NEWS

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APPLICATION SPOTLIGHT

IMPROVED ACCURACY IN THE ANALYSIS OF PRECIOUS METALS BY ICP-OES

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Introduction

This study investigates the intensity variations and measurement accuracy of precious metals at different spray chamber temperatures, compared with a conventional spray chamber at ambient temperature. The aim of the exercise was to investigate the feasibility of analysing precious metal solutions using ICP techniques to replicate the high level of accuracy and precision obtained using conventional wet chemical techniques.

The investigation was carried out using an IsoMist Programmable Temperature Spray Chamber connected to a Varian Vista Radial ICP-OES as shown in Figure 1. Figure 1 also shows the Capricorn Argon Humidifier, which is used to help prevent salt build-up inside the sample introduction system when running samples having a high concentration of dissolved solids. The IsoMist allows the user to control the temperature of the spray chamber, whether it be for cooling or heating, to finely tune the conditions for optimal sample introduction and ensure long term stability of the signal. Therefore, any change in laboratory temperature is not reflected in the results. The IsoMist also has a number of other benefits depending on the type of sample matrix. For example; setting the IsoMist at low temperatures reduces the solvent load on the plasma and facilitates the analysis of highly-volatile organic samples that, under normal laboratory conditions, tend to promote plasma instability, or in some cases completely extinguish the plasma. Thus, even highly volatile solvents like naphtha can be run undiluted if the IsoMist temperature is set at -10°C. Another advantage is that when samples are very limited in volume, they can be analyzed at very low uptake rates with a heated spray chamber without the ensuing loss in detection limits as would be realized on a conventional spray chamber without temperature control.

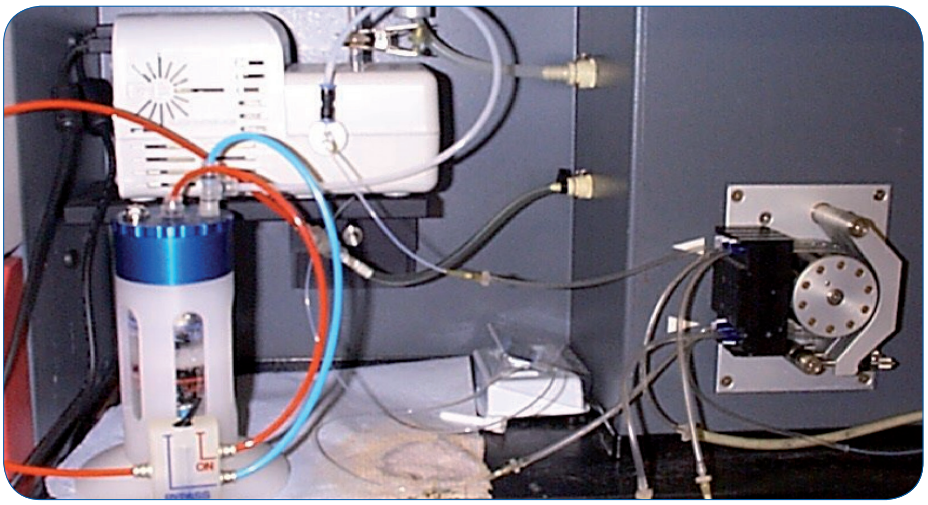
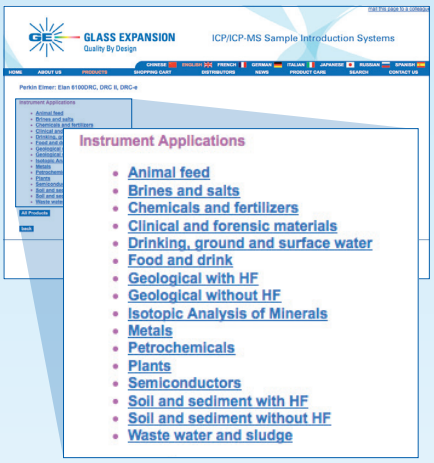


Figure 1: Experimental setup for the Varian Vista Radial ICP-OES with a Glass Expansion IsoMist Programmable Temperature Spray Chamber and the Capricorn Argon Humidifier attached.

GE NEWS

Website upgrade – simplified selection of products to suit your application

With our new product selection feature, finding the optimum sample introduction system for your particular application has never been easier. Simply select your ICP model and your application, and the most suitable sample introduction components are immediately displayed. Information is available to suit the most common applications of the most common ICP-OES and ICP-MS models. **Click here** to check it out. We will be expanding this product selection feature to other models over time. If your model or your application is not currently covered, please send an email to **enquiries@geicp.com** and we will happily provide you with details of the most suitable sample introduction system.



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GLASS EXPANSION
Quality By Design

Results for Aqueous Solutions

In this study, the IsoMist was used to precisely control the spray chamber conditions during the analysis of various precious metal elements in solution to investigate the effect of temperature on precious metal signal intensities and analytical accuracy. The elements analysed included: gold [Au]; iridium [Ir]; palladium [Pd]; platinum [Pt]; rhodium [Rh]; and ruthenium [Ru]. Figure 2 shows the average signal intensity [n = 10] of various precious metals at specified spray chamber temperatures using the IsoMist.

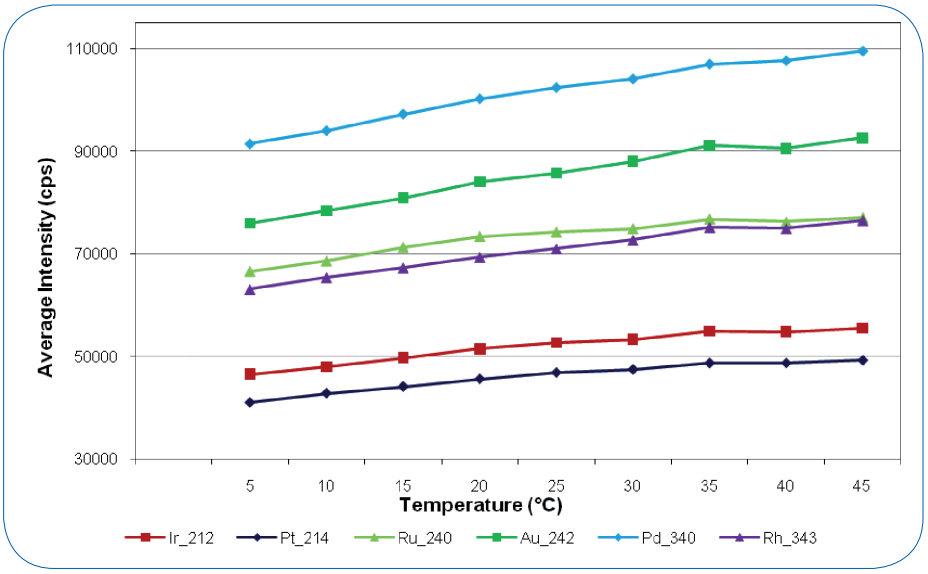


Figure 2: Graph of signal intensity versus spray chamber temperature for several precious metals.

In order to gauge the effect of temperature on the accuracy of the precious metal measurements, a solution of platinum was analysed at a range of temperatures and compared to the concentration determined gravimetrically. The results shown in Figure 3 show that, at higher spray chamber temperatures, the ICP determinations of the platinum concentration are closer to the gravimetric or true value. Again this demonstrates that the IsoMist enhances the sample introduction environment to help provide the conditions necessary for accurate and reproducible platinum measurements.

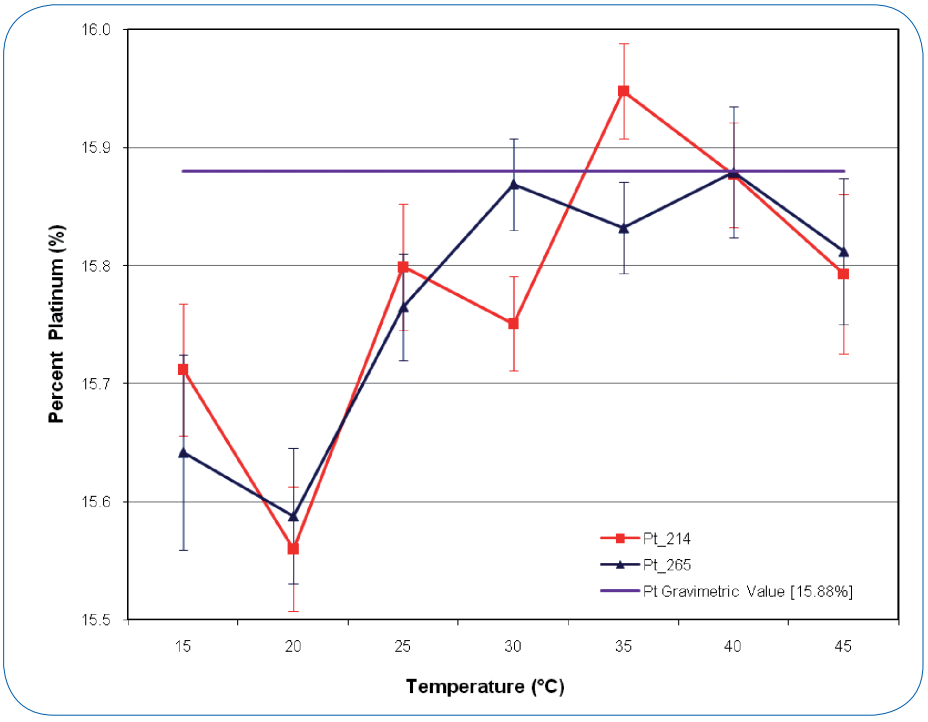


Figure 3: Plot of average ICP measurement of the percentage of platinum in solution [n = 10] versus temperature. The error bars are 1σ.

GE NEWS

Website upgrade – choice of language

Most of the Glass Expansion website is conveniently available in English, French, German, Italian, Spanish, Chinese, Japanese and Russian. The non-English pages have recently been updated so all of the latest information is now available in your choice of language. Navigation is simple and you can switch languages at any time using the language buttons at the top of each page.

At higher temperatures the data is more accurate according to Figure 3; both platinum wavelengths measured by ICP are closest to the wet chemistry gravimetric figure when the IsoMist is at 40°C. But what is the effect of the IsoMist on the precision of the measurements?

Figure 4 show the frequency distribution of the RSD (%) for each of the elements analysed with a conventional spray chamber and those analysed with an IsoMist set at 30°C. A higher proportion of low RSD values implies that the data is more "precise". Although the data set is small there is an indication that the IsoMist has improved the accuracy and precision of precious metal measurements.

