



GLASS EXPANSION NEWSLETTER

Quality By Design

February 2005

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APPLICATION SPOTLIGHT

Wastewater Analysis by ICP-AES

INTRODUCTION

The determination of metals in wastewater is one of the most common applications of ICP spectrometers, both atomic and mass. It is also one of the most regulated applications since the results of the analyses may be used in litigation. There is a number of government agencies around the world involved in the establishment of environmental analytical protocols. In the US, the Environmental Protection Agency (USEPA), created in 1970, has been at the forefront of this effort. Government agencies outside the US often base their protocols on those promulgated by the USEPA. As an agency of the EPA, the Office of Solid Waste (OSW) is responsible for developing analytical methods and protocols for the analysis of wastewater and solid waste. The OSW document, SW846, is a compendium of methods for this purpose. Method 6010b is the ICP-AES method for wastewater samples. The Environmental Monitoring & Support Laboratory (EMSL) in Cincinnati, Ohio, another branch of EPA, also develops methods for similar samples. The EMSL method for ICP-AES is 200.7. EPA has established a host of private laboratories as part of their Contract Laboratory Program (CLP) to help monitor and regulate pollution. These laboratories must follow a stringent protocol (ILMO 5.2D) to comply with EPA regulations. Rather than go into minute detail as to the nature of this protocol, suffice it to say that if an instrument is not kept in top shape, a contract laboratory would be hard pressed to make a profit. EPA continually monitors instrumental performance developments and strives to keep Contract Required Detection Limits (CRDL) barely above Instrument Detection Limits (IDL) for many potential pollutants.

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 perhaps Al, almost any element may be found at high concentration due to specific process pollution. Organic matter is destroyed in the nitric acid digestion step and any particulates are removed prior to analysis by filtration.

ICP INSTRUMENTATION

ICP-AES spectrometers have routinely been used for this application although they must be carefully configured to meet the demanding detection limit criteria. Although some manufacturers recommend high resolution systems for this application, most espouse the need for axial or dual view spectrometers. Axially viewed, or end-on-plasma, spectrometers permit a greater cross-section of the analytical zone in the plasma to be viewed by the detector than does a radial view spectrometer, thus increasing raw intensity counts. Various means of lowering background, such as simultaneous spectral background correction, help lower detection limits. However, for the same reason that axial viewing increases intensities, interferences may be enhanced. The radial system detector views a smaller region of the torch plume but it is one that is least prone to interferences, whereas the axial system detector views some regions of the plume that are more prone to interferences, such as easily ionizable element effects, and poor linearity. For this reason, dual view systems have become popular so that only the low concentration elements are viewed axially and the rest radially. It should be mentioned that some manufacturers maintain that an axial configuration by itself can be properly configured to avoid the interferences listed above.



VISIT GLASS EXPANSION AT PITTCON 2005
BOOTH 3013
FEBRUARY 27 - MARCH 2, 2005.

SAMPLE INTRODUCTION SYSTEM

The goal of the sample introduction system for this application is to produce the highest intensity emission signal with the most stable response and the lowest background, and to do this for long periods of time with minimal maintenance.

Torch

We recommend a fully demountable torch (if available) primarily to minimize the cost per sample. This application is a high volume one that is sensitive to the cost of laboratory consumables. The axial and dual view torches often employed tend to degrade more rapidly than their radial counterparts. Because the outer tube is exposed to fairly high dissolved salt levels, devitrification of the quartz is more pronounced. This can lead to relatively short lifetimes for the torch outer tube. A fully demountable torch allows the operator to replace only the outer tube as needed to minimize consumable costs.



Fully Demountable Torch for Thermo Iris

Injector

A wider bore (2 to 2.5mm) quartz injector is recommended for two reasons. First, it is able to stand up longer to exposure to dissolved salts without clogging. In addition, a wider bore injector has been found to lower detection limits for an axially viewed torch. This is because the wider bore actually enlarges the analytical zone within the plasma, permitting greater signal intensities to reach the detector.

Spray Chamber

We recommend the glass Tracey spray chamber for this application. Whereas the Twister (with its central baffle) does a good job of filtering the large droplets from the torch, the Tracey provides the highest intensities. When the EPA protocol to determine detection limits is followed, high intensity provides lower detection limits as long as the noise of a blank is not increased. We also recommend the Helix for three reasons. First, the Helix eliminates the need to change worn o-rings, a process which is onerous and time consuming. Second, the Helix is the ideal nebulizer/spray chamber interface, reducing dead space inside the spray chamber where solution can build up and saving precious seconds in rinse times. Thirdly, its simplicity ensures accurate nebulizer positioning whilst the

Teflon seal prevents jamming, possibly saving both nebulizer and spray chamber.



Tracey Cyclonic spray chamber with Helix for Perkin-Elmer Optima

Nebulizer

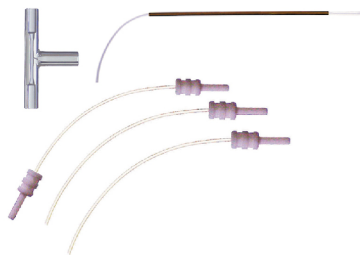
The SeaSpray concentric glass nebulizer is the nebulizer of choice for this application. Its high nebulization efficiency combined with its ability to nebulize high dissolved solids samples makes it the ideal choice for wastewater analysis. As with most Glass Expansion nebulizers, the SeaSpray is equipped with the EzyFit sample connector. The EzyFit comprises a capillary tube connected to a Teflon plug that is inserted into the back of the nebulizer. The plug and nebulizer are designed so that the plug leaves zero dead volume in the nebulizer sample port to minimize washout times.



SeaSpray nebulizer with EzyFit sample connector and EzyLok argon connection arm

Internal Standard

To achieve financial and analytical success in this type of laboratory, an in-line addition assembly is recommended. An in-line addition kit provides for the simultaneous addition of two necessary reagents. An internal standard is added to compensate for the matrix effects of dissolved salts on the analytes. An ionization suppressant (usually Cesium) is added to compensate for the effects of easily ionizable elements on each other. Typically, a solution of 5 or 10 mg/L Yttrium or Scandium is doped with 1% Cs. The resulting solution is added to all samples blanks and standards with an in-line addition assembly resulting in a 3 to 10X dilution of the reagent and a 10 to 30% dilution of the samples. Glass Expansion offers such a kit, P/N 60-808-1185, which is 100% modular so that only those parts that are worn need be replaced. We also provide flared-end pump tubing to facilitate the connection of narrow bore internal standard tubing to the capillary lines.



Modular Internal Standard Kit

Argon Humidification

Typically, laboratories involved in wastewater analysis also deal with digested soil samples, which present even higher levels of dissolved solids. For these types of samples and for particularly dirty wastewater samples, an in-line argon humidifier is recommended for maintenance-free operation. The humidified argon reduces the likelihood of salting out within the nebulizer or injector tip, which can lead to poor performance and eventually a clogged sample introduction system. However, when running cleaner samples, it may be preferable to turn off the humidifier. Adding moisture to the argon may unnecessarily cool the plasma and may render some of the hard lines less intense. Running the torch hotter may compensate for this effect but may also result in more plasma noise. In addition, running the humidifier when not needed leads to more frequent refilling of the reservoir. Glass Expansion offers an argon humidifier called the Capricorn™ which has a built-in bypass toggle that allows the operator to turn off humidification with the flip of a switch. The Capricorn also has a refill port so that it does not require any disassembly to add more water. In addition, it is encased in plastic for safe operation under pressure.



Capricorn™ Argon Humidifier with bypass switch

Conclusion

With the right sample introduction components, a very demanding analysis can be rendered as close to a routine analytical procedure as possible. In the next issue of the Glass Expansion newsletter, we will focus on the analysis of wastewater and drinking water by ICP-MS.

NEW PRODUCTS

NIAGARA RAPID RINSE ACCESSORY

Most ICP systems incorporate an autosampler. With these systems there is a significant delay between the time when the autosampler probe enters the sample and the time when the sample reaches the nebulizer. There is a similar delay between the time when the probe enters the rinse solution and the time when the rinse solution reaches the nebulizer. If these delays could be eliminated, the analysis time per sample could be reduced significantly and the sample throughput increased.

This is what the new Glass Expansion Niagara Rapid Rinse Accessory achieves.

The Niagara begins the rinsing of the nebulizer and spray chamber the instant the sample measurement is completed and continues to rinse until the next sample is ready. Thus the rinse is carried out in the time that is usually wasted waiting for the sample and the rinse solutions to flow from the autosampler to the nebulizer.

The Niagara incorporates an electronically controlled switching valve. During the sample measurement time, the rinse solution is pumped through the valve to waste (see Fig.1).

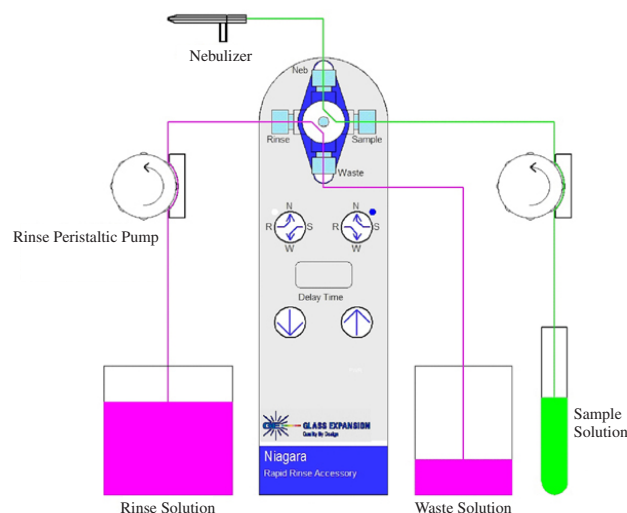


Fig.1. Sample to Nebulizer, Rinse to Waste

At the completion of the sample measurement, the valve switches instantly and the rinse solution is directed to the nebulizer (see Fig. 2).

The valve stays in this position while the autosampler probe moves to the rinse position and then on to the next sample. Only when the next sample has made its way through the full length of the uptake tubing does the valve switch back to the position shown in Fig. 1. The time that is

saved is around 30% for a typical analysis (see Table 1).

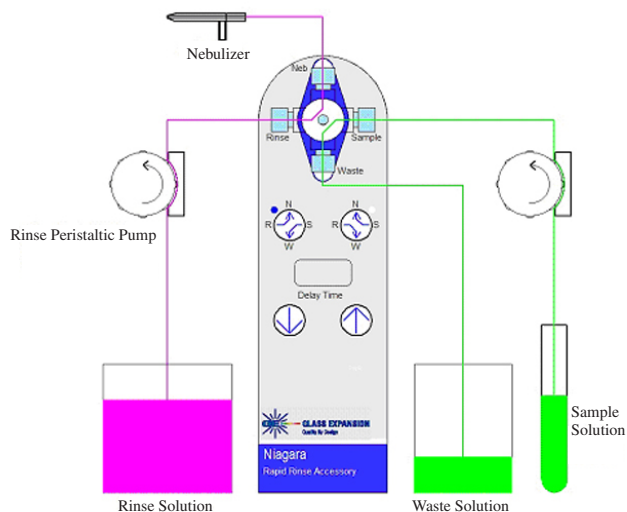


Fig. 2. Rinse to Nebulizer, Sample to Waste

The benefits of the Niagara for the busy ICP laboratory are:

- Faster sample turnaround time
- More productivity for your ICP
- Argon costs are reduced

Contact enquiries@geicp.com for more information on how the Niagara can save you time and money on your ICP analysis.

TEFLON COATED RF COILS FOR THE VARIAN VISTA

Glass Expansion has been supplying high-quality gold-plated and silver-plated RF coils for the Varian Vista for some time. We have now expanded our range to include Teflon-coated coils. These are available with either gold or silver plating. Tests have shown that the Teflon coating increases the lifetime of the coil by approximately a factor of three. We also have Teflon-coated coils for the Thermo

Iris and gold or silver-plated coils for a wide range of ICP-AES and ICP-MS models. Contact enquiries@geicp.com for more information on the Teflon-coated coils for the Varian Vista or to check the availability of RF coils for your ICP.

HINTS FOR THE OPERATOR

Care and maintenance of Quartz Torches


We are sometimes asked how long a quartz torch normally lasts. The answer can be more than a year if it is treated well or a few minutes if it is not treated well. Some of the factors that contribute to a reduced lifetime are:

Temperature: If the torch reaches a temperature above the melting point of quartz then clearly it will melt. The most common cause of a melted torch is incorrect argon flow. It is critical that the plasma not contact the quartz and it is the flow of argon that holds the plasma in position and prevents it from contacting the torch. If the argon flows are not set correctly, or if there is an interruption to the flow, or if there is a leak in the argon lines, then it is possible to see an instant torch meltdown. Glass Expansion recommends the use of GazFit connectors for reliable connection of the argon lines to the torch. Improper alignment of the torch within the RF coil is another potential cause of torch degradation. Therefore it is important to ensure proper alignment of the coil when it is replaced. Glass Expansion offers a coil installation and alignment tool to be used with its line of RF coils. If the correct argon flows are maintained and the torch is positioned correctly in the RF coil, then the torch should not melt. However, high temperatures will still contribute to devitrification of the quartz over time. So devitrification is likely to occur faster with those applications where the torch is run at

Table 1. Typical Analysis Cycle

	Standard System		With Niagara	
	Cycle Time, sec	Rinse Time, sec	Cycle Time, sec	Rinse Time, sec
Probe to Sample	5	0	5	5
Uptake Delay	15	0	15	15
Stabilisation	10	0	10	0
Read	15	0	15	0
Probe To Rinse	5	0	5	5
Rinse	30	30	5	5
Total	80	30	55	30

With this example, the total cycle time with the Niagara (including 30sec rinse) is 55sec compared with 80sec for the standard system without the Niagara – a saving of 25sec or 31%.

