GLASS EXPANSION NEWSLETTER

Quality By Design

June 2005

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APPLICATION SPOTLIGHT

Wastewater Analysis by ICP-MS

INTRODUCTION

In the last issue, this column was devoted to the analysis of wastewater by ICP-AES. This issue will focus on the ICP-MS analysis of the same. Until a few years ago, ICP-MS was not a USEPA approved technique for wastewater. There are now several EPA protocols that describe the use of this technique for wastewater. The Office of Solid Waste (OSW) has issued methods 6020 and 6020a for this application. The Environmental Monitoring and Support Laboratory (EMSL) modified its 200.8 drinking water method to include wastewater. Even the litigious Contract Laboratory Program (CLP) has approved ICP-MS as a valid method for wastewater in its statement of work (SOW), the most recent of which is ILM05.3. The next revision, ILM06.X, which is currently in the proposal stage, includes the use of ICP-MS for soils analysis as well. The use of ICP-MS complements existing technology by enabling lower levels of detection (by a factor of ten in many cases) and the ability to determine isotope ratios.

SAMPLES

Due to the nature of the samples, wide variations in matrix are found. Although the typical high concentration elements found are Na, K, Ca, Mg, Fe, and sometimes Al, almost any element may be found at high concentration due to specific process pollution. Organic matter is destroyed in the acid digestion step and any particulates are removed by filtration prior to analysis. Since ICP-MS is less tolerant of dissolved solids than ICP-AES, a dilution may be called for to ensure that the total dissolved solids (TDS) concentration does not exceed 0.3%.

ICP-MS INSTRUMENTATION

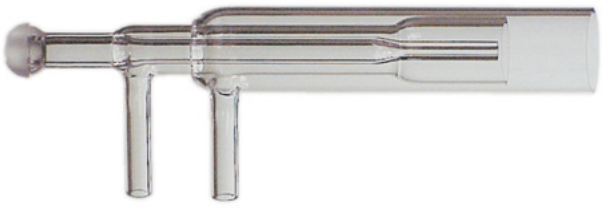
Two categories of ICP-MS instrument are typically used for this application; high resolution magnetic sector and quadrupole detector systems. Although the high resolution systems are significantly more expensive, they offer greater freedom from isobaric interferences than the quadrupole systems. For wastewater samples, polyatomic species consisting of Ar, Na, and/or Cl are particularly troublesome. Analytes of environmental concern that suffer from these include Cu, As, Se, V, Ni, and Fe.

Although the use of "cold plasma" conditions initially showed promise for quadrupole systems, interferences were still severe in many cases. Today, several quadrupole manufacturers offer reaction or collision cell accessories to destroy the polyatomic species before they reach the detector. It should be noted, however, that these cells dilute the analytes somewhat, resulting in lower signals. Quadrupole systems without cells rely on isobaric correction factors to compensate for many of the interferences.

SAMPLE INTRODUCTION SYSTEM

Torch & Adaptor

A standard quartz injector is adequate for this application since HF is not normally added to samples and silicon is not an analyte of environmental concern. It may be preferable to choose a wider bore injector, such as 2.0 to 2.4mm to reduce the likelihood of clogging with matrix salts. Likewise, a single-piece quartz torch is the most economical torch for environmental analysis.



Single-Piece Quartz Torch with 2mm Bore Injector

Nebulizer

The 0.4ml/min MicroMist concentric glass nebulizer is the nebulizer of choice for this application. This design has a high ratio of gas flow to liquid flow resulting in a finer aerosol mist than higher uptake nebulizers. This in turn produces a more uniform droplet size, lower oxides, and better ion production. Although, this nebulizer has an aspiration rate five times less than a standard flow (2ml/min) nebulizer, the amount of sample delivered to the plasma is reduced only by 20% or so. Due to the design of

the nebulizer which has a machined internal capillary and uniform sample channel, the nebulizer is capable of handling high matrix environmental samples without clogging.

MicroMist Nebulizer Designed for 0.4ml/min Uptake

Spray Chamber

We recommend the glass Twinnabar spray chamber for this application. The Twinnabar has a small internal volume to accommodate the low-flow MicroMist nebulizer. The combination of smaller internal volume and reduced internal surface area reduces the required washout times between samples. The Twinnabar also has an internal baffle which acts as a cutoff filter, reducing the mean droplet size by approximately 20%. We also recommend the Helix o'ring-free nebulizer fitting for two reasons, both of which are related to productivity increases. First, the Helix obviates the need to change worn o'rings, a process which is both onerous and time consuming. Second, the Helix eliminates the build-up of solution around the o'ring, saving precious seconds in rinse times. For added stability, the spray chamber can be thermally controlled via a recirculation bath. For this approach, a jacketed spray chamber is available.

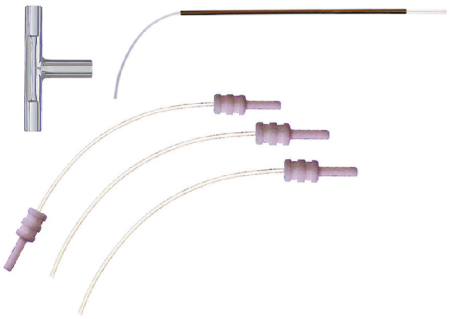
Jacketed Twinnabar Spray Chamber with Helix Fitting

Internal Standard

The use of an internal standard for ICP-MS analyses compensates for more than one potential inaccuracy. First, as in ICP-AES, it can compensate for physical matrix effects such as variations in viscosity and dissolved solids content. In addition, internal standardization is used to compensate for mass-space effects. In essence, ions of varying mass will be affected differently by the presence of matrix ions. To compensate for this, typically a range of internal standards that covers the mass range of interest is employed.

The manual addition of internal standards to each sample, standard, and blank is not only time consuming and laborious, it is also fraught with potential dangers. The presence of an air bubble in the pipette tip can contribute a significant error to the final concentration of the internal standards. Furthermore, the handling of samples and the act of adding another reagent to them is always a potential source of contamination that should be avoided if possible.

We therefore recommend the use of an in-line addition kit such as Glass Expansion's Internal Standard Kit (P/N 60-808-1185). This kit is completely modular so that any component can be replaced as needed. The availability of Flared-End pump tubing supplements the modularity of this kit. The heart of the kit is the glass tee/mixing chamber. The inlet arms of the tee have zero dead volume connections for both the sample and internal standard lines. The outlet arm, however, has a miniature built-in mixing chamber which guarantees that the internal standard elements are intimately mixed with the sample prior to introduction to the nebulizer.



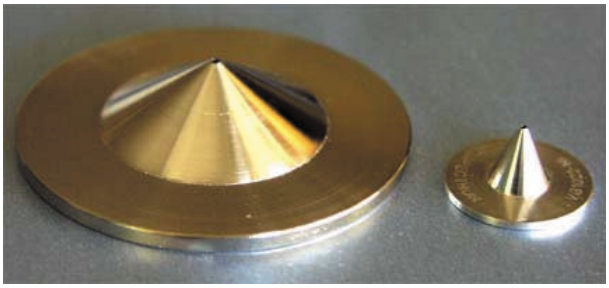
In-line Modular Internal Standard Kit

Cones

Over the past few years, ICP-MS spectrometers have been more customized for specific applications. Such has been the case for environmental analyses. The sample interface is a major area of focus in this endeavor. The two aspects of improvement have been in the temperature of the cones and the size of the orifice. If the sampler is run at a higher temperature, it is usually less susceptible to clogging by matrix components. However, higher temperature tends to shorten the useful life of the cone. The temperature can be controlled by the design of the cooling system and both the geometry and material of the cone. Nickel cones will deteriorate more quickly than those made of platinum but are less expensive. Because Glass Expansion offers a free

lifetime refurbishment policy for its platinum cones, the cost of using platinum is about the same as for nickel in the long-term. As an additional benefit, the frequency of changing cones will be reduced with platinum.

The orifice of the cones used for environmental configurations is in some cases enlarged to prevent or reduce the likelihood of clogging. This varies between instrument manufacturers and is an integral component of the spectrometer design. For example, the vacuum capacity of the instrument is dependent upon the orifice of the cones.



Ni/Cu Sampler (left) and Ni Skimmer Cones

ENHANCED PRODUCTIVITY

Niagara Rapid Rinse Accessory

Environmental service laboratories are faced with financial challenges as well as analytical ones. Because of the competitive nature of this business, labs are driven to reduce sample turnaround times and increase the number of samples successfully analyzed each day. The recently introduced Niagara Rapid Rinse Accessory is capable of increasing a laboratory's productivity by as much as 40%. The Niagara was the focus of the New Product column in the last issue of this newsletter and a full description will be found there and on our web site. In essence, the Niagara allows the operator of either an ICP-AES or ICP-MS instrument to eliminate the programmed rinse time and still achieve adequate rinsing. To measure the "Niagara Benefit" for your methods, please use the calculator on the Niagara page of our web site.

CONCLUSION

ICP-MS instrumentation has matured dramatically in the past several years. Although not yet a turn-key system, it now approaches that end to a much greater extent. The introduction of more modular components, more sophisticated software, more rugged sample interface, and customized sample introduction components have all contributed to this. Environmental analyses place a special burden on this instrumentation. However, an understanding of the interferences and the options available to resolve them make ICP-MS a viable technique for this application.

PRODUCT DESIGN FOCUS

The Capricorn Argon Humidifier

When samples with a high concentration of dissolved salts are being analyzed, it is not uncommon for salt particles to be deposited at various points in the sample introduction system. These salt particles can partially block the nebulizer and injector and can also accelerate de-vitrification of the outer tube. It is possible to reduce the problem by choosing a salt-tolerant nebulizer such as the SeaSpray, OpalMist or VeeSpray; a wide bore capillary injector and a reduced-length outer tube, but it may not be possible to eliminate the problem entirely. This is where the argon humidifier can help. With this device, the argon flowing to the nebulizer is saturated with water vapour and this causes the salts in the sample to remain in solution rather than depositing in the sample introduction system.

Argon humidifiers have been around for many years. However, the early versions were little more than a tube in a vessel of water. They were cumbersome to install, remove and fill; often did not address the hazards inherent in a pressurized glass vessel; and did not efficiently saturate the argon. They have improved over the years and the Capricorn is the culmination of the evolutionary process, effectively addressing all of the shortcomings of earlier versions:

1. In the Capricorn, the argon is passed through a rugged frit which disperses the argon into the water, ensuring maximum saturation.

2. The water vessel is thick-walled glass which is housed in a solid plastic container for added safety.

3. There is a water level indicator which clearly shows when water needs to be added and how much to add.

4. Water is added through a simple fill port so the Capricorn does not need to be disconnected before refilling.

5. A pressure indicator shows when the vessel is pressurized.

6. Quick-connect gas fittings enable the Capricorn to be quickly and easily installed.

7. For samples that do not require humidification, an optional bypass valve allows the argon to pass directly to the nebulizer without the Capricorn being disconnected.

8. A stable base enables the Capricorn to be positioned in a convenient location close to the nebulizer.

If you would like more information on the Capricorn Argon Humidifier please contact enquiries@geicp.com.

Capricorn with Bypass & Capricorn without Bypass

NEW PRODUCTS

HF-RESISTANT INTERNAL STANDARD KIT

An HF-resistant version of the Internal Standard Kit is now available. Internal standards are often used to improve stability. The internal standardization process involves the addition of a known concentration of a particular element to every sample, which can be a very time-consuming procedure. The Glass Expansion Internal Standard Kit allows the internal standard to be automatically mixed with each sample during sample introduction, thus saving considerable sample preparation time. The new HF-resistant version is made entirely from HF-resistant polymer materials. It is compatible with the Glass Expansion OpalMist and PolyCon nebulizers and our range of HF-resistant spray chambers. We can also supply packs of special peristaltic pump tubing with flared ends to facilitate easy connection to the sample tubing. Contact enquiries@geicp.com for more details.

INSTRUMENT NEWS

FROM AGILENT

Agilent has introduced a chromium speciation kit (G3268A), which is a new, simple and reliable method to separate and measure Cr (III) and Cr (VI) using an Ion Chromatography pump (Metrohm Ltd.) coupled to an Agilent 7500ce ICP-MS with Integrated Sample Introduction System (ISIS). The speciation of Cr (III) and Cr (VI) is possible in less than 4 minutes run time with ppt detection limits on this system. Furthermore, the new revision of ICP-MS ChemStation software (rev.B.03.02) is released to enhance the capabilities of speciation. It allows Agilent GC/LC ChemStation (32bit) and ICP-MS ChemStation to co-reside on the same PC. LC/GC methods can be run from the ICP-MS sequence table – making speciation studies easier and more powerful. There are more features on this new revision such as "Pre-emptive rinsing", which shortens rinsing time and improves sample throughput. More information about the Agilent 7500 Series ICP-MS can be found at www.agilent.com/chem/icpms.

GLASS EXPANSION NEWS

NEW ICP SPECIALIST

Vesna Dolic has joined the Australian office as ICP Specialist. Vesna was previously employed by Perkin Elmer as Inorganic Application Specialist and she has over 15 years of ICP experience. Vesna has taken over the running of Glass Expansion's ICP laboratory and she will enhance our ability to solve problems for ICP users and to deliver innovative products to make life easier for our customers. Vesna can be contacted at vdolic@geicp.com.

JAIMA SHOW 2005

A wide selection of Glass Expansion products will be on display at the JAIMA Show, Tokyo, Japan, August 31 to September 2, 2005. The display will include nebulizers, spray chambers, torches, RF coils and accessories (including the new Niagara Rapid Rinse Accessory). Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP.