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A Field Procedure To Screen Soil for Hazardous Lead

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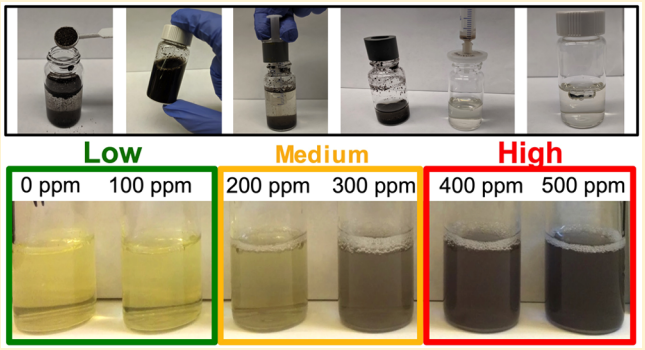
Supporting Information

ABSTRACT: Soils retain lead contamination from possible sources such as mining, smelting, battery recycling, waste incineration, leaded gasoline, and crumbling paint. Such contamination is often concentrated in toxic hot spots that need to be identified locally. To address this need, a simple field procedure was designed to screen soil for hazardous Pb for use by the general public. The procedure is a modification of the *in vitro* soil Pb extraction described by Drexler and Brattin (*Hum. Ecol. Risk Assess.* **2007**, *13*, 383) and EPA Method 1340, and uses a 0.4 M glycine solution at pH 1.5. A higher soil-to-solution ratio of 1:10 allows for classifying soil samples based on extractable Pb concentrations of <200 mg/kg (low), 200−400 mg/kg (medium), and >400 mg/kg (high) using sodium rhodizonate as a color indicator. The 1:10 soil-to-solution ratio also makes it possible to measure Pb concentrations in the glycine extract solutions on a continuous scale using a portable X-ray fluorescence analyzer. The procedure rather consistently extracts about one-third of the Pb extracted by the standard method across a wide range of Pb concentrations. Manufacturing the kit in larger quantities could reduce the cost of the materials well below the current $5/test.



Lead is known to be highly toxic to all organ systems and is especially harmful to cognitive development in children.[1−4] Ingesting soil contaminated with Pb is an important pathway for child exposure,[5−8] and soil remediation has been linked to decreased Pb exposure.[9,10] Soils in industrial and urban areas have accumulated Pb from deteriorating Pb-based paint, atmospheric deposition from leaded gasoline and municipal waste incinerators, as well as past and present industrial activities such as mining, smelting, and battery recycling.[10−13]

Poisoning from environmental contamination with Pb often goes undetected and is unevenly distributed:[12,14] 82% of deaths from Pb poisoning occur in lower- and middle-income countries (LMICs).[11] Lead poisoning accounts for 63% of the disability-adjusted life years (DALYs) of toxic waste sites in India, Indonesia, and the Philippines, and contributes to a greater burden of disease than malaria in these countries.[15] Globally, the disease burden from Pb exposure is estimated to have caused 13.9 million DALYs in 2012 and 0.5−0.7 million deaths.[16] The disease burden and loss of cognitive function due to Pb poisoning have serious economic implications: Pb poisoning in urban Peru is estimated to cost the country 0.5% of its GDP.[17] In the U.S., each annual cohort of children born after 1980 is estimated to add $110−300 billion in economic productivity gains due to an increase of 2−5 IQ points from reduced Pb exposure.[18]

Several studies have demonstrated a clear correlation between soil-Pb concentrations and child blood lead levels (BLL).[5−7,19,20] In some cases, Pb exposure has been correlated with the degree of urbanization because soils retain past Pb contamination.[10,21,22] Children under age 6 are the most vulnerable to Pb poisoning because they are in a crucial neurological development phase; they are also at higher risk for Pb exposure because they often crawl on the ground and place dusty toys and hands into their mouth. Although U.S. child BLLs declined by more than 90% after Pb was no longer added to gasoline and paint, the U.S. Centers for Disease Control and Prevention (CDC) recognizes that "no safe Pb level has been identified." In 2012, the CDC replaced its BLL of concern of

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10 μg/dL with a reference level of 5 μg/dL for children.[23,24] Reflecting this change, some U.S. states have updated their soil reference level to as low as 80 mg/kg,[25] but the federal soil hazard standards remain at 400 mg/kg for bare soil where children play and 1200 mg/kg for other residential soils.[26]