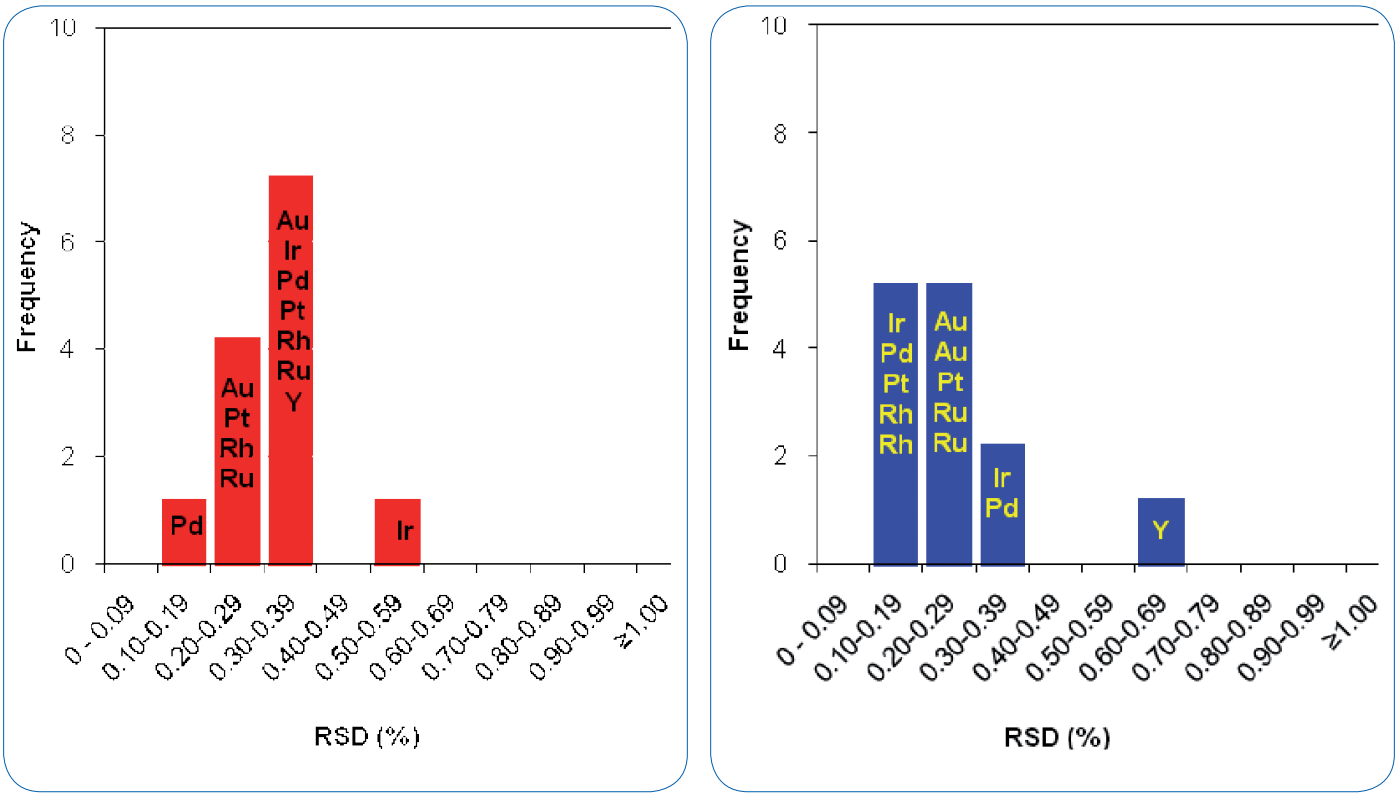
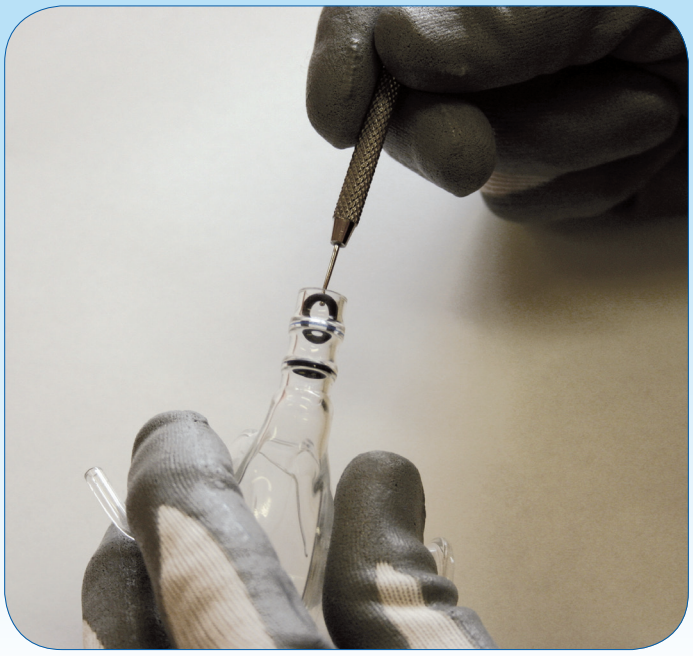


Figure 4: Frequency histogram of RSDs of precious metal measurements [n = 10] taken with a conventional spray chamber [left figure] and those measurements taken using an IsoMist set at 30°C [right figure]. A higher proportion of measurements in the lower bins suggests high precision.

HINTS FOR THE OPERATOR

How to remove stubborn o-rings

Several years ago, Glass Expansion introduced the spray chamber with Helix, in which the nebulizer is held in place by an inert PTFE seal. The Helix eliminates the problems with o-rings and it has become the preferred system. However, some of the older-style spray chambers with o-rings are still in use. It is common for the o-rings to bond to the glass, making them very difficult to remove. The removal of o-rings is now much easier thanks to the introduction of the Glass Expansion O-ring Tool. It can be ordered as part number **50-100-1636**.



Results for Organic Solutions

The ability of the ICP analyst to control the temperature of the spray chamber improves their ability to analyse elements in matrices that would otherwise require further chemical treatment and the potential loss of analyte, for example, the analysis of ruthenium in an organic solvent matrix. The organic solvent matrix is common with pharmaceutical samples as these are analysed with liquid chromatographic techniques using organic solvents as the carrier. Conventional methods would involve wet oxidising the sample in a mixture of nitric and sulphuric acid to remove the organic solvent, however there is the possibility of ruthenium loss during the oxidation process. Another potential method would be to dry ash the sample, but loss of ruthenium occurs with the volatile species; and then there is the problem of dissolving ruthenium metal which can only be done in alkali hypochlorite solutions. The fused alkali dissolution process results in a solution containing sodium salts which unfortunately degrades the detection limits.

It is possible to directly measure precious metals in an organic matrix using the IsoMist set at low temperature. In this experiment the spray chamber was set to temperatures between -10°C and +10°C in order to minimise the amount of vapour going into the plasma and two solvents, methanol and isopropanol, containing various precious metals, were analysed. Note that, if a methanol matrix is run at temperatures above 10°C, then the plasma is extinguished and, for an isopropanol matrix, the plasma is extinguished at temperatures above 15°C. Additional steps were also taken to reduce plasma loading such as increasing the power and argon flow rates, as well as using smaller diameter peristaltic pump tubing.

Table 1 shows the plasma conditions used with the organic solutions compared with those for the aqueous solutions. The results for methanol and isopropanol are shown in Figures 5 and 6. Figure 5 shows that there is a general decrease in intensity as the temperature is increased. This is most likely due to an increase in plasma loading. Figure 6 indicates that, as the temperature is increased from -10°C, there is an initial increase in intensity due to increased sample transport but, above zero, plasma loading becomes the dominant factor and the intensity decreases.

These results clearly demonstrate that the IsoMist facilitates the measurement of analytes in volatile organic solvents at temperatures below 15°C.

	Organic	Aqueous
Power (W)	1600	1300
Plasma flow (L/min)	19.5	15.0
Auxiliary flow (L/min)	2.25	1.50
Nebuliser flow (L/min)	0.60	0.60
Integration time (s)	10.0	10.0
Pump tubing [ID mm]	black-black [0.76]	grey-grey [1.30]
Waste tubing [ID mm]	red-red [1.14]	blue-blue [1.65]
Injector diameter (mm)	1.5	1.8
Pump rate (rpm)	25	15
Sample uptake (mL/min)	1.0	1.5

Table 1: Plasma conditions for the analysis of precious metals in an organic matrix compared with those for aqueous solutions.

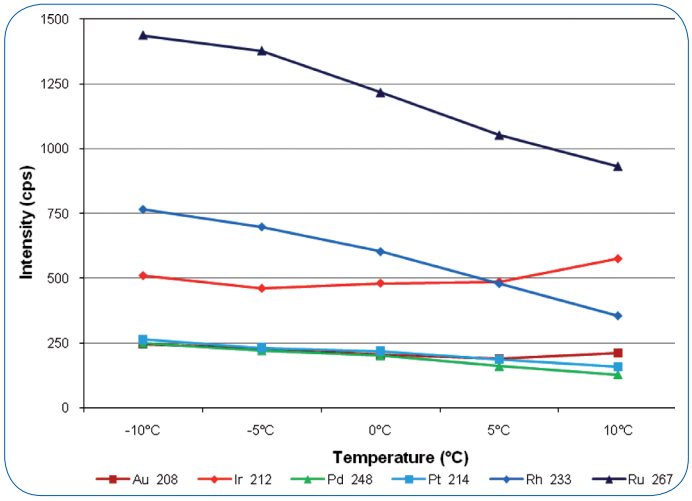


Figure 5: Average [n = 5] intensity of various precious metals in a methanol matrix versus temperature. All species shown are ionic. Note that at spray chamber temperatures above 10°C the plasma was extinguished.

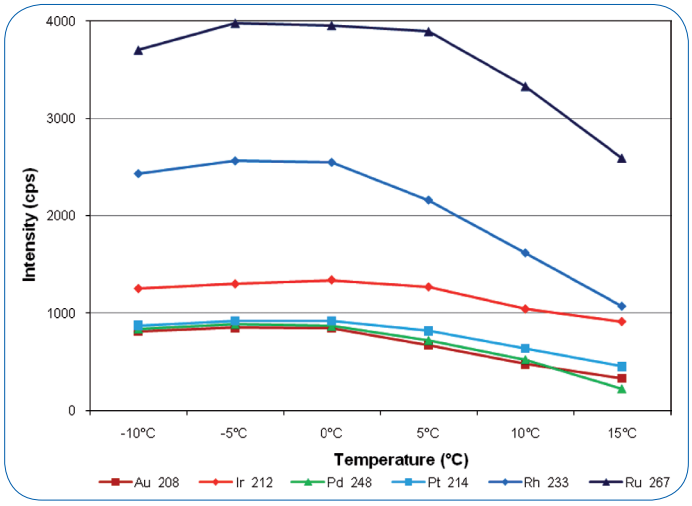


Figure 6: Figure 6: Average [n = 5] intensity of various precious metals in an isopropanol matrix versus temperature. All species shown are ionic. Note that at spray chamber temperatures above 15°C the plasma was extinguished.

Conclusion

The benefits of the IsoMist for the analysis of precious metals are:

(1) control of the temperature of the spray chamber to enhance signal intensity;
(2) reduced signal instability and improved measurement accuracy and precision;
(3) direct analysis of elements in organic solvents at temperatures below 15°C.

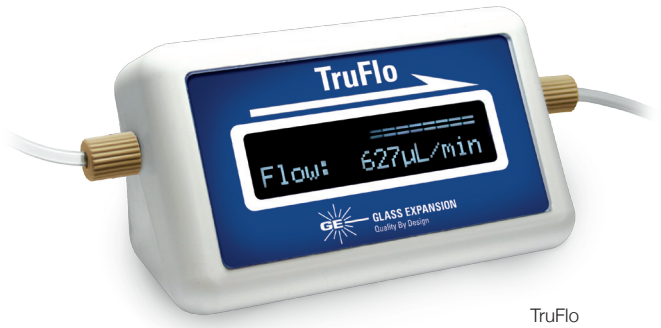
NEW PRODUCTS

HF-resistant TruFlo Sample Monitor

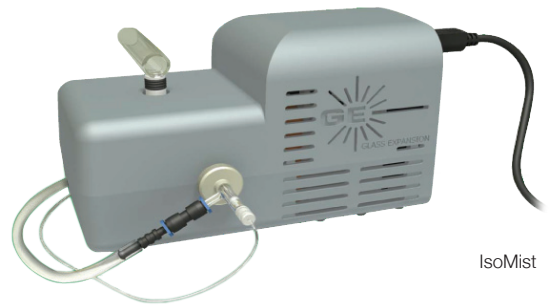
The TruFlo range has been expanded to include HF-resistant versions. Released in 2009, the TruFlo remains the simplest and most effective way to monitor sample uptake. The sample uptake has a significant effect on ICP performance but is rarely measured. In many cases, the operator can only rely on guesswork to determine the uptake. With the TruFlo Sample Monitor, you always know the actual rate of sample uptake to your nebulizer. This enhances the day-to-day reproducibility of your results and reduces the need to repeat measurements due to a blocked nebulizer, worn pump tubing or incorrect clamping of the pump tube. The TruFlo can even sound an alarm if the sample uptake is outside your specified limits. It is simple to install and is compatible