Sampling the Soils around a Residence Containing Lead-Based Paints: An X-ray Fluorescence Experiment

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Chemical educators need to instruct students on the process of sampling since this is a critical step in chemical analysis. This experiment ties sampling to a societal issue: minimizing exposure to lead in soils from lead-based paints. Considering that lead-based paints were used on residential constructions for more than five decades in the United States, this is an issue that exists nationwide. The removal of lead from gasoline, which was hailed an epidemiological success, may have generated the societal perception that the lead exposure problem has been solved. Exposure to lead from soil and dust still appears as a critical issue *(1)*. Linking fundamental sampling concepts to this societal environmental issue should make students more aware of this exposure pathway.

This experiment utilizes field portable instrumentation and applies an EPA soil screening method. Students gain insight into the utility of cost effective screening technologies. A report in this *Journal* discusses the use of a screening method; however, in that lab, students were comparing and contrasting the results from different methods and were not focusing on sampling *(2)*. A number of articles in this *Journal* discuss sampling activities and error analysis *(3–6)*. Two other new experiments in this *Journal* highlight screening technologies and this lab complements them with an emphasis on field sampling *(7, 8)*. This X-ray fluorescence (XRF) experiment is designed as a general science lab; however, it could be adapted for analytical or environmental chemistry labs.

Field portable X-ray fluorescence (FP-XRF) instruments were developed through the EPA Superfund Innovative Technology and Evaluation and Housing and Urban Development (HUD) lead exposure minimization programs *(9, 10)*. Instrumental field sampling has been employed as a cost effective methodology in industry (mining, environmental science, etc.) *(11)*. With the increased availability of field portable instruments, it is desirable to have experiments that demonstrate how the quality of the sampling yields cost savings by minimizing the number of samples sent for confirmatory lab analysis *(11, 12)*. A sampling laboratory connected to a societal issue should be especially valuable to chemical educators since sampling has previously been raised as a critical topic. In response, a few educators have written articles on transforming analytical labs to be more project oriented *(13–15)*.

Figure 1. Student collecting spectral data in the field.

Experimental

This sampling experiment involves in situ nondestructive XRF lead soil analyses. The soil sampling lab follows the U.S. EPA Method 6200 *(10)*. The limitation of this method is that the soil can not be too wet, so rainy weather must be avoided *(10)*. Students know the relative accuracy of the data because NIST standard reference materials are used to calibrate the XRF instrument. Students collect spectral data in the field during one three-hour lab period (Figure 1). Since students average the four spectral readings at each of the in situ sampling locations and compute the standard deviations, students directly observe sample variability. To complete the experiment, students make comparisons to values in public lead risk documents and write letters communicating the results and potential risks to the homeowner *(16)*. An extension of the experiment is to have students perform an ex situ XRF method and compare the results evaluating their sample preparation process *(17)*. If an educational institution does not own an XRF, then renting an instrument is an option since FP-XRFs are routinely rented.

The sampling site is a pre-1950 residential construction in Oakland, California. The residence is far from major highways and known to have lead-based paint on the exterior. The owner is a colleague who offered the site prior to repainting and presumably many residential sites throughout the country are available. The site size facilitates students working in teams to prepare sampling locations including the field background. Students can complete the lab in one three-hour lab period; however, two lab periods provides for more detailed instruction and flexibility for proper field sampling.

Hazards

Faculty obtained radiation safety training from the instrument manufacturer through a one day seminar. Before field sampling began, students were given radiation safety information as a part of the prelab assignment and were provided with an informed consent form. No student was required to actually trigger the instrument if they felt that radiation exposure risks were too high, based on their own health situations, but all students were required to evaluate the spectral data. All students collected data with the instrument, since students were provided sufficient information to evaluate their radiation risk as very low. Students were also educated on minimizing their contact with soil by wearing gloves, safety glasses, and dust masks.

Residential Sampling and EPA Method 6200

Student teams selected their sampling locations following the U.S. EPA's Lead Safe Yards program information *(18)*. The Lead Safe Yards documents define the dripline and background sampling locations; all dripline locations were within 30 cm of the building. In some cases, sampling locations occurred under windows that had peeling paint. Since the lab was limited to three hours and there was some travel time to the site, the students sampled five locations. A background sampling location that was more than 8 m from the residence, in a garden, was selected. In addition, XRF data were collected on paint chips, which demonstrated peeling paint as a source of lead in the dripline soils.

Each designated sampling location, including the background, was carefully cleared so 100 cm^2 of soil was exposed and the soil was packed firm with a wooden trowel following Method 6200. The students collected four 60 s exposure spectra selecting different places in the exposed area. For each in situ sampling location, students averaged the lead content from four spectra. More than 50 spectra were collected in one afternoon and all the numerical results were provided to the students.

Instrumentation Specifics and Standards

Soil lead content was quantified using a Niton 796 XLt (other instruments could be rented) *(9, 19)*. The X-ray tube output was first quantified and then the spectral output was validated by recording spectra on a NIST standard reference material (SRM). The instrument's specifications listed the accuracies within 10% for the NIST 2711 SRM and reference spectra were recorded in triplicate at the beginning of sampling. The FP-XRF has a bracketed spectral shoe that attaches to the instrument so when placing it into soft soil, the proximity safety will be depressed. This shoe ensured that X-rays are only emitted when the instrument's trigger is pulled and in contact with the soil (Rental XRFs have different safeties for field use).

Data Analysis and Results

Students averaged the numerical results of four spectra at each sampling location including the background and computed the standard deviations. The average sample soil lead values were compared to the background and to EPA's preliminary remediation goals (PRGs) *(16)*. Measurements for each sampling location are given in Table 1. The background sample is just above the instrumental detection limit (40 mg/kg for lead) for a 60 s exposure *(19)*. The dripline sample spectra clearly have lead Lα and Lβ X-ray lines as shown in Figure 2. Reading #42 (the lower spectrum) shows less intense lead X-ray lines and reading #52 (the upper spectrum) shows more intense lead X-ray lines. Both spectra show iron X-ray fluorescence lines with nearly identical intensities. The instrument's computer calculates the lead content from the peak intensities. Dripline locations 1–3 show lead content at least five times greater than the background, indicating the deteriorating paint as the source. The average soil lead at a given location shows large standard deviation, which reflects inhomogeneity in the sample. The homogenous NIST 2711 SRM spectral result shows a small standard deviation and the instrumental values are accurate to within 8.0%. For this experiment, the focus is to have students gain insight on the process of field sampling and have sufficient data to see general trends without performing a rigorous statistical analysis.

Discussion

This sampling lab experiment is directly related to an environmental societal issue. The author has been affiliated with the SENCER project, which has recognized that teaching through societal issues can yield more student interest *(20)*. The FP-XRF instrumental methods are designed for rapid data collection, so multiple sampling locations can be measured in one lab period. The students gained a better appreciation for the number of sample locations that can be sampled in an afternoon of work using EPA Method 6200. Students in the field were critical of sample site 4 data where the organic debris yielded very low values since the instrument was not truly in contact with the soil. Students were motivated to obtain quality data and inform the homeowner where to find useful lead

Table 1. In Situ Lead Content Field Average at Selected Sampling Locations

Soil Samples	Lead Content in Soil (mg/kg)	Standard Deviation (mg/kg)
NIST 2711	1069	96
Background	58	21
Site 1	314	84
Site 2	651	382
Site 3	751	448
Site 4[a]	32	2

[a]Organic debris interfered with the site reading.

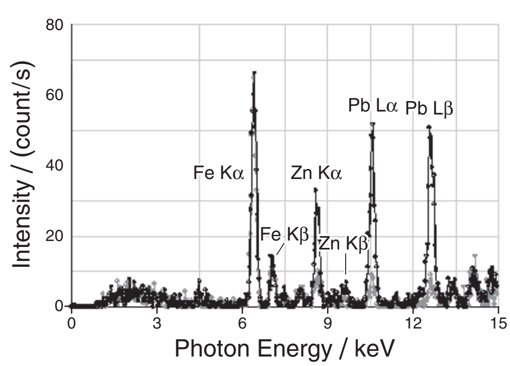

Figure 2. XRF spectra of soil samples from two dripline sampling locations.

poisoning prevention documents. A follow-up class discussion, where students explained points in their draft letters to the homeowner, gave the class insight on risk assessments. The class explored that topic more deeply since the students had data to apply to the discussion. Student lab notebooks and letters were evaluated with respect to the computed average soil lead values, data comparisons to EPA PRGs, and the recommendations to minimize contact with the soil. Students completed a survey (Student Assessment of Learning Gains) and their responses to a question on adequately maintaining painted surfaces for similar aged houses demonstrated a point on minimizing exposure to lead.

Acknowledgments

This XRF field sampling experiment was developed as an outcome of a SENCER-AACU grant and additional College funding to support innovative curriculum. The Niton 796 XLt instrumentation was purchased by the Chemistry Department with the support of the Camille and Henry Dreyfus Foundation (Special Grant # SG-04-083). The access to the colleague's Oakland residence was appreciated.

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