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Chemical Extractability of Pb in Field-Contaminated Soils: Implications for Estimating Total Pb

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Abstract

Lead (Pb) is frequently present in urban soils at concentrations of concern for human health. Regulations for this metal are based on total soil concentrations as determined by acid digestion, but a less expensive screening test for Pb would be useful in facilitating more thorough soil testing of urban areas if it could be shown to correlate strongly to total soil Pb. In this study, three extractants (0.1 M citrate, Modified Morgan, 1 M nitric acid) were evaluated for their ability to estimate the total Pb in contaminated soils. Nitric acid not only extracted a greater fraction of total soil Pb, but also produced the strongest correlation to total Pb and is concluded to be the superior extractant for a soil Pb screening test. As the spatial distribution of Pb was observed in selected soils to be highly heterogeneous on the micron scale, thorough soil homogenization prior to testing is recommended.

Introduction

Urban soils around the world have been contaminated significantly by human activities such as transportation, construction and manufacturing (Peltola and Astrom, 2003; Norra et al., 2001; Biasioli et al., 2007). As a consequence, urban community and private gardens can be moderately to severely contaminated by one or more toxic heavy metals, with lead (Pb) commonly being the most serious soil contaminant of concern to human health (Stillwell et al., 2008; Preer et al., 1980; Chaney et al., 1984).

Soil tests for Pb usually entail an acid-digestion procedure followed by ICP-emission spectrometry to determine total Pb (e.g., EPA method 3051). This test can cost gardeners and homeowners from \$20 to \$50 or more for each soil sample analyzed. Given the high spatial variability of Pb in soils adjacent to buildings in urban developed areas, a single soil analysis, whether based on a composite or discrete sample, may not be sufficiently informative in assisting the gardener or homeowner with how to best minimize exposure during gardening or other activities. A less expensive Pb test could enable the gardener or homeowner to test more locations for the same cost, and obtain more location-specific test results to inform planning of the garden or other site activities.

In this study, we compared some simple extractions using less chemically aggressive reagents with the standard concentrated nitric acid digestion (EPA method 3051) to determine whether a less costly and simpler screening test could be used to estimate total soil Pb. The extractants chosen for comparison were 0.1 M Na citrate (pH 6), Modified

Morgan solution (1 M ammonium acetate at pH 4.8) and 1 M HNO₃. The reasons for choosing these extractants were the Pb-chelating ability of citrate, the existing widespread use of Modified Morgan solution for soil testing in northeastern states, and the reported effectiveness of 1 M nitric acid in dissolving a large fraction of soil Pb (Chaney et al., 1984). A range of Pb contamination levels and types were included in the soils extracted to evaluate the universality of the extraction methods being tested. A preliminary investigation of the problem of spatial heterogeneity of Pb contamination in soils was also undertaken using Pb mapping by micron-scale X-ray fluorescence (XRF) and measures of reproducibility of extractable Pb in replicate samples.

Methods

Soil properties

Lead-contaminated soils were sampled from an upstate NY apple orchard with a history of insecticidal Pb arsenate use, an industrial smelting site near Montreal, an urban community garden, and two sites heavily amended with sewage-sludge 25–30 years ago. Included for testing were control soils (with no significant Pb contamination, and several soils experimentally Pb-spiked and aged for about 1 year. All soils were dried, well-mixed and put through a 2 mm Teflon sieve. Important properties of the soils included in the comparison of extraction tests are listed in Table 1. A second set of 15 garden and park soils from New York City was included in this study for the purpose of determining the extent of heterogeneity and statistical variability in measuring extracted Pb of urban soils. Soil pH was determined on a 1:1 soil/distilled water slurry using a glass electrode, while organic carbon was estimated using the standard Walkley-Black chemical oxidation method. Total soil Pb was determined using the EPA 3051 digestion procedure with measurement of Pb in acid digests at 220.35 nm using ICP spectrophotometry.

