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Geochemical indices allow estimation of heavy metal background concentrations in soils

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[1] Defining background concentrations for heavy metals in soils is essential for recognizing and managing soil pollution. However, background concentrations of metals in soils can vary naturally by several orders of magnitude. Moreover, many soils have also been subject to unquantifiable anthropogenic inputs of metals, in some cases, for centuries. Hence determination of heavy metal background concentrations in soils has to date been fraught with difficulty. Here we demonstrate that there are associations between the background heavy metal and Fe or Mn contents in soils which appear to be consistent for seven important heavy metals of environmental concern. The relationships are remarkably independent of both soil type and climatic setting. These observations provide the basis for a series of general equations from which it is proposed Southeast Asian including Australian, and possibly worldwide background concentrations for As, Cr, Co, Cu, Ni, Pb, and Zn in soils can be derived.   INDEX TERMS: 1803 Hydrology: Anthropogenic effects; 9810 General or Miscellaneous: New fields (not classifiable under other headings); 6324 Policy Sciences: Legislation and regulations; KEYWORDS: baseline, heavy metal contaminant, risk assessment, soil pollution

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1. Introduction

[2] Regional background values for metals in soils have been typically estimated from the geometric mean, median or the 95th percentile concentration ranges of extensive survey data sampled from sites with presumed negligible anthropogenic inputs [*Kabata-Pendias et al.*, 1992]. Given the naturally high variability of trace element concentrations in the environment, the robustness of the values are proportional to the sampling intensity. Moreover, the values are only assumed to be "reasonable" (i.e., free of anthropogenic contamination) if they do not exceed the "normal" concentration range of metals found in the soil parent rock materials [*Kabata-Pendias et al.*, 1992] or other comparative databases from the region. Recommendations have been made [*Darnley*, 1997; *Darnley et al.*, 1995] for the development of a global geochemical baseline data set to provide consistency to this somewhat arbitrary approach. Comprehensive background data would also help dispel the widespread misconception that trace element concentrations above "background" values

are necessarily anthropogenic in origin [*Rasmussen*, 1998]. However, major anthropogenic emissions of heavy metals resulting in significant contamination of soils have occurred, particularly in the Northern Hemisphere [*Nriagu and Pacyna*, 1988] where much of the work to date to elucidate background concentrations has been focused. This makes the task of establishing background concentrations for these metals using the traditional reconnaissance sampling methods extremely difficult [*Chen et al.*, 1999].

[3] It has long been recognized that geochemical associations exist between trace elements [*Goldschmidt*, 1937]. However, it has been widely assumed that the biogeochemical and hydrologic processes that lead to the genesis and weathering of soils have resulted in differential dispersion of elements throughout different soil types [*Rose et al.*, 1979; *Palumbo et al.*, 2000]. At the localized level, mineral explorationists have used observed associations between elements to normalize pathfinder element concentrations in order to enhance anomaly contrasts [*Fletcher et al.*, 1986]. However, there have been no attempts to extrapolate these relationships to a more global scale, or to consider the value of such relationships in the derivation of background concentrations for heavy metal pollutants. The aim of this study was to examine whether relationships which have been observed at the local level between different elements and semi-conservative properties of soils [*Fletcher et al.*, 1986; *Baize*, 1997] could be extrapolated to a wider scale to

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provide a useful index for establishing background concentrations for heavy metals in soils.

2.   Materials and Methods

2.1.   Soils

[4]   Soils with wide ranging characteristics (Table 1 and Figure 1) were sampled at different depths from 129 sites scattered across Australia, 11 sites in Namibia, 301 sites across Malaysia, and 317 sites across Thailand. Of the sites in Australia, 94 were from extremely remote areas (RA) as were the sites in Namibia, and were considered likely to have received negligible anthropogenic heavy metal inputs. The remaining soils originated from agricultural areas (AG) with various cultivation histories and hence may have been subject to different degrees of contamination, in particular by As, Cd, Pb, Zn, and Cu [McLaughlin et al., 2000].

2.2.   Soil Analyses

[5]   Soil samples were air-dried and sieved to <2 mm prior to analyses. Soil pH was determined in water with a soil:solution ratio of 1:2.5. Aqua regia soluble metals were extracted following the procedure of Zarcinas et al. [1996]. Briefly, subsamples (∼0.5 g) of the soils were placed in 5 mL of a solution containing concentrated HNO_3 (Mallinkrodt Ultrapure) and HCl (BDH AR Grade) (1:3 v:v) and progressively heated until approximately 1 mL of acid remained in the digestion tubes. The samples were then diluted to 20 mL with deionized H_2O (MilliQ) and filtered. Samples analyzed by ICP-MS (RA soils) were further diluted (1:20, v:v) to minimize interferences. The remaining samples (AG soils) were analyzed by ICP-AES, with metal concentrations below the detection limit for this instrument assessed using F-AAS or GF-AAS where necessary. Certified reference material (Stream Sediment Reference Materials, CANMET), replicate samples, and blanks were included with each of the digests to verify accuracy and reproducibility of the results. All equipment was cleaned prior to use by soaking in 10% HNO_3 and rinsing in deionized H_2O. Clay content was determined using the pipet particle size distribution method [Gee and Bauder, 1986]. Organic C content was determined using a LECO total C analyzer following acidification with HCl. Amorphous Fe-oxide content was assessed following sample extraction with citrate/dithionite [Rayment and Higginson, 1992] and crystalline Fe-oxide content assessed using an oxalate extraction method [Rayment and Higginson, 1992]. The cation exchange capacity was determined using an NH_4Cl exchange procedure [Rayment and Higginson, 1992].

2.3.   Statistical Analyses

[6]   Regression analyses were performed to evaluate relationships between metal concentrations and other soil parameters. Regression fits obtained for the AG and RA data sets were compared to determine evidence of non-parallelism and differences in intercepts. These statistical analyses were conducted using Genstat 5.

3.   Results and Discussion

[7]   Despite soil pH and organic carbon being well recognized as playing a significant role in heavy metal binding in

soils [McBride, 1989], regression analysis failed to demonstrate any relationship between these factors and total metal concentration in the soils. Similarly, no relationship was observed between total metal concentration and clay concentration from the Australian soils data set (RA and AG), versus Fe oxide content from the Australian AG soils data set, or versus cation exchange capacity (CEC) from the Malay AG soils data set.

[8]   For the RA soils, highly significant correlations (p < 0.001) were found between (correlation coefficient given in brackets): As (0.69), Cr (0.82), Cu (0.60), Ni (0.48), Pb (0.66), and Zn (0.61) and aqua regia soluble Fe content, or, in the case of Co (0.71) versus aqua regia soluble Mn content, for log transformed data. Comparison of RA with AG soils showed that As, Cr, Cu, and Ni were slightly but significantly (p < 0.001) enriched, particularly at high metal contents, with respect to Fe in the RA soils (Figure 2). Most likely, this reflects the fact that the RA soils were sampled from areas of potential mineral exploration interest. In contrast, the AG soils were found to be significantly (p < 0.001) enriched in Pb and Zn compared to the RA soils (Figure 2). For the former element, this is possibly indicative of widespread contamination which has occurred as a result of use of Pb additives in petrol [Heyvaert et al., 2000], and for the latter element, due to the necessity to maintain adequate inputs of Zn for crop growth in agricultural soils [Holmgren et al., 1993]. The relationship between Co and Mn was found to be the same for the AG and RA soils (slope, p > 0.9, intercept, p > 0.09). In contrast to a commonly expressed contention, no significant relationship was found between Cd and Zn or between Cd and any of the other soil parameters assessed in this study.

[9]   Accounting for the above considerations, the data sets were refined as follows. Regression relationships between Fe and As, Co, Cr, Cu, and Ni were determined from the linear correlations of the whole data set, following removal of 5% of observations with the highest positive residuals. For Pb and Zn, only the RA soil data set was used to define the relationship. The parameters describing these regressions are shown in Table 2. Predictions of the background metal concentrations (Table 3) were then made using the equation derived from the line encompassing the upper 95th percentile of the data. This line is described by an equation having the form

$$\log[M] = a \log[Fe] + c, \tag{1}$$

where [M] is the predicted background concentration of metal (mg kg^{-1}) in the soil, a is the slope coefficient of the regression equation (Table 2), [Fe] is the concentration of Fe in the soil (%), and c is the respective constant for each metal as shown the last column of Table 2.