 Rinse Period

higher temperatures.

Contamination: Contamination of the surface of the quartz, combined with high temperature can lead to rapid devitrification. Contamination is most commonly due to deposits of salts or oils from the samples near the end of the outer tube. If this is occurring the torch should be cleaned regularly. Carbon deposits from organic samples can best be removed by baking the torch in a muffle furnace at 450 deg C for 30 minutes. Salt deposits are best removed by soaking the torch in 10% HCl. If contamination of the outer tube is a persistent problem it may be advisable to use a shorter outer tube. Although this may result in some loss of sensitivity, it can also lead to a significant improvement in torch life. Glass Expansion can supply several different types of torch with shorter outer tubes. A fully demountable torch will also reduce costs, since only the outer tube need be replaced rather than the whole torch.

Contamination can also be caused by handling of the quartz with bare hands. It is important to always use safety gloves when handling quartz torch components. If the quartz components are handled with bare hands, body oils may be deposited on the surface. These oils can accelerate devitrification of the quartz and significantly reduce the torch lifetime.

In summary:

1. Ensure the torch is correctly positioned with respect to the RF coil.
2. Ensure the correct gas flows are maintained at all times.
3. Ensure that there is no argon leakage at the tubing connection points.
4. Never touch the torch with bare hands.
5. Use the lowest power consistent with your application.
6. Do not allow contaminants to build up on the quartz.
7. Consider a shorter outer tube when running samples with high dissolved solids.
8. Consider a fully demountable torch to reduce torch replacement costs.

INSTRUMENT NEWS

FROM AGILENT

Agilent Technologies celebrated the shipment of the 1000th 7500 Series ICP-MS. More than seven hundred 4500s were shipped over six years, while the 7500 reached 1000 units in just over four and a half years. One of the strengths of the 7500 Series is its flexibility so that new technologies can be added at a later date to meet the evolving requirements. Now all 7500 Series users can benefit from the significant analytical benefits of the Octopole Reaction System (ORS) by taking advantage of these on-site

upgrades. Upgrade paths are available to take any 7500 mainframe right up to the current 7500ce or 7500cs ORS design. All ORS upgrades also include an upgrade to the newest version of the ICP-MS ChemStation software (B.03). Contact your local Agilent representative for a quote on upgrading your 7500 to the latest ORS performance. More information about the Agilent 7500 Series ICP-MS can be found at www.agilent.com/chem/icpms.

FROM THERMO

The new XSeriesII ICP-MS from Thermo Electron Corporation is a major update to the highly successful X Series ICP-MS. Utilizing a new ion extraction system, the XSeriesII significantly improves the detection limits of the quadrupole ICP-MS technique with a further improvement to the already class leading signal to background performance. The XSeriesII also features a new Protective Ion Extraction (p) ion optics design, which dramatically reduces blank levels and improves the interference removal capabilities in combination with Thermo's new Third Generation Collision Cell Technology. A new Xt interface, which is featured as standard in the X SeriesII ICP-MS, utilizes the ground breaking cone geometry and analytical benefits of the previous Xi Interface but improves the matrix tolerance capabilities and reduces user maintenance requirements. Ensuring maximum ease-of-use and high productivity, the new interface as well as the ion optics design require no user cleaning or replacement of components in the high vacuum area - a benefit unique to Thermo ICP-MS. The new XSeriesII ICP-MS presents enhanced benefits across the whole range of common ICP-MS applications. The ease of use and practical design of the XSeriesII means that laboratories can achieve their analytical objectives faster, with greater confidence and less hands-on time from the operator.

GLASS EXPANSION NEWS

PITTCON 2005

Glass Expansion will once again be exhibiting at Pittcon in Orlando, Florida, February 27 – March 4, 2005. You are invited to visit us at Booth 3013. A full range of our products will be on display and our experts will be available to assist you.

EUROPEAN WINTER CONFERENCE

Glass Expansion successfully exhibited a full range of products at the European Winter Conference on Plasma Spectrochemistry which was held in Budapest, Hungary, January 29 to February 2, 2005. We would like to thank all the conference attendees who visited our stand to view the latest developments in ICP sample introduction. We appreciate your interest in our products.