NEWS

Glass Expansion Newsletter • June 2009 • issue 19

APPLICATION SPOTLIGHT

ADVANCING QUALITY CONTROL IN ICP SPECTROMETRY

Introduction

Both inductively coupled plasma optical emission spectrometry (ICP-OES) and inductively coupled plasma mass spectrometry (ICP-MS) have become workhorses in today's inorganic laboratories. Most parameters of the spectrometer are tightly controlled and easy to monitor. For example, the detectors employed are often temperature controlled. Gases used are pressure and/or flow regulated. The RF power supply has a built-in feedback loop, etc. However, there are two areas of the sample introduction system where recent advances have enhanced the control of quality for these spectrometers and these are the subject of this article. Figure 1 is a schematic of the ICP sample introduction system. The sampler and skimmer cones are present only for the ICP-MS, otherwise the sample introduction systems for ICP-OES and ICP-MS are in essence the same.

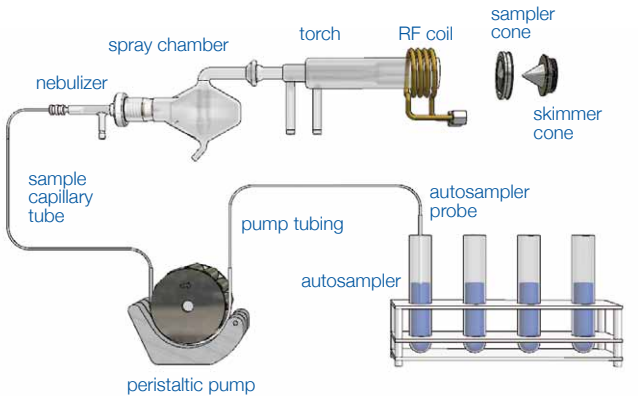

Figure 1 - Schematic of a typical sample introduction system for an ICP spectrometer.

Sample Uptake Rate

Sample is delivered to the nebulizer of the sample introduction system typically via a peristaltic pump. The operator chooses the appropriate pump tubing internal diameter and pump speed to achieve the desired sample flow rate. This is typically between 1 and 2 mL/min for ICP-OES and somewhat lower for ICP-MS. In order to achieve the best analytical performance, sample delivery must be consistent over both the short and long term. Failure to consistently deliver sample shows up in terms of

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GLASS EXPANSION
Quality By Design

KEY EVENTS

JAIMA SHOW 2009

A full range of Glass Expansion products will be on display at the JAIMA Show, Tokyo, Japan, September 2 to 4, 2009 and Glass Expansion specialists will be on hand to assist you.

ICASS CONFERENCE 2009

Glass Expansion is sponsoring the atomic spectroscopy symposium at the ICASS conference in Kingston, Ontario, Canada, August 9-12, 2009. Jerry Dulude of our US office will be presenting a paper in that session entitled, "The Effect of Spray Chamber Temperature Control on Environmental Analyses by ICP"

GE NEWS

NEW 2009 CATALOG

If you do not yet have your copy of the 2009 Glass Expansion catalog, please send your mailing address to **enquiries@geicp.com** and we will send you a copy immediately.

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poor precision and inaccuracy but may not become evident until a QC sample is measured. Potential causes of this failure are as follows:

- Worn pump tubing; as the pump tubing wears its compressibility degrades and the sample uptake rate slows and becomes inconsistent.
- Incorrectly adjusted pressure on the tubing clamp; a peristaltic pump includes a clamp which places a variable pressure on the tubing that is wrapped around the rollers. This optimum pressure is that which is high enough to completely close the tubing when it is between the clamp and roller but not so high that it will shorten the lifetime of the tubing.
- Worn pump rollers; as a peristaltic pump is used the rollers may with time develop a low spot. A low spot on any roller will cause erratic sample delivery.
- Clogged nebulizer; when the sample line of a nebulizer becomes clogged either from an errant particle or salt build up, it produces significant back pressure and reduces the sample uptake rate.

