



GLASS EXPANSION NEWSLETTER

Quality By Design

October 2007

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blood (or blood serum), urine, and tissue. Each of these sample types has its own unique challenges. Blood samples have the advantage of being consistent yet very complex. Relatively high concentrations of iron, magnesium, calcium, sodium, potassium, copper and zinc result in significant isobaric and mass/charge interference potential. Furthermore, the high organic content produces its own challenges. However, once a method has been developed to conquer or at least control these challenges, it is usually a robust method. On the other hand, urine samples vary widely in element and organic content. Though not as complex a matrix as blood, urine samples often present a more difficult challenge in the creation of a robust methodology. Tissue samples, since they are not in liquid form, pose the challenge of dissolution or digestion with the associated risk of contamination.

APPLICATION SPOTLIGHT

Clinical Applications of ICP-MS: Optimizing the Front End

Jerry Dulude, Glass Expansion, Pocasset, MA

The following is based on a paper published in the October 2007 Applications Primer of Spectroscopy magazine. In the paper below, we have included information about products and models that was omitted from the original Spectroscopy paper.

INTRODUCTION

Two of the most significant areas of advancement in ICP-MS, with respect to clinical applications, have been the evolution of the sample introduction system and the interface of liquid chromatography (LC). The complexity of the sample matrix creates challenges for a number of components involved with the introduction of ions into the mass spectrometer including the nebulizer, spray chamber, torch, and interface cones. The development of LC-ICP-MS methods enables the analyst to quantitate not only the total metal content but the form of the metal as well, a distinction which in many cases is crucial. Although the refinement of reaction and collision cell technology has been important for this application, much has been written elsewhere and it will not be addressed here.

SAMPLES

The types of samples discussed here will be limited to

Due to sample volume limitations (particularly for infants and small animals), typical sample preparations must be scaled down to avoid large dilutions which would degrade detection limits unacceptably.

SAMPLE INTRODUCTION SYSTEM

A low sample uptake sample introduction system is recommended due to the limited sample available, the need for replicate measurements, and the expense of disposing of biological waste.

Nebulizer: The salient characteristics of a nebulizer used for clinical applications are the material of composition, the uptake rate, ruggedness, and consistency. Borosilicate glass, quartz, or perfluoroalkoxy (PFA) polymer are all well suited. Although PFA nebulizers are the most inert and may be preferred for applications requiring ultra-trace determinations of certain elements including boron, glass nebulizers are adequately free of trace metals and, because of their lower cost, may be preferable for clinical applications. Concentric nebulizers provide the best performance but have a reputation for being easy to clog which can be a problem with undigested biological samples. Glass Expansion has designed concentric glass nebulizers which have a constant bore internal capillary reducing the likelihood of clogging (Figure 1). A specific model of concentric nebulizer known as the SeaSpray™ is particularly tolerant of high dissolved solids. Depending upon the sample size available, uptake rates on the order of 0.1 to 0.4ml/min are preferred. The mass transport to the torch of a 0.4ml/min nebulizer is only slightly less than that

of a higher uptake nebulizer since it generates a more efficient aerosol. The SeaSpray nebulizer has recently been re-engineered to create a model with a natural uptake rate of 0.4ml/min, ideal for clinical applications of ICP-MS.

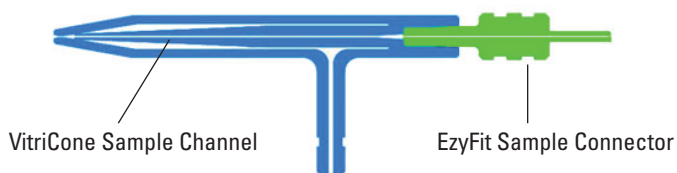


Figure 1. Concentric glass nebulizer with internal capillary made from a machined thick wall glass tubing (VitriCone™ technology)

