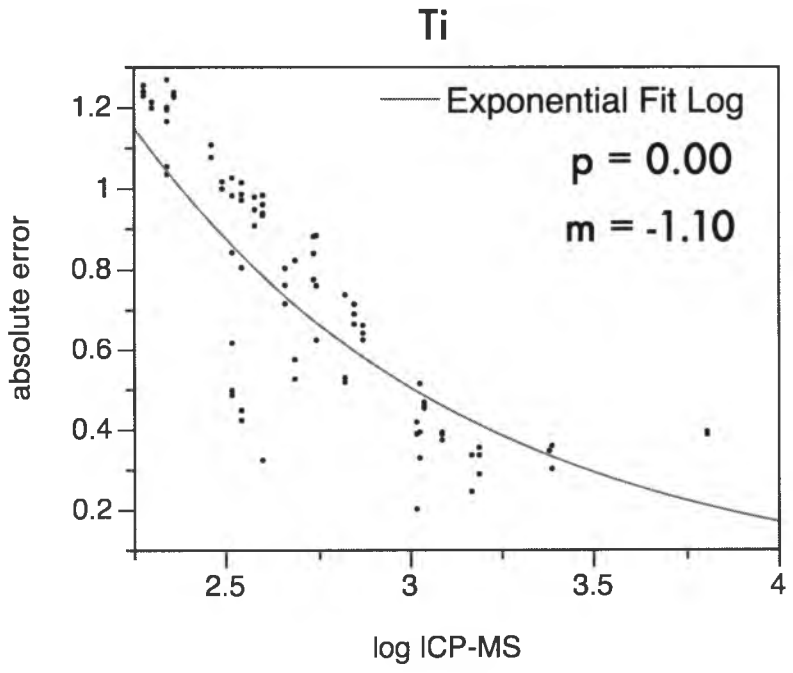

Pb
Exponential Fit Log
p = 0.00
m = -0.56
absolute error
log ICP-MS
0.3
0.25
0.2
0.15
0.1
0.05
0
0.5
1
1.5
2
2.5
3

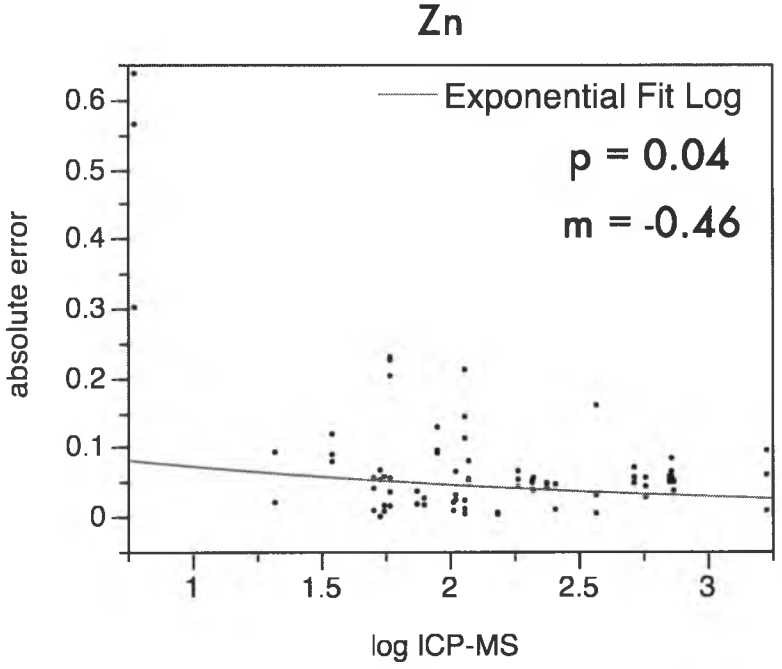

Rb
Exponential Fit Log
p = 0.00
m = -0.24
absolute error
log ICP-MS
1
0.9
0.8
0.7
0.6
0.5
0.4
0.6
0.8
1
1.2
1.4

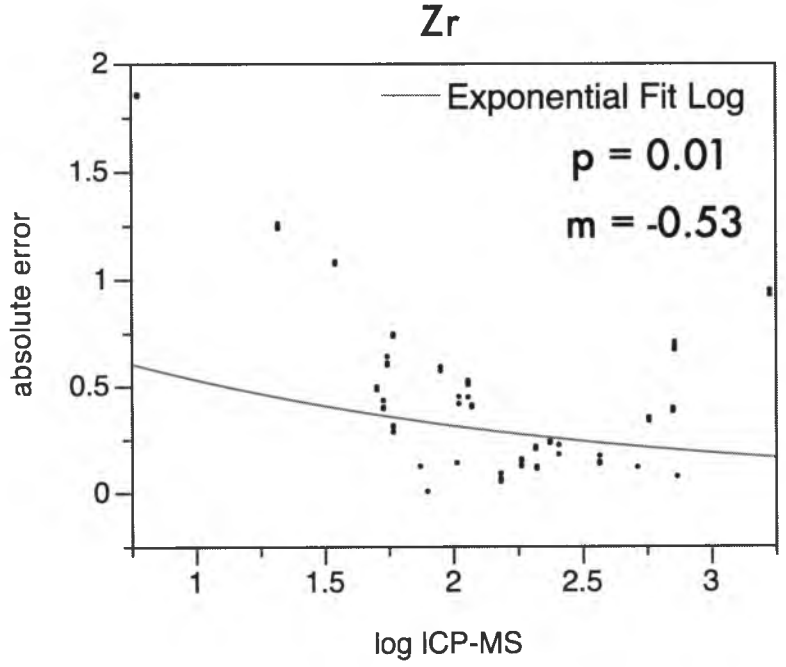

Sb
Exponential Fit Log
p = 0.00
m = -0.63
absolute error
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.5
1
1.5
2
2.5
log ICP-MS


Sn
Exponential Fit Log
p = 0.00
m = -1.61
absolute error
1
0.8
0.6
0.4
0.2
0
1
1.5
2
2.5
log ICP-MS


Sr
Exponential Fit Log
p = 0.00
m = -1.70
absolute error
log ICP-MS
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
1.4
1.6
1.8
2
2.2


Ti
Exponential Fit Log
p = 0.00
m = -1.10
absolute error
log ICP-MS
1.2
1
0.8
0.6
0.4
0.2
2.5
3
3.5
4


Zn
Exponential Fit Log
p = 0.04
m = -0.46
absolute error
0.6
0.5
0.4
0.3
0.2
0.1
0
1
1.5
2
2.5
3
log ICP-MS


Zr
Exponential Fit Log
p = 0.01
m = -0.53
absolute error
2
1.5
1
0.5
0
1
1.5
2
2.5
3
log ICP-MS