[10]   The composition of highly weathered soils often bears little resemblance to the underlying rock material due to both physical mixing and chemical sorting processes [Rose et al., 1979; Palumbo et al., 2000]; hence the observed correlations are unlikely to be due to original chemical stratification during the evolution of the parent rock material. However, Fe and Mn compounds are directly involved in scavenging metals, with Fe-oxides contributing to a significant proportion of the heavy metal binding

Table 1. Ranges of Selected Properties for the Different Soils[a]

Property	Remote Soils Range	Agricultural Soils		
		Australian Range	Malay Range	Thai Range
pH$_{water}$	4.2−9.5	4.5−8.5	2.5−7.0	3.5−8.1
Organic carbon, %	0.04−1.5	0.2−7.0	0.01−12	0.06−13
Clay, %	0.5−40	1−59	n.a.	n.a.
CEC	n.a.	n.a.	1.7−79	n.a.
Fe-cit/dit, mg kg^{-1}	n.a.	700−110400	n.a.	n.a.
Fe-ox, mg kg^{-1}	n.a.	100−11200	n.a.	n.a.

[a]Note: n.a. denotes "not assessed."

capacity of soil [*Fergusson*, 1991]. The lack of correlation between the trace elements and the Fe-oxide content of the soils does not preclude this as being the primary mechanism linking Fe and trace element concentration as it is well known that extraction methods for determining the Fe-oxide content of soils are somewhat non-selective [*Hall and Pelchat*, 1999]. Hence it is possible that aqua regia soluble Fe provides a better estimate of the total capacity of Fe compounds for metal binding in soils than the standard oxalate/citrate dithionite extraction procedures which were used here to assess the Fe-oxide fraction.

[11] The pH of the soils collected in this study tended toward the more acidic end of the pH scale (Figure 1a); hence further investigation is required to assess whether the observed relationships are valid across calcareous soils. Also, the soils were sampled primarily from areas subject to a high degree of weathering. Therefore the soils considered in this study cannot necessarily be defined as a globally representative sample. Nevertheless, the soils were collected from a highly diverse range of environments and, as is clearly shown in Table 1, encompassed a very wide range of soil properties. Studies reporting reliable background concentrations of heavy metals in soils from other parts of the world have rarely provided associated information on the total or aqua regia soluble content of Fe in the soils. There are a few exceptions, including *Chen et al.* [1999], who also observed significant positive correlations between total Fe and a range of heavy metals including As, Cr, Cu, Ni, Pb, and Zn for a suite of soils from Florida. Data from this and two other studies in the Northern Hemisphere, where care was taken to select soils at low risk of contamination, are shown overlain on data from the current study (Figure 2). These studies reported mean values for different heavy metals and total Fe content in 441 soils from Florida [*Chen et al.*, 1999], in podzols (31 samples), luvisols (34 samples), cambisols (51 samples), and fluvisols (8 samples) from Poland [*Kabata-Pendias et al.*, 1992], and individual data from two soil horizons at six sites in Italy [*Palumbo et al.*, 2000]. A similar relationship between heavy metal and Fe concentration in the soils can be clearly seen in these samples (Figure 2), suggesting that the regression equations are likely to be relevant for predicting background concentrations on a more global scale.

[12] The inability to accurately define background concentrations of metals in soils has significant environmental and economic consequences. In many countries an index value is used to assess the presence of contamination by a given metal and triggers a decision for either no action or further investigation/remedial intervention. Expected back-