Figure 2 – TruFlo in-line sample flow monitor

A sample uptake monitor is now available that can measure and record the sample uptake in real time and display the value both digitally and graphically. Known commercially as the TruFlo™ (Glass Expansion, Melbourne, Australia), the device uses a thermoelectric sensor to cover the range of 0.05 to 4.0 mL/min (Figure 2). The TruFlo tells the operator instantaneously when the sample uptake rate deviates significantly from the desired level. In fact, a range can be programmed so that an alarm is triggered when the sample uptake falls outside the range (Figure 3). Figure 4 shows the effect of pump clamp tension on signal. Note that there is an optimum tension for maximum flow and minimum pump noise. Additional tension will only prematurely wear the tubing and may even produce a lower sample flow. Figure 5 plots the relationship between pump speed and sample uptake rate. As expected, pump speed is proportional to uptake rate.

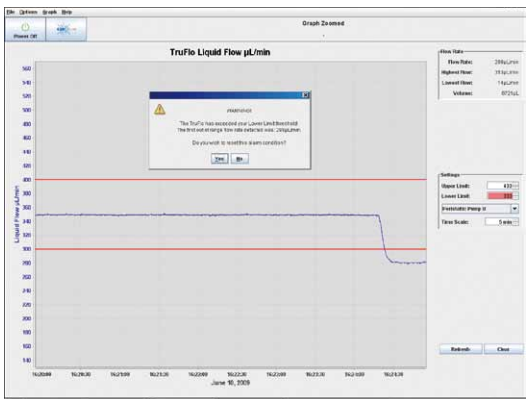


Figure 3 – TruFlo screen display showing an out of range shift in sample flow rate

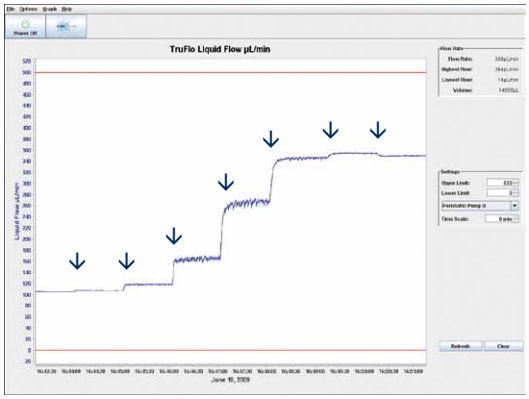


Figure 4 – TruFlo screen display showing the effect of tightening the pump tubing clamp. The clamp was tightened by 1/2 turn at each arrow

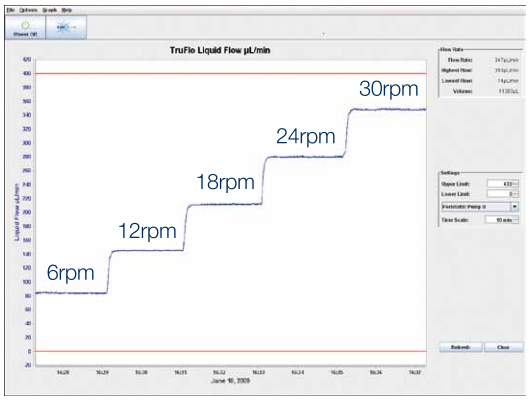


Figure 5 – TruFlo screen display showing the relationship between pump speed and sample flow rate

Spray Chamber Temperature

The temperature of the spray chamber in an ICP spectrometer has a direct influence on the sample transport and the signal stability. However, even though the detectors are thermally controlled, the spray chamber temperature (in many cases) is incorrectly assumed to be constant. The importance of spray chamber temperature control is more accepted for ICP-MS for two reasons. First, optimizing both the spray chamber temperature and the nebulizer gas flow allows oxide interferences to be minimized[1]. Also, stabilizing the spray chamber temperature for an ICP-MS is generally accepted to significantly improve precision. Historically, application of a controlled temperature spray chamber for ICP-OES has been restricted to reducing the plasma load when analyzing volatile solvents[2-4]. It is likely that this restriction is due to the complexity and expense of adding a temperature controlled spray chamber. Traditionally, this involved the acquisition and installation of an external chiller in combination with a jacketed spray chamber (Figure 6). Recently, a free standing Peltier driven device has been introduced which significantly simplifies temperature control. The IsoMist™ Programmable Temperature Spray Chamber (Glass Expansion, Melbourne, Australia), also shown in Figure 6, has a range of -10 to 60°C in 1°C increments and a stability of +/- 0.1°C.

The applications of a temperature controlled spray chamber are diverse and have been described elsewhere[5]. From a quality control viewpoint, the ability to both control and monitor the spray chamber temperature provides an added layer of confidence in the analytical results. Figure 7 demonstrates the signal drift of an ICP-OES system with and without temperature

Figure 6 – Two ways to control the temperature of the spray chamber; (a): jacketed spray chamber plus external chiller; (b): IsoMist Programmable Temperature Spray Chamber

control. Note that the drift range has been reduced from 6% to less than 1%. The benefit of this improvement comes into play in a number of applications. For environmental analyses, the added stability means reducing QC failures and decreasing the number of standardizations. For precious metal assays, it means higher accuracy, which translates into higher profits. With the IsoMist, a plot of measured temperature vs. time can be displayed and saved as a record of performance during the sample run (Figure 8).

Optimization of the sample mass transport is another advantage to controlling the temperature of the spray chamber. Figure 9 shows the effect of spray chamber temperature on ICP-OES signal for a wide range of element lines. Note the large and varied effect of temperature on the individual lines selected. In the most dramatic case (Ca317.9), 1°C results in a 5% change in signal, while in the least dramatic case (Al396.1), 1°C causes only a 2.4% change in signal. The conclusions are threefold as follows:

- Spray chamber temperature is a critical parameter.
- Due to the varied effect of temperature, internal standardization may not be an accurate means of compensation.
- To achieve the best detection limits, the instrument should be operated at the maximum mass transport that will sustain a robust plasma.

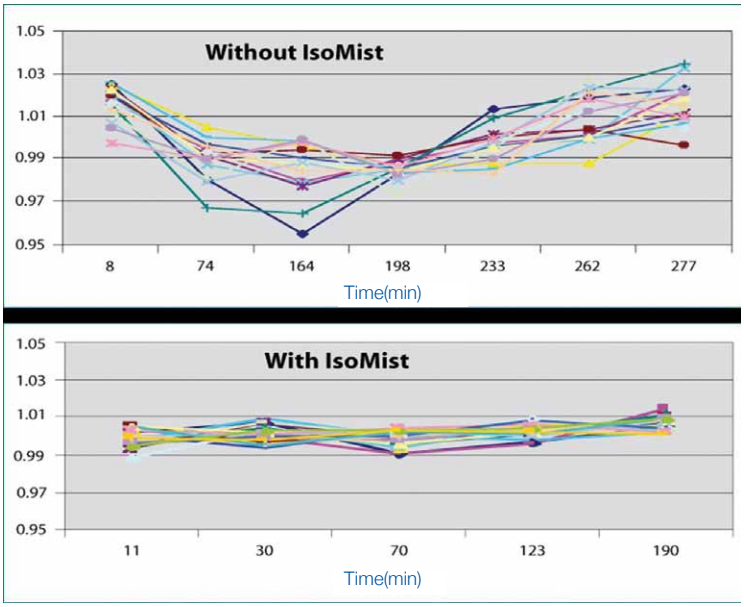


Figure 7 – Effect of spray chamber temperature control on signal drift in ICP-OES

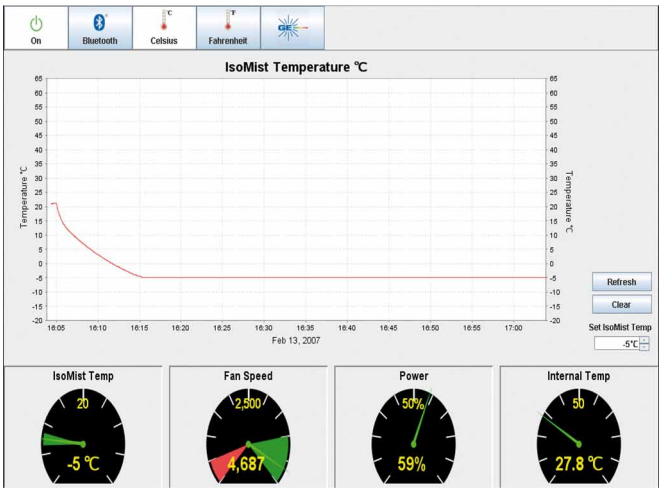


Figure 8 – IsoMist screen display showing recordable plot of temperature vs. time

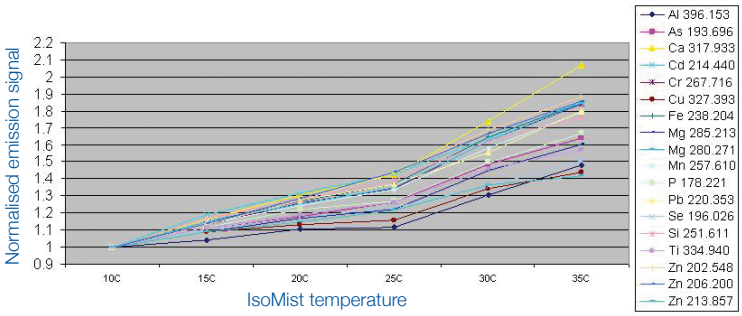


Figure 9 – Effect of varying the spray chamber temperature on signal intensity by ICP-OES (1mL/min uptake rate)

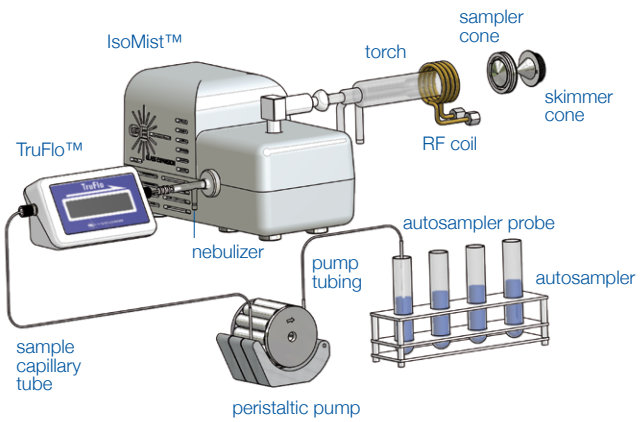


Figure 10 – Schematic of a sample introduction system with both TruFlo and IsoMist

Summary

The control of two additional parameters adds even more credibility and robustness to ICP spectrometry as an analytical tool. Figure 10 illustrates the ICP-MS system showing the positioning of both the TruFlo and IsoMist. Both sample flow rate and spray chamber temperature have profound effects on the performance of this technique. In the first instance, a sample flow monitor assures the operator of acceptable performance and provides a digital graph of the performance over the length of the sample run. In the second case, a programmable temperature spray chamber not only monitors but controls the temperature of the spray chamber to achieve optimum, consistent, and reproducible performance.

This article is based on one published in *Canadian Laboratory Product News* (December 2008, Vol. 38, No. 7) for the section on Test & Measurement and was modified for this newsletter.

NEW PRODUCTS

U-series nebulizer

The U-Series™ is a new style of glass concentric nebulizer. Instead of the EzyFit™ sample tube connector, it has a UniFit type of sample tube connector. The benefits of the U-Series are:

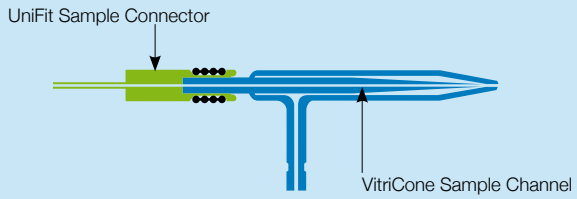
- The sample channel is uniform from the entry point to the tip. This helps avoid blockages since there is nowhere for particulates to be trapped.
- Since there is nowhere for sample to be trapped, washout time is reduced and sample throughput increased.
- The UniFit connector is even easier to use than the EzyFit connector.

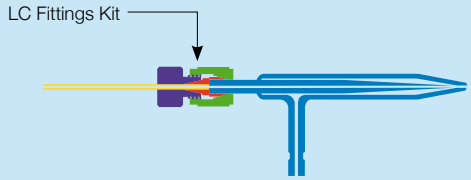
SeaSpray, Conikal, MicroMist and Slurry U-Series nebulizers are available to suit all common models of ICP-OES and ICP-MS.

The part numbers for U-Series nebulizers are the same as those for nebulizers with EzyFit, except that the letter “F” is replaced by the letter “U”. Examples of U-Series part numbers are AR30-07-USS2E for SeaSpray, AR35-1-UM04E for MicroMist, AR40-1-UC3E for Conikal or AR30-07-US6E for Slurry.

For speciation measurements, the U-Series nebulizer can be quickly and easily connected to an LC using the LC fittings kit, part number FT-16-8.

You can find information on the U-Series nebulizers in our 2009 catalog and price details are available on our website. Or contact **enquiries@geicp.com** for advice on the best U-Series nebulizer for your application.





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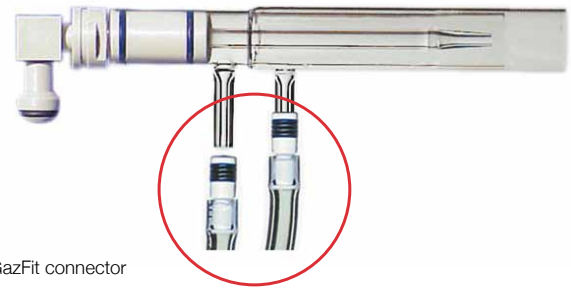
3. S. J. Hill, J. Hartley, and L. Ebdon, *J. Anal. At. Spectrom.* 7, 23-28 (1992).
4. A. S. Al-Ammar, R. K. Gupta, and R. M. Barnes, *J. Anal. At. Spectrom.* 14, 793-799 (1999)
5. J. Dulude, R. Stux, and V. Dolic, Pittcon, *Paper #1370* (2007).

HINTS FOR THE OPERATOR

Using GazFit connectors

Since the GazFit™ connector expands slightly to fit the torch side arm and there is a small variation in the diameter of the side arms, the GazFit connector may appear loose when it is moved from one torch to another. If this is the case, let the GazFit connector rest for 24 hours and it will relax to its original diameter. This process can be speeded up by putting the GazFit connector in hot water. The connector may also deteriorate or harden over time due to exposure to strong UV light from the plasma. If the connection is still loose after the hot water treatment, replace the GazFit connector with a new one.

Make sure you have the correct sized GazFit for your torch. GazFit connectors are available to suit 8mm, 6mm, 5mm and 4mm side arms.



GazFit connector

New EzyLok connector

We have found that, if there is acid in the nebulizer when the argon is turned off, it is possible for it to leak into the argon line and we had some reports of the EzyLok™ connector getting corroded. For this reason, we have changed the material of the connector from Delrin (black) to acid-resistant Ertalyte (white). Replacement connectors are available as part number 70-808-0735, EzyLok 6mm hose adaptor.

EzyLok connector

INSTRUMENT NEWS

From Agilent Technologies – Next generation ICP-MS offering unmatched data integrity, ease of use, smallest footprint

Agilent Technologies has introduced the 7700 Series ICP-MS. This successor to the world's best-selling ICP-MS, the Agilent 7500 Series, provides unmatched data integrity, is simple to operate and occupies the smallest amount of bench space by far of any ICP-MS instrument.

The most obvious feature of the new 7700 Series is its small size, occupying just 73 cm of bench space. Mainframe footprint is more than 30% smaller than any other ICP-MS. Despite its small footprint, the 7700 Series sets a new standard for ICP-MS performance and ease-of-use in a rugged package.

Collision/reaction cells have been adopted as the industry-standard technology for removing spectral interferences in ICP-MS. Agilent's Octopole Reaction System (ORS), featuring helium collision mode, provides the most reliable and effective interference removal, especially for complex and unknown sample types. The 7700 Series features a new, third generation cell (ORS3) which further improves the efficiency of helium mode, eliminating the need to use reaction mode (with reactive cell gases such as hydrogen) in almost all applications. This simplifies operation and eliminates false positives that frequently occur with reaction mode, thereby improving data quality.

The new collision/reaction cell, plus Agilent's unique High Matrix Introduction technology which is standard on the 7700, make the instrument particularly well suited for complex sample types such as food, wastewater and soil digests, while a redesigned ion lens improves sensitivity and reduces background noise across the mass range.

From PerkinElmer – Nine new analytical platforms available for biodiesel and bioethanol laboratories

PerkinElmer has announced a major expansion of its EcoAnalytix™ biofuels analytical solutions portfolio. The Company has expanded its biodiesel and bioethanol portfolio to include nine analyzers and systems, crossing six technologies: ICP-optical emission spectroscopy (ICP-OES), gas chromatography (GC), liquid chromatography (LC), infrared (IR), differential scanning calorimetry (DSC), and liquid scintillation counters (LSC).

A full suite of systems for testing bioethanol, a sugar-based fuel made from such crops as sugar-cane, corn or wheat, is available. This line consists of an EcoAnalytix Trace Metals Analyzer based on the Optima™ 7000 ICP-OES for testing Group I and Group II metals and phosphorus, Alcohol system for determining the right blend of ethanol in gasoline by GC, and Bioethanol Fermentation HPLC system for monitoring fermentation broth.

Both the biodiesel and bioethanol platforms also include PerkinElmer's LABWORKS™ greenLIMS™, a pre-configured software application specifically for the biodiesel industry, as well as consumables, application notes, methods, standard operating procedures (SOPS) and onsite training. All biofuels analyzers, instruments and configured systems are available globally for purchase. For more information visit **www.perkinelmer.com/biofuels**.

From Spectro – Smart Analyzer Vision 4.0: New software for ARCOS and GENESIS ICP-OES instruments

SPECTRO Analytical Instruments introduced its new SMART ANALYZER VISION 4.0 system software for the SPECTRO ARCOS and SPECTRO GENESIS ICP-OES instruments at Pittcon 2009. SMART ANALYZER VISION has been designed along the lines of the current Microsoft software environment to enable intuitive operation. The new release guarantees users simplified operation despite its greatly increased range of functions; making it easy for new operators to become acquainted and maximize their SPECTRO ICP-OES analysis output.

SMART ANALYZER VISION's graphical interface is based on the familiar design of the Office group of products: SMART ANALYZER VISION uses a central navigation bar with six job oriented views. Users can open each view with a single mouse click on the navigation bar; the instrument functions are then immediately available for use: For example, automatic and manual analysis, spectral viewing or method development.

NEW PRODUCTS

D-Torch for PerkinElmer Optima 2/4/5/7000 DV

The D-Torch is a new demountable torch design that provides the benefits of a fully demountable torch at a significantly lower cost. Interchangeable outer tubes made of quartz or ceramic are available. The ceramic outer tube has a much longer lifetime, greatly reducing interruptions and downtime due to torch failure. It is also of particular benefit for the determination of elements such as Si which suffer from high background levels with quartz torches.

The D-Torch is now available for the PerkinElmer Optima 2/4/5/7000 DV. We will soon be releasing the D-Torch for other ICP models. Please contact **enquiries@geicp.com** for information on the availability of the D-Torch for your ICP.

In addition to a new look for the SMART ANALYZER VISION interface, SPECTRO has increased the range of functions. There are many new import, sorting, filter and configuration options now available for the SPECTRO ARCOS and SPECTRO GENESIS systems; allowing for increased sample throughput and efficient automation of the analyses.

The SMART ANALYZER VISION 4.0 software is now standard in all new SPECTRO ARCOS and SPECTRO GENESIS ICP-OES instruments.

From Thermo Fisher Scientific – Thermo Fisher Scientific Cambridge facility wins The Queens Award for Enterprise in 2009

Thermo Fisher Scientific Inc. has announced that its Cambridge Facility has been awarded a Queens Award for Enterprise 2009 in the Outstanding Innovation category for the iCAP 6000 Series ICP-OES instrument. This award, the most prestigious for UK industry, recognizes the iCAP 6000 as one of the year's most outstanding technical innovations.

The Queens Award for Enterprise recognizes the novel design of the iCAP 6000 which delivers outstanding performance, ease of use and low cost of ownership, all in a uniquely compact instrument. Specifically developed for environmental, metallurgical, petrochemical and food safety applications, this compact instrument provides users in laboratories with increased analysis speed, exceptional stability and trace (parts per billion) level detection capabilities up to five times lower than comparable older designs.

The Thermo Fisher Scientific team in Cambridge started work on the design of the iCAP 6000 in 2002 and has been manufacturing the product and distributing it world wide since 2006. Since its launch, the iCAP 6000 has captured a significant share of global markets in a number of important applications segments.

The Queen's representative in Cambridge will make a formal presentation of the award at the Thermo Fisher Scientific facility, later in 2009.

For more information about the Thermo Scientific iCAP 6000 Series ICP-OES spectrometers please email **analyze@thermofisher.com** or alternatively please visit **www.thermo.com/icap**.

From Varian – Determination of trace metals in > 99.999% gold using the Varian 820-MS

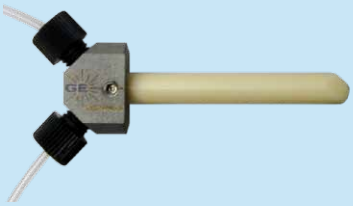
The strong demand for gold in recent times has seen the metal's market value grow to levels not encountered since the early 1980's. Although the jewelry industry still makes up for the majority of the world's total demand for gold, one area that has seen significant growth is its use in electronics, particularly within telecommunications, information technology and safety critical applications. The unique physical, electrical and chemical properties of gold make it a valuable material in the manufacture of semiconductor devices, including printed circuit boards, automotive electronics and smart card technology, to name a few.

Wire bonding is the method used to attach very fine gold wire (typically thinner than a human hair at 10-200 microns) from one connection pad to another, completing the electrical connection in an electronic device. Nowadays literally billions of wires are bonded every year in the world and most are used in the integrated circuits (ICs) we take for granted in all manner of electronic goods.

If you are interested in reading an application paper about the determination of trace metals in high purity 5N-gold (> 99.999% gold) using Varian ICP-MS, please select the following link: **http://www.varianinc.com/cgi-bin/nav?applications/icpms**

NEW PRODUCTS

New Ceramic VeeSpray™ nebulizer design



The Ceramic VeeSpray Nebulizer is the most versatile and durable nebulizer available. The ceramic material provides the same chemical resistance as plastics such as PFA and PTFE, including resistance to HF. But unlike plastic materials, it does not wear out and, under normal use, it will maintain optimum performance indefinitely. It can also handle a wider range of samples than any other nebulizer, including samples with high salt content (up to 30%) and samples with particulates (up to 300µm).

We have upgraded the design to make it simple to replace the sample or gas tubing. Now that the tubing can be easily replaced, your Ceramic VeeSpray nebulizer will virtually last forever.
Click here for more details.

Nebulizer tip view

GE NEWS (CONT.)

CUSTOMER SUPPORT TEAM

Our customer support team has a wealth of ICP experience and is ready to provide advice on the best procedure and sample introduction system for your application. We also have four ICP spectrometers (both OES and MS models) in our own ICP laboratory to assist us with quality control and product development.

Click here for more information.

INTERNATIONAL

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