Comparison of soil extraction methods

Sodium citrate (0.1 M) at pH 6 was prepared from reagent-grade Na citrate. After weighing 2.00 g of air-dry soil into a 125 ml Erlenmeyer flask, 20 ml of the Na citrate solution was added, and the flask was agitated on a rotary shaker for 30 minutes. The soil slurry was then paper-filtered (Whatman #42) and the filtrate was put through a 0.2 µm cellulosic membrane filter in order to obtain a completely clear extract. Pb was measured in the filtered extracts by flame atomic absorption at 217 nm using Pb nitrate standards (1–20 mg/L) made up in 0.1 M Na citrate.

Modified Morgan solution (1 M ammonium acetate at pH 4.8) was prepared by combining glacial acetic acid (143.8 ml) and concentrated NH₄OH (91.25 ml) with about 1 L of distilled water in a 2 L glass beaker. After stirring to allow reaction, distilled water was added to create a total volume of just under 2 L, and the pH adjusted to 4.8 with CH₃COOH or NH₄OH. The final volume was then adjusted to 2.00 L with distilled water.

After weighing 5.00 g of air-dry soil into a 125 ml Erlenmeyer flask, 20 ml of Modified Morgan solution was added, and the flask was agitated on a rotary shaker for 15 minutes. The soil slurry was then paper-filtered (Whatman #42) to obtain a clear extract. Pb was

measured in the filtered extracts by FAA at 217 nm using Pb nitrate standards (1–20 mg/L) made up in Modified Morgan solution.

M HNO₃ was prepared by dilution of trace-metal grade concentrated nitric acid with distilled water. After weighing 5.00 g of air-dry soil into a 125 ml Erlenmeyer flask, 50 ml of 1.0 M HNO₃ was added, and the flask was agitated on a rotary shaker for 1 hour. The final pH of the extracts were below 1.2. The soil slurry was then paper-filtered (Whatman #42) to obtain a clear extract. Pb was measured in filtered HNO₃ extracts by both FAA at 217 nm and axial-view ICP spectrometry at 220.4 nm.

Variability of extractable Pb in soil samples

The 15 urban garden soils were mixed well and put through a 2mm Teflon sieve before extraction for Pb with 1M HNO₃ by the method described above. In this case, however, 4 replicate extractions were conducted on each of the 15 mixed samples in order to assess the inherent variability of the Pb test for urban garden soils. Three of the soil samples showing substantial variability were further subjected to pulverization using a mortar and pestle in an attempt to reduce the variability in replicate extractions. Subsequent 1M HNO₃ extractions were conducted on these three soils in both the unpulverized and pulverized state, using 5 replicates to determine the effect of this homogenization on variability in extracted Pb. Total Pb in these soils was determined by EPA Method 3050B with determination of Pb in the acid digests using flame AA at 283.2 nm as well as by EPA method 3051 with ICP-emission analysis of the digests at 220 nm. A standard reference material (NIST Marine Sediment 2702) containing 132.8 mg/kg Pb was included in the 3050B digestions, with Pb recovery determined to be acceptable at 94%. There was reasonable agreement between the estimates of total Pb by the 3050B and 3051 methods ($r = 0.934$), with soil subsample heterogeneity probably accounting for much of this variability.

Mapping of Pb in Soil by μ -XRF

From one of the sampling sites in the apple orchard, which had been contaminated by heavy metals from both Pb arsenate sprays applied prior to the 1960s and a heavy sewage sludge application in the late 1970s, a dried soil sample was passed through a 300 mesh sieve and the fine fraction used for synchrotron-based micro-x-ray fluorescence mapping (μ -XRF) at beamline 2–3 of the Stanford Synchrotron Radiation Laboratory (SSRL). Soil samples were mounted on tape. The pixel size was set at 5 microns and the count time was 250 ms using continuous scanning and a 100 mA operating current. The μ -XRF data were analyzed and element maps were generated using the software package SMAK version 0.50 (<http://www-ssrl.slac.stanford.edu/~swebb/smak.htm>).

Results and Discussion

Citrate and Modified Morgan Methods

Both the citrate (pH 6) and Modified Morgan (MM) tests extracted Pb approximately in proportion to total Pb. Although the correlations for citrate and MM to total Pb, shown in Figure 1, have highly significant r^2 values of 0.58 and 0.84, respectively, these correlations are heavily influenced by the results for only 4 soils – the highly contaminated industrial-site

soils. In fact, if these 4 soils are excluded from the comparison, citrate-extractable Pb is better correlated than MM-extractable to total Pb (data not shown). The calcareous industrial soils show unusually high extractability of Pb by the strong MM buffer, but low extractability by the much weaker citrate buffer. The MM buffer is known to be very effective in dissolving carbonates, and it is likely that Pb carbonates are present in the industrial soils.

Excluding the industrial-site soils, the citrate and MM extracted Pb are more strongly correlated to one another than to total Pb, as shown in Figure 2. This result suggests that the fraction of total soil Pb that is chemically labile is different in soils with different sources of Pb contamination, with both citrate and MM tending to dissolve *this* fraction. However, except for the industrial soils, the fraction of total Pb extracted by both methods is rather low, only several % of total (see Table 2). Because the efficiency of Pb extraction by MM and citrate varied depending on the type of soil contamination, it is inevitable that the overall correlation between extractable and total Pb is not strong enough for these soil tests to estimate total Pb reliably. This may be partly due to the effect of soil organic matter in suppressing Pb dissolution as shown in Figure 3, with extractability by MM inversely correlated to soil organic carbon.

HNO₃ extraction for Pb

For the 32 soils extracted separately by 1 M HNO₃ to determine acid-soluble Pb by both FAA and ICP, there was very good agreement between the two measurements. The best-fit equation for the two spectroscopic methods was:

$$(\text{Pb})_{\text{FAA}} = 12.7 + 1.11 (\text{Pb})_{\text{ICP}} \quad r^2 = 0.980$$

where (Pb)_{FAA} and (Pb)_{ICP} represent the extracted soil concentrations of Pb (mg/kg) in 1 M HNO₃ as determined by FAA and ICP, respectively. Note, however, that FAA on average gave an 11% higher value than ICP as indicated by the slope in the best-fit equation. This may have been due to small differences in the reaction and filtration process on separate subsamples. A direct comparison of FAA and ICP analysis of the same 1 M HNO₃ extracts showed very good agreement between the two methods, indicating that the bias did not arise from inherent differences between atomic absorption and plasma emission measurements. Although Fe and Al in the extracts can produce spectral interferences, particularly for ICP, concentrations of these two metals were both in the range of 100–300 mg L⁻¹, much lower than the concentrations in acid digests of soils that cause severe interferences for Pb determination by ICP (Asfaw and Wibetoe. 2009). At very high concentrations in solution (e.g., 10,000 mg/L), Fe can produce a serious interference for Pb determination by FAA (Price, 1979). However, in preliminary testing of Pb standard solution in 1 M HNO₃, we found that Fe concentrations from 100 to 1,000 mg/L had no effect on absorption by Pb at 283.3 or 217.0 nm (unpublished data).

Nevertheless, FAA has an inherently lower sensitivity for most trace metals in water samples than ICP-emission. For Pb, the reported detection limit for the 217 nm absorption of FAA is 0.19 mg/L, whereas the ICP emission line at 220.35 nm has a detection limit near

0.002 mg/L. Nevertheless, this theoretical advantage in sensitivity for ICP may be offset by severe ICP-specific spectral interferences when solutions contain high levels of other elements (Zbaral, 1996; Asfaw and Wibetow, 2009). In fact, both background continuum (non-spectral) and interelement (spectral) interference affect the preferred Pb 220.3 nm ionic analytical line in ICP, with the former arising from easily ionized elements and the latter largely from Al and Fe in acid digests and extracts from soils (Zarcinas, 2002; Zbaral, 1996). Asfaw and Wibetow (2009) reported poor precision for the Pb 283.3 and 217 nm (overestimation) and 220.4 nm (underestimation) lines when Fe was 2,000 mg/L. Acid soil digests typically contain even higher concentrations of Fe, usually exceeding 10,000 mg/L for mineral soils. Consequently, for strong acid extracts or digests with dissolved Pb above the FAA detection limit, FAA may be a preferred method to ICP-emission, and has been used successfully to measure total Pb in urban Pb-contaminated soils (Granadillo et al., 2006).

The 1 M HNO₃ method extracted a much higher fraction of the total Pb than either citrate or MM. Table 2 shows the extraction efficiencies for the different soils included in the comparison. Averaged over all soils, about 83% of the total Pb was extracted, but the highest fractions were extracted from the laboratory-spiked and industrial-site soils, and the lowest from the uncontaminated (control). The apple orchard soil with historical Pb arsenate use had an intermediate extraction efficiency of 74%. It may be that the relatively low solubility of Pb arsenate contributes to this somewhat lower extractability compared to most of the other contaminated soil types, since Pb and As from these soils are associated spatially at the micron scale as shown by μ -XRF microprobe (see next section). These Pb and As-enriched particles are ubiquitous in the soil, but not numerous, and it is informative that they persist despite the many decades since the contamination occurred.

Based on this comparison, most types of anthropogenic Pb contamination encountered in this study appear to have about 80–90 % of the Pb extractable by 1 M nitric acid. Geogenic Pb in uncontaminated soils is somewhat less extractable, presumably because a substantial fraction of this native Pb is entrained within primary minerals that do not readily dissolve.

Because of the consistently high extraction efficiency, there was an excellent correlation between 1M HNO₃ -extracted Pb and total Pb over all soils, as shown in Figure 4. This correlation to total Pb was much stronger than that for either citrate or MM, meaning that the 1M HNO₃ extraction appears to be the most reliable estimate of total and the best choice for a “Pb screening” test. Even if the soils with Pb > 500 mg/kg (industrial-site soils) are excluded from the comparison to reduce the bias that these create in the regression analysis (these few data points strongly influence the best-fit lines), 1 M HNO₃ remains clearly superior to MM as a soil screening test to predict total Pb (compare Figures 5 and 6). The 1 M HNO₃ -extractable fraction of Pb from soils with less than 500 mg/kg total Pb averaged about 71% based on the slope of the best-fit line in Figure 6, which is somewhat lower than that observed for all soils combined.

A study by Hamel et al. (2003) is highly relevant to the present evaluation of soil tests for Pb. That study compared three extraction methods, Modified Morgan (MM), Mehlich-1

(M1) and Mehlich-3 (M3), for their ability to estimate total soil Pb as determined by EPA Method 3050. For soils with total Pb up to 720 mg/kg, the best-fit relationships were:

$$\begin{aligned}\text{EPA 3050 Pb} &= 5.3 \text{ Pb(M1)} + 96 & r^2 &= 0.49 \\ \text{EPA 3050 Pb} &= 1.3 \text{ Pb(M3)} + 39 & r^2 &= 0.60 \\ \text{EPA 3050 Pb} &= 4.7 \text{ Pb(MM)} + 64 & r^2 &= 0.74\end{aligned}$$

Thus, while M-3 extracted Pb from soils in that study with almost as high an efficiency as 1 M HNO₃ in the present study, the predictive accuracy was lower than that of the 1 M HNO₃ extraction, which in our study correlated to total Pb with an r^2 of 0.976. Conversely, MM extracted Pb with the least efficiency, but provided the most reliable estimate of total Pb of the three methods tested by Hamel et al. (2003). In our study, excluding the industrial-site soils, MM extracted a much smaller fraction of total Pb than measured by Hamel et al. (2003), and the r^2 for the correlation of MM-Pb to total Pb was only 0.302, clearly too low for using MM extraction to predict soil total Pb.

Variability of extractable Pb in urban soil samples

The 1M HNO₃-extractable Pb not only showed a wide range (from 46.0 to 748 mg/kg) among the 15 urban soils tested, but also a high degree of variability among replicates (see Figure 7). The relative standard deviation (RSD) of soil test Pb varied from 1.0% to 47% among the soils, with an average of 13.6%. There was no clear trend between the magnitude of the average measured soil test Pb and the RSD. Although the two highest RSDs (47% and 37%) were measured for soils which averaged less than 100 mg/kg extractable Pb, RSDs less than 5% were found for soils with both high and low soil test Pb values. More thorough homogenization of soils by pulverizing them with a mortar and pestle showed a reduction in the RSD in only two of the three soils tested (based on 5 replicates). The correlation between 1M HNO₃-extractable Pb (mean value) and estimated total Pb (determined by EPA Method 3050B) was strong, with Figure 7 showing a regression r -value of 0.970.

Mapping of Pb in Soil by μ -XRF

Synchrotron XRF microprobe element maps of the sludge-contaminated orchard soil (not shown) revealed distinct particles rich in both Pb and As (believed to be relict lead arsenate particles surviving from historical pesticide application) and separate particles rich in Pb, Fe, Cu and Zn but low in As (possibly residual particles of sludge). These two distinct forms of Pb resulted in a strong correlation between the count intensities for Pb and As where Pb is at very high concentration, and a low Pb-As correlation in particles with much lower Pb concentration. The particles rich in both Pb and As ranged in size from a few microns to 50 microns, and while not numerous, appeared to be ubiquitous as one or two appeared in each map created from the soil.

The heterogeneity of Pb distribution in the soil, as suggested by the variability in soil test results for replicates (discussed above) and by the observed spatial heterogeneity at the sub-millimeter scale, may have important implications for soil testing, as non-representative results may be obtained from samples that are not thoroughly homogenized prior to conducting extractions for Pb. Furthermore, it appears that the chemical form of some

fraction of the Pb, at least in the case of the orchard, persists for decades, and may account for differences in Pb extractability by soil tests.

Conclusions

The 1 M HNO₃ extraction is superior to both MM and citrate in estimating total soil Pb because of a strong correlation of the quantity of Pb dissolved in this test to total soil Pb regardless of form of Pb contamination. The MM and citrate tests extract a much smaller fraction of the total Pb, a fraction which varies substantially among soil types and may represent a chemically more labile and bioavailable “pool” of Pb in soils. However, knowledge of total soil Pb is important in conducting risk assessments of human health effects from soil Pb exposure because a high fraction (70% or more) of the total Pb in contaminated soils has been shown experimentally to be bioaccessible in solutions simulating the gastric stomach (Ellickson et al., 2001; Bosso and Enzweiler, 2008). Consequently, the more chemically aggressive test using 1 M HNO₃ is the preferred candidate for a soil Pb screening test. This test is less time-consuming and less expensive than the measurement of total soil Pb by acid digestion. In addition, it is a relatively easy matrix to analyse for Pb, with both flame atomic absorption and ICP-emission able to quantify Pb concentrations in the 1 M HNO₃ extracts without the serious spectral or matrix interferences reported for acid soil digests.

For soils with <500 mg/kg total Pb, the correlation between HNO₃-extractable and total Pb was:

$$\text{Pb (total)} = 1.317 \text{ Pb(1 M HNO}_3\text{)} + 0.606 \quad r^2 = 0.933$$

The slope of this linear function indicates that about 76 % of total soil Pb is dissolved by the 1 M HNO₃ extraction, comparable in magnitude to the bioaccessible fraction of soil Pb measured in simulated gastric solutions.

In summary, 1 M HNO₃ extracts Pb efficiently from contaminated soils, and analysis of the extract by either ICP or flame atomic absorption spectrometry provides a reliable estimate of total soil Pb without the necessity to digest the soil in concentrated acid under high-temperature conditions. This method of extraction can potentially be used to provide a less expensive alternative to the standard EPA Methods 3050, 3051 and 3052. The highly heterogeneous spatial distribution of Pb at the scale of 1–100 microns, observed in the orchard soils, suggests the need for thorough soil homogenization prior to testing. Repeated extraction by 1 M HNO₃ of subsamples taken from the same well-mixed urban soil samples frequently produced relatively large differences in measured extractable Pb, an indication of a ‘nugget effect’ in subsamples taken from a larger sample.