ground concentrations of heavy metals in soils with a given Fe/Mn content were calculated using equation (1) and are shown in Table 3. The constant used in the equation was based on the 95th percentile of the data. As can be seen from Figures 2a−2g, there was up to 2 orders of magnitude variation in background metal concentrations at a given soil Fe concentration. Hence it is important to recognise that the values provided in Table 3 are the maximum metal concentration that would be predicted for a given soil Fe content: The actual background concentration of metal in a given soil could be significantly lower. The index values used in
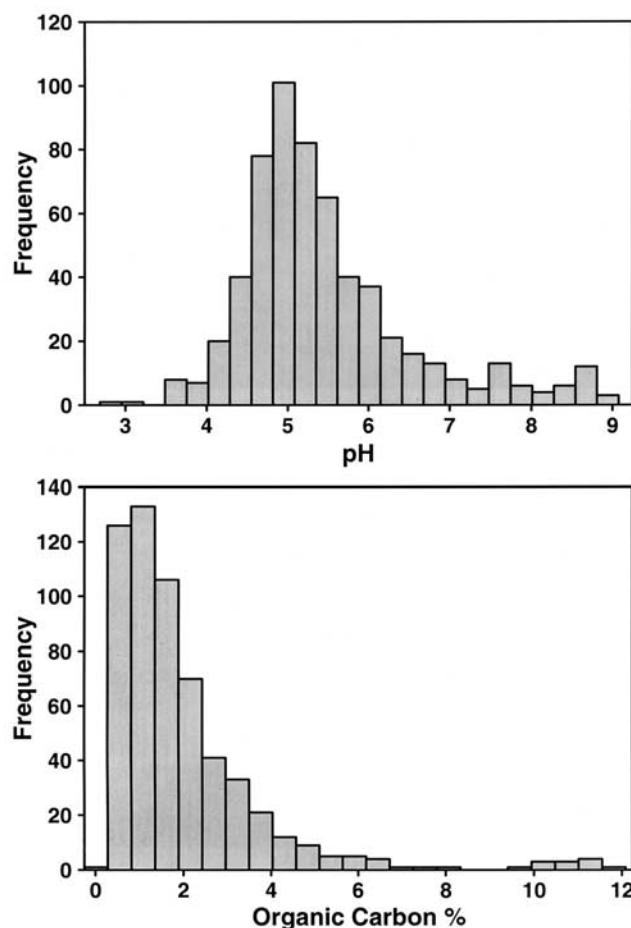

Figure 1. Distribution histograms showing the range and frequency of (a) soil pH and (b) organic carbon (%) in the soil sample data set.
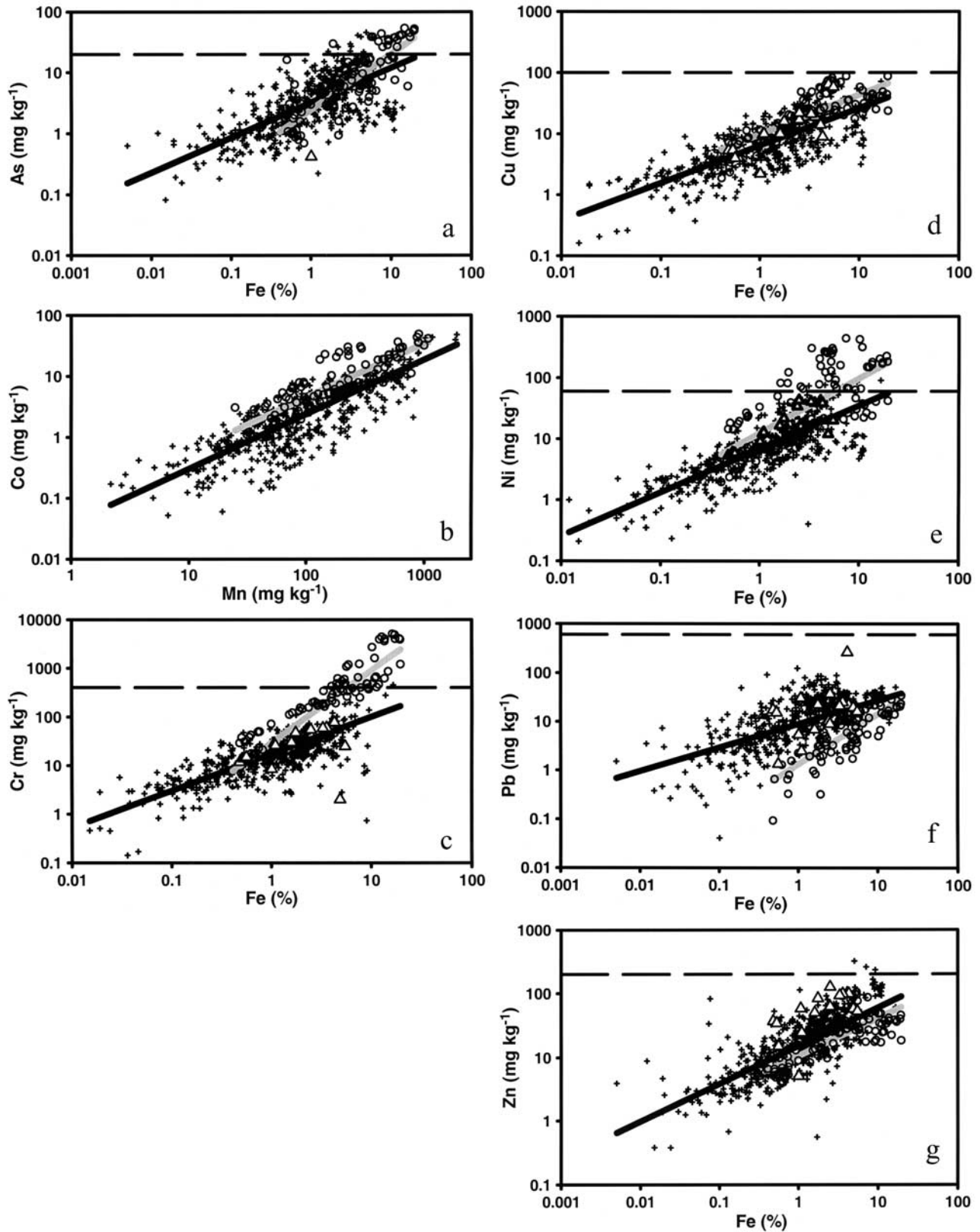
Figure 2. Relationship between heavy metal concentration and Fe or Mn concentration in the soils. AG soils data are represented by a cross, RA soils data are represented by an open circle, and Florida [*Chen et al.*, 1999], Polish [*Kabata-Pendias et al.*, 1992], and Italian [*Palumbo et al.*, 2000] data by an open triangle. The black line represents the regression fit for 95% of observations for the whole data set, the shaded line represents the regression fit for the RA soils only, and the dashed horizontal line shows the index value for Australian Ecological Investigation Levels [*NEPC*, 1999] (note, no index value currently exists for Co).

