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## Particle induced X-ray emission of lake sediment

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Three recent ion beam analysis research projects are described that have been undertaken by the Hope College Nuclear Group. Success in the routine ion beam analysis of lake sediment as well as the elemental analysis of lake sediment for environmental fingerprinting is reported. In addition, a novel ion beam analysis detection method for organohalogen flame retardants in polyurethane foam is proposed, and in each of these research projects significant student training has occurred. Building on the success of prior research published in both nuclear physics and applied physics, these projects involve undergraduate students in every aspect of the sample collection, data acquisition and data analysis. The combination of publishable scientific inquiry and undergraduate training using ion beam analysis is suggested as a model that could be done wherever there are appropriate accelerator facilities.

*Keywords:* PIXE; sediment analysis; radiometric.

Se describen tres proyectos de investigación relacionados con análisis de materiales con haces de iones realizados por el grupo nuclear del “Hope College”. Se reporta el éxito en el análisis de sedimentos lacustres así como del análisis elemental de esos sedimentos como huella digital ambiental. Adicionalmente proponemos un método novedoso de análisis con haces de iones de compuestos organohalógenos retardadores de llamas en espuma de poliuretano. Cada uno de estos tres proyectos de investigación ha involucrado un intenso entrenamiento de estudiantes de nivel licenciatura en lo que toca a: colección de muestras, adquisición y análisis de datos. La combinación de una investigación científica publicable y el entrenamiento de estudiantes utilizando análisis con haces de iones, se sugiere como un modelo que puede llevarse a cabo en cualquier sitio que cuente con un laboratorio con un acelerador de partículas.

*Descriptores:* PIXE; análisis de sedimentos; radiometría.

PACS: 29.30.Kv; 93.85.Np

### 1. Introduction: The hope college ion beam analysis laboratory

For the past 18 years the Hope College Nuclear Group has been engaged in undergraduate research – focused on publishing research and training undergraduate students in experimental nuclear science. Hope College is unique in its combination of research and education at the undergraduate level in nuclear science, as demonstrated by the more than 100 peer-reviewed publications that include over 150 student co-authors over the last 18 years. Over 90% of the students who have joined the Nuclear Group have continued on to graduate programs and the two principle investigators of the Hope College Nuclear Group are Paul DeYoung (Physics) and Graham Peaslee (Chemistry & Geological and Environmental Sciences).

While much of our work involves radioactive nuclear beam studies conducted at nearby experimental facilities at Michigan State University and Notre Dame University, a key component to the successful undergraduate research program has been the installation and operation of a 1.7 MV tandem pelletron accelerator at Hope College in 2004. Funded by the National Science Foundation, this ion beam analysis facility has been instrumental in attracting students into the Nuclear Group and providing local research projects that have involved dozens of research students each year. Not only do the ion beam analysis experiments provide an excellent experimental opportunity for the students, but it has been pos-

sible to find new research areas in applied nuclear science where fundamental research methods and results can be published. This presentation will focus just on three recent areas where ion beam analyses have resulted in novel research projects that will lead to future scientific publications, and where they already have yielded valuable training opportunities for future scientists.

The Hope College Ion Beam Analysis Laboratory consists of an NEC 5SDH tandem pelletron, plus three beamlines – one of which houses an NEC nuclear microprobe. It is supplied with light ions (H and He) from an NEC Alphatross source, with an RF ionization bottle and a Rb-vapor ion exchange chamber that yields  $H^-$  and  $He^-$  ions. The central column is pressurized with  $SF_6$  and can reach 1.7 MV in routine stable operation. Nitrogen is bled into the central cavity as a stripper gas, and  $H^+$ ,  $He^+$  or  $He^{2+}$  is accelerated on the high-energy end of the tandem. A water-cooled analyzing magnet can put ion beams on target in one of three chambers currently, and on the nuclear microprobe line, a set of quadrupole quadruplets can focus a collimated beam of ions to a beam spot size of less than 50 microns easily, and to a spot size of less than 20 microns with effort. The scattering chambers are equipped with Si(Li) x-ray detectors at backward angles, fixed and rotating surface barrier detectors for particle scattering measurements, and in the nuclear microprobe beamline a 5-axis goniometer that can move targets reproducibly to within a micron or two. Pictures of the facility are shown in Fig. 1.



FIGURE 1. The Hope College Ion Beam Analysis Laboratory (tandem pelletron and alphasource ion source on above, control and data acquisition stations on below).

