GLASS EXPANSION NEWSLETTER

Quality By Design

June 2007

CONTENTS

APPLICATION SPOTLIGHT

Applications of a Temperature Controlled Spray Chamber

INTRODUCTION

We have known for many years that the temperature of a sample introduction system has a profound effect on the performance of an inductively coupled plasma optical emission spectrometer (ICP-OES) and an inductively coupled plasma mass spectrometer (ICP-MS). We use cooled spray chambers to reduce the volatility of certain solvents so that the plasma is sustained. We know that heating a spray chamber results in higher sample transport. We know that controlling the temperature of a laboratory can be critical to long-term stability. Yet only a small percentage of the instruments in the field either monitor or control the temperature of the sample introduction system.

The IsoMist™ Programmable Temperature Spray Chamber is a convenient and universal device for both controlling and monitoring spray chamber temperature (see Figure 1). Its characteristics are listed below:

- It uses a solid-state Peltier device to control temperature in the range of –10°C to +60°C in 1 degree increments.
- It incorporates a baffled Twister™ cyclonic spray chamber which is encapsulated in a conductive material for intimate contact with the Peltier heat transfer block (see Figure 2). This enables it to go from room temperature to –5°C in less than 15 minutes.
- Configurations are customized for each make and model of ICP-OES or ICP-MS.
- The IsoMist is controlled through a standalone software application with a single interface screen through which spray chamber temperature can be set and monitored.
- Communication to the IsoMist is performed either by a standard USB cable or through wireless BlueTooth® technology. Once programmed, a PC connection is not required.

Figure 1. IsoMist Programmable Temperature Spray Chamber

Figure 2. Encapsulated Twister Spray Chamber

Because of its unique features, the IsoMist can be used to facilitate a number of applications, two of which are described below.

DIRECT ANALYSIS OF VOLATILE SOLVENTS

Volatile organic solvents can be troublesome due to the high transport efficiency to the plasma which creates excessive load and typically results in plasma instability or, in the worse case, cessation. One of the most difficult commonly analyzed solvents is light naphtha. The petrochemical industry is interested in the metal content of naphtha for many reasons. These metals can interfere with the "cracking" process during the refining of oils. They can also cause serious degradation of the expensive catalysts used in the process. Furthermore, there are environmental concerns related to the release of metals during combustion. And lastly, the specific metal or metals found and their relative concentrations are informative markers in the determination of both the origin and migration of oil reserves.

Because of the high volatility of light naphtha, it is usually diluted a factor of 10 with a more "friendly" solvent such as kerosene or xylene. However, analysts would often like to reach lower detection limits than this dilution will allow. The IsoMist run at –10°C allows the direct analysis of light naphtha without dilution.

Instrument Conditions

This work was performed on a Perkin-Elmer Optima 2100DV using the axial mode and the conditions listed in Table I.

Forward Power	1500Watts
Coolant Flow	20L/min
Auxiliary Flow	1.8L/min
Nebulizer Gas Flow	0.35L/min
Injector	1mm capillary bore
IsoMist Temperature	-10°C
Nebulizer	SeaSpray™ glass concentric
Sample Uptake Rate	0.3mL/min

Table I. ICP-OES parameters for the analysis of naphtha.

Several of the instrument parameters invite explanation. The nebulizer gas flow was significantly lower than that commonly used for this nebulizer in order to decrease sample transport. The torch injector had a small 1mm bore for similar reasons. The IsoMist temperature of –10°C was required to achieve stability. A temperature as close as –5°C was not low enough. The sample uptake rate was critical and higher rates resulted in instability.

The IsoMist mounts on the ICP as shown in Figure 3.

Figure 3. IsoMist mounted on the Optima 2100DV

Sample Preparation

Blanks and standards were made from 100% kerosene and S-21 Conostan organometalic standards (ConocoPhillips, Ponca City, OK). Samples were 100% naphtha. All blanks, standards, and samples were spiked with 0.5ppm cobalt as an internal standard in the form of an organometalic (also Conostan).

Results

To measure the reproducibility of the technique, a naphtha sample was twice analyzed for 11 elements 90 minutes apart. The results are shown in Figure 4. With the exception of phosphorus, reproducibility was very good. Phosphorus demonstrated severe instability in naphtha.

The sample was spiked at a level of 0.1ppm for all metals monitored and the results are shown in Figure 5.

Figure 5 shows good recoveries for all elements except P, Ni, and Pb. Phosphorus was discussed above. Nickel and lead yielded poor recoveries suggesting that the response in naphtha is very different from the response in kerosene, so much so that internal standardization was inadequate. Although further investigation is under way, it may be that the forms of nickel and lead used are rendered more volatile by the naphtha matrix resulting in premature losses of the metals.

ANALYSIS OF LIMITED SAMPLE VOLUMES

Sample size can be quite small for many ICP applications. Clinical and pharmaceutical laboratories are often faced with the daunting task of achieving trace measurements of several metals in small samples such as neonatal blood or serum. Forensic labs must sometimes deal with micro samples which can be traced through their metal content. Another application where micro sampling is desirable is in the analysis of radioactive samples. Here the goal is to minimize or eliminate waste due to the expense of disposal.

Typical ICP methods use sample uptake rates of 0.5 to 2ml/min., which is unsatisfactory for micro samples. Therefore, micronebulization is recommended. However, sensitivity and hence limit of detection are often unavoidably sacrificed with this technique.

We used a MicroMist™ nebulizer configured to run at an uptake rate of 20ul/min. and compared the results taken at different temperatures using the IsoMist. All instrument conditions and gas flows and pressures were standard. Figure 6 compares the relative sensitivity at 3 different temperatures for 17 different analytical wavelengths. On average, sensitivity is enhanced by a factor of 2 to 2.5 by going from 21°C to 40°C and a factor of 3 to 3.5 at 60°C.

We also evaluated the effect of temperature on detection limit using the same conditions as above and the results are shown in Figure 7. In this case the normalized value is the detection limit obtained at 2 ml/min uptake at 21°C. Plotted against this are the results obtained at 20ul/min uptake at both 21°C and 60°C. The results at 21°C represent the type of degradation in detection limit expected using micronebulization and range between a factor of 5 and 10. The results at 60°C demonstrate a degradation of on average only a factor of 2. To put this in perspective, we used an uptake rate of 1% of the standard uptake rate yet sacrificed only a factor of two in performance.

SUMMARY

Controlling the temperature of an ICP sample introduction system has numerous benefits. This paper illustrates two such benefits, one at low temperature and another at elevated temperature. Additional applications of this device will be discussed in future issues of the newsletter.

Figure 4. Reproducibility of naphtha results taken 90 minutes apart.

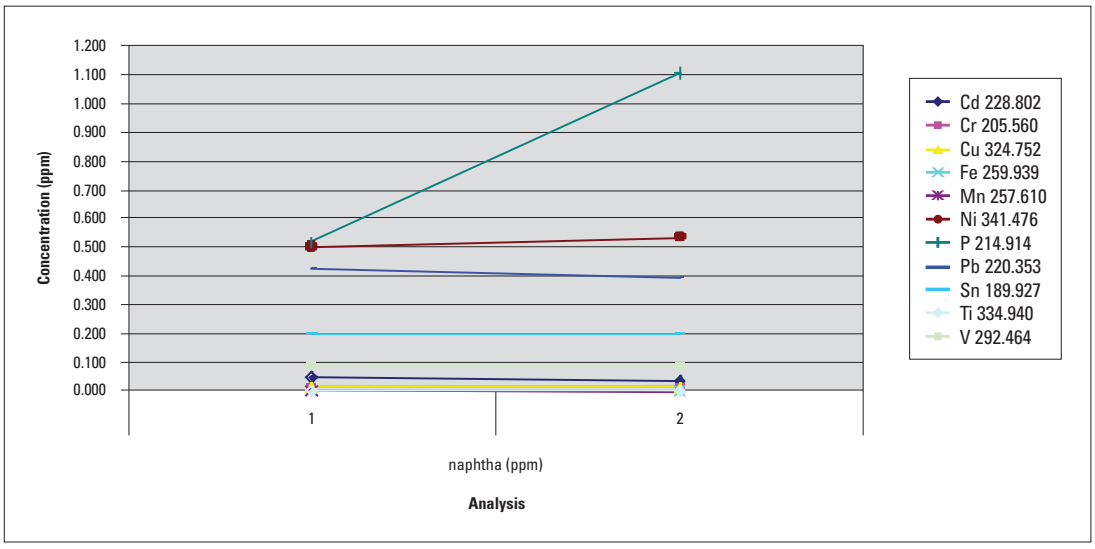


Figure 5. Recovery of spiked metals in naphtha.

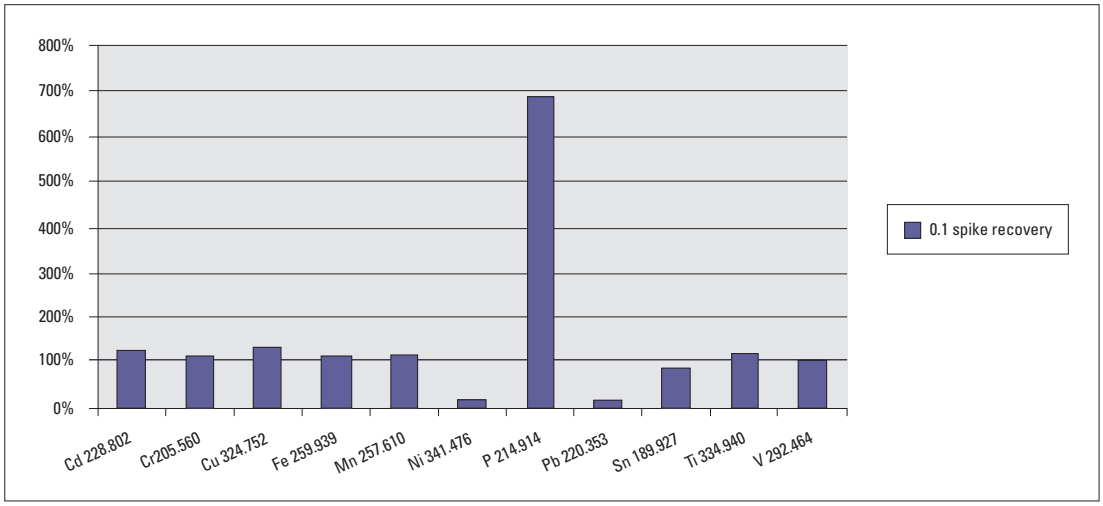


Figure 6. Sensitivity comparison of 20ul uptake rate at different temperatures.

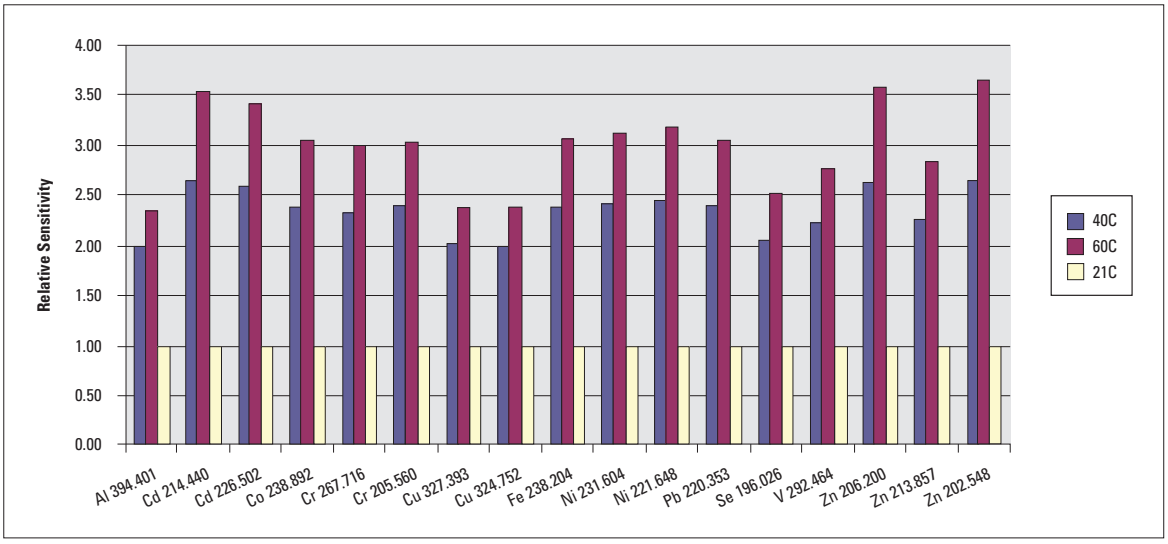
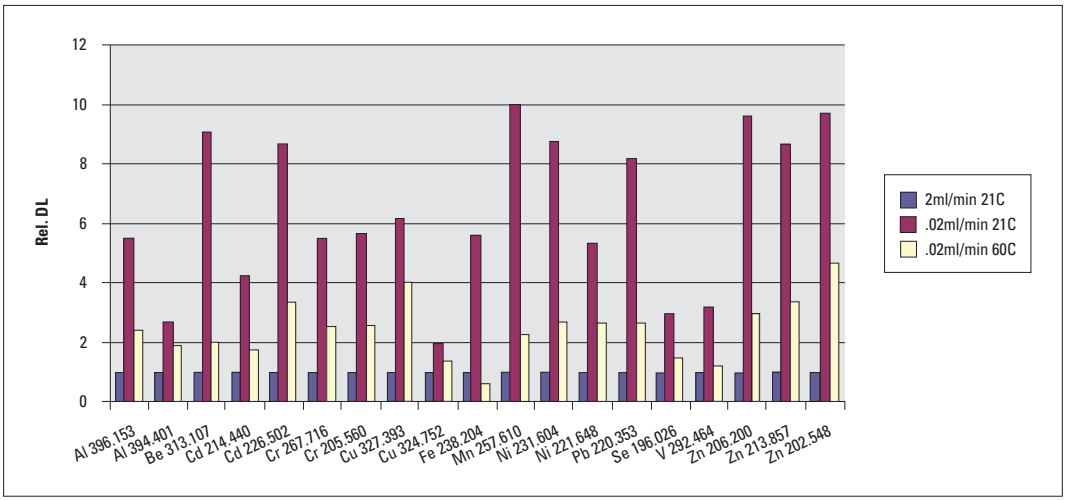


