



GLASS EXPANSION NEWSLETTER

Quality By Design

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APPLICATION SPOTLIGHT

Determination of Trace Metals in Rat Serum by ICP-MS

SAMPLES

Due to sample volume limitations, typical sample preparations must be scaled down to avoid large dilutions which would degrade detection unacceptably. Usually, for animal studies, 0.5mL is readily available. Therefore, a typical sample preparation would be to dilute 0.5mL of blood serum with 4.4mL of deionized water containing 0.05% Triton X-100 and 1ppb of the appropriate internal standards and vortex. Subsequently, add 0.1mL of 20% (v:v) nitric acid and vortex again. The nitric acid concentration is selected to be high enough to stabilize the metals in solution to prevent losses to the container walls but not so high as to cause precipitation of the protein.

ICP-MS SYSTEM

Many potential interferences exist due to the complex nature of the sample. Physical interferences due to the viscosity of the sample should be lessened by the 10-fold dilution and the use of a concentric glass nebulizer with a uniform VitriCone™ capillary. Any remaining physical interferences will be corrected by the appropriate selection of internal standards. Isobaric and chemical interferences may be more of a challenge. The direct analysis of serum has been accomplished via several means:

- * High resolution systems have been used to separate isobaric interferences via spectral resolution.
- * Reaction/Collision cells have been used to separate interfering species via chemical and physical means.

With a standard resolution ICP-MS without a separation cell (depending upon the analytes of interest) an added protein precipitation step or even an acid digestion may be required to strip away many of the interferences prior to presentation of the sample to the instrument.

RECOMMENDED SAMPLE INTRODUCTION SYSTEM

A small volume sample introduction system is recommended in this instance due to the limited sample available, the need for replicate measurements and the potential desire to resample. In addition, because of the expense of disposing of biological waste, it is advantageous to minimize the volume of waste collected.

Nebulizer: The MicroMist Concentric glass nebulizer designed to operate at 0.4mL/min will provide the best performance. This nebulizer comes standard with the EZT-050 EzyFit sample line connector, which not only simplifies sample line connection but also eliminates sample dead volume, hastening washout.

Spray Chamber: The Twinnabar Cyclonic spray chamber will provide the best performance in terms of producing a steady state signal with excellent filtering of larger droplets and biological particles. Combining the characteristics of the Twister and the Cinnabar, the Twinnabar is a small volume spray chamber (20mL internal volume compared with 50mL for standard cyclonic spray chambers) equipped with a central channel that serves as a baffle to create a circuitous route for the sample vapor (please see photo on page 3). The smaller internal volume of the Twinnabar results in faster washout times yet is large enough to generate the homogeneous sample vapor necessary to produce high precision when used with a low-flow nebulizer.

* The **Helix version** of the above spray chamber may provide added benefits. With standard O-ring based spray chambers, the protein may accumulate in the nebulizer entry port around the inside of the O-ring, causing sample occlusion and longer washouts. The Helix O-ring free design uses a Teflon ferrule to seal the inside of the spray chamber so that sample comes into contact only with glass and Teflon, preventing any opportunity for protein deposits.

Additional stability is provided via the use of a jacketed spray chamber. With this design, the central body of the spray chamber is surrounded by an external chamber containing a liquid that can be cooled or heated via a thermostatically controlled chiller. Creating temperature uniformity within the spray chamber enhances long term reproducibility.

Torch: A semi-demountable torch is recommended. The semi-demountable torch has fixed inner and outer tubes but demountable injectors. With samples containing significant concentrations of proteinaceous material, it is beneficial to be able to easily remove the injector for periodic cleaning and even replacement.



Semi-Demountable Torch

Torch Injector: Since protein deposits are more likely to develop at the tip of a tapered ID injector, a capillary straight bore injector is recommended.

Cones: For a couple of reasons, platinum cones might be preferable to nickel for this application. First, if nickel is of analytical significance, platinum offers an advantage. Second, with respect to nickel, platinum reduces the degree of salt pitting of the cone and carbon buildup within the orifice. Although platinum cones are more expensive, Glass Expansion offers a no charge refurbishment policy for the life of the cones.

OTHER CONSIDERATIONS

Nebulizer Maintenance: To prevent protein and/or salt deposits at the tip of the nebulizer, use the Eluo nebulizer cleaner to back-flush the nebulizer with methanol at the end of each run or at the end of each day.

Nebulization Efficiency: Because the MicroMist nebulizer is operating at a lower uptake rate, a proportional decrease in detection limit intuitively might be expected. However, because of the greater nebulization efficiency of the MicroMist nebulizers, this is not the case. Furthermore, the lower flows of the MicroMist result in two conditions that enhance detection limits:

- * The lower concentration of sample in the RF plasma yields greater ion generation efficiencies.
- * The smaller mean droplet size generated by the MicroMist reduces the amount of water associated with the sample and may reduce oxide interferences.

Boron Measurements: If trace boron determinations are of interest, it is best to steer clear of borosilicate glass sample introduction components. Spray chambers and nebulizers as described above are available in quartz which is boron-free. Alternatively, a PFA nebulizer designed to operate at 0.4ml/min, such as the Glass Expansion OpalMist would be a good choice for this application. Spray chambers made of PFA or polypropylene are also available.

PRODUCT DESIGN FOCUS

The Twister Cyclonic Spray Chamber

Early ICP spectrometers mostly used large volume (>100mL) double pass spray chambers. These generally provided good stability and detection limits. However they had some limitations, including low efficiency and long wash-out times. The availability of the cyclonic spray chamber in the 1990's helped overcome these limitations.



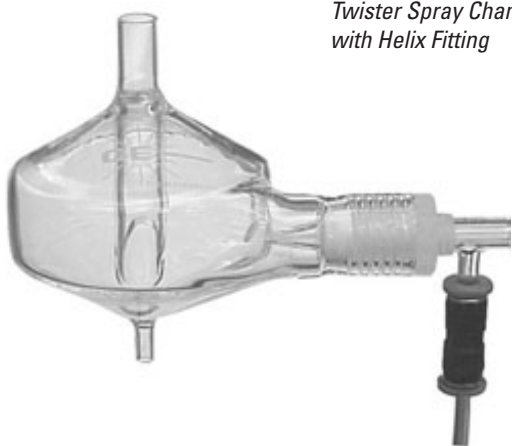
Cyclonic Spray Chamber

With the cyclonic spray chamber, the aerosol is introduced tangentially by a concentric nebulizer. The aerosol flows in a vortex about the axis of the spray chamber and the larger droplets are eliminated by centrifugal action. These larger droplets strike the wall of the spray chamber and flow to the drain while the smaller droplets are swept into the torch by the argon stream.

Compared with the double pass spray chambers, the newer cyclonic spray chambers provide improved efficiencies and detection limits. Since they are of much lower volume (usually about 50mL), they also provide much faster washout times and reduced carryover from one sample to the next. At the same time, they are able to match the excellent precision of the double pass spray chambers.

As the cyclonic spray chambers became more widely used it was apparent that, with some samples and torch designs, they were simply too efficient. For example, with organic solvents, the higher amount of solvent being delivered to the torch had a destabilizing effect on the plasma. This led to the introduction of the Twister design, where a central baffle provides a secondary droplet filter, resulting in a finer aerosol reaching the torch.

The Twister spray chamber is slightly less efficient than the standard Tracey cyclonic, but the improvement in plasma stability leads to even better precision and equivalent detection limits. The low carryover and fast washout characteristics are retained, enabling a rapid sample throughput.



*Twister Spray Chamber
with Helix Fitting*

Jacketed versions of the Twister spray chamber enable the temperature to be stabilized or the chamber cooled for use with volatile solvents.

The Twister is also available with the unique Glass Expansion Helix o-ring free nebulizer fitting. This fitting is totally inert and allows the smooth insertion and removal of the nebulizer every time.

NEW PRODUCTS

RF COILS

Replacement of a corroded RF coil increases energy transfer and reduces the load on the RF generating system, resulting in a more robust plasma and higher analytical line intensities. The high quality and consistent plating of the Glass Expansion coils promotes extended coil life. Also, each coil is supplied on a plastic former, ensuring correct dimensions are maintained during transport and simplifying installation. A re-usable installation kit, which includes instructions on CD-ROM, is also available. Glass Expansion RF coils are available for a number of commonly used ICP models and we are regularly adding to our range. Check our website at www.geicp.com to see the current range.

FULLY DEMOUNTABLE TORCH FOR VARIAN VISTA

The new Glass Expansion Fully Demountable Torch for the Varian Vista ICP enables the outer, inner and injector tubes to be replaced in seconds. This means that you no longer need to throw away an entire torch when one tube has deteriorated, saving you money on replacement torches. You can also easily change tubes to suit your application needs. Quartz and alumina injectors in varying bore sizes are readily available, as are ceramic outer and inner tubes. Fully Demountable Torches are also available for many other ICP models. Check the "Products to suit your ICP" section of our website or send an email to enquiries@geicp.com.

INSTRUMENT NEWS

FROM AGILENT TECHNOLOGIES

Agilent Technologies has introduced the new 7500cs high sensitivity reaction cell ICP-MS specifically for semiconductor and research facilities that demand the widest elemental coverage and ultimate detection power in high purity matrices. Superseding the 7500s, the semiconductor industry standard, the new 7500cs combines the high sensitivity of the previous model with the interference removal power of Octopole Reaction System (ORS) technology. Agilent's highly successful cool plasma mode is also available if preferred - offering the analyst complete flexibility in operation. An enhanced ChemStation for the 7500 ICP-MS using the Windows 2000 and Windows XP operating systems has also been released. And furthermore, the User Access Control Pack security software for the 7500 ICP-MS has been released in response to requests from users who require enhanced security from their ChemStation for auditing and access management. Please visit www.agilent.com/chem/icpms for more information.

FROM LEEMAN LABS

Stability in automated instrumental analyses means better quality control, less replication, more productivity. The Prodigy inductively coupled plasma spectrometer from Leeman Labs demonstrates exceptional long-term stability, typically less than 0.5% RSD on repetitive readings of a control sample over a five hour period. The system design employs an image stabilized plasma torch mounted directly to the polychromator to keep the image peaked on the entrance aperture, a thermostatically controlled polychromator to eliminate wavelength drift, and a temperature stabilized L-PAD (large format programmable array detector). These results were achieved using a radial torch equipped with Glass Expansion's high precision Conikal nebulizer and Twister cyclonic spray chamber. For more information, please visit www.leemanlabs.com.

FROM JOBIN YVON

The ULTIMA 2 family of ICP Spectrometers features systems tailored to your needs. The NEW ULTIMA 2000 combines affordability and performance better than competitive ICP spectrometers at far higher prices. The ULTIMA 2 provides the ultimate performance in resolution (0.005 nm at 200 nm) and detection limits, while the ULTIMA 2C is a complete solution combining speed, flexibility and precision, with environmental (ULTIMA 2CE) and high resolution (ULTIMA 2CHR) models available. JY also produces Made-to-Measure Custom ICP spectrometers made to order for your exact laboratory setting - radioactive, mobile, shipboard, etc. All products

are backed by the ALLIANCE Service program featuring an online catalog, E-News Application updates and the World Link User Forum. For more information visit <http://www.jobinyvon.com/emission/products/ULTIMA2Family.htm>.

HINTS FOR THE OPERATOR

Getting the Best Results from your Spray Chamber

Although polymer and quartz spray chambers are available for special applications, most Glass Expansion spray chambers are made from high-quality borosilicate glass. Glass should be treated with the care that is due to a brittle material. It can fracture and produce sharp edges, so handle glass spray chambers carefully and don't apply large mechanical forces to them, especially when connecting drain tubes, aerosol tubes or nebulizers. Don't knock a glass spray chamber against hard objects or leave it unprotected when not in use. Soft plastic caps are provided to protect spigots of Glass Expansion's spray chambers. They should be used to protect the ends of the spigots when the spray chamber is placed in storage.

Do not use metal or ceramic brushes or scraping tools. These are likely to damage your spray chamber.

It is good practice to always start and finish use of a glass spray chamber by nebulizing a mildly-acidic blank solution for several minutes. This ensures that sample deposits or crystals don't form when the solvent inside the chamber dries out.

Don't wash glass spray chambers in an ultrasonic bath. If a spray chamber becomes contaminated with sample deposits, it should be soaked in a cleaning solution such as FLUKA 'RBS-25' at 25% strength for a full day. If this doesn't remove the contamination, acidic baths can be used, but never use hydrofluoric acid.

If you see droplets collecting on the internal surfaces of your spray chamber, this is a sign that stability may be suffering. Droplets in the spray chamber are the most common and visible indication of spray chamber instability, and they should be removed. A long soak in 25% RBS-25 solution is recommended. Avoid touching any internal surfaces of the spray chamber as this may damage its wetting properties. If there are still drainage problems, adding a small amount of a surfactant such as Triton X-100 to your samples usually helps. Be sure that you do not add too much – a concentration of 0.01% Triton in your samples should be sufficient. Adding Triton to your samples is also

useful with the polymer spray chambers recommended for samples containing hydrofluoric acid, since the polymer materials generally do not wet as easily as glass.

Does the sensitivity of your ICP change regularly with the time of day, or the season? Remember that a spray chamber temperature change of 1 degree can cause some line sensitivities to change by 1%. Try to insulate your chamber thermally or upgrade to a jacketed version.

Hydrofluoric acid (HF) should never be used with glass. Glass Expansion has a wide range of polymer spray chambers specially designed for use with HF solutions. Use of even a small amount of HF may damage a glass spray chamber. Glass Expansion spray chambers are supplied clean and ready to use. Do not treat glass spray chambers with HF. For your protection, always use safety gloves and glasses when handling glass.

GLASS EXPANSION NEWS

NEW WEBSHOP

Glass Expansion has upgraded its website (www.geicp.com) to include an online ordering facility. If you know the part number you require, you simply select the SHOPPING CART button, enter the part number, and fill in your details. If you do not know the part number, select the PRODUCTS button and then "Products to suit your ICP." You will then be presented with instrument and product options. Even if you do not wish to order online you will be able to access the current Glass Expansion prices.

ORDERING FROM EUROPE

European customers are now able to order directly from the factory in Australia. We hold stock of approximately 1000 parts and are able to offer immediate shipment of most parts. Shipment to most European countries takes approximately three days by Federal Express.

JAPANESE WEBSITE

Most of the information on the Glass Expansion website is now available in Japanese. Simply go to www.geicp.com and select JAPANESE VERSION.

NEW CHIEF FINANCIAL OFFICER

Glass Expansion is pleased to announce the appointment of Ben Kaiser as Chief Financial Officer. Ben has filled several international finance roles, most recently with Ernst & Young in Singapore.