Table 2. Regression Parameters for the Relationship Between Heavy Metal and Fe, or Mn (for Co) Concentrations in the Soils and Also the Constant Calculated for the Line Which Describes the Upper 95th Percentile of the Data[a]

Element	Slope Coefficient	Constant for Regression	Standard Error of Estimate	r^2	Constant for 95th Percentile Line
As[b]	0.574	0.507	0.3550	0.50[b]	1.064
Co[b]	0.894	−1.409	0.3063	0.71[b]	
Cr[b]	0.750	1.242	0.3640	0.58[b]	1.916
Cu[b]	0.612	0.808	0.2840	0.61[b]	1.235
Ni[b]	0.702	0.834	0.3110	0.64[b]	1.381
Pb[c]	1.039	0.118	0.3014	0.66[c]	0.558
Zn[c]	0.589	1.024	0.2220	0.61[c]	1.529

[a]All parameters based on log transformed data.
[b]Observations from 95% of whole data set.
[c]Observations from the RA soils data set only.

Australia [*National Environment Protection Council (NEPC)*, 1999] are also shown in Table 3. It is obvious from this table that use of a single value has the potential to both seriously underestimate and overestimate the presence of metal contamination in soils. The single value obscures significant contamination that may have occurred in soils with low Fe/Mn contents, and can lead to costly, unachievable remediation targets in soils with high Fe/Mn contents. In contrast, use of the information provided in Table 3 to define the likelihood of contamination in a given soil provides a basis for enabling rational decisions to be taken that better identify true contamination across a wide range of soil types.

[13] The Netherlands, recognizing the inadequacy of referencing allowable metal concentrations to a single value, has produced more realistic values for background metal concentrations by using a sliding scale defined by the quantity of clay and organic matter in the soil. These properties were found to be related to background concentrations of As, Cd, Cr, Cu, Hg, Ni, Pb, and Zn in the Netherlands soils [*Lexmond and Edelman*, 1992]. This study did not report Fe and Mn contents, but these elements are likely to be correlated to clay content in Netherlands soils. As discussed above, clay concentration was not related to background metal content in our suite of soils. However, the data presented in our study shows that aqua regia soluble Fe/Mn can be used to provide an estimate of background soil metal concentrations in a more global context.

