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Preliminary studies for the standardization of a pXRF analyzer *via* ICP-OES for the accurate quantification of Pb in new paint†

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Determining lead (Pb) concentrations in new paints using spectroscopic methods such as Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) requires technical expertise, consumables, equipment for method preparation, and instrumentation that can be cost prohibitive and difficult to maintain in low and middle-income countries (LMICs). Although portable X-ray Fluorescence (pXRF) analyzers are less expensive and simple to operate, their inaccuracy has limited their use to screening for the analysis of Pb in new, dried paint. To determine the limits of pXRF analyzers, new paint samples were purchased, dried, homogenized, and analyzed *via* pXRF and ICP-OES. Pb concentrations determined *via* pXRF were ~27% lower than those determined by ICP-OES. Interestingly, the concentrations determined by both methods exhibited a strong linear correlation. The resulting equations were used to calibrate an individual pXRF analyzer, improving the accuracy of the pXRF result to within ±4.4% of verified concentrations of two certified reference materials. Additionally, eleven new paint samples from Benin and Türkiye were analyzed *via* pXRF, and their calculated concentrations were similar within ±10% of concentrations as verified by ICP-OES. This method requires no acid digestion or dilutions and provides an alternative method for the determination of Pb in new paint in LMICs.

Introduction

In spite of their toxicity, compounds of lead (Pb) have been employed as paint additives for millennia because they are often vibrantly colored and increase both durability and the rate of drying.[1,2] Pb exposure from Pb-based paint results from the inhalation or ingestion of Pb-contaminated particulate matter generated from paint degradation.[2,3] Children are at greater risk than adults for Pb poisoning originating from Pb-based paint because they often put their hands in their mouths, transferring Pb from contaminated surfaces to their bodies. Exposure to Pb during childhood has been associated with behavioral issues, lower IQ scores, limited academic achievement, and life-long cognitive deficits.[2–8] Despite this, the majority of countries around the world, particularly low- and middle-income countries (LMICs), currently have no laws limiting the manufacture, import, or sale of Pb-based paints.[9,10] Thus, the identification and quantification of Pb in new paint is critical, particularly in LMICs working towards legislation limiting Pb in paint.

The World Health Organization (WHO) recently updated guidance on selecting analytical methods for the determination of Pb in new, dried paint, including methods employing flame atomic absorption spectroscopy (FAAS), graphite furnace atomic absorption spectroscopy (GFAAS), and inductively coupled plasma-optical emission spectroscopy (ICP-OES).[11] These instruments are accurate and precise and can determine concentrations below the 0.009 weight percent or 90 mg total Pb/kg of dry paint (ppm) threshold recommended by the United Nations Environment Programme's (UNEP's) Model Law and Guidance for Regulating Lead Paint.[12] However, they also require a dedicated laboratory and properly trained operators; are expensive to purchase, maintain, and operate; and require extensive and expensive sample preparation. Alternatively, portable X-ray fluorescence (pXRF) analyzers can identify and quantify Pb in paint in a variety of matrices and painted surfaces, requiring no sample preparation and returning results in real time.[13–19] Unfortunately, the accuracy of this technique is highly variable between individual spectrometers and is dependent upon several parameters including positioning of the device, thickness of paint, matrix sensitivity, and substrate effects, all of which can result in over- or under-reported concentrations of Pb.[11,20–22] In addition, most pXRF analyzers employ proprietary "modes" for analyzing samples that employ a fundamental parameters calibration methodology that attempts to provide the "total chemistry" of a sample by normalizing to 100%.[23–27] As a result, light elements such as

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carbon are often estimated and elements present in the sample but not included in the mode can introduce error, complicating the determination of Pb in organic rich matrices such as paint.

Manufacturers' modes that are specifically designed for analyzing Pb in paint often report concentrations in terms of surface loading (mg cm^{-2}), which requires knowledge of both the paint thickness and density of the dried paint to estimate the concentration of Pb in ppm.[11,20] Difficulties in accurately converting surface loading to weight percent preclude these modes from being used for the determination of Pb in new paint. As a result, modes specifically designed for the analysis of other materials including soils,[10,19] alloys,[13] and plastics[17] that return concentrations of Pb in ppm or weight percent are frequently employed in the analysis and quantification of Pb in paint. This in turn leads to instrument hardware diagnostic checks that are independent of the specific chemistry of the sample and the use of certified reference materials (CRMs) confirms only that the instrument is operating within the manufacturer's parameters, not that the determination of metals in a given paint sample will be accurate. Although the percent relative standard deviation (% RSD) of this technique can be relatively low, accuracy can be poor.[21,28,29]

There remains ongoing and continued support for the elimination of Pb in consumer products, especially in new paint. A direct and accurate method employing pXRF analyzers to determine Pb concentrations in new paint that replaces the more expensive, time-consuming, complex, "gold-standard" methods employing ICP-OES would be valuable in LMICs, especially in those countries looking to develop legislation restricting Pb in paint and increasing analytical capacity. Herein a direct method for the accurate determination of Pb in new paints *via* pXRF is reported.

Materials and methods

Safety

Gloves and safety glasses were worn during sample preparation, digestion, and analysis. Paint samples were homogenized in a fume hood while wearing appropriate personal protective equipment, including a disposable particulate respirator (3M 8511, N95).

Collection and preparation of samples

New paints from multiple manufacturers were purchased in Guyana and sampled using a modification of the method described by Gottesfeld and coworkers.[30,31] New paint was poured onto clean glass louvers (∼10 cm × 45 cm) purchased in Bartica, Guyana, and dried in a conditioned room for four days. Samples were scraped from the glass and stored in 50 mL centrifuge tubes, transported to Mercer University, and dried in an oven at 105 °C until no change in mass was noted. Paint samples (Sample ID: 1–10) collected in 2019 were difficult to homogenize and thus were first cut into small pieces with acid-washed, stainless-steel scissors and then homogenized with a mortar and pestle. An additional 11 new paint samples collected in Türkiye and Benin were received and similarly dried at 105 °C. Samples were placed in a stainless steel SPEX SamplePrep grinding vial (8009 SS), cooled in liquid nitrogen for 1 minute, and homogenized using a SPEX SamplePrep 8000M mixer/mill. All samples were homogenized to a size less than 500 μm (sieve #35).

Reagents and standards

Trace metals basis (99.999%) nitric acid (HNO_3) was purchased from Sigma-Aldrich. A standard stock solution was purchased from PerkinElmer (26 multi-element Std Pure: PE no. N9301721). Lead (Pb) standards (0.1, 0.5, 1, 5, 10, and 15 ppm) were prepared through dilution of the stock solution with 2% HNO_3 acid to construct the calibration curve. A reagent blank of 2% HNO_3 was used. National Institute of Standards and Technology (NIST)-Standard Reference Materials (SRMs) 2711a (Montana II Soil), 2580 Powdered Paint (nominal mass fraction of 4% Pb), and 2582 Powdered Paint (nominal mass fraction of 200 mg kg^{-1} Pb) were used for quality assurance/quality control (QA/QC). 18.2 MΩ cm ultrapure water was obtained from a Siemen's ELGA LabWater purification system and used as the solvent for all standards and samples.

Analysis of new paint *via* pXRF

New paint samples were analyzed in triplicate using an Olympus Vanta C pXRF analyzer on a field stand test kit employing the manufacturer's GeoChem(2) mode with no modification (10 s Beam 1, 40 kV; 20 s Beam 2, 10 kV). The instrument calculates 3-σ as error. As a result, samples detected below 3-σ return a determination of "<LOD" (limit of detection). The manufacturer's stated LOD under ideal conditions is 3 ppm. The GeoChem(2) mode utilizes a fundamental parameters calculation method.[23–27] The L_β line (12.6 keV) is used in the quantification of Pb.

Homogenized paint samples were transferred into polyethylene sample cups fitted with 4 μm thin-film proline windows (Chemplex Industries, Inc.). The paint sample height in the cup was measured to exceed 14 mm to ensure infinite thickness was reached, unless otherwise noted in Table S1.† The sample cup was rotated in between each individual measurement. Samples collected from the preliminary trip in 2019 (sample ID: 1–10) were analyzed in triplicate per 3 rotations, totaling 9 measurements per sample. All other samples were analyzed in triplicate.

QA/QC for pXRF analysis: instrument performance check and the certified reference material

A hardware check was conducted at the beginning and end of every day of analysis for pXRF. A blank and calibration check standard (NIST SRM 2711a, Montana II Soil) were analyzed at the beginning, after every tenth sample, and at the end of analysis to ensure that the mode was operating within the manufacturer's specifications. The analysis of NIST 2711a returned concentrations within ±5% of the known value.