The Pb in soil that is bioaccessible or bioavailable provides a better estimate of health hazard than the total Pb concentration.[27] *In vivo* bioavailability tests require complex animal trials whereas *in vitro* bioaccessibility (IVBA) tests are less expensive substitutes based on laboratory extractions that simulate gastric conditions. Bioaccessibility methods measure the amount of Pb released from the soil in a simulated gastric solution and determine a relative bioavailability (RBA) that reflects the chemical form and matrix of the soil Pb.[28−30] Absolute bioavailability (ABA) is generally defined as the fraction of ingested Pb that is absorbed in the gastrointestinal system and enters the bloodstream. Relative bioavailability (RBA) is the ratio of the ABA of a soil sample to the ABA of a soluble form of Pb such as lead acetate.

There is increased interest in developing alternative methods to ease the determination of bioaccessible Pb.[31] One challenge of the existing methods for measuring bioaccessible Pb in soil is the need for costly laboratory equipment to process soil samples and measure Pb concentrations in the soil extract. The high spatial variability of total and bioaccessible Pb in soils[32−36] means that many measurements are needed. Many LMICs do not have the resources needed to identify soil Pb contamination with the same methods used by high-income countries, nor do they conduct routine screening of Pb in blood. A rapid and affordable field procedure could therefore considerably reduce human exposure by identifying hot spots of Pb contamination to avoid or address.

To the best of our knowledge, there is no field procedure to test for Pb in soils reliably, and no test kit for Pb in soil has been approved by the U.S. Environmental Protection Agency (EPA).[37] The commercially distributed 3M LeadCheck swabs can only be used to test paint, which has a much higher Pb content than even the most contaminated soils.[38] The field procedure for soil presented in this study is derived from the IVBA method of Drexler and Brattin reported in 2007[29] and EPA Method 1340,[39] but uses a higher soil-to-solution ratio. The modification allows the detection of elevated bioaccessible Pb concentrations with sodium rhodizonate, the same indicator used to detect Pb in gunshot residue.[40−42] The modified procedure also makes it possible to use a portable X-ray fluorescence analyzer to measure bioaccessible Pb concentrations in the liquid extracts on a continuous instead of a categorical scale. Importantly, these modifications enable the procedure to be used in the field.

EXPERIMENTAL SECTION

Materials and Methods. *Soil Samples.* All 137 soil samples were collected in the field and passed through a kitchen sieve with a 1 mm mesh size. The samples represent a variety of contamination types and come from six different countries, including 65 urban soil samples from New York City and 31 rural soil samples from Peru. The New York samples were collected from residential backyards (n = 42), urban farms (n = 3), and publicly accessible parks and tree pits (n = 20). The Peruvian soil samples were collected from small mining-impacted communities along the Carretera Central east of Lima (n = 10), the city of Cerro de Pasco built around a gaping open-pit mine (n = 11), and the infamous smelter town of La Oroya (n = 10). Researchers from Pure Earth (formerly Blacksmith Institute) contributed an additional 41 soil samples from the following locations: a secondary lead smelter in India (n = 8), a large secondary lead smelter of lead-acid batteries in the Philippines (n = 4), an electronics-waste site in Uruguay (n = 12), and informal lead-acid battery recyclers in Indonesia (n = 10) and the Philippines (n = 7).

Instrumental Measurements. Total Pb concentrations in soil were measured in the laboratory with a portable hand-held X-ray fluorescence (XRF) analyzer (Innov-X Systems DELTA Premium) in a benchtop stand. Sieved soil samples in 20 mL scintillation vials were sealed with plastic wrap, inverted on the benchtop XRF stand, and analyzed using the instrument's standard soil mode for 20 s at each of the three incident-beam energies. Soil samples were analyzed three times, shaking the vial end over end between each analysis in an attempt to capture soil heterogeneity. Results from XRF analysis of metals in soils, especially Pb, have been found to highly correlate with laboratory acid digestions,[43,44] and XRF is approved for use by EPA Test Method 6200.[45]

Concentrations of Pb in the field-procedure extract solutions were also measured by XRF and confirmed in a subset by a high-resolution inductively coupled plasma mass spectrometer (ICP-MS) (Thermo Scientific Element II or PerkinElmer DRCe). For XRF analysis, 10 mL of the field-procedure extract solution was analyzed similarly to the soil samples, by inverting the 20 mL vial sealed with plastic wrap on the benchtop stand and using the instrument's soil mode for 20 s at each incident-beam energy. For samples containing <10 mg/kg Pb in solution, the analysis time was tripled, which lowered the detection limit calculated by the instrument's software from 5 to 1.5 mg/kg Pb. Concentrations of Pb in the Drexler and Brattin (DB) extract solutions were measured exclusively by ICP-MS, because the dilution of 1 g soil in 100 mL solution results in concentrations below the XRF detection limit.

The XRF's accuracy for soil Pb measurement was verified with U.S. National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) soils including SRM 2710 with 5532 mg/kg Pb (mean 93 ± 1% SD of the reference value, n = 27), SRM 2710a with 5520 mg/kg Pb (103 ± 2% n = 3), SRM 2711 with 1162 mg/kg (98 ± 5%, n = 86), and SRM 2711a with 1400 mg/kg (104 ± 2%, n = 27). At much lower Pb concentrations, XRF readings were too high at 128 ± 17% (n = 67) of the reference value for SRM 2709 of 18.9 mg/kg Pb. Soil XRF Pb data were not adjusted for this bias at low concentrations. Data for field-procedure extract solutions measured by XRF were corrected by subtracting 3.5 mg/kg Pb based on the repeated measurements of a blank extract solution. For ICP-MS analysis, data were accepted when concentrations of Pb obtained for NIST reference materials 1640A (mean recovery 104 ± 3%, n = 8) and 1643F (99 ± 4%, n = 8) were within 10% of their published values. All ICP-MS method and vial blanks contained <1 μg/L Pb.

Field Procedure. The field procedure for assessing bioaccessible Pb was developed by modifying various steps of the IVBA method described by Drexler and Brattin in 2007 and used in EPA Standard Operating Procedure (SOP) 9200.2-86 and EPA method 1340.[28,29,39] Soils are sieved in the field, if possible, and otherwise dried overnight until sieving is achievable. For this study, three level scoops of sieved soil (0.5 mL each, approximately 1.5 g total) are weighed and added to 15 mL of a 0.4 M glycine solution adjusted to pH 1.5 with hydrochloric acid in a polyethylene scintillation vial with a

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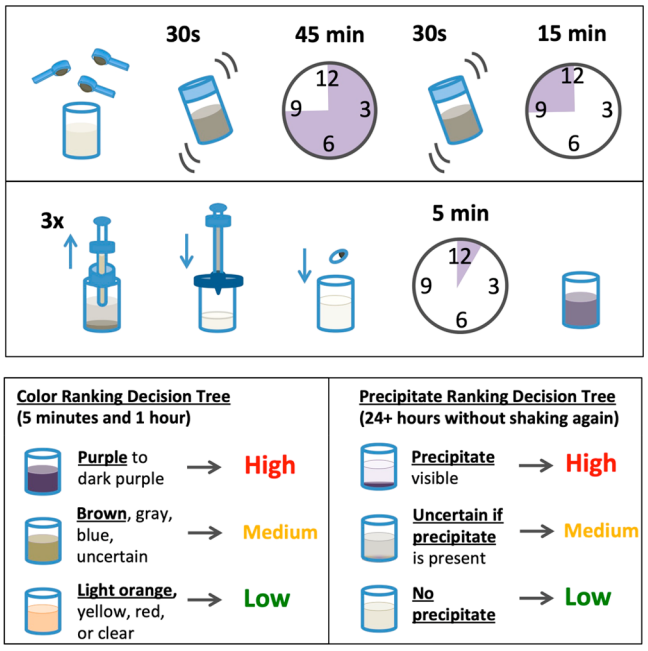

Figure 1. Schematic of the field procedure steps and a decision tree for the color ranking. A more detailed visual instruction sheet as given to those using the procedure in pilot studies can be found in Figure S1.

Polyseal cone cap (Figures 1 and S1). While weighing is not required for a field deployment, not doing so can add additional variability. The vial is agitated for 30 s at the beginning of the extraction to ensure that no sediment adheres to the bottom and again after 45 min to promote mixing. To achieve a total extraction time of 1 h and to allow soil particles to settle, the vial sits for an additional 15 min. Next, about 10 mL of the supernatant is filtered through a 0.45 μm disposable syringe filter into a clear vial using a syringe that fits within the opening of the vial. A 1 cm plastic spacer is placed on top of the vial to ensure that the tip of the syringe remains in the supernatant and does not take up soil that could clog the filter (Figure S2). In a subset of analyses, different amounts of soil were extracted while maintaining the same soil-to-solution ratio (0.5 g in 5 mL for 12 samples in 2014 and 1 g in 10 mL for 50 samples in 2015).