Spray Chamber: This is a critical component of the sample introduction system, the significant characteristics of which are its composition, design, and temperature. Borosilicate glass provides adequate purity and a surface that is conducive to the smooth liquid sheeting action required for precise performance. The cyclonic design of the Twister™ spray chamber efficiently removes large droplets while the central baffle acts as a droplet size cutoff filter, resulting in a fine and consistent tertiary aerosol, thereby enhancing ion production in the plasma. Temperature control of the spray chamber is important in several respects. Setting the spray chamber at close to zero degrees Celsius reduces the water transport to the plasma causing a commensurate reduction in potentially interfering oxides (Figure 2). Moreover, stabilizing the chamber temperature produces a more uniform response over the long term. For those samples with extremely limited volumes requiring uptake rates of less than 0.1ml/min, elevating the temperature of the spray chamber is a good way to increase sample transport to optimize detection limits. Figure 3 shows the effect of spray

chamber temperature on sensitivity for a 20 micro liter per minute sample uptake rate. Data shown in figures 2 and 3 were generated with a programmable temperature spray chamber which can be employed with virtually all commercially available ICP-OES and ICP-MS systems and covers the range of -10 to 60 degrees Celsius (IsoMist™ from Glass Expansion). Figure 4 shows the IsoMist mounted in an ICP-MS. The IsoMist incorporates a Twister spray chamber encapsulated in a heat conductive resin for responsive temperature control. It is also available with an inert PFA spray chamber for ultra-trace measurements.

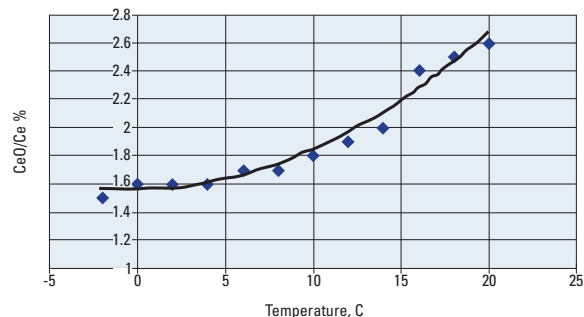


Figure 2. Effect of spray chamber temperature on oxide ratio

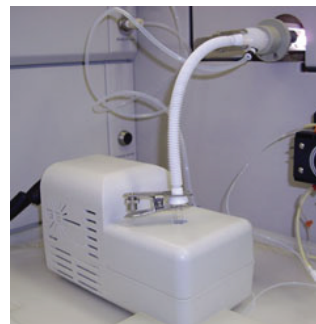
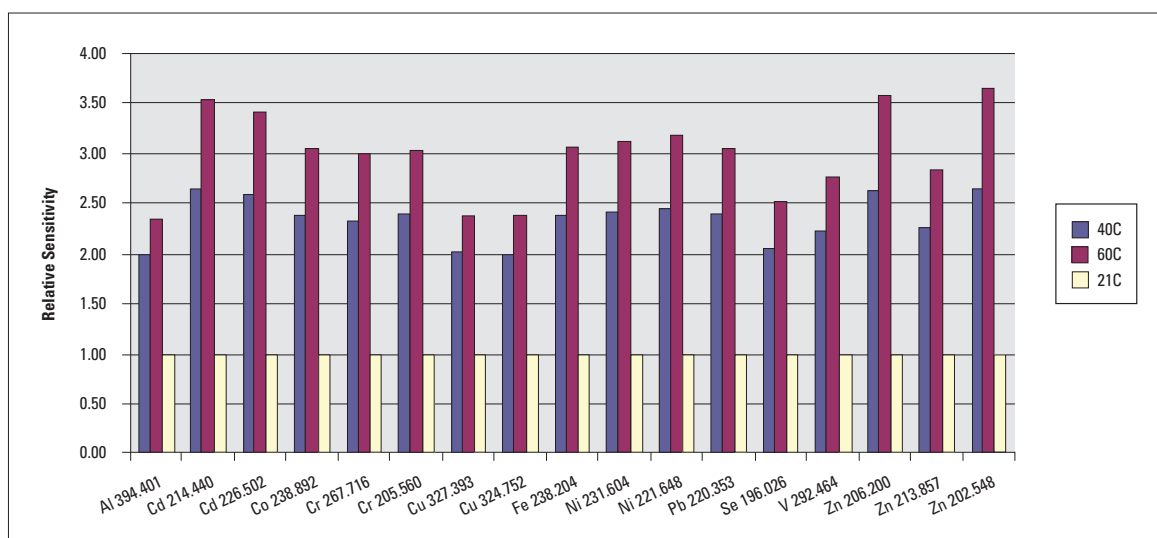