Figure 7. Detection limit comparison at different temperatures.



INSTRUMENT NEWS

From Agilent Technologies:

SIMPLER. FASTER. MORE ACCURATE

Agilent Technologies has introduced the new 7500cx model with Octopole Reaction System (ORS). The 7500cx removes interfering species using only inert helium gas (He mode). He only mode simplifies operation and vastly improves speed and accuracy under a single set of operating conditions. The Agilent 7500cx also allows the user to achieve the full potential of ICP-MS for semiquantitative elemental screening of a wide range of sample types. Complex, unknown samples can be analyzed with better speed, accuracy, and data integrity than ever before, since all matrix interferences are removed in the ORS using helium collision mode. More information about the Agilent 7500 Series ICP-MS can be found at www.agilent.com/chem/icpms.

From PerkinElmer:

SCIEX™ ELAN® v3.4 SOFTWARE

PerkinElmer's new SCIEX™ ELAN® v3.4 software for ICP-MS instrumentation has fully automated features to help increase consistency of performance and efficiencies in laboratories with high-productivity demands, including environmental, clinical and geochemical laboratories, and laboratories in the food, nutrition and semiconductor industries.

It features enhanced productivity tools such as a new Scheduler that allows laboratories to pre-set daily performance checks and to automatically load samples and conduct unattended testing procedures, eliminating the need to manually schedule each test. Procedures such as auto start and warm-up can be set up to run over night, or early in the morning, resulting in significant time and

cost savings for laboratories. Automated tests also help improve data reliability due to the reduction in operator intervention.

The software also includes enhancements to PerkinElmer's patented Dynamic Reaction Cell™ (DRC) technology with new fully automated method development optimization that minimizes analysis time for users. It also simplifies DRC method development by integrating a script, to determine the appropriate testing process. Also included is an improved, user-friendly SmartTune feature for unattended multi-parameter optimization of the ICP-MS instrumentation. A SmartTune window added to the workspace offers improved layout, expanded and improved functionality and more comprehensive and consistent data results.

From Spectro:

ARCOS - THE ULTIMATE ICP-OES FOR SAMPLE THROUGHPUT AND SENSITIVITY

Its name tells it all: ARCOS (Advanced Rowland Circle Optical System). The new optics offer up to 2-times better resolution, making it the ideal ICP-OES for line-rich matrices and for ultra-trace analysis.

The SPECTRO ARCOS is especially suited for such demanding analytical applications as ultra-trace analysis for environmental applications or line-rich spectra for metal applications or the analysis of organic materials in petrochemistry. The new instrument's high performance results from the two completely new major components: the optics and the generator.

In the optic, SPECTRO utilizes 32 linear CCD detectors in an optimized Paschen-Runge mount ORCA (Optimized Rowland Circle Alignment) for the simultaneous recording of the wavelengths between 130 and 770 nm. However, what makes the optic so special is the enormous focal length of 750 mm, which improves the resolution by a factor of two. Coupled with the higher resolution is an improvement in the signal to background ratio, which has also been bettered by a factor of up to 2.

The other major development in the new SPECTRO ARCOS is its generator. Here too, major technological advances are combined that result in significant economic improvements. The generator uses a tube with a maximum input power of 5 kilowatts; however, the maximum load during an analysis is only 1.7 kilowatts. There are enough high-power reserves for even peak loads such as for the analysis of volatile organic materials, so that the generator has an excellent lifetime. The low-temperature ceramic tube is forced air cooled by an internal ventilation system, eliminating the need for uneconomical liquid coolants.

NEW PRODUCTS

Kits for the IsoMist Programmable Termperature Spray Chamber

We have established an IsoMist Kit for common ICP-OES and ICP-MS models. Each kit contains the IsoMist module, encapsulated Twister spray chamber, torch interface and mounting bracket (if required). Please select the kit to match your ICP from the following:

KT-1010 - IsoMist for Agilent 7500
KT-1011 - IsoMist Kit for Perkin Elmer Optima 4300/5300V
KT-1012 - IsoMist Kit for Perkin Elmer Optima 2000/4000/5000DV
KT-1013 - IsoMist Kit for Perkin Elmer 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 Duo
KT-1020 - IsoMist Kit for Thermo Finnigan Neptune
KT-1025 - IsoMist Kit 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-1019 - IsoMist Kit for GBC Integra

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

HINTS FOR THE OPERATOR

Care and Maintenance of Inert Spray Chambers

Glass is the most common material used for ICP spray chambers. It is cost-effective, impervious to most samples and provides excellent analytical performance. However, there are some applications for which glass is unsuitable – samples containing HF for example, or when determining elements such as B and Si where the glass spray chamber may contribute to the background signal. For these applications, inert polymer materials are often used. Materials such as PTFE and PFA provide the required purity and chemical resistance but historically have not provided very good analytical performance. This is because these materials do not wet evenly and therefore do not provide consistent drainage.

In 2006, Glass Expansion released a new range of PTFE and PFA spray chambers which have an internal surface that is specially treated to ensure that it wets evenly and provides consistent drainage. These spray chambers provide excellent analytical performance, similar to that provided

by the glass spray chambers. The treatment turns the surface a characteristic brown colour. It should be noted that the treatment actually changes the molecular structure of the PTFE and PFA. It is not a coating and it does not introduce any potential contaminants.

While the surface treatment is long lasting, operators should be aware that it may degrade after prolonged use. The lifetime of the treated surface depends on the type of samples used and could range from several months to several years. To ensure that you get the best performance from your PTFE and PFA spray chambers, we recommend the following:

- Do not use H_2O_2 for cleaning the spray chambers as this will accelerate the degradation of the surface.
- Do not make physical contact with the chamber interior surface with any instrument, including your hands or a brush.
- Do not be concerned if the brown colour fades over time. This is normal and does not necessarily lead to a degradation in performance.
- If you notice a degradation in performance (such as poorer precision or detection limits), then clean the spray chamber with Fluka 'RBS-25'. In the first instance, aspirating a 2.5% Fluka solution for 15 minutes will probably be sufficient to recover the performance. However, if this is not effective, the spray chamber should be soaked overnight in a 25% Fluka solution.
- Eventually the surface may degrade to the point where it does not recover after soaking in Fluka. At this point the spray chamber needs to be returned to Glass Expansion where the surface can be re-treated for a nominal cost.

Please contact enquiries@geicp.com if you have any questions regarding the care of your Tracey TFE or Tracey PFA44 spray chamber.

GLASS EXPANSION NEWS

NEW 2007 CATALOG NOW AVAILABLE

The new Glass Expansion catalog is now available. This 140-page full color catalog lists nebulizers, spray chambers, torches, RF coils, ICP-MS cones, accessories and consumables for over 70 ICP-OES and ICP-MS models. If you would like your personal copy, please send your mailing address to enquiries@geicp.com and we will rush a copy to you.

JAIMA SHOW 2007

A full range of Glass Expansion products will be on display at the JAIMA Show, Tokyo, Japan, August 29 to 31, 2007, Booth 6-305 and Glass Expansion specialists will be on hand to assist you.

FACSS 2007

Our products will also be on display at the 34th FACSS Conference, Memphis, TN, USA, October 14 to 18, 2007, Booth 42. You are invited to visit our booth and discuss your ICP application with our specialists.