## 2. Results

### 2.1. Lake Sediment Characterization by PIXE

The (Particle Induced X-ray Emission) PIXE analysis of aerosols has been a well-established application of ion beam analysis for decades, but a second area where PIXE can be very useful is in the quantitative analysis of sediments and soils. We have already established a method for routine PIXE analysis of dried sediment samples [1], where they are simply dried, homogenized mechanically and pressed into a self-supporting solid target with a hydraulic pellet press. Using this technique we have measured anthropogenic metal concentrations in Lake Michigan sediment cores as a function of

sediment depth, where we use  $^{210}\text{Pb}$  and  $^{137}\text{Cs}$  radiodating analyses to correlate the sediment depth to year of deposition (shown in Fig. 2). The measured metal concentrations from a core taken from the 65 m depth contour in Lake Michigan have been analyzed with thick-target GUPIX [2] and are shown to have a characteristic increase around the time that industrialization increased around Lake Michigan at the turn of the nineteenth century (see Fig. 2). Important in these data is the observation that the introduction of the US EPA Clean Air Act in 1972 had a significant effect on the introduction of anthropogenic metals into Lake Michigan sediment. These findings support the assertion that most of the anthropogenic metal contributions in deep Lake Michigan sediment arise from continuous aerosol deposition on the cold lake surface.

### 2.2. Environmental forensic analysis of sediments

In addition to routine analysis of total metal content in historical sediments, it is possible to analyze recent sediment for elemental composition. Since the mid-western US suffers from extensive non-point-source pollution of its lakes and streams from sediment and attached nutrients, we have adopted a low-cost approach to monitoring sediment and we are developing methods for fingerprinting the origin of these sediments. The sediment collection method was adapted from the literature [3] and consists of PVC tubing suspended above the low-water mark in small streams, with small inlet and outlet apertures. The sediment collection tubes are shown in Fig. 3, together with the standard method to harvest the sediment after each rain “event”. As the stream level rises during a rain event, suspended sediment enters the sampling device, and because of the relative diameters of the stainless-steel inlet tube to the PVC tubes, suspended sediment will settle in the PVC tubing with a known efficiency. After the rain event, these sediments can be harvested quantitatively from the tubes as shown in Fig. 3.

The collected sediment from these low-cost samplers can be dried and weighed to give a measure of the absolute sediment load in the stream during the rain event, and we have begun an ambitious monitoring program in the Lake Macatawa watershed with 129 separate sediment sampling tubes in 2011. Not only will the sediment load estimates be useful to help pinpoint the sources of sedimentation within the watershed, and to ground-truth various hydrological models of the watershed, but the collected dried sediment will be saved and analyzed for 10 common elements by PIXE analysis. Approximately 100-200 mg of homogenized dried sediment from each of the events collected in each tube is pressed into a self-supporting target and measured with 3.4 MeV protons with an integrated beam current under  $\sim 1.5 \mu\text{C}$ . A thick-target GUPIX analysis yields. These quantitative elemental analyses for P, K, Ca, Ti, Cr, Mn, Fe, Cu, Zn, Sr, and Zr yield a potential method to identify geological point sources for the sediment within the watershed. While soil mineralogy is more complex than just the relative yield of elements, the elemental analysis of sediment in Lake Macatawa has provided

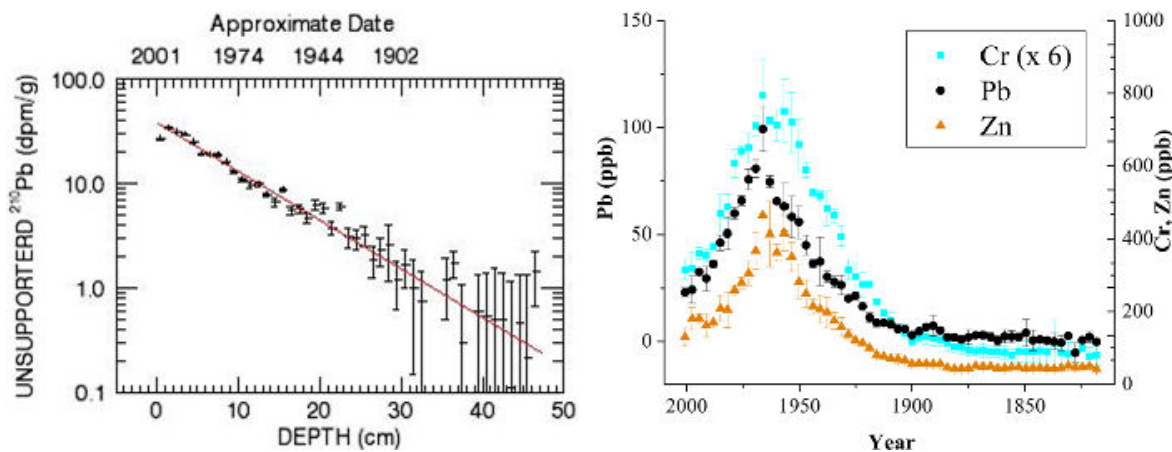


FIGURE 2. Radiometric (left) and elemental (right) analyses of sediment core slices from the 65m depth contour of Lake Michigan. The dates derived from the <sup>210</sup>Pb analysis for each core slice are used as the x-axis in the elemental analysis.



FIGURE 3. Sediment sampling tubes (left) and students harvesting collected sediment (middle) and transferring the sediment quantitatively to a collection bucket (right).

11 common elemental signatures that can be used as part of a larger analysis to fingerprint the origins of the sediment. By combining the elemental analyses with microscopic size and shape analysis of the sediment, reflected light color analysis, radioisotopic analysis and trace biological analysis of pollen and diatoms, it may be possible to uniquely define sources of collected sediment to very precise locations. This environmental forensic geology method is still under development, but shows a lot of potential promise.

**2.3. Detection of Flame Retardants in Foam by PIXE**

A third area where ion beam analysis can have a very practical application is in the elemental analysis of polyurethane foams that have been treated with flame retardants. Because the state of California has established very strict flame-retardant standards for home furnishings under Technical Bulletin 117 of the California Bureau of Home Furnishings, most furniture manufactured for sale in the US has added chemical flame retardant to the polyurethane foams and fabric coverings of office and home furniture. These flame retardants include multiple forms of polybrominated diphenyl ethers (PBDE's) and a more recent substitute for the brominated compounds: tris(chloropropyl/ethyl)phosphate (TCPP/TCEP). These flame retardants are known bioaccumulators in mammalian systems and persist in the environ-

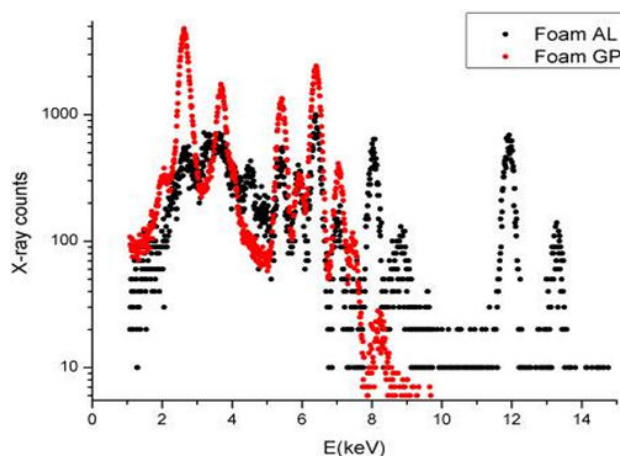


FIGURE 4. Representative PIXE spectra of two foam samples extracted from different couches, one treated with chlorinated compounds such as TCEP (red), and one treated with brominated compounds such as PBDE (black). The x-ray peaks at 2.6 keV (Cl) and 11.9 keV (Br) are unambiguous indicators of the presence of the halogens at percent levels within the foam.

ment as well. They have been linked to developmental disorders and cancer, and are prevalent in many commercial products [4]. One characteristic of these organohalogen flame retardants is that they contain either chlorine, bromine or both at weight-percent levels in the foam products to which they

are applied. Although they can be identified unambiguously by gas chromatograph-mass spectrometric methods in an elution of foam samples with organic solvents, ion beam analysis methods are much faster to identify the presence or absence of these compounds in foam samples. Several hundred milligrams of foam samples are cryogenically frozen in liquid nitrogen and then mechanically ground into a fine powder while frozen. These powders are then applied to a double-sided carbon tape and adhered to a standard PIXE target ladder. Semi-quantitative measurements of thick-target PIXE samples are then performed, with typically 1 or 2 nA of 3.4 MeV protons for less than 120 seconds on target. This rapid analysis allows positive and unambiguous identification of both halogens in the large carbonaceous samples, as shown in Fig. 4. Because of the simplicity of sample preparation and data analysis, over 100 samples can be analyzed in a day, while precise GC/MS analysis would require over a two weeks for the sample set of samples.

### 3. Conclusions

In this short overview we hope to demonstrate that there are many environmental applications of PIXE beyond the standard aerosol analyses that are done routinely. The use of ion beam analysis techniques on both historical lake sediment and relatively modern sediment collected as part of an environmental forensic science project are the first two areas

where we have demonstrated a recent practical application. PIXE analysis, which has been so good for aerosols, can also be applied to soils, minerals, and even chemical contaminants as shown in the flame retardant studies for many of the same reasons. It is a sensitive, non-destructive technique that involves relatively simple sample preparation and data analysis. In addition, the idea of teaching undergraduates by “doing” research – actively engaging undergraduates in the development and use of ion beam techniques – is a powerful teaching tool.

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