Figure 4. IsoMist Programmable Temperature Spray Chamber mounted on an Agilent 7500 ICP-MS

Figure 3. Effect of spray chamber temperature on sensitivity for 20ul/min uptake using ICP-OES



Torch: The most important torch characteristic for this application is the design of the injector tube. To tolerate complex samples without a buildup, either a capillary bore (no internal taper at all) or a gradual taper is recommended. Also, a larger bore decreases the likelihood of interfering deposits. Although injectors are available in a variety of materials including alumina, sapphire, and platinum, quartz is sufficiently pure and tolerant of the samples encountered in this application.

Sampler and Skimmer Cones: Both the material of composition and the orifice size are important considerations. For a few reasons, platinum cones might be preferable to nickel for this application. First, if nickel is of analytical significance, platinum offers an obvious advantage. Second, as compared to nickel, the more noble platinum reduces the degree of salt pitting of the cone, thereby extending its useful lifetime. A slightly larger orifice in the sampler cone than that recommended for cleaner samples helps to reduce residue buildup in the orifice.

OTHER CONSIDERATIONS

Nebulizer maintenance: To prevent protein and/or salt deposits at the tip of the nebulizer, the nebulizer should be removed at the end of each day and back flushed with methanol. Nebulizer cleaning tools, such as the Eluo™ (Glass Expansion) are available to facilitate this task.

Internal standardization: Rather than pipetting an internal standard "soup" into each sample standard and blank separately, it may be more efficient to use an in-line additions kit instead. Glass Expansion offers the Trident™ in-line reagent addition kit that minimizes carryover while intimately mixing the sample with internal standard.

Nebulizer to spray chamber interface: Many spray chambers use o-rings to seal the nebulizer in the chamber port. For clinical samples, this interface provides a location for buildup of protein which can degrade precision. Spray chambers are available with the proprietary Helix™ nebulizer interface which incorporates a specially tapered Teflon seal that eliminates dead volume between the nebulizer and spray chamber.

Helium as auxiliary gas: Adding helium gas to the argon flow has been shown to result in fewer argon oxide interferences and greater ion production particularly for difficult to ionize elements such as arsenic, selenium, and lead². Either the spray chamber or the torch adaptor can be configured with a port to accommodate the addition of an auxiliary gas.

LC-ICP-MS

The interfacing of an LC to the ICP-MS requires certain modifications to the sample introduction system. The

nebulizer natural uptake rate should be matched to the eluent rate of the LC to maintain laminar flow. In addition, the interface of the LC eluent line to the nebulizer must be as short as possible with minimum dead volume and be capable of withstanding high pressures. Glass Expansion supplies a distinct nebulizer configuration and a separate interface kit to accomplish this (see the NEW PRODUCTS section of this newsletter). In addition, the spray chamber volume should be minimized to limit peak broadening. Although 20ml internal volume spray chambers are available, even smaller volume chambers may result in sharper peaks and experimentation is ongoing in this regard.

REFERENCES

1. Data courtesy of David Jones of ALS Chemex, Brisbane, Australia, taken on a PE Sciex Elan 6000 using an IsoMist™ Programmable Temperature spray chamber.
2. B. S. Sheppard, W. L. Shen, T. M. Davidson, and J. A. Caruso, Helium-Argon Inductively Coupled Plasma for Plasma Source Mass Spectrometry, *J. Anal. At. Spectrom.* 5, 697-700 (1990).

NEW PRODUCTS

LC Interface for Speciation Measurements

The chemical effect of some elements varies dramatically depending on the species present. Therefore the concentration of each species needs to be determined rather than the total concentration of each element. In order to measure the concentration of different species, they must first be separated using chromatography - most commonly LC - and then detected using ICP-MS or ICP-OES.

Glass Expansion has released a special nebulizer with a capillary sample inlet arm plus an LC fittings kit (Figure 1)

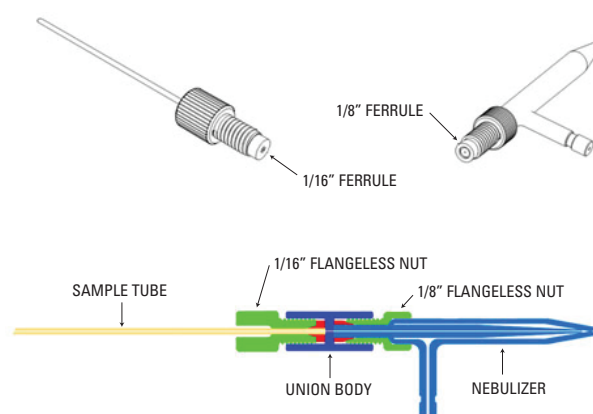


Figure 1. Nebulizer with capillary sample inlet arm plus LC fittings kit.

to allow quick and easy connection of your LC to your ICP. SeaSpray, MicroMist and Conikal versions of the new nebulizer are available to suit all common models of ICP-MS and ICP-OES. And the LC fittings kit can be configured to interface to all common LC outputs.

Contact enquiries@geicp.com to find out how easy it is to connect your LC and ICP.

Applications Kits for Thermo Fisher iCAP 6000 Series

Application-specific kits are now available for the Thermo Fisher iCAP 6000 Series. The components of each kit have been specially selected to provide optimum performance for the specified application. Each kit includes a complete sample introduction system – nebulizer, spray chamber, torch and peristaltic pump tubing. Select the most appropriate kit for your application from the following:

[KT-1032](#) - Standard Kit for iCAP 6000 Duo

For normal aqueous samples with relatively low (< 3%) levels of dissolved solids and no particulates. Suitable for drinking water and relatively clean environmental samples.

[KT-1033](#) - Organics Kit for iCAP 6000 Duo

For samples containing non-volatile organic solvents such as xylene and kerosene.

[KT-1034](#) - Volatile Organics Kit for iCAP 6000 Duo

For samples containing volatile organic solvents such as alcohol, gasoline and naphtha. Includes the IsoMist Programmable Temperature Spray Chamber.

[KT-1036](#) - High Solids Kit for iCAP 6000 Duo

For samples containing up to 20% total dissolved solids. Suitable for sea water, ground water, brine, geochemical samples and fusions. Includes the Capricorn Argon Humidifier (Figure 2).

[KT-1035](#) - HF Resistant Kit for iCAP 6000 Duo

For samples containing hydrofluoric acid (HF), typically geological samples, soils and some alloys.

[KT-1031](#) - Standard Kit for iCAP 6000 Radial

For normal aqueous samples with relatively low (< 3%) levels of dissolved solids and no particulates.

[KT-1037](#) - Wear Metals Kit for iCAP 6000 Radial

This kit has been specifically configured for the determination of wear metals in lubricating oils.

[KT-1038](#) - Volatile Organics Kit for iCAP 6000 Radial

For samples containing volatile organic solvents such as alcohol, gasoline and naphtha. Includes the IsoMist Programmable Temperature Spray Chamber.

[KT-1040](#) - High Solids Kit for iCAP 6000 Radial

For samples containing up to 20% total dissolved solids. Suitable for sea water, ground water, brine, geochemical samples and fusions. Includes the Capricorn Argon Humidifier.

[KT-1039](#) - HF Resistant Kit for iCAP 6000 Radial

For samples containing hydrofluoric acid (HF), typically geological samples, soils and some alloys.



Figure 2. KT-1036, High Solids Kit for iCAP 6000 Duo

If you have a specific application which is not covered by one of these kits, please send details to enquiries@geicp.com and we will recommend the most appropriate system for you.

Additional Kits for the IsoMist Programmable Temperature Spray Chamber

We have extended the range of IsoMist Kits to cover more ICP-OES and ICP-MS models. Kits with quartz or PFA spray chambers are also now available for the most common ICP-MS models. Each kit contains the IsoMist module, spray chamber, torch interface and mounting bracket (if required). The currently available kits are as follows:

IsoMist Programmable Temperature Spray Chamber

[KT-1010](#) - IsoMist Kit for Agilent 7500

[KT-1044](#) - IsoMist Kit with Quartz Spray Chamber for Agilent 7500

[KT-1019](#) - IsoMist Kit for GBC Integra

[KT-1042](#) - IsoMist Kit for GBC OptiMass

[KT-1043](#) - IsoMist Kit for Leeman Prodigy Axial

[KT-1011](#) - IsoMist Kit for PerkinElmer Optima 4300/5300V

[KT-1012](#) - IsoMist Kit for PerkinElmer Optima 2000/4000/5000DV

[KT-1013](#) - IsoMist Kit for PerkinElmer Elan (Figure 3)

[KT-1051](#) - IsoMist Kit with Quartz Spray Chamber for PerkinElmer Elan

[KT-1015](#) - IsoMist Kit for Thermo iCAP 6000 Duo

[KT-1021](#) - IsoMist Kit for Thermo iCAP 6000 Radial

[KT-1018](#) - IsoMist Kit for Thermo Iris Axial/Duo

[KT-1020](#) - IsoMist Kit for Thermo Finnigan Element

[KT-1047](#) - IsoMist Kit with Quartz Spray Chamber for Thermo Finnigan Element

[KT-1023](#) - IsoMist Kit for Thermo (VG) PlasmaQuad

[KT-1025](#) - IsoMist Kit for Thermo X Series

[KT-1046](#) - IsoMist Kit with Quartz Spray Chamber for Thermo X Series

[KT-1014](#) - IsoMist Kit for Varian 700-ES or Vista Axial
[KT-1022](#) - IsoMist Kit for Varian 700-ES or Vista Radial
[KT-1017](#) - IsoMist Kit for Varian 800-MS
[KT-1052](#) - IsoMist Kit with Quartz Spray Chamber for Varian 800-MS
[KT-1024](#) - IsoMist Basic Kit ROL (for Horiba Jobin Yvon)
[KT-1027](#) - IsoMist Basic Kit LOL
[KT-1029](#) - IsoMist Basic Kit EROL (for Spectro)

IsoMist Programmable Temperature Inert PFA Spray Chamber

[KT-1028](#) - IsoMist Kit with Inert PFA Spray Chamber for Agilent 7500
[KT-1050](#) - IsoMist Kit with Inert PFA Spray Chamber for PerkinElmer Elan
[KT-1049](#) - IsoMist Kit with Inert PFA Spray Chamber for Thermo Finnigan Element
[KT-1048](#) - IsoMist Kit with Inert PFA Spray Chamber for Thermo X Series
[KT-1026](#) - IsoMist Kit with Inert PFA Spray Chamber for Varian 800-MS
[KT-1030](#) - IsoMist Basic Kit LOL with Inert PFA Spray Chamber (for PlasmaQuad)



Figure 3. KT-1013, IsoMist Kit for PerkinElmer Elan

Please email us at enquiries@geicp.com if your model is not listed.

