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### Field Sampling with a FP-XRF: A Real-World Lab Experience

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## Field Sampling with a FP-XRF: A Real-World Lab Experience

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### ABSTRACT

A field sampling laboratory experiment was developed so students would gain experience sampling on a field site and have an introduction to XRF spectrometry. The experiment used a rented field portable XRF instrument (FP-XRF) to quantify the lead in soil samples collected adjacent to an urban highway and explored aspects of the USEPA Method 6200. Rainy weather conditions eliminated the possibility to record spectra in the field, so sample preparation procedures were modified to model the typical in situ and intrusive mode of spectral investigations stated in the method. The lead content in the soil samples collected at 15 ft from the highway were determined to be greater than 2000 ppm. The soil lead content decreases as the distance perpendicular to the highway increases. The site background sample collected from 300 ft away is nearly equal to the instrumental lead detection limit of 20 ppm.

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The experiment demonstrated the need to collect replicate spectral data for in situ sampling and that sample homogenization is a critical step in the intrusive sample analysis mode.

**Key Words:** Field sampling; XRF; Spectroscopy; Soil lead; Urban highway; Sample homogenization.

## INTRODUCTION

The traditional chemistry laboratory curriculum has students learning many standard instrumental methods to quantify various analytes. Students in instrumental analysis or analytical chemistry courses are typically introduced to numerous electrochemical, chromatographic, and spectroscopic methods to adequately prepare them for industry, graduate and professional programs. The analytical and instrumental chemistry texts clearly explain that proper sampling and sample preparation is a necessity for meaningful analyses in the chemical sciences,<sup>[1]</sup> but in numerous laboratory manuals the emphasis on sampling does not receive as much attention. Some analytical chemistry faculty have developed more problem-based learning experiences to begin to attend to this issue.<sup>[2]</sup> The laboratory experiment reported here represents an example where students explore the true challenges of sample collection and sample preparation. This experiment should be adaptable to many institutions.

Analytical chemistry faculty at the 1998 American Chemical Society (ACS) National Meeting—Boston symposium expressed the need for more instruction with a focus on the “generic components of an analytical problem-solving approach.”<sup>[3]</sup> This group emphasized that getting students to be more adept at defining the analytical problem, recognizing how to sample properly, along with separating the analyte from interfering substances, were more important issues than training on newer instrumentation.<sup>[3]</sup> Margaret Merritt of Wellesley College who was a participant at 1998 ACS meeting, developed a problem-based analytical laboratory experience involving pigment identification of art objects. In this laboratory experience, students also recognized the additional constraint that art conservators face which is applying nondestructive techniques.<sup>[4]</sup> Our new field sampling experiment has been developed to address one of the fundamentals noted above. Field sampling with portable instrumentation has become a common cost-saving methodology for environmental and redevelopment work so this type of experiment can be developed around numerous real world problems (e.g., monitoring contaminant plumes, soil surveys for pollutants, etc.). The new



instrumental analysis experiment reported here utilizes field portable x-ray fluorescence (FP-XRF) instrumentation. The use of a rented FP-XRF highlights to students that institutions may consider leasing or renting instruments or contracting out for expertise on future projects instead of fully developing expertise in house. In particular, numerous manufacturers rent FP-XRF instrumentation, which various industrial and environmental firms use for project work. The FP-XRF laboratory experience facilitated a focused discussion on proper sampling and allowed us to incorporate this type of instrumentation more easily into the course. The field sampling takes some additional preparation (filing access permits, arranging instrument rental, etc.) however, the students' interest more than compensates for the faculty member's extra efforts.

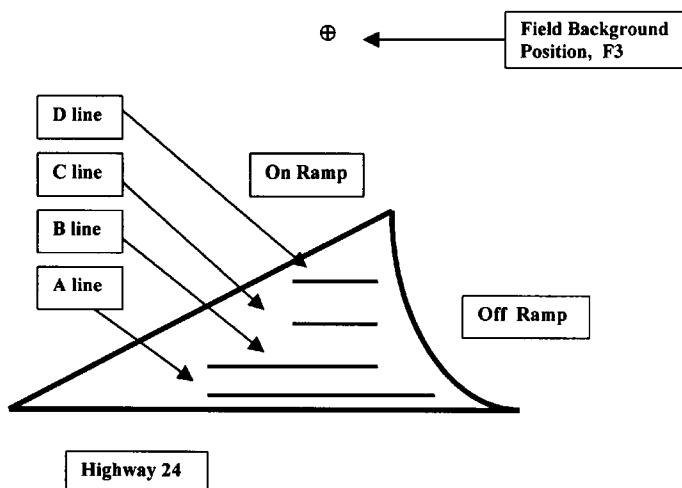
## EXPERIMENTAL

### Site Selection

The field sampling experiment had a relatively straightforward objective: quantify the lead in the soils adjacent to a major highway. The selected highway (California Highway 24) has been in use for more than 50 years and is a major transit freeway in the San Francisco Bay Area. The highway should have received significant deposition of lead over the years from leaded gasoline. The selected site is an on/off ramp property that is roughly triangular in shape, as shown in Fig. 1. The parcel is nearly linear adjacent to Highway 24 so sampling was easily organized. The site is within a mile of the Caldecott Tunnel and the tunnel limits the flow of traffic during commute hours. Periodically, the trees and shrubs are trimmed and grasses and weeds are mowed, however the site appeared relatively undisturbed, so it should be good repository of lead deposition from the use of leaded gasoline. To have access to the site required an encroachment permit from the state highway authority (CAL-TRANS) which was obtained in advance.

### Safety

Safety protocols required a great deal of attention for both the faculty member and students. The field work was done adjacent to a heavily travelled freeway and the soils samples were predicted to have high values of lead. Furthermore, the class was using the FP-XRF instrument which contained a radioactive source for generating the excitation x-rays to analyze the soils. As a requirement of the CAL-TRANS permit, all personnel were required to wear



**Figure 1.** Schematic drawing of the Highway 24/Gateway Boulevard field site. The site was defined by the west bound highway lanes and the on ramp and off ramp. There were five sampling positions on the A line, four sampling positions on the B and three sampling positions on both the C and D lines. The background sample position, F3 was 300 ft from the highway and adjacent to a frontage road further away from the highway.

reflector vests and hard hats on site and no work was authorized in the traffic or breakdown lanes. All personnel wore safety glasses, hand gloves, sturdy shoes, and appropriate clothing for collecting soil samples. Dust masks were provided to the class initially. However since it was the rainy season, the soils were very damp and dust was not an issue. The FP-XRF instrument was a Niton 700 XLI, which had two radioactive sources ( $Cd^{109}$  and  $Am^{241}$ ). In the State of California, the rental of this instrument did not require the user to have an additional radiation safety license from the Department of Health Services. However, the regulatory paperwork must be kept with the instrument during transit (including leak test paperwork) and regulatory procedures must be followed. The faculty member attended an all day workshop, which included safety instruction on the instrumentation. In preparation for the lab, an instructional video on safe use of a FP-XRF instrument with radioactive sources was incorporated into the student's pre-lab. The students were introduced to the instrument and safety protocols by the Niton representatives and were supervised by the faculty when the instrument was in use. Since the instrumentation generates x-rays, a human subject protocol was also filed with the College. Students who felt that the lab would potentially expose them to undue risk were allowed to opt out without penalty and only evaluate the collected data.



### **Sampling and Instrument Calibration**

The instrumental analysis laboratory handout informed the students that they would establish a simple sampling grid upon viewing the site using the hypothesis that the lead concentration should, in general, decrease upon moving perpendicular to the main highway. No formal sampling plan was prepared prior to the day of field sampling in this experiment since the class had only limited access to the site. This educational component could be incorporated in other field studies. To demonstrate the utility of the field portable instrumentation, the lab handout stated that any areas with relatively high concentrations (i.e., hot spots) identified on the sampling grid would receive further investigation. The students constructed a grid recognizing the limitations of the off ramp and on ramp using two 150 ft and one 50 ft field tape measures and numerous stakes, as shown in the schematic drawing of the site in Fig. 1. Five sampling positions were selected 15 ft from the roadway and the positions were 50 ft apart from each other. This line was the sample line A, and the positions were numbered 1 to 5 with the lowest at a position nearest to the Caldecott Tunnel, which is west of the site. Three additional lines B, C, D were 30, 60, and 90 ft from the roadway. The sample positions on the B, C, or D lines corresponded directly to the line A numbers, therefore mapping perpendicular to the roadway. There were four positions on line B, three positions on both line C and D. Due to the triangular shape of the parcel, certain perpendicular positions could not be sampled. As well, near the edge of the roadway and on/off ramps the soils contained more gravel and rock most likely by design of roadway engineers, so the grid was somewhat truncated. One position, F3, was 300 ft from the roadway and served as a background sample.

The students were directed to read portions of USEPA Method 6200, which the laboratory sampling experiment was designed to mimic, applying both *in situ* and some intrusive sampling.<sup>[5]</sup> Our initial plan was to obtain four spectra at each position on the grid in the field and then collect soil samples, homogenize them in a plastic bag and then record the spectra, similar to the procedure outline by Stephen Shefsky.<sup>[6]</sup> This procedure should demonstrate the need for sample homogenization when applying XRF instrumentation or the collection of numerous replicate spectra *in situ*, as noted in Shefsky's analytical report and the EPA verification reports.<sup>a</sup> The students were not directed to record formal sample blanks. However, the Niton XLi 700 documentation states a detection limit of 20 ppm for lead using an intru-

<sup>a</sup>EPA Environmental Technology Verification Report, Field Portable X-ray Fluorescence Analyzer; Niton XL Spectrum Analyzer, EPA-VS-SCM-06, 8-21, 64-98.



sive sampling procedure using NIST SRM materials.<sup>b</sup> Method 6200 also specifies that the moisture content must not be greater than 20% so that the overall error from moisture is minimal.<sup>[5]</sup>

Our sampling experiment occurred during the rainy season and a fine mist was present while the students collected samples in the field, therefore only the intrusive sampling procedure could be applied. Following the EPA protocol, surface soil samples were collected from a 4 × 4 inch square of soil with removal of obvious organic debris and large rocks.<sup>[5]</sup> The collected soil samples were oven dried in the laboratory. The dried soils were ground with a mortar and pestle, however the samples were not sieved. To simulate the field sampling that would normally occur before the formal intrusive sampling, the oven dried samples were placed in a plastic bag and spectral data was collected from four different positions on the bag. The soil samples were then homogenized with added sodium fluorescein, by kneading the sample in the plastic bag for a minimum of 2 minutes following the directions in the EPA verification report.<sup>a</sup> These homogenized soil samples were analyzed again recording the spectra twice by positioning the instrument window somewhere near the center of the bag. As noted above, none of these samples were sieved as specified in formal intrusive sampling procedure of Method 6200 since the EPA verification report indicated that good precision can be obtained from simple sample homogenization in the plastic bags.<sup>a</sup>

The Niton XLi instrument Cd<sup>109</sup> source was used to analyze the soil samples for lead. As a part of the instrument start up, a source energy calibration was performed with the instrument's tungsten shutter covering the sample window. To further validate the analysis, two NIST SRM soil standards (2710 and 2711) were analyzed. After the two calibration steps, the spectral data was recorded, subsequently downloaded from the instrument, and the numerical results for lead were tabulated in a spreadsheet.

## RESULTS

The Spring 2003 Instrumental Analysis students sampled the Highway 24 field site in late February. Due to the rainy conditions, the soils were too wet to measure the lead content while in the field therefore site characterization was limited to the selected sample grid positions. The instrumental analysis students collected soil samples using only the top few centimeters of soil as

<sup>b</sup>Niton XLi 700 Series Instruments — Element Limits of Detection in Soils, mg/kg (ppm), XLi700-LOD-V1.01.pdf; contact Niton representatives.



described above. The soil samples were labeled and oven dried in the laboratory (oven temperature set to 110°C for 4 days). The dried soils were placed in new plastic bags after a small amount of grinding with a mortar and pestle but without thorough mixing to simulate the sampling that would have occurred in the field. As stated above, four spectra were recorded from different positions on the bag. Before studying the samples, the spectral data of two NIST SRM samples was recorded. The results were  $5548 \pm 387$  ppm and  $1151 \pm 223$  ppm, respectively, for NIST SRM 2710 and 2711 with an error of less than 1% from the NIST values.<sup>[7]</sup>

All bagged soils contained generally fine particles due to the initial grinding. The average lead content for the unhomogenized soil samples are listed in Table 1. All spectra from sample positions on line A gave lead content values greater 2000 ppm lead. The four replicate spectra at position A4 gave lead content values of 10,100, 7591, 10,600, and 7286 ppm and yielded an average soil lead content of 8894 ppm which was the highest of all the field positions. The four different replicate values demonstrate the instrument sensitivity dependent on alignment of the sample in front of the instrument sampling window. The x-ray fluorescence spectral data shows the lead L $\alpha$  and L $\beta$  lines with nearly equal intensity, which is consistent with theoretical predictions. The

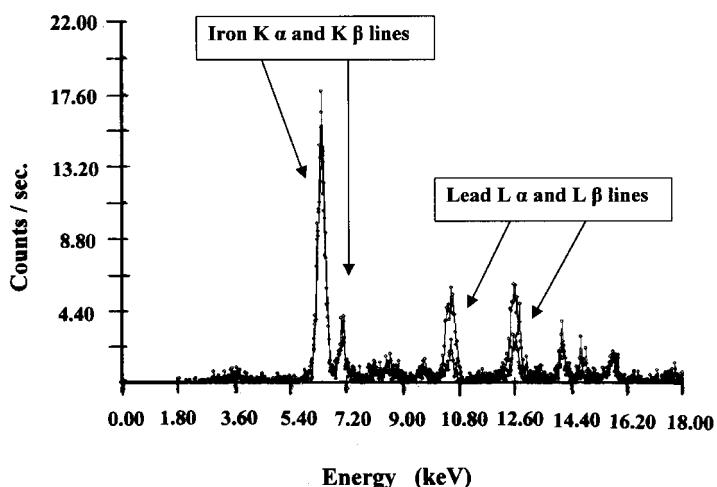
**Table 1.** Lead content in Highway 24 Gateway Boulevard on/off ramp median.

Sample position	Average unhomogenized lead content		Average homogenized lead content	
	ppm	Standard deviation	ppm	Standard deviation
A1	2,517	791	2,187	485
A2	4,815	1,205	4,449	436
A3	4,742	887	4,650	23
A4	8,894	1,698	7,756	293
A5	3,731	577	2,850	13
B1	1,080	32	860	2
B2	706	126	722	52
B3	786	83	903	48
B4	663	137	482	214
C2	439	145	446	240
C3	410	49	452	98
C4	337	46	308	81
D2	95	45	22	4
D3	410	45	482	31
D4	269	56	278	63
F3	32	21	42	10



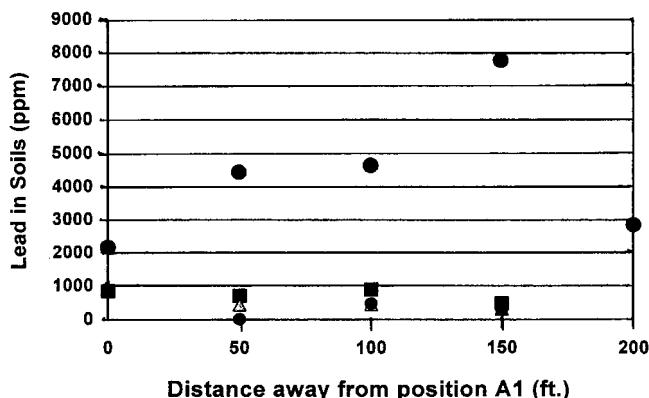
content of iron appeared reasonably uniform in the soils and the iron  $K\alpha$  and  $K\beta$  lines are nearly coincidental for most of the sample spectra. Two spectra which are representative of the collected spectra data are shown in Fig. 2. The A line lead content values are comparable to soil lead values reported by Harrison et al. for heavily travel urban roadways, too.<sup>[8]</sup>

There is a general decrease on each of the numbered lines perpendicular to the highway, which is shown in Fig. 3. The lead content values on the A line are the highest for each numbered lines. Due to the wet soil conditions, sampling in the field did not occur so the position A4, hot spot was not well characterized. The differences in the values on the B and C lines do not show any obvious trends. The D line values show a wider range of values than B and C line data. In particular, the D2 sample lead content is very low. However, the D2 sampling position was very close to the on ramp and the soil appeared noticeably different to the students when the sample was collected. The F3 position lead content values are approaching the instrument detection limit, which was recorded using standardized materials by the manufacturer.



**Figure 2.** Representative spectra: The top spectra (Reading #212) is from position A1 and shows higher counting rates for the lead lines; The bottom spectra (Reading #232) is from position B1 and shows lower counting rates for the lead lines. There is a noticeable difference in the counting rate at the energies of 10.55 and 12.61 keV which coincide with the  $L\alpha$  and  $L\beta$  lines for lead. The iron content in the soil appears nearly constant for these soils and the  $K\alpha$  (6.40 keV) and  $K\beta$  (7.06 keV) lines for iron are essentially the same for the two spectra.





**Figure 3.** Highway 24 site soil lead content. The soil lead content is the highest for sampling positions closest to the highway. The line A (15 ft from highway), line B (30 ft from highway), line C (60 ft from highway), and line D (90 ft from highway) values have the shapes ●, ■, ▲, ● respectively. Each numbered line at given distance away from position A1 shows the largest decrease between the line A and line B sample positions. The line B soil lead values are just slightly above the EPA's industrial soil PRG values. The line C and D lead content values nearly coincide for each numbered line with four sampling positions (A,B,C,D).

Looking only at the averaged unhomogenized soil lead values, the trend of higher lead content with closer proximity to the highway was clearly demonstrated even though the on/off ramps could have complicated the analysis. The lead in the soils was non-uniformly distributed across the field site. The students made additional observations that a storm drain was near the off ramp and in between sampling position A5 and B4, however, this feature did not give any insight on the numerical results. The F3 sampling position gave a result of  $32 \pm 21$  ppm and indicates that the onsite background sample is below the detection limit since the EPA verification report states the detection limit would be 3 times the standard deviation on precision based standard and the quantitation limit would be 10 times the standard deviation.<sup>a</sup> With a field detection limit of 60 ppm lead for this study and a quantitation limit of 200 ppm, the lead content at positions F3 and D2 are not considered quantitative, but the other 14 positions are sufficiently high to quantify the lead content.

The unhomogenized bagged soil samples were more thoroughly mixed after the addition of the sodium fluorescein.<sup>a</sup> To estimate the extent of mixing, a handheld UV lamp was illuminated on the bagged soils. If the sample gave uniform molecular fluorescence then it was considered



sufficiently homogenized to record the x-ray fluorescence spectra. This homogenization step was performed by a number of students. In the EPA verification report, the homogenization step was noted as a key quality control factor that could affect the results. To minimize any sample preparation variations, the same personnel was used in the EPA test. For our experiment, each student needed to be exposed to this sample preparation step. Unfortunately, lower quality data may result since a quality control factor under the EPA testing conditions has become a variable factor in our experiment. This sample homogenization part of the protocol appeared to be only partially successful for the students in this class and their reported observations indicated that the sodium fluorescein seemed to adhere to the plastic bag, too.

After this homogenization step, the XRF spectra were recorded twice for each soil sample. The average homogenized lead content values are also listed in Table 1 and are evenly split with eight higher than and the other eight lower than the averaged values of the unhomogenized samples. In our treatment, 11 of the unhomogenized samples gave larger standard deviation values than the homogenized soil values at the same sample positions, which shows the importance of homogenization of the soils. Although the decrease in standard deviation was not observed for all the sampling positions, this may reflect a level of inexperience in homogenizing the soil samples. A more thorough sample homogenization procedure could be implemented where samples are ground in a ball mill and the resulting homogenized soils loaded into XRF sampling cups for analysis. However, since this lab experience was designed to model sampling in the field, a more rigorous sample homogenization procedure was not implemented. In the EPA Method 6200, the soils being analyzed following an intrusive mode of sampling would be homogenized by grinding, sieved through a number 60 sieve, and packed in an XRF solid sample cups with a mylar film window.<sup>[5]</sup> This was not done since the class was only utilizing the instrument for 1 week.

After the homogenization process with added sodium fluorescein, the precision in the replicate samples improved for 11 of the 16 samples. In our procedure, these spectra were collected sampling the center of the plastic bag, however, students did not attempt to measure the exact same spot on the sample bag. The lead content at position A4 was still the highest at 7756 ppm. All A line soil lead values were still above 2000 ppm and there are rough incremental decreases on each line moving perpendicular to the major highway, as noted above. In a *t*-test of differences between the homogenized and unhomogenized lead content values in Table 1, the averaged values are not statistically different at the 95% confidence level. Therefore, our data set supports that individuals sampling in the field should collect many replicate spectra at a sampling position which is the proper *in situ* method or analyze a homogenized sample collected from the sampling



position representing an intrusive method. These results are in general agreement with results reported in the EPA verification report.<sup>a</sup>

## DISCUSSION

This field sampling experiment provided multiple student learning outcomes: gaining experience performing field sampling, while being introduced to XRF spectroscopic instrumentation and an EPA method. When the students observed the field site, they recognized the challenge of making measurements in the field. The weather conditions and constraints of the physical site gave the class a realistic situation, which they are likely to confront working in the industrial setting. Since the data set was limited to sampling grid positions, students expressed the need for a hierarchical sampling to more fully characterize the site and the identified hot spot. The students also recognized with more clement weather, that the site characterization could have been completed in one visit to the site. So although the students did not get to use the FP-XRF in the field, the utility of the portable instrument was learned at least on a conceptual level. The student assessment of the experiment demonstrated that they recognized the need for replicate sampling using this field portable instrumentation even though the sample spectra were not recorded in the field as originally planned. The students expressed on the Student Assessment of Learning Gains (SALG) survey that the lead content values were much higher than they had expected and the key educational point was that sample homogeneity was critical to obtain high quality data in the analysis of soils.<sup>[9]</sup>

The Instrumental Analysis class was introduced to XRF spectroscopy through this experiment. The basic concepts of the instrumentation were explained so the students gained a richer exposure to an instrument that utilizes a multi-channel analyzer to detect and quantify various elements. Since the instrumental details of the Compton-normalization and the Fundamental Parameters analysis are performed automatically, the students were not necessarily confident how the instrumentation obtained the resulting values.<sup>[10]</sup> The students recognized the necessity to follow the EPA protocols as they reviewed their own data. The fair improvement in the precision of the homogenized soil samples made the students more critical of their own qualitative assessment. This lack of a strong correlation gave the students insight on how critical this minimal sample preparation step is in the EPA Method 6200. The long oven drying time may have yielded a minor error in the lead content values since the EPA Method 6200 specifies a moisture content between 5% and 20%.<sup>[5]</sup> An improvement in this experiment would be a shorter drying time. The validation of data set using the NIST standards (2710 and 2711)



did give students confidence that instrumental values were indeed of high quality. Working within the constraints of the weather and limited access time to the rented instrumentation, the students also became aware of the practical challenges that workers in the environmental field face everyday.

In addition to analyzing the data and formulating conclusions, students were required to write a letter to Cal-Trans summarizing the data. To address potential risks in an appropriate context, the students were directed to evaluate their data set in comparison to USEPA Preliminary Remediation Goals (PRG's—the lead in industrial soil PRG is 750 ppm) with the recognition that this highway does not represent a residential use but might be somewhat appropriate as an industrial setting at least for highway construction workers.<sup>[11]</sup> U.S. EPA Preliminary Remediation Goals, are values used as guidelines for remediation of sites. This risk assessment was not necessarily easy for the students to complete since the class had no formal training on environmental risk assessment. The letter writing adds to the reality aspect of the lab, since the lab handout states that a few student letters will be enclosed with the report to Cal-Trans.

A number of manufacturers produce field portable XRF instruments. The instruments typically have a radioactive source to excite the samples and are routinely applied in soil analyses, confirmation of alloys, and other applications.<sup>[10,12]</sup> For other XRF vendor info provided by US EPA see Ref.<sup>[13]</sup>. Some manufacturers have also produced instruments with compact x-ray tubes, which obviously have certain advantages.<sup>[10,12]</sup> The devised experiment did not attempt to compare these different type of instruments, just introduce the instrumental analysis students to a field portable instrument available for rent. This exposure to renting instrumentation may also be a useful introduction for students entering the workforce since many firms will have to balance the costs of instrumentation and adequately training their personnel to operate appropriate instrumentation. This experiment demonstrates that appropriately trained technical personnel can become competent and productive with this type of instrumentation with relative ease.

The field sampling experiment for the instrumental analysis course was developed after performing previous field sampling experiments in an Environmental Chemistry course and reviewing appropriate documents in the literature.<sup>[1,2,4,6]</sup> The instrumental analysis students' learning was assessed using the laboratory results and written documents. Probing questions on what student perceived that they learned were asked anonymously using the SALG which is a web-based tool which can give the instructor more insight to improve the laboratory experiment.<sup>[9]</sup> Future experiments could incorporate some additional quality control steps to further enhance the learning experience. This type of experiment provides information for students on the



utility of methods used in screening sites for elements or compounds of concern.

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