Color Indicator. After filtration of the extract, a dissolvable gelatin capsule containing 10 mg of sodium rhodizonate is added to the vial. Sodium rhodizonate forms a purple precipitate with Pb, as described by Feigl and Suter (1942), and is still used today in forensic applications to identify lead gunshot residue.[41,42] The intensity of the violet-purple color for each sample was ranked by two researchers independently as low, medium, or high after 5 min, based on the color chart (Figure 1). All vials had tape over the labels and were randomly ordered to ensure an unbiased reading.

All samples that were clearly purple were ranked high, and all samples that were orange, yellow, clear, or red were ranked low. Samples that were brown, gray, blue, or appeared darker but not purple were ranked medium. In addition, one researcher ranked the color after 1 h, based on photos that were taken during the procedure. One researcher also ranked the actual vials after 24 h based on the amount of precipitate that was visible on the bottom of the vial. Samples were ranked highest if the precipitate covered the vial bottom and ranked high if any precipitate was visible. If no precipitate was visible, the sample was ranked low. Samples were ranked medium if the precipitate was barely visible, often appearing like a wisp of smoke in the solution.

Standard Bioaccessible Pb Method. For comparison, bioaccessible Pb was determined in a subset of 50 soil samples and two NIST soil standards following the standard Drexler and Brattin (DB)[29] method and EPA Method 1340[39] by extracting 1 g of dried soil <250 μm in 100 mL of 0.4 M glycine adjusted to pH 1.5 at 37 °C for 1 h. We used an incubator with a shaker table at 37 °C and 30 rpm, instead of the prescribed end-over-end rotator in a water bath; samples were turned end over end every 10 min by hand to simulate the end-over-end rotator. The pH was measured at the end of the extraction to confirm that it remained within 0.5 units of 1.5. The DB method establishes IVBA and can be used to estimate RBA by calculating RBA = 0.8782 × IVBA − 0.028.[28,29] The EPA Method 1340 was updated in 2017 to use soil sieved to <150 μm; however, the majority of our field and lab work was conducted prior to this EPA update and followed the previous method. Recoveries were within the expected range for NIST 2710a (96%, n = 1), NIST 2711a (115%, n = 1), a duplicate (98% recovery, n = 1), and a matrix spike (92% recovery, n = 1). For a subset of 13 samples contaminated by different industries analyzed in 2014, a 1:100 soil-to-solution ratio was maintained by using 0.2 g soil in 20 mL glycine solution, instead of the standard 1 g in 100 mL, and a hot water bath on a shaker table was used instead of the incubator.

Statistical Analysis. Statistical analyses were performed with R Studio 1.0.136 using R 3.2.3 (R Core Team 2015) to assess the difference between (a) the XRF and ICPMS Pb results for extracts and (b) the field procedure extracts and the DB IVBA extracts. Since the data are not normally distributed, we used a Spearman rank correlation coefficient. Correlation between data sets were considered not statistically significant if $p > 0.05$.

RESULTS

XRF Analysis of Field Procedure Extracts. A total of 114 liquid samples were analyzed by both ICP-MS and XRF, including five Pb solution standards and the field-procedure extracts from a subset of 107 soil samples and two NIST soil standards. Concentrations of Pb determined by XRF after blank correction were consistent with ICP-MS analyses across a range from <0.1 to 930 mg/L (Spearman's r_s = 0.99, p < 0.001). The XRF analyzer overestimates Pb concentrations in the glycine solution by about 16% (Figure 2).

Field Procedure Extracts. The concentrations of Pb extracted by the field procedure and the original DB method for 52 soil samples are well correlated (Spearman's r_s = 0.92, p < 0.001) (Figure 3). Across the range of concentrations and contamination types, the field procedure extracted about one-third as much Pb (mean 37 ± 15% SD, range 4−73%). The three soil samples from a smelter in India are outliers for which the field procedure extracted 70% of the Pb extracted by DB method (Table 1). The amount of Pb extracted was corrected for the dilution of the soil into solution by weighing the soil for both methods. However, no large difference is seen in the best-fit line across the subset of samples that contained both soil weight and scoop volume (Figure S4).

Total soil-Pb concentrations in the 137 soil samples from different countries and two NIST soil standards measured by XRF ranged from 40 to 100 000 mg/kg (Figure 4). The field procedure extracted between 12 and 10 700 mg/kg Pb from

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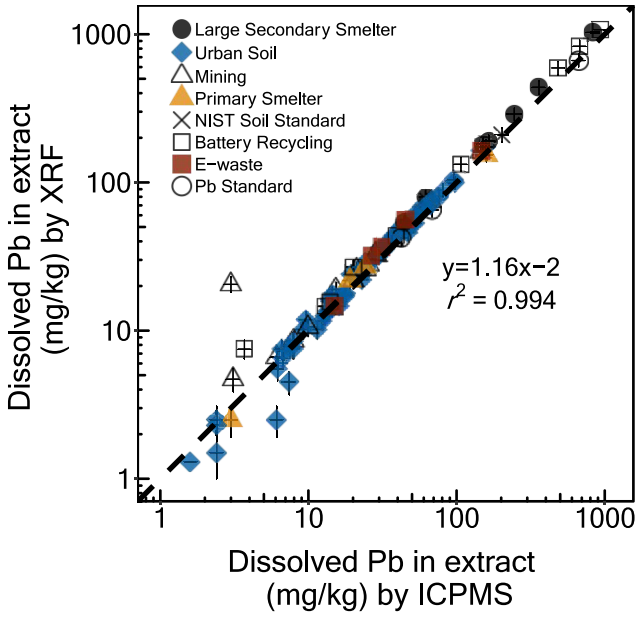

Figure 2. Blank-corrected XRF vs ICP-MS measurements of dissolved Pb (mg/kg) in solution for the field-procedure extracts of 107 soil samples and two NIST soil standards, as well-dissolved Pb standards. XRF and ICP-MS error bars are 10%. Samples with <10 mg/kg Pb by XRF were analyzed again tripling the analysis time per beam energy. One non-detect sample by XRF is plotted at 1.2 mg/kg, half the instrument's estimated detection limit. Different types of contamination are shown in the legend.

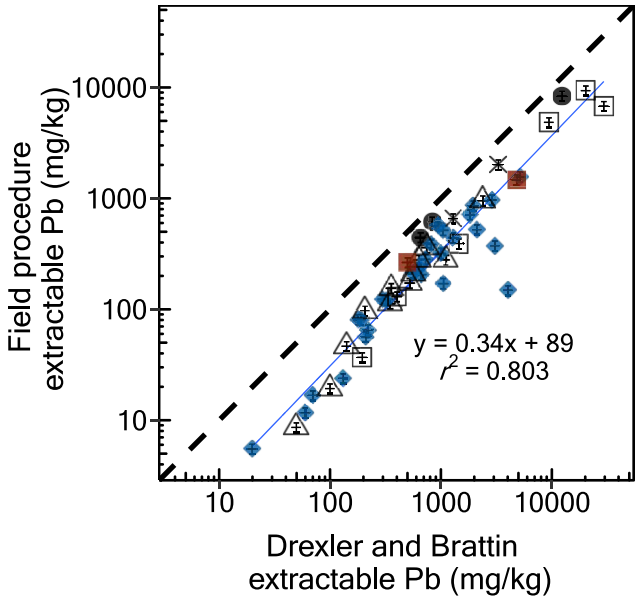

Figure 3. Amount of Pb extracted by the field procedure and the original Drexler and Brattin (DB) method for n = 52 soil samples (including two NIST standards) after accounting for soil-to-solution dilution. Extracted Pb was analyzed by ICP-MS and error bars are 10%. Samples from large secondary smelters, battery recycling, and e-waste were analyzed by the early procedure, which maintained the 1:10 and 1:100 soil-to-solution ratios but used less soil. For the 1:10 field-procedure method 1 g of soil was added to 10 mL of a glycine solution, and for the 1:100 DB Pb method 0.2 g of soil was added to 20 mL of a glycine solution. Symbols indicating contamination type are the same as in Figure 2.

Table 1. Proportion of Pb Extracted by the Field Procedure Compared to the Original Drexler and Brattin (DB) Method,[a] and Proportion of Total Soil Pb Extracted by the Field Procedure[b]

	% Pb extracted by the field procedure compared to DB		% total soil Pb extracted by field procedure	
contamination type	mean (%) (±1 SD)	n	mean (%) (±1 SD)	n
large secondary smelter	70 (±3)	3	78 (±20)	12
urban soil	33 (±12)	27	34 (±14)	65
mining soil	35 (±11)	12	17 (±12)	21
primary smelting			22 (±13)	10
battery recycling	33 (±13)	6	53 (±46)	17
e-waste	42 (±16)	2	42 (±38)	12
standards	56 (±7)	2	41 (±11)	2
total	37 (±15)	52		139

[a]See Figure 3. [b]See Figure 4.

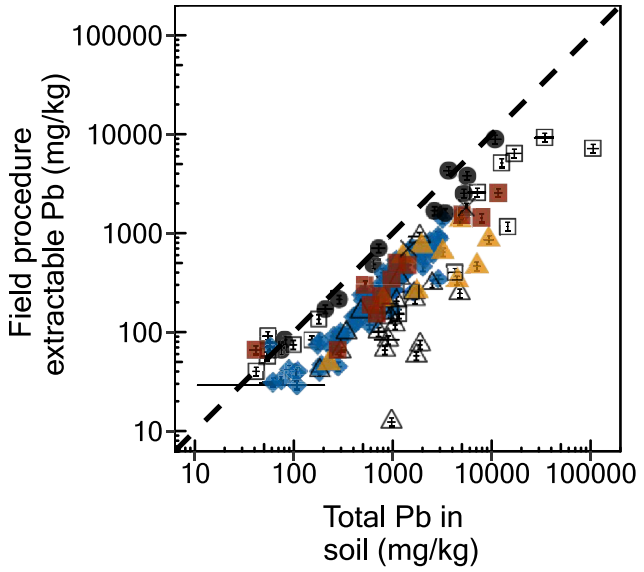

Figure 4. Concentration of Pb extracted by the field procedure for 139 soil samples of varying contamination types (symbols as in Figure 2). Total Pb in soil was analyzed by XRF and error bars are the standard deviation of three measurements. Extracted Pb was analyzed by XRF and blank and slope corrected based on the equation in Figure 2; also shown are 10% error bars.

these same samples. Thirty-one solutions from the field procedure were checked by a pH test strip, of which 28 confirmed the pH remained below pH = 2, and three indicated the pH was between 2 and 3. The field procedure extracted the highest proportion of Pb from soils around large secondary smelters, followed by soil near used lead-acid battery recyclers, e-waste sites, NYC urban areas, a primary ore smelter, and mining areas (Table 1).

Color Readings. Lead concentrations in 79 field procedure extracts (65 urban soil samples from New York City and 10 mining-impacted soils from Peru) were ranked as high/medium/low based on the intensity of the purple color after 5 min and the amount of precipitate after 24 h (Figure 5). After 5 min, all 23 samples that ranked high extracted more than 200 mg/kg Pb, and all 38 samples that ranked low extracted less than 220 mg/kg Pb. The detection limit is reduced after 1 h, as

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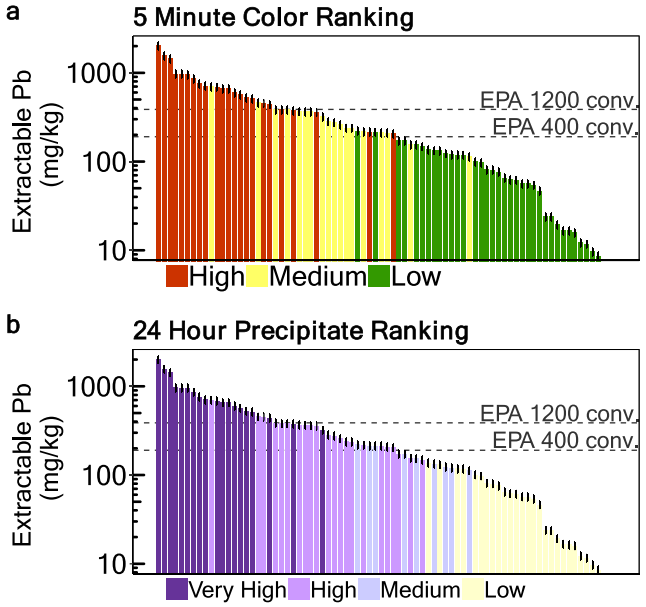

Figure 5. Ranking of field procedure extracts by extractable Pb concentration (dissolved Pb measured by ICPMS and multiplied by dilution factor) after (a) 5 min based on color and (b) 24 h based on precipitate amount. Rankings are compared to EPA soil screening levels of total Pb converted to the amount of Pb extracted by the field procedure based on 71.5% IVBA and the equation in Figure 3 (400 to >190 and 1200 to >387 mg/kg Pb).

33 samples ranked high and all had extracted more than 150 mg/kg Pb (SI Figure S3). Of the 34 samples ranked low, 31 extracted less than 150 mg/kg Pb. Assessing the amount of precipitate formed after 24 h further reduced the detection limit: all 38 samples that ranked high extracted more than 150 mg/kg Pb, and all 29 samples that ranked low extracted less than 140 mg/kg Pb.

To compare the amount of Pb extracted by the field procedure to EPA soil standards, federal total Pb soil screening levels were converted to bioaccessible Pb concentrations, where $IVBA_{EPA}$ = (0.6 + 0.028)/0.8782 = 71.5%, with the EPA's assumption of 60% (0.6) RBA (or 30% ABA).[28,39] At 71.5% IVBA, the EPA soil screening levels of 400 and 1200 mg/kg total Pb convert to 286 and 858 mg/kg Pb extracted by the DB IVBA method. These levels were then converted to the Pb that field procedure would extract by using equation from Figure 3, resulting in 190 and 387 mg/kg Pb, respectively.

Comparing the visual color rankings to these reference levels, we see that after 5 min, samples that extracted more than 387 mg/kg Pb ranked either medium (n = 2) or high (n = 18), while samples that extracted less than 190 mg/kg Pb ranked either low (n = 36) or medium (n = 2) (Figure 5). After 24 h, all samples extracting more than 387 mg/kg Pb ranked high (n = 20). Of the 38 samples extracting <190 mg/kg Pb, a larger fraction ranked medium (n = 8) and high (n = 1) due to the lower detection limit.

DISCUSSION

Trade-Offs between Field and Laboratory Testing. The field procedure and the DB method produced remarkably consistent results even if only about one-third as much Pb was extracted by the field procedure (Figure 3, Table 1). Likely reasons that the field procedure releases a lower fraction of total Pb include the following: larger particle size (~1 mm instead of <250 or 150 μm), re-adsorption due to the higher soil-to-solution ratio (1:10 vs 1:100), lower extraction temperature (22 to 37 °C), and possibly less frequent shaking. Reducing the soil-to-solution ratio from 1:100 to 1:10 is crucial, however, for reaching a Pb concentration in solution that is high enough to (a) form a color precipitate with sodium rhodizonate and (b) analyze the extract solution by XRF. It would be impractical for a wide group of users in the field to follow the DB method, which stipulates sieving soil to <250 μm, or the updated EPA 1340 method, which sieves soil to <150 μm. Similarly, using ambient temperature (10−30 °C) instead of an incubator set to 37 °C is a requirement for adapting the method for use in the field. Another simplification is that the field procedure relies on a volumetric method, a small scoop, to measure the amount of soil added to glycine solution. The density of soil can vary widely, however; the mass of three scoops of dried soil analyzed for this study ranged from 0.5 to 2.8 g (mean = 1.6 ± 0.4 g SD). However, across the subset of samples that contained both soil weight and scoop volume (Figure S4), the field-procedure still extracted a similar proportion of Pb. When increased accuracy can be obtained with an XRF analyzer, bringing a portable balance to the field for maintaining a more consistent soil mass could be justified. Finally, although the pH did not increase much in these samples, a pH test is also recommended to confirm that the pH of the field-procedure extract remains around or below 2, especially when testing soils with a high pH buffering capacity. Site-specific confirmation of pH stability and average scoop mass could be a simple way to increase accuracy without measuring these parameters on all samples.

Results from the simplified field procedure admittedly may be less directly relatable to child exposure, or at least the relationship established between absorption of Pb in juvenile swine and the DB procedure. Larger particles, for instance, are less likely to stick to hands and be ingested. Such limitations are more than compensated, in our opinion, by the benefit of providing concerned parents or community members with the means of testing soil wherever a child is likely to play. Furthermore, the field procedure outlined here still manages to give a reasonable estimate of bioaccessible Pb in soils, which may be a better measure of actual health risk from Pb in soil than total Pb measured by XRF in bulk soil. The current thresholds of <200, 200−400, and >400 mg/kg extractable Pb in soil could be adjusted if needed by varying the amount of soil added to the extraction solution. However, these values do not differ greatly from the calculated 190 mg/kg and 387 mg/kg field-procedure extractable Pb that correspond to the current EPA screening levels of 400 and 1200 mg/kg total soil Pb.

The field procedure estimates bioaccessible Pb by measuring the concentration of Pb released into the extract, which is directly related to health risk. Results from the field procedure are therefore sufficient to start prioritizing and addressing the most hazardous areas. Since soil contamination is often very spatially heterogeneous, once the location of Pb hotspots is known, individuals and communities can avoid these areas and take additional actions to reduce exposure. Possible actions include covering the area with clean soil or another barrier, ensuring children do not play in the high-Pb area, ensuring high-Pb soil is not transported out of the area or into homes, and maintaining clean environments with low dust and dirt levels where children play and eat.[10,46]

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Extension of the Method. In addition to identifying areas of hazardous Pb in soil with the colorimetric method, the field procedure could be used to estimate IVBA as described by Drexler and Brattin in 2007 and EPA Method 1340[39] by measuring the Pb in the field-procedure extract solution by XRF or ICP-MS and estimating the Pb extracted by the DB method by $Pb_{DB} = (Pb_{field\_procedure} - 89)/0.34$ (Figure 3) and applying corrections to the XRF readings if needed (Figure 2). One could consider developing a site-specific or updated relationship, especially if samples are from soils near large secondary smelters or unknown types of contaminations (Table 1).

Pairing the field-procedure extraction with an XRF in the field could also help select samples for more detailed laboratory analysis. In situations where laboratory analysis is not planned or available, local health departments or technicians with access to a portable hand-held XRF analyzer could still assess the health risk of soil Pb contamination more accurately by applying the field procedure than by only measuring total soil-Pb concentrations. One would need to measure the total Pb concentration in the soil in order to determine the proportion of soil Pb that is bioaccessible.

Potential Color Interference. Our observations show that in addition to analyzing the color at 5 min, waiting for 24 h to assess the amount of precipitate amount enables us to confirm the presence of Pb and lowers the detection limit. When glycine extracts turn brown, blue, or gray instead of purple, they should be categorized as medium and analyzed for the presence of precipitate after 24 h. While the use of sodium rhodizonate to detect Pb has been well documented, Feigl and Suter (1942) pointed out that complexes are formed with other cations including zinc (brown-violet), tin (violet), strontium (red-brown), barium (red-brown), cadmium (brown-red), mercury (brown-red), bismuth (red-brown), calcium (red-brown), and iron (blue). We tested the following solutions for potential interference: zinc (30 mg/kg and 1000 m/kg), barium (30 mg/kg), copper (30 mg/kg), calcium (30 mg/kg), and iron (30 mg/kg and 500 mg/kg), created from high-purity standards in 1−2% nitric acid for ICP-MS. Only iron solutions resulted in a potentially interfering dark solution, although this solution was dark blue and not purple; all other solutions remained light yellow in color. To further check for the likelihood of these interferences in our soil extracts, we examined elemental concentrations of these elements in the extract solutions. Besides Pb, the only element detected by XRF that was significantly correlated with the color ranking of our samples was zinc. Extract solutions from two Peruvian mining samples contained 2700 and 1500 mg/kg Zn; all other samples contained <300 mg/kg Zn. While the two samples with >1500 mg/kg Zn were a darker, brown color at 5 min, the presence or absence of a precipitate after 24 h accurately indicated the level of Pb. The Zn standard of 1000 mg/kg had not changed color. The Fe standard of 30 mg/kg turned blue; however, all our extracts contained Fe below the XRF detection limit of 15−20 mg/kg.

CONCLUSION

Screening soils for Pb is not common in residential areas and urban gardens, neither in the U.S. nor internationally, although the negative health impacts of Pb are well established and many studies have shown elevated Pb levels in soils, often due to historical contamination. As studies continue to confirm negative health impacts at lower Pb exposures in children and cardiovascular impacts later in adults,[4,23] testing soil for Pb is likely to receive more attention, especially in urban and previously industrial areas. The cost of materials and supplies per analysis of the current procedure is around US$5/sample. This could be reduced significantly if a kit derived from the procedure is produced in large quantities. Due to its modest cost and simplicity, the field procedure is well suited for deployment by local health departments, citizen scientists, concerned parents, or community groups without access to a laboratory.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.analchem.9b00681.

Figure S1, field-procedure instruction sheet and color guide as provided to people using the procedure in pilot studies; Figure S2, close-up photos of filtration step (step 9 in Figure S1); Figure S3, color ranking of field procedure results at 5 min, 1 h, 24 h, and precipitate ranking at 24 h; Figure S4, comparison of Figure 3 with dilution corrected by soil mass vs scoop volume; Table S1, verview of sample type and origin by figure (PDF)

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Notes

The authors declare no competing financial interest.

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