INSTRUMENT NEWS

From Agilent Technologies:

HIGH MATRIX INTRODUCTION (HMI) ACCESSORY

Agilent Technologies has introduced the new "High Matrix Introduction (HMI) accessory" for the Agilent 7500 Series ICP-MS. Agilent's 7500 Series was designed to routinely handle TDS levels up to 0.3% TDS. In order to meet the TDS requirements of ICP-MS, higher matrix samples must normally be diluted prior to analysis. This dilution step has several disadvantages: increased influence of reagent blank, possibility of analyst error, and additional sample prep time. To avoid the drawbacks of conventional dilution,

Agilent has developed a new technique for the handling of high matrix samples - "aerosol dilution". The new High Matrix Introduction (HMI) accessory enables the 7500 Series to analyze 1% TDS samples directly – eliminating the dilution step and its associated drawbacks. Plasma robustness is significantly improved over conventional ICP-MS, greatly reducing matrix suppression and making the analysis of high matrix samples more reliable and accurate than ever before.

More information about the HMI accessory will be available on www.agilent.com/chem/icpms.

From Horiba Jobin Yvon:

NEW APPLICATION NOTE: GEOLOGICAL SAMPLES BY ACTIVA-M ICP-AES

This application note describes the analysis of several elements (Ba, Be, Co, Cr, Cu, Nb, Ni, Rb, Sc, V, Y, Zn and Zr) in geological samples, with the ACTIVA-M ICP-AES instrument. Analysis of geological samples requires an ICP-AES instrument with good resolution and robustness, to compensate for the variability of matrix and the influence of the major elements.

The material was dissolved by fusion with LiBO_2 in a Pt95Au5 crucible. The calibration was performed with 3 different certified reference materials (GSR-1 (Granite), GSR-2 (Andesite) and GSR-3 (Basalt)) from IGGE, China. A Kimberlite sample (SARM 39) was then analyzed to validate the analytical methodology. For the determination of low concentrations of Rb, a specific optimization was realized and is detailed.

The work was done on the ACTIVA-M ICP-AES spectrometer. For the determination of elements in geological samples, which are dissolved by alkaline fusion with LiBO_2 , it is strongly recommended to use a nebulizer suitable to high salt concentration, to provide freedom from clogging. We used the OpalMist nebulizer, which is made of PFA and is then resistant also to HF solutions.

For copy of the complete application note, please send your request to info@jobinyvon.fr.

From PerkinElmer:

UPDATED S10 AUTOSAMPLER FOR ATOMIC SPECTROSCOPY

PerkinElmer has announced an improved S10 Autosampler, a computer-controlled, multi-purpose sampling system for atomic absorption (AA), inductively coupled plasma-optical emission spectroscopy (ICP-OES) and inductively coupled plasma-mass spectrometry (ICP-MS). The autosampler automates standard and sample introduction for instrument calibration and sample analysis, extending the

spectrometer's capabilities to those of a fully automated analytical workstation.

The new instrument features an advanced drive system with high-precision motors that move the sampling arm of the S10 in the X and Y coordinates simultaneously to minimize changeover time between samples. It includes random-access programming for flexibility and more than 200 vessel positions for maximum capacity. Easily interchangeable sample racks allow for faster analysis setup.

For more information, visit www.perkinelmer.com/s10.

From SPECTRO:

SPECTRO CAMPUS ONLINE: SPECTRO ADDS A FREE E-LEARNING PLATFORM TO ITS NEW WEBSITE

SPECTRO Analytical Instrument's new website is now online at www.spectro.com. The amount of information available has been greatly increased and has become more interactive with this relaunch: In addition to a large amount of specialized applications and product information, visitors to the website now find an e-learning section – the SPECTRO Campus.

