ENVIRONMETAL ANALYSIS AT THE CENTRE FOR ENVIRONMENTAL AND INFORMATION TECHNOLOGY

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INTRODUCTION

A state of the art trace metal clean room facility containing 4 variations of mass spectrometer is being constructed in the Centre for Environmental and Information Technology. The mass spectrometer is the most widely accepted detector of major- and trace-elements for environmental analysis. We will be using mass spectrometry for geochemical and isotope analyses of groundwater collected from metal-contaminated mining, power generation and industrial sites, and also from field and laboratory investigations of groundwater remediation.

Methods based on mass spectrometry are among the most powerful, sensitive, and reliable available for water science research. Inductively coupled plasma mass spectrometry (ICP-MS) is an analytical technique which requires the sample (usually liquid) to be introduced to a high temperature plasma (a gas consisting of ions, generated by radio frequency magnetic fields), commonly argon, which dissociates molecules and ionizes atoms. The ions are passed into vacuum where a lens focuses the ion beam into a mass spectrometer. Here, the ions are sorted by mass and detected.

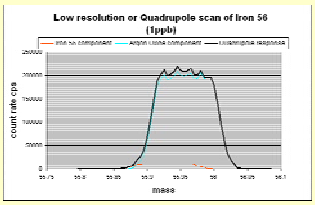
Left: Permeable reactive barrier installation for treatment of Cr(VI)- & TCE-contaminated groundwater.

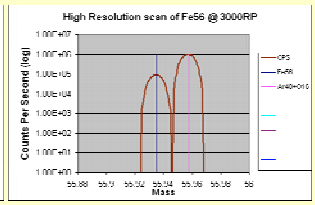
Right: Surface discharge of acidic metal-bearing water at a mine tailings site.

INSTRUMENTATION

Three variations of mass spectrometer, with several sample introduction interfaces will be used in this facility. **Quadrupole** mass spectrometers (Q-ICP-MS) have excellent stability, sensitivity, and resolution, and are fast and efficient to use. Q-ICP-MS instruments are routinely used in in commercial and research facilities for analyzing high volumes of samples at ppm and ppb levels of detection. A Q-ICP-MS instrument will be our choice for routine analysis of our water samples.

The evaluation of environmental samples and isotopes often requires higher levels of sensitivity (ppt, ppq) than Q-ICP-MS provides. **Magnetic sector** instruments are more expensive and are slower to operate than Q-ICP-MS instruments, but they offer greater sensitivity and mass resolution and provide rapid and sensitive multielement and isotope analysis of environmental materials. Many samples that are difficult to analyze with a Q-ICP-MS because of interferences can be resolved with HR-ICP-MS. Magnetic sector instruments (also called **high resolution**, HR-ICP-MS) are available as **single collector** or **multicollector**. Simultaneous measurement of multiple isotopes provides better precision in isotope ratio measurement, in comparison to single isotope collection. Because plasmas are relatively unstable, simultaneous measurement of the isotopes reduces the noise in the measurement.





The Thermo-Finnigan ELEMENT 2 is the only high resolution single collector (HR)-ICP-MS currently manufactured. The Element 2 is a powerful tool for analysis of trace element concentrations and measurement of isotopic composition in geological and environmental samples.

Element concentrations down to ppt and ppq level can be measured. The determination of elements such as Fe, V, As, and Se has been problematic with conventional quadrupole ICP-MS because of the limited mass spectral resolution of that instrument. However, the high resolution capability of a HR-ICP-MS allows most of these elements to be distinguished from the interfering masses.

Element 2 High Resolution ICP-MS

A high resolution ICP-MS that uses multicollector technology (HR-MC-ICP-MS) will be selected from one of several manufacturers. Such an instrument will offer

- precise and accurate isotope ratios of most of the periodic table
- multicollection with high mass resolution

These features will further enhance our ability to investigate the processes that control the distribution of dissolved metals in the environment.

CLEAN ROOM FACILITY

Mass spectrometers are frequently used to detect metal concentrations at parts per trillion range. Because of this high sensitivity, it is critical that the sample, standard and blank preparation areas are free of contamination. Low blank levels are critical for the measurements of ultra-trace concentrations and isotopic composition. To maintain the blanks at the lowest levels possible, standard and sample preparations will be accomplished in a positively pressured Class 100 clean lab with HEPA filtered air. The 154 m^2 (1650 ft^2) clean room is equipped with metal-free (PVC and polypropylene), fully exhausted MicroZone vertical laminar flow (VLF) fumehoods, drying areas, a Millipore water filtration system, and analytical balances.

Where unfiltered building air contains in excess of 1,000,000 particles per cubic meter, the fumehoods will offer workspace that has <100 particles/m^3, and the general space will contain <10,000 particles/m^3.

Metal-free VLF fume hoods.

The clean room is divided into several working spaces each with different levels of air purity. The quadrupole ICP-MS and LC-MS will be operated in the traffic area of the Class 10,000 room. The two HR-ICP-MS will be operated in the low traffic area of the Class 10,000 room. Sample/standard preparation will be conducted inside the laminar flow fumehoods in the Class 100 room.

INSTRUMENT VARIATIONS

There are several interfaces for sample introduction into the ICP-MS. Solution nebulization is the standard practice. In our facility we will also use laser ablation (LA) and liquid chromatography (LC) interfaces.

LA-ICP-MS: Earth scientists generally deal with solid samples that are heterogeneous on a variety of scales. Hence microbeam techniques are popular for investigating compositional variations on small scales. However, the analysis of trace elements on such scales presents challenges that, until the development of LA-ICP-MS, required the use of expensive ion-probes or similar tools. With LA-ICP-MS we now have a technique that can give precise analyses on the scale of a few tens of microns, not quite the resolution offered by electron and ion probes, but an excellent compromise considering the ease of operation and cost. The EIT clean room will have a laser ablation system such as the New Wave UP 213, which is modular and can be connected to the quadrupole and HR-ICP-MS instruments.

In LA-ICP-MS, a laser is directed to the surface of a solid sample and the resultant ablated material is swept up into the plasma of the ICP-MS.

LA-ICP-MS has been widely used as a powerful analytical technique for solid micro sampling analyses in geological, biological, environmental, nuclear, and metallurgical applications. It is a rapid and moderate cost technique for analytical chemists. The technique combines the advantages of the high sensitivity multi-element capability by ICP-MS and in situ micro solid sampling by LA. It is particularly attractive to scientists who want to study dissolution-resistant solid materials or study spatial distributions of trace elements and isotopic composition in a micro scale area on sample surfaces.

View of the sample chamber of a laser ablation probe.

LC-MS: The toxicity and metabolism of many components in water often depends on the form of those species. In order to measure the concentration of different species, typically organometallics or different valence states of a given element (e.g. As^{3+} or As^{5+}), the species must first be separated. LC-MS is able to separate a target component from a mixture and to detect it sensitively. The separation is conducted in a liquid chromatography (LC) column. Positive identification of compounds can then be provided with mass spectrometry. LC-MS is especially useful for research directed toward understanding the fate of environmental contaminants found in water and soils. Some of the compounds that can be analyzed by environmental mass spectrometry include:

- organometallic compounds
- pesticides and their transformation products
- gasoline additives
- pharmaceutical compounds, including antibiotics and other drugs
- endocrine disrupting compounds, including hormones, surfactants, and plasticizers
- biomarkers, or compounds characteristic of certain microorganisms

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