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# An Undergraduate Experiment for the Measurement of Trace Metals in Core Sediments by ICP-AES and GFAAS

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## Objectives

The objective of this experiment was to utilize the techniques of inductively coupled plasma atomic emission spectroscopy (ICP-AES) and graphite furnace atomic absorption spectroscopy (GFAAS) to analyze five metals (lead, manganese, iron, copper, and cadmium) contained in sediment core sections. Microwave extraction was used to digest the sediments and extract the target metals. To validate the techniques incorporated, sediment samples were spiked with a known concentration of the metal and recovery was determined. Replicates and blanks were also included for quality control. Particular emphasis was placed on the interpretation of the results obtained relative to the sediment depth, and hence the age of sediment deposition (1). Certain anthropogenic activities can be correlated to the stratigraphic shifts in metal concentrations (2, 3).

## Methods

**Sampling.** A core was obtained from the central position within Crawford Lake, Ontario, using a cold finger corer (4). Crawford Lake is meromictic with varved sediment easily dated by counting individual varves back for ca. 700 years. The results from this lake were compared with those obtained from Small Lake, a pristine lake located in the Canadian High Arctic. The core samples were kept frozen until analysis could be carried out. The thawed Crawford Lake core was subsectioned according to year, on the basis of lamina contained within the vertical profile; Small Lake was sectioned by depth into 1-cm subsamples, as no laminae were visible. In total, 47 subsamples were randomly assigned to 8 groups of students.

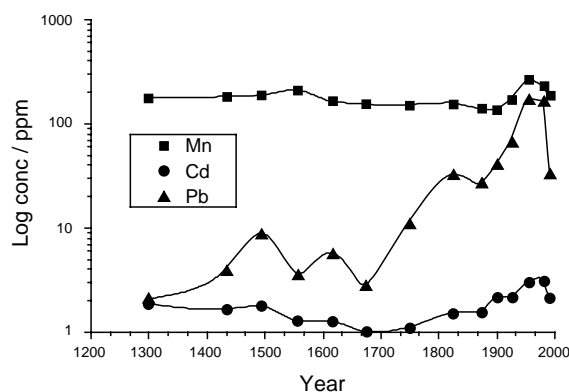


Figure 1. Variation in the concentrations of Pb, Cd, and Mn in Crawford Lake sediment over a 700-year period.

**Extraction.** Prior to extraction all glassware was acid-soaked for 3 days (10%  $\text{HNO}_3$ ) and all acids used in this experiment were UHP grade. The samples were ground and taken up in 5 mL of a 4:1 solution of concentrated nitric/hydrochloric acids. Microwave digestion (5) was carried out using an MDS-2000 extraction system. The samples were then diluted to 25 mL using 2% nitric acid.

**Analysis.** Analysis of the samples was carried out using a PE Optima 3000 DV ICP-AES equipped with a PE AS-90 autosampler for determination of Pb, Mn, Fe, Cu, and Cd (6); Pb was additionally determined by GFAAS using a PE EPL System 2 equipped with a PE AS-72 autosampler, a PE AA analyst 100, and a PE HGA 800.

**QA/QC.** Spike and recovery experiments were carried out for Pb, Cd, and Cu, through the addition of a known amount of each of the metals to six separate sediment samples; replicate analyses was carried out on seven of the samples. Six reagent blanks were carried through the extraction and analysis to determine contamination and metal carry-over.

## Hazards

Proper attention and caution should be exercised when handling concentrated acids.

## Results and Discussion

The stratigraphic concentrations of lead, cadmium, and manganese in the Crawford Lake core sample are shown in Figure 1. Recoveries for lead, cadmium, and copper were 84% (RSD = 6.1%), 84.2% (RSD = 6.5%), and 73.5% (RSD = 7.5%), respectively ( $n = 6$ ). Replicate measurement for all five metals showed a variance not greater than 8% ( $n = 7$ ).

As can be seen from Figure 1 the concentrations of all three metals remain relatively constant until the late 19th century, when lead, manganese, and cadmium increase significantly. The concentrations of these metals then undergo a marked reduction toward the end of the 20th century, indicating a possible change in anthropogenic input; these data are similar to recent literature reports (1). Data from Small Lake, in the Arctic, showed sediment concentrations of all metals largely invariant, with no defined temporal trends observed.

An evaluation of the methods for the analysis of Pb by GFAAS and by ICP-AES was carried out to show the linear working range of both instruments. Students were required to assess the quality of the data produced from both instruments with respect to linear range, limits of detection, and quantitation. An assessment of rapid multi-element analysis by ICP relative to GFAAS was clearly demonstrated. This experiment provides students the opportunity for hands-on

experience with the most advanced methods for metals analysis, with samples that are of current scientific interest. The very large data set produced necessitates familiarity with spreadsheet programs and encourages the development of data presentation skills.

### Acknowledgments

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### <sup>W</sup>Supplemental Material

The instructions for students are available in this issue of *JCE Online*.

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