"User behavior in Internet has changed a lot in the last few years. The Web is not just for reading anymore. Users want more than just dry information, they want material that is presented well and they want to become actively involved," says Tom Milner, Marketing Director at SPECTRO. Using multimedia presentations, the analytical company's new website now makes complex interactions easier to understand. "We placed a large emphasis on interactivity. The e-learning seminars give our visitors the opportunity to deepen and test their knowledge," continues Milner.

SPECTRO has made three interesting online seminars dealing with basic analytical theory and practice available to customers and other interested parties in this secure portal. There are basic courses for X-ray fluorescence and optical emission spectroscopy as well as a course for the new SPECTRO ARCOS high-end ICP spectrometer.

Interactive e-learning in the SPECTRO Campus is not the only innovation in SPECTRO's website: The analytical company's relaunch also includes the addition of many new application reports in its free-of-charge download platform. Information packages specific to various industries are also available; dealing with the analysis of fuels in the petrochemical industry or RoHS conform analysis in electrical and electronics production companies, for example.

From Thermo Fisher:

THERMO FISHER SCIENTIFIC SHIPS ITS 500TH iCAP 6000 ICP EMISSION SPECTROMETER TO BOEING

Thermo Fisher Scientific Inc. is pleased to announce that the 500th iCAP 6000 ICP emission spectrometer produced at its facility in Cambridge, UK has been shipped to Boeing. The instrument has been purchased by Boeing for use at its Frederickson facility in Washington State, USA. It will be used in the quality assurance laboratory to monitor the contents of chemical tanks used in the production of aluminum wings and other structural components. The Thermo Scientific iCAP 6000 will be analyzing traces of metals including copper and iron in the tanks, which range in capacity from 34,000 to 59,000 gallons. This analysis is indicative of the "health" of the tanks and is a key step in the QA processes.

Paul Gillyon, product group director of Elemental Analysis, Thermo Fisher Scientific, comments: "We are extremely proud to have Boeing as our 500th customer and I am sure that the iCAP 6000 will provide excellent service in their laboratory for many years".

For more information on the Thermo Scientific iCAP 6000 Series of ICP emission spectrometers, please email analyze@thermofisher.com or alternatively visit www.thermo.com/elemental.

From Varian:

NEW VERSION OF VARIAN ICP EXPERT™ II SOFTWARE NOW AVAILABLE

A new version of ICP Expert II software for Varian 700-ES series and Vista-PRO ICP optical emission spectrometers is now available. Version 1.1.1 software includes many new features, especially for Vista-PRO users. ICP Expert II now supports operation of the A.i. Scientific AIM3600 high capacity autosampler, which can hold 630 sample and 22 standard/QC solutions. The AIM3600 accommodates up to four separate rinse stations, making it ideal for analyzing sample batches of varying matrix. Autosampler travel time is also reduced, improving productivity. Comprehensive multimedia Help plus efficient semi-quantitative worksheets ensure you are quickly up and running.

ICP Expert II is also available to pharmaceutical laboratories operating under 21CFR11 environments including full validation (IQ/OQ) and re-validation (OQ) options.

ICPEXPERT II includes a range of performance and productivity enhancing options:

- Automax auto-optimization wizard eliminates manual optimization, providing fast, automated, method

optimization

- Smart rinse increases washout efficiency and analysis productivity
- MultiCal extends the linear range allowing measurement from parts-per-billion to percent levels in a single read
- Fitted background correction provides real-time background correction for fast and simple method development
- F.A.C.T allows real-time spectral deconvolution, solving spectral interference problems in difficult samples.

To read more about ICP Expert II, click on the following link
<http://www.varianinc.com/products/spectr/icpoes/software/index.htm>

GLASS EXPANSION NEWS

WINTER CONFERENCE

A wide selection of Glass Expansion products will be on display at the 2008 Winter Conference on Plasma Spectrochemistry, Temecula, California, USA, January 7 - 12, 2008. The display will include nebulizers, spray chambers, torches, RF coils and accessories (including the new IsoMist Programmable Temperature Spray Chamber). Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP.