

On-site teaching with XRF and XRD: training the next generation of analytical X-ray professionals

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There is a growing need for environmental scientists, geoscientists, and analysts skilled in the use of X-ray fluorescence spectrometry and X-ray diffractometry. The challenge for educators is how to inspire, teach, and make the next generation of professional X-ray users and analysts ready for employment. In this paper, we present vignettes from teaching applications of X-ray analytical techniques at three scaffolded levels, from senior high school students, undergraduate science students, to postgraduate researchers. At each of these levels the pedagogical complexity increases, from simple data use at high school, to observing how data are generated and being able to constrain analytical uncertainty at the undergraduate level, to generating high-quality data at the postgraduate level. In all cases, transportable equipment is used in on-site analytical programs to inform the experimental design, level of sampling required, and research outcomes. © 2014 International Centre for Diffraction Data. [doi:10.1017/S0885715614000876]

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I. INTRODUCTION

Educators should always seek more efficient ways of teaching subjects which, rightly or wrongly, seem to be difficult or unpopular among students. More than ever, society relies on evidence-based knowledge through improved numeracy, physics, and chemistry information. Paradoxically, our knowledge in these critical traditional disciplines appears to be falling (Wyatt and Stolper, 2013), providing a significant challenge of how to inspire and teach these subjects effectively, so that there are sufficient skilled industry and research professionals for the next generation. This is certainly the case, in our experience, in X-ray analytical methods, and more particularly, X-ray fluorescence spectrometry (XRF) and X-ray diffractometry (XRD).

Modern pedagogical theory advocates “active learning” techniques, which engage students more effectively (e.g. Day, 2012). Under this model, students “learn by doing” (Gibbs, 1988) rather than the traditional approach of preparatory lectures and reading followed by examination of examples drawn from the literature or teaching texts. A subset of this approach is inquiry-based learning, which can be question- or research-driven. Real benefits for student critical thought and extent of understanding can accrue from this approach (Miller *et al.*, 2010). There are similar benefits for the incorporation of on-site, and particularly field-based, teaching, and learning (Fuller, 2006, 2012; Day, 2012; Mogk and Goodwin, 2012).

The pedagogical approach we have adopted is one of structured teaching, targeted at three different learner audiences. The first involves presenting high school students with their own environmental data, from samples they have

collected themselves, and that we have analyzed using XRF spectrometry. These students do not see, or necessarily understand how X-rays or the analytical data are generated. The aim is simply to illustrate an environmental problem with real-world data, and we choose to use XRF for that purpose. The more capable students might retain part of an explanation as to what XRF spectrometry is, but many more students might simply become inspired by the ability to address real-world problems with analytical X-ray techniques.

The second audience consists of undergraduate science students who receive a demonstration of the use of XRF and XRD, with an introduction to the principles of environmental X-ray technology. Those students then have to use the data for assessment tasks, and so they need to understand not only a little of how the methods work, but also issues of data reliability and quality, and how to constrain these. This teaching approach is applied normally in the field at derelict mines or contaminated sites, using a transportable spectrometer and diffractometer operated out of a mobile laboratory.

The third, most advanced approach to our structured X-ray training program is applied to our postgraduate research students, who become trained and certified in the use and operation of ionizing radiation analytical equipment. This learner group generate their own data and learn to calculate and present analytical precision and accuracy, and in so doing develop into early career professionals in their own right. For this we use both transportable and laboratory-scale spectrometers and diffractometers, according to the data quality objectives of the research.

The motivation for this paper was the request from industry professionals to document some of our teaching activities, to act as inspiration for other educators and industry X-ray analysts. Already in some industry sectors, there is a shortage of skilled X-ray spectrometrists and diffractionists. Although we recognize that many other universities teach X-ray analytical

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techniques, we are reminded frequently how unusual and refreshing it is to have on-site and field-based teaching of environmental sciences and geosciences using these techniques. Therefore the aim of this paper is to document how we use XRF spectrometry and XRD in the field to train and, hopefully inspire, the next generation of industry and research professionals for the environmental, geoscientific, and analytical X-ray-related industries.