[14] The values presented in Table 3 may be used to infer that a soil is contaminated. However, one factor which needs to be considered to prevent a false conclusion that a soil is not contaminated (i.e., to minimize the risk of a Type II error) is that aqua regia soluble Fe/Mn are only quasi-conservative tracers of contamination: In some contaminated sites, these elements are co-contaminants with heavy metals. This is a particular issue for sites which have been contaminated with relatively "clean" materials, for example soils to which high-quality biosolids (i.e., those produced since the introduction of regulations restricting industrial discharges of heavy metals into wastewater systems [*Epstein*, 2003]) have been applied. An in-house survey of 24 biosolids produced across Australia in 1999 found aqua regia Fe contents ranging from <10g kg^{-1} up to 110 g kg^{-1} with a median value of 1.44 g kg^{-1} (I. Oliver, personal communication, 2003). The median value for Fe in biosolids from the United States was reported to be 11 g kg^{-1}

[*Epstein*, 2003]. Values for Fe in 10 biosolids and 28 other waste materials from Japan were found to range from 0.02 to 28 g kg^{-1} [*Zhang et al.*, 2001], and in eight biosolids from Poland, ranged from 17 to 51 g kg^{-1} [*Bodzek and Janoska*, 1999]. Assuming from these data that the concentration of Fe in biosolids would rarely if ever exceed 150 g kg^{-1} (i.e., 15%), Table 3 conservatively suggests that a Type II error could be incurred at sites receiving biosolids if metal concentrations in the biosolids were less than (mg kg^{-1}): As 55, Cr 630, Cu 90, Ni 160, Pb 60, and Zn 165. The concentrations of As, Cr, and Ni in biosolids produced today are typically lower than these values [*Epstein*, 2003]. The metals Pb and Cu are often, but not always, present at concentrations higher than these values [*Epstein*, 2003]. However, one metal which is consistently present in high concentrations in biosolids is Zn. The concentration of Zn in recently produced biosolids has been found to range from 320 to >2300 mg kg^{-1} in Australia (I. Oliver, personal communication, 2003), 545 to >7900 mg kg^{-1} in Poland [*Bodzek and Janoska*, 1999], and mean values of Zn reported to be (mg kg^{-1}) 830, 823, 670 in the United States, Canada, and Japan, respectively [*Epstein*, 2003; *Zhang et al.*, 2001]. Hence, while for many of the metals there is the potential to incur a Type II error on sites amended with biosolids, evidence that the site was not pristine would be revealed through interpretation of the soil Zn data. If necessary, this information could be used to trigger further investigation to establish the source of the contamination, and therefore identify whether other metals are likely to be above background concentrations.

[15] This study demonstrates that although natural background concentrations for heavy metals of environmental concern can vary in soils by over 3 orders of magnitude, background concentrations of As, Co, Cr, Cu, Ni, Pb, and Zn in a given soil can be defined by use of aqua regia soluble Fe/Mn as a quasi-conservative tracer. Moreover, background concentrations determined in this way do not require high sampling densities for verification. This approach provides a mechanism allowing environmental authorities and researchers to recognize when elevated concentrations of metals in soils are not the result of anthropogenic inputs. From this will come a greater appreciation that "natural" soils are not automatically hazard free and an understanding that soils which may be hostile to traditional human pursuits can nevertheless host significant biodiversity. Similarly, the data in Table 3 reveal the

Table 3. Expected Concentrations of Heavy Metals in Uncontaminated Soils (mg kg^{-1}) at Different Levels of Soil Fe, With Current Legislative Values (Australian Ecological Investigation Levels (EILs)) Shown for Comparison[a]

Soil Fe%	As	Cr	Cu	Ni	Pb	Zn
0.1	<3	<15	<4	<5	<0.3	<9
0.5	<8	<50	<10	<15	<2	<25
1	<10	<80	<15	<25	<4	<35
5	<30	<275	<45	<75	<20	<85
10	<45	<465	<70	<120	<40	<130
15	<55	<630	<90	<160	<60	<165
20	<65	<780	<105	<195	<80	<195
25	<75	<925	<120	<230	<100	<225
Australian EILs	20	400	100	60	600	200

[a]Calculated from the 95th percentile of samples (see text); values greater than 10 are rounded to the nearest 5.

high degree of caution that must be exercised in the use of a single index value to identify the presence of metal contamination as, unless set unrealistically low, such a value can obscure significant metal contamination that may be occurring in soils with inherently low background concentrations.

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