Analysis of new paint *via* ICP-OES

Approximately 0.150 g of each paint sample was digested using a modified ASTM E1645-16 microwave digestion procedure[32] using a PerkinElmer Titan MPS™ microwave sample preparation system. A sample of NIST 2580 was digested with each batch of digested samples. HNO_3 (5.0 mL) was the only acid employed during digestion. The microwave digestion system was programmed to reach 180 °C in 5 minutes and then held at this temperature for 15 minutes. Digested samples were centrifuged in an Allegra X 14-R Beckman Coulter centrifuge at 4000 rpm at 21 °C for 15 minutes, final volumes are noted, diluted using 2% HNO_3, and analyzed *via* ICP-OES.

Pb concentrations were determined using a PerkinElmer (PE) Optima 8300 Concentric ICP-OES equipped with an SC-4 DX autosampler. Samples were introduced using a SeaSpray nebulizer with a cyclonic spray chamber. The Optima 8300, the autosampler, and the peristaltic pump were fully automated and controlled by the Windows ICP Syngistix Controller software. Operating conditions are noted in Table 1.

QA/QC: instrument performance check and the certified reference material

The solution for the instrument performance check (IPC) was prepared similarly to the standard calibration solutions for the ICP-OES. A 1.0 mg L^{-1} Pb solution was used as an IPC concentration as suggested by EPA method 200.7.[33] Yttrium was added as an internal standard *via* split-injection, and 5% nitric acid used as a rinse solution between standards and samples. The limit of detection for Pb was calculated to be 0.01 ppm. NIST 2580 was digested with each batch of digested samples, centrifuged, diluted in 2% HNO_3 and analyzed *via* ICP-OES. The IPC and CRM were analyzed at the beginning of analysis, after every tenth sample and at the end of analysis. The CRM and IPC recoveries were 96.68% and 95.95%, respectively.

Results and discussion

I: Pb concentrations in new paint purchased in Guyana

Sixty unique cans of paint in a variety of colors were purchased from multiple stores in Bartica and Georgetown, Guyana (Table S1†). Where possible, paint origin was noted as was the type of paint (synthetic enamel, quick-dry enamel, primer, *etc.*). Of the paints analyzed, 33% exceeded the Guyanese limit of 600 ppm [Pb][34] and 35% exceeded the UNEP's recommended 90 ppm Pb in the paint limit as determined by ICP-OES.[12] The maximum value of Pb in paint determined in this study was greater than 2600 times the recommended 90 ppm limit. Three of the 60 paints that were labeled "lead free" had Pb concentrations over 13 700 ppm, with the highest concentration being 55 205 ppm.

Table 1 Operating conditions for ICP-OES

Parameter	
RF generator power (W)	1500
Frequency of the RF generator (MHz)	40
Plasma gas flow rate (L min^{-1})	Ar, 10
Auxiliary/shear gas flow rate (L min^{-1})	0.2
Nebulization gas flow rate (L min^{-1})	0.70
Sample uptake rate (mL min^{-1})	1.50
Type of detector	Dual state solid
Injector tube diameter (mm)	2.0
Plasma view, distance (mm)	15.0
Read	Peak area
Measurement replicates	3
Read delay (s)	120
Rinse time (s)	150 (no delay)
Internal standard	10 ppm Y in 2% HNO_3

Paint samples with determined non-zero Pb concentrations and low % RSDs *via* both pXRF and ICP-OES range in concentration from 13.3–236 163 ppm as determined by ICP-OES. A regression analysis of the 27 sample measurements with non-zero concentrations of Pb (Fig. 1) from Table S1† reveals poor agreement between individual measurements, indicating that the pXRF analyzer underestimates Pb by ~27% in comparison to ICP-OES analyses. Yet, a very strong linear relationship ($R^2 = 0.9977$) exists between the measurements collected *via* pXRF and ICP-OES. The shaded blue area represents the prediction interval for the regression line; hence, the narrow band of the prediction interval indicates the usefulness of using pXRF measurements to predict ICP-OES measurements.

When the equation generated in Fig. 1 is forced through a y-intercept of zero ($y = 1.2651x$), Pb concentrations in new, dried paint as determined *via* pXRF can be corrected and their accuracy improved. In essence, this new calibration curve can be defined as $pXRF_c = 1.2651 \times pXRF_m$, where the term $pXRF_m$ is the Pb concentration measured *via* pXRF and $pXRF_c$ is the Pb concentration corrected to ICP-OES. To demonstrate the validity of this new relationship two lead-in-paint certified reference materials were analyzed in triplicate *via* pXRF, CRM 2582 (certified [Pb]: 208.8 ± 4.9 ppm) and CRM 2580 (certified [Pb]: 43 400 ± 100 ppm Pb). When analyzed *via* pXRF directly ($pXRF_m$), CRM 2580 returned a concentration of 36 400 ± 318 ppm Pb and CRM 2582 returned a concentration of 181.3 ± 4.6 ppm, 16.1% and 13.2% lower than the known concentrations of the CRMs, respectively. However, when applying the equation $pXRF_c = 1.2651 \times pXRF_m$ the corrected concentrations ($pXRF_c$) both fall within 10% of the known concentrations, with $pXRF_c$ for CRM 2580 returning a concentration of 46 100 ppm (6.2% higher than the known value) and the $pXRF_c$ for CRM 2582 returning a concentration of 229.4 (9.9% higher than the known value).

Despite the strong coefficient of determination found in Fig. 1, with only 27 data points spanning several orders of magnitude of Pb concentrations, it is likely that the curve itself is not truly linear across all concentrations. It was hypothesized that generating equations from sequential data points that were subsets of the full calibration curve would improve the accuracy of $pXRF_c$. When a curve is generated from concentrations between 8–1115 ppm as measured *via* pXRF (Fig. S1†), the resulting equation ($pXRF_c = 1.1791 \times pXRF_m - 8.387$) returns an estimated concentration of 205.4 ppm Pb for CRM 2582, which is 1.6% of the known concentration. Similarly, when a curve is generated between 24 054–169 680 ppm as measured *via* pXRF, the resulting equation ($pXRF_c = 1.3156 \times pXRF_m - 2616.5$) (Fig. S2†) returns an estimated concentration of 45 300 ppm Pb, which is 4.4% of the known CRM 2580

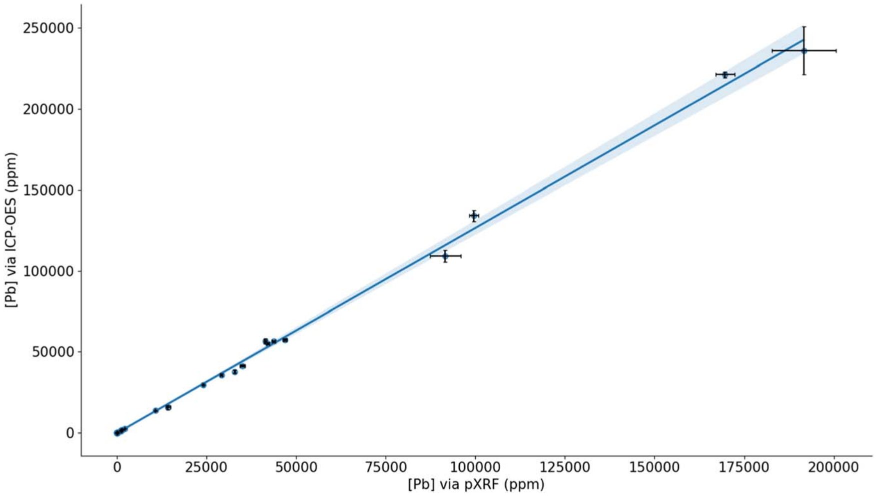

Fig. 1 Direct comparison of Pb concentrations (ICP-OES, ppm) *vs.* Pb concentrations (pXRF, ppm). ($y = 1.268x - 281.329$; $R^2 = 0.9977$). The shaded blue area indicates the 95% prediction interval for the regression line.

concentration. In both cases, accuracy was improved by using subsets of the full calibration curve. These results indicate that one calibration curve over a wide range of concentrations of Pb in paint limits the accuracy of this method, particularly at the lower end of the curve. The improvement of accuracy for CRM 2582 to within 1.6% of the known value is specifically noteworthy, as this approached the 90 ppm proposed limit for Pb in paint. These results further indicate that additional data will continue to refine and improve the accuracy of the corrected pXRF value and perhaps allow for pXRF to be used for the determination of Pb in new paint, supplanting more expensive and time-consuming spectroscopic methods.

In order to confirm the utility of the generated calibration curves and their applicability to paint in other regions, 11 new paint samples collected in Benin and Türkiye were analyzed *via* pXRF and ICP-OES (Table 2). All concentrations determined *via* pXRF were corrected using the general equation generated in Fig. 1 and the appropriate equations generated in Fig. S1, S2, or S3.† In all cases the % difference is determined. All calculated Pb concentrations were within ±10% when using the equations generated in Fig. S1, S2, or S3.† As noted previously, it is anticipated that accuracy will improve as additional data are incorporated into the pXRF/ICP-OES calibration curves.

The results presented here open the possibility that pXRF analyzers can be used to accurately determine Pb concentrations in new, dry paint. While more work must be completed to understand the limitations and scope of this relationship, and each pXRF analyzer employed in the analysis must generate its own calibration curve from paint analyzed *via* a valid spectroscopic method such as ICP-OES, the analysis of dried and homogenized new paint samples in sample cups using pXRF can be used to inform decisions made by laboratory technicians *prior* to analysis with conventional methods. It is possible that pXRF analysis can supplant analysis *via* ICP-OES for the determination of Pb in paint, allowing for the rapid and accurate analyses of multiple new paint samples without the need for expensive and

Table 2 Calculated concentrations of Pb in new paint samples from Türkiye and Benin

Sample #	pXRF_m [Pb], ppm	ICP-OES measured [Pb], (ppm)	pXRF_c (full curve[a]) [Pb], (ppm)	% Difference from ICP-OES	pXRF_c (subsets[b]) [Pb], (ppm)	% Difference from ICP-OES
B1	50 102 ± 1256 (2.51%)	66 471 ± 173 (0.26%)	63 384	4.6	63 298[c]	4.8
B2	58 016 ± 422 (0.73%)	71,104 ± 351 (0.49%)	73 396	3.2	73 709[c]	3.7
B3	1693 ± 89 (5.3%)	1961 ± 16 (0.81%)	2142	9.2	1956[d]	0.2
B4	384 ± 2 (0.40%)	403 ± 4 (1.1%)	486	21	444[e]	10
T1	<LOD	<LOD	N/A	N/A	N/A	N/A
T2	34 235 ± 1994 (5.8%)	44 025 ± 903 (2.1%)	43 311	1.6	42 423[c]	3.6
T3	29.7 ± 3.2 (11%)	<LOD	37.6	N/A	26.6 [e]	N/A
T4	47 469 ± 1309 (2.8%)	59 223 ± 963 (1.6%)	60 053	1.4	59 834[c]	1.0
T5	41 662 ± 1393 (3.3%)	54 167 ± 273 (0.50%)	52 707	2.7	52 194[c]	3.6
T6	9.7 ± 0.6 (6.0%)	<LOD	12.3	N/A	3.1 [e]	N/A
T7	41 433 ± 896 (2.2%)	48 411 ± 1019 (2.1%)	52 417	8.3	51 893[c]	7.2

[a] pXRF_c is calculated from the equation generated from all data, $\text{pXRF}_\text{c} = 1.2651\text{pXRF}_\text{m}$ for $[\text{Pb}]_\text{ICP}$: 13–236 163 ppm. [b] pXRF_c is calculated from equations generated from sequential data points that are subsets of the full calibration curve as described below. [c] $\text{pXRF}_\text{c} = 1.3156 \times \text{pXRF}_\text{m} - 2616.5$ (Fig. S2). [d] $\text{pXRF}_\text{c} = 1.2781 \times \text{pXRF}_\text{m} - 207.45$ (Fig. S3). [e] $\text{pXRF}_\text{c} = 1.1791 \times \text{pXRF}_\text{m} - 8.387$ (Fig. S1).

time-consuming acid digestions of paint samples, the expense of maintaining and operating sensitive instrumentation, or the expense of shipping and analyzing paint samples to laboratories with the capacity to analyze samples.

Data availability

The data supporting this article have been included as part of the ESI.†

Author contributions

Caryn S. Seney: conceptualization, data curation, formal analysis, funding acquisition, investigation, methodology, project administration, supervision, writing – original draft, writing – review and editing. Adam M. Kiefer: conceptualization, data curation, formal analysis, funding acquisition, investigation, methodology, project administration, supervision, writing – original draft, writing – review and editing. Samantha T. Brown: investigation, methodology, writing – review and editing. Evan R. Stair: investigation, methodology, writing – review and editing. David G. Nelson: data curation, formal analysis, visualization, writing – review and editing. Lloyd L. Bandoo: writing – review and editing, resources, investigation, writing – review and editing. W. Benjamin Stewart: data curation, formal analysis, investigation, methodology, writing – review and editing. Nuren Z. Lara: visualization, writing – review and editing. Clare Donaldson: funding acquisition, resources, writing – review and editing.

Conflicts of interest

There are no conflicts of interest to declare.

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