II. HIGH SCHOOL STUDENTS

A challenge with teaching senior high school students is that any efforts must contribute to the various school curricula in some way. Students would not be called on to learn about analytical methods, so demonstrating the use of equipment would have little point. Similarly, students rarely learn about mineralogy in high school so there would be little point in demonstrating XRD or presenting data collected from that technique. However, students do learn about the scientific method, and environmental investigations, so we choose to use XRF to illustrate actual or potential environmental problems.

To date we have visited high schools on four occasions, using environmental lead as the focus. Three visits have been in a large city (Sydney in this case) and the other visit has been to a mining and industrial town remote from large cities. On another occasion students came to the university and worked on campus over two half-days. In each case, we introduced the notions of urban or industrial heavy-metal pollution via a lecture in a science class, and helped the students create a defensible urban sampling design. Then under the direction of their teachers, the students collected and oven dried their soil samples, and using a 2 mm sieve discarded the >2 mm gravels as part of their school science practical. We then returned to the schools to analyze the <2 mm fraction of the soils, present the data, and discuss the findings in the context of national soil-quality guidelines.

The soils were analyzed as loose powders, using the <2 mm fraction. The data are not meant to be reportable, and so further sample preparation is not conducted in order to minimize dust production in school-science laboratories. For the analysis of soils we use a transportable energy-dispersive XRF spectrometer (EDXRF) using a 29 kg PANalytical MiniPal 4 with 9 W, 30 kV Rh tube, and a 12-position sample carousel, or using an Olympus Delta Premium handheld XRF with 4 W, with 40 kV Rh or 50 kV Ta tubes. The MiniPal 4 and Delta Premium operated in a test stand are safe for use on-site, being fully enclosed and interlocked, and because the school students are not being taught about the analytical method the spectrometer can be operated and samples can be measured remote from them. The main analyte of interest is Pb, although in some instances Cu or Zn can also have large concentrations in urban soils.

In the example of the remote metal-mining town, we have examined Broken Hill in far-western NSW. We used XRF spectrometers to generate data for high school science students, for the measurement of environmental soil-chemistry associated with industrial point-sources. The program of learning enriched the NSW “Earth and Environmental Science” curriculum and enabled students to understand more intimately their local landscape, by way of analyzing soil and environmental dust samples. The project examined changes in surface-soil metal concentrations in contemporary

samples vs. historical and background values. Although only trained university staff operated the spectrometers, the high school students were able to view and chart the data soon after collection, providing almost “real-time” information. We re-sampled locations that had been analyzed in 1893 by NSW Government scientists (Thompson, 1893), who showed there was “no soil lead” at that time. The school students dug and sampled the soil profiles under direction (Figure 1), then we analyzed the soils to see if after a century of mining and industry, there was still “no soil lead” at the soil surface. The deeper soils were believed to be natural and intact, allowing us to rely on samples at depth as a proxy for background values. The students thus had two checks on historical soil quality – the 1893 data and the soils at depth. Surface soils were found to be enhanced with lead and zinc, in contrast to deeper soils reflecting “background” values (Figure 1). Consequently, the students could conclude, on the balance of field and historical information that mineral extraction and processing had resulted in a significant impact on soil chemistry.

The objectives of our high school-related teaching exercises are to expose students to the scientific method, experimentation, and environmental science. By “personalizing” their learning experience with samples they have collected themselves, we believe that students have enhanced learning outcomes and in so doing, we hope that students have become inspired to attend university, follow careers in science in general, and environmental or geological sciences in particular.

III. UNDERGRADUATE UNIVERSITY STUDENTS

Training undergraduate science students is constrained by the students not having radiation safety training, so they are rarely able to use the equipment directly. The reason undergraduate students at Macquarie University do not receive radiation-safety training is generally one of cost but also of need, as student classes typically range from 20 to 40 in number and the courses generally have an environmental or geological focus rather than being analytical methods courses. However, the students are able to collect and prepare the samples, see how they are measured, and learn a little about the fundamentals of XRF and XRD. The teaching also allows them to be led through the important issues of quality assurance (and be provided with quality assurance/quality control data for calculation of precision and accuracy), and to consider

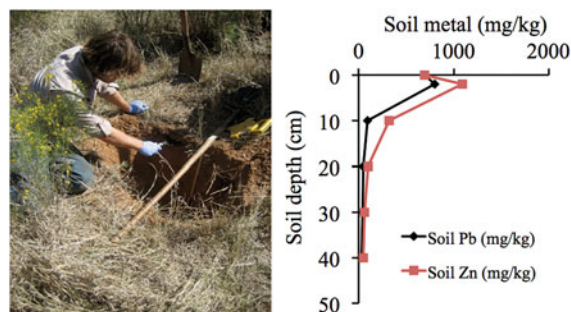


Figure 1. (Color online) Left: Soils at a remote mining town in western NSW were sampled at 0–2, 2–10, 10–20, 20–30, 30–40, and 40–50 cm, with the deepest samples assumed to be a proxy for ‘background’ values. Right: Soil lead and zinc concentrations through the undisturbed soil profile showing the shallow nature of metal contamination.

other aspects of data quality. Undergraduates can also be led through problems with high-quality standards and the creation of robust, matrix-appropriate calibrations, although it generally is not worthwhile in terms of the time taken, and alignment with the course objectives, having the students create the calibrations. In the following example, we illustrate how transportable EDXRF is used at a derelict base metal mine in northeastern NSW, Australia, to train undergraduate students in ore geology and contaminated site remediation.

Students are transported to the site in buses and then taken onto the site in four-wheel drive vehicles. There, they meet the owner and are inducted into the site, and the use of appropriate Personal Protective Equipment (PPE), in this case, boots, high-visibility clothing, long sleeves, long pants, hat for sun protection, nitrile gloves, and when preparing samples dust masks, safety glasses, and decontamination station. The mobile laboratory, consisting of an off-road camper trailer towed by a four-wheel drive vehicle, provides a robust platform for equipment transport, as well as providing a focus for student activity, rest, and a place for sample preparation (Figure 2). A 2 kW generator provides electrical power for the mill, spectrometer, and laptops, and no filtering or other “cleaning” of the power is required. A portable oven, suitably labeled “no food”, is used to dry samples in small oven-proof containers, before comminution in a small shaker mill (such as the Retsch MM301). The Retsch mill we use is ~20 kg and operated at 25 Hz for 3 min. It has twin tungsten carbide jars and balls, allowing two samples to be processed at a

time. To lighten preparation equipment, a press is not used and instead samples are analyzed with a “loose powder” calibration. In reality, the sample is lightly pressed in an X-ray sample cup with about 14 hPa (100 g over the 7 cm² of the cup) pressure to prevent dust migration into the analytical area of the spectrometer. The X-ray cup typically has a 3.6 μm Kapton[®] film, but other films would also be appropriate. For this fieldwork, the same EDXRF spectrometer (MiniPal 4) as used in the previous high school example, provides enough X-ray flux and thus element sensitivity to make measurements within 60 s (for demonstrations of sample chemistry) to 300 s (for accurate quantification of abundant elements). Again, it is also safe, being fully enclosed and interlocked. As base-metal ores are of interest for our teaching, the elements most usually quantified are V, Cr, Fe, Co, Ni, Cu, Zn, As, and Pb. More rarely, Mo, Sn, and Sb are of concern. All of these elements have penetrating enough K or L shell emissions for quantification in air, so that He gas bottles are not required.

At this field site, each student collects and prepares about two samples per day, with 40–80 samples being analyzed per day for a typical class size. This amount of information can be adequate for environmental investigations, and given that an experienced XRF operator is in charge of data quality, the results can be good enough for peer-reviewed publication (e.g. Gore *et al.*, 2007). In this example, students were able to assess the post-site rehabilitation risk posed by a former base-metal mine, through a spatial study of samples of ore, slag, tailings, waste rock, and channel and floodplain sediments



Figure 2. (Color online) The derelict Conrad base metal mine, northeastern NSW. Top left: Students learn about the functions of derelict mine structures from the site owner. Top right: Towing the portable laboratory onto the site. Bottom left: Portable laboratory being set up on the slag heap. Outside the laboratory, students are setting up tables to be used for milling samples during the day. Bottom right: At night, students use a field hut with good lighting to mill samples prior to analysis. Source of photographs: K. Fryirs.

downstream. Notions of chemostratigraphy can be examined down natural sections incised into floodplain sediments. In places, precipitates covering channel gravels could be examined by placing the whole clast into the measurement area of the spectrometer. Fragments of ore rock can also be examined in the same way. In the case of analysis of rock fragments, the data are semi-quantitative but can be good enough for environmental science students to reconstruct common primary ore minerals such as galena (PbS) and many secondary precipitate minerals downstream such as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). One common question we pose to students is to determine whether or not the white precipitates downstream of arsenic-rich base metal ores are arsenical salts, gypsum, or salts of some other composition, which leads them into deeper considerations of environmental chemistry using Pourbaix diagrams and simple measurements of pH and Eh. This can be done rapidly and on-site, allowing students to focus more on sedimentological and mineralogical materials of real concern, wherever they might be found.

When a more nuanced understanding of mineralogy is required, we also use a transportable X-ray diffractometer to quantify the mineralogy of selected materials. The smallest and lightest fully-enclosed diffractometer available is an In Situ (now Olympus) “Terra” which operates in transmission mode using 30–50 mg of sample. Minerals are crushed in a steel hammer and anvil, and are then sieved at $63 \mu\text{m}$. The diffractometer can accommodate grains up to $150 \mu\text{m}$ but for good particle-statistics we prefer a finer grain size. With sample preparation at its simplest, we take stream sediments, sieve

at $63 \mu\text{m}$ and analyze for longitudinal trends in stream sediment mineralogy. An ultrasonic vibrator in the sample holder periodically re-aligns the grains, and in so doing allows a more random measurement of grains in the sample. The data are visible and the diffractometer is controllable via Bluetooth to a browser on a portable device such as laptop or iPad, allowing the user to decide when sufficient resolution of minor peaks has been achieved and when the measurement can be terminated. The “Terra” is battery or mains powered, and so X-ray flux is weak and typical measurements take 15–30 min or longer to capture minor phases. Data are exported wirelessly to a laptop for search/match using PANalytical’s HighScore+ software with ICDD PDF2 and PAN ICSD databases. Each night at the field camp, we use a data projector to work students through the challenges of mineral identification and genesis. For almost real-time data viewing, we use OziExplorer (2013) software, which allows any photographs (but usually aerial photographs), to be georectified on site with data from hand held GPS systems (Figure 3). Certified reference materials are also measured periodically, and students are required as part of the assessment tasks to understand and constrain analytical uncertainty via the calculation of precision and accuracy.

We have used this analytical capability at derelict base-metal mines and other sedimentary deposits where we can use sediments from different catchments as tracers (e.g. Fryirs and Gore, 2013) from far-western NSW to the eastern coast, allowing students to understand a range of different environments from the arid to the temperate zone. The real

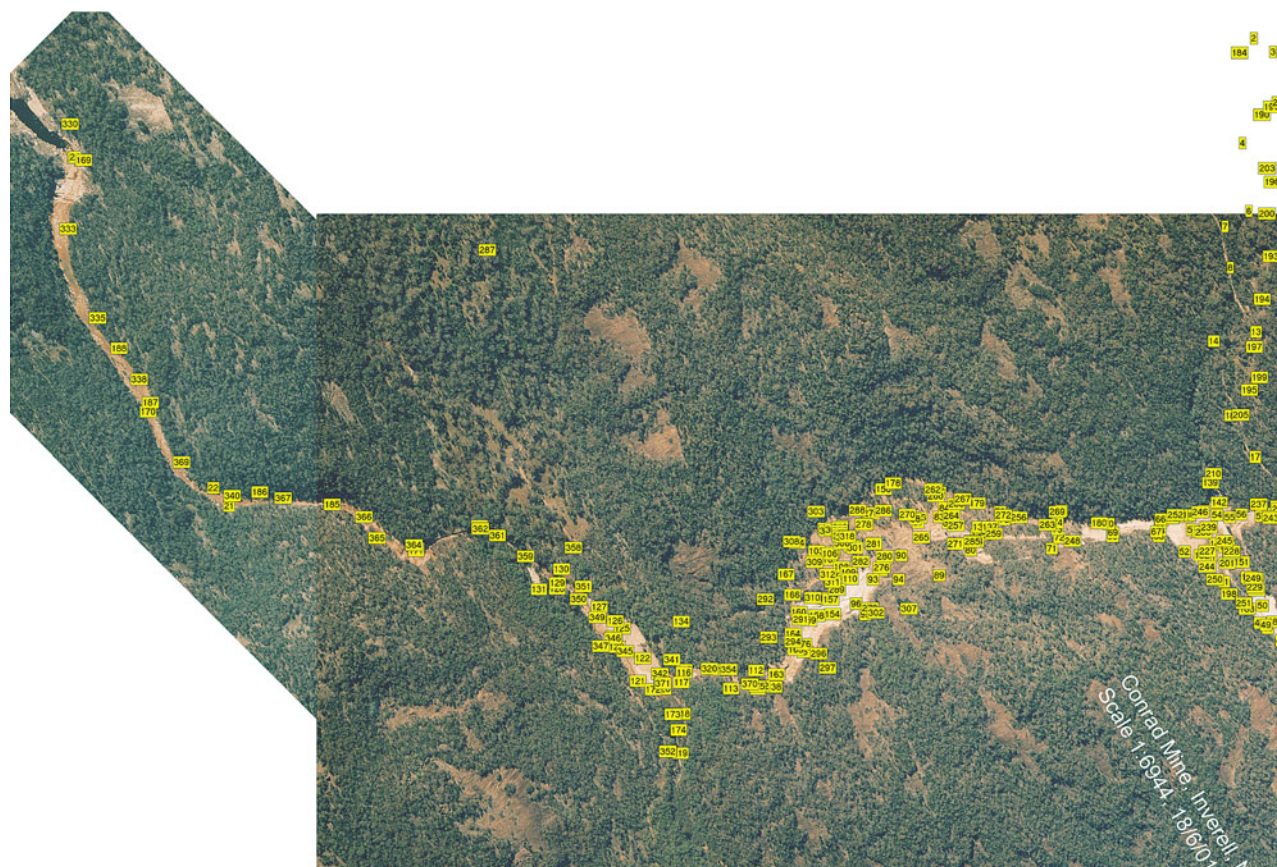


Figure 3. (Color online) OziExplorer image of the derelict Conrad base metal mine in NSW. The hundreds of small labels illustrate the sampling density undertaken by the students over the different mining landforms, and sediments downstream. Field of view is 1 km left to right.

attraction of this on-site analytical capability is: (1) the students are able to take part in the creation of the data they will use in their assignments; and (2) the students can have a dynamic work plan which targets areas of interest to the questions that are posed in their assessment tasks. For example, if the assessment question is to identify how far downstream contaminated sediments have travelled from the mine site, rapid feedback with respect to sample composition can save a lot of wasted “null” results with more effort being focused on the materials of interest. In this way, students are kept engaged, interested, and have a sense of ownership of both the project and the data. The students also get a strong sense that this is making them more “job ready”, which leads to enhanced learning outcomes.

IV. POSTGRADUATE UNIVERSITY RESEARCHERS

Training of postgraduates in the field results in a shift from observation and minimal hands-on analysis of samples to the direct use of the equipment, processing of samples, and analysis of data. The activities of research students are different from the previous two examples as their aims and results address directly their individual research projects rather than being a pedagogical exercise. Postgraduate training provides an “apprenticeship”, where the student is responsible for all aspects of the scientific process and implementation of the experiment in the field with the guidance of a supervisor. Most critically, postgraduate training on X-ray related projects requires skills in experimental design and quality assurance/quality control. Formal training and certification, understanding the use of the instruments, workplace health and safety, radiation, and PPE is also required by the student to ensure that they are responsible for the experiment and the safety of those working.

Postgraduate environmental scientists often have the requisite skills to undertake research in a range of different environments that require multiple investigative techniques. They often have significant track records of working in specific areas of interest. However, the transferability of these skills to new projects in new environments is a significant component of professional development. This transition often requires that new technology be used and new ways of analyzing data pursued. This is particularly the case for the example given here.

Here we describe the methods used during a contaminated site assessment at the abandoned Wilkes Station in East Antarctica (including the Wilkes landfill) where a postgraduate environmental scientist was responsible for undertaking the assessment. This student was responsible for all aspects of the project including learning how to use XRF, undertaking the experimental and sampling strategy, fieldwork preparation before leaving Australia, fieldwork organization in Antarctica, fieldwork execution (including the difficulties of sample collection and equipment transportation), data analysis, and publication. This example project spanned 4 years, highlighting the often-longitudinal nature of environmental science in remote locations.

For this fieldwork, the same instrument described in Sections II and III (transportable EDXRF MiniPal 4, with Rh tube) was used. This instrument was transported to Antarctica and then to Wilkes Station in a reinforced polycarbonate case with a total weight of 45 kg. This was initially transported by aircraft to Wilkins Aerodrome, then by

Häggglunds oversnow vehicle to Casey Station, then by Inflatable Rubber Boat (IRB) to the shore near the Wilkes Station area, and finally by sled to the hut (Wilkes Hilton) which became the field-based laboratory (Figure 4).

In field-based projects, a balance needs to be reached between the number of samples collected, the spatial and temporal distribution of sampling, the time taken to collect and prepare samples, and the reliability, replicability, and rigor of the data required to test hypotheses of scientific significance. The ultimate aim is for postgraduate students to produce data that can withstand the peer-review required for international publication. In remote environments, these considerations are particularly important (and often challenging) where sampling is often time-limited. In this case study a targeted sampling strategy was employed whereby sediment samples were collected from known areas of waste disposal or storage covering a spatial area of about 1.5 km² (including background sampling). The study area was traversed on skis, with equipment loaded onto a sled that was person-hauled with all sampling gear, samples and survival kits. PPE is particularly challenging in a contaminated polar environment, where special issue thermal clothing is required as well as gloves and safety glasses. Tyvek oversuits, face masks (to guard particularly against asbestos), and nitrile gloves were used at each sampling location. Samples were collected with a trowel and double bagged for safe return to the hut on the sled. Sample weight was required to be kept to a minimum.

Each evening a small amount of each sample was placed in an X-ray cup with a 3.6 μm Kapton film. The MiniPal 4 was run on a small generator that was transported to Wilkes with the field gear. This limited the sample run time to 90 s per sample. The hut had no drying oven for contaminated samples, so the samples were at field moisture condition when analyzed. The possible presence of asbestos precluded milling the samples in this field location. Given that no data existed on the contamination at Wilkes Station, analysis of samples in the hut was undertaken as a baseline study and to direct the next day’s sampling. Samples were then stored in plastic bins and buried in snow outside the hut. At the end of fieldwork, all equipment, samples, and waste were transported back to Casey Station, and the samples refrigerated at 4 °C before being returned to Australia for further analysis.

To undertake a more comprehensive contaminated site assessment, reanalysis of the samples in the laboratory was undertaken (Fryirs *et al.*, 2014). At Macquarie University, each sample was oven dried for 24 h at 105 °C and sieved to <2 mm to remove the gravel fraction. The fraction <2 mm is most appropriate for metals analysis as it gives a realistic estimate of metals in the whole sediment and not just in a concentrated fine fraction such as the <63 μm fraction. Twenty grams of the <2 mm fraction was milled for 90 s in a Retsch MM301 mill with tungsten carbide jars and balls. Between each sample the mill was cleaned with acid-washed quartz sand and ethanol. Nine grams of powdered sample was well mixed with 1 g of Licowax (C₃₈H₇₆O₂N₂) binder. This sample was pressed at 60 kN for 45 s using a Herzog press.

Samples were analyzed in a vacuum using a PANalytical Epsilon 5 cartesian geometry EDXRF, with a dual-anode W/Sc tube, and six measurement conditions. Measurements were made in triplicate and averaged for each site. For elemental quantification we used “Auto Quantify”, PANalytical’s automated qualitative spectrum-analysis combined with a



Figure 4. (Color online) Contaminated site assessment at Wilkes Station, Antarctica. Top left: Transporting the XRF to Wilkes via IRB before being carried by hand over rocks to the snow. Top right: Transporting equipment by hand-drawn sled to the field hut. Lower left: Sampling waste among rusty steel fuel drums that are over 50 years old. Lower right: On-site laboratory at a field hut. MiniPal 4 spectrometer is visible at lower right. Source of photographs: K. Fryirs.

fundamental parameters matrix-correction model. All data are reported as elements, and the data were normalized to 100%. The accuracy of the XRF calibrations was assessed using four U.S. Geological Survey certified reference materials.

The real attraction of this on-site analytical capability is that the equipment can be transported into a remote environment relatively easily and can provide on-the-spot results that can inform the science, the experimental design, and the level of sampling required. In a remote locations such as Antarctica, it is impossible to just ‘duck back to the site’ to take more samples! However, the project is more than just about postgraduate research; first and foremost it is postgraduate training. The on-site analytical environmental programs using XRF and XRD are highly successful in training postgraduate researchers.

V. DISCUSSION AND CONCLUSION

The aim of this paper was to present teaching vignettes of how Macquarie University scientists have developed on-site environmental analytical programs using transportable EDXRF spectrometers and X-ray diffractometers to help train the next generation of industry and research professionals. An important aspect of our educational approach is to avoid didactic teaching, and instead engage students to “learn by doing” through incorporation of their own samples, collected from their own fieldwork, into analytical programs designed to illuminate environmental problems and solutions. The examples we have used in our teaching include derelict

mines, a landfill, industrial areas, and urban environments. We have deliberately sampled Australian environments from the arid zone to the temperate coast, so that students (and teaching staff) become experienced and remain fresh and engaged with environmental problems and possible solutions.

We have used XRF on-site since the year 2000 and XRD on-site since 2011. Our analytical capability has evolved since 2000, and from a humble beginning with a single PANalytical MiniPal 2, we now use a PANalytical MiniPal 4 EDXRF, PANalytical Epsilon 3 EDXRF, and three Olympus Delta handheld EDXRF, each operable in interlocked test stands for students or in hand-held mode for legally trained users. An Olympus “Terra” diffractometer allows on site mineralogical determinations.

The total value of this transportable laboratory and contained equipment is of the order of \$450,000 AUD for the mobile laboratory, sample preparation, and all of the analytical equipment mentioned. Given that the same equipment can be used for other research projects, or for use in the classroom when not being used in the field, this arrangement constitutes a powerful yet flexible analytical solution for teaching students from senior high school to training postgraduate-level researchers.

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