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Pb Extracted By Morgans and Citrate - All Soils

Figure 1.
Relationship of citrate- and Modified Morgan-extractable Pb to total Pb in all soils tested.

Figure 2.
Relationship of citrate- to Modified Morgan-extractable Pb in all but the highly Pb-contaminated industrial soils.

Figure 3.
Fraction of total soil Pb extracted by Modified Morgan method as a function of soil organic carbon content.

Pb Extraction by 1 M HNO₃ (Anna)-All DataFinal

Figure 4. Relationship between concentration of Pb extracted from all soils by 1 M HNO₃ and total soil Pb.

Figure 5.
Relationship between total soil Pb and concentration of Pb extracted by Modified Morgan from soils with less than 500 mg/kg total Pb.

Figure 6. Relationship between total soil Pb and concentration of Pb extracted by 1 M HNO₃ from soils with less than 500 mg/kg total Pb.

Figure 7. Relationship of mean 1M HNO₃-extracted Pb (4 replicates) to total soil Pb in urban soils as measured using method 3050B (acid digestion). The error bars depict one standard deviation unit.

Figure 8.

Tricolor map of element distribution in a sample of the apple orchard soil, showing an apparent relict lead arsenate particle (magenta particle at lower left). Red = arsenic, green = zinc, blue = lead. Inset graph shows the linear correlation between As and Pb count intensities for all pixels in this image. Pixel size is 5 microns.

Table 1

Measured properties of soils contaminated by Pb from various sources included in the comparison of Pb extraction methods.

Soil	pH	Organic carbon (%)	Total Pb (mg/kg)
Community garden	7.11	4.96	112
Community garden	6.96	13.8	27.4
Community garden	6.68	7.51	69.6
Community garden	7.07	4.50	152
Community garden	6.93	5.72	114
Apple orchard	5.84	2.85	44.7
Apple orchard	4.51	2.50	107
Apple orchard	6.42	2.81	135
Apple orchard	6.63	2.94	249
Apple orchard	5.69	3.11	260
Apple orchard	6.77	3.27	316
Apple orchard	6.52	3.45	289
Apple orchard	6.62	3.55	410
Spiked soil	5.67	2.43	34
Spiked soil	5.82	2.74	60
Spiked soil	5.69	2.36	112
Spiked soil	5.53	2.43	213
Sludged soil	7.51	1.39	35.5
Sludged soil	6.80	2.36	76.6
Sludged soil	6.97	1.39	66.6
Sludged soil	6.83	3.11	68.9
Sludged soil	5.72	1.99	96.0
Sludged soil	5.42	4.78	221
Sludged soil	5.52	3.57	203
Sludged soil	6.75	4.34	184
Sludged soil	6.13	4.87	259
Control soil	5.17	1.30	10.4
Control soil	5.69	2.90	12.3
Industrial site	7.33	0.70	152
Industrial site	7.31	1.94	1182
Industrial site	7.34	1.80	654
Industrial site	7.49	0.88	507

Table 2

Efficiency of Pb extraction (expressed as fraction of total Pb) from the variously contaminated soils as measured by each extraction method.

Soil Contamination Type	Citrate	Modified Morgan	1 M HNO ₃
Control (N=2)	0.0138 ± 0.0086	0.0087 ± 0.0054	0.570 ± 0.010
Old Orchard (N=8)	0.0862 ± 0.0457	0.0529 ± 0.0331	0.744 ± 0.127
Spiked (N=4)	0.118 ± 0.069	0.0957 ± 0.0625	0.912 ± 0.122
Community Garden (N=5)	0.0509 ± 0.0151	0.0177 ± 0.0159	0.805 ± 0.071
Old Sludged (N=9)	0.0430 ± 0.0100	0.0266 ± 0.0072	0.782 ± 0.107
Industrial (N=4)	0.0531 ± 0.0419	0.224 ± 0.0767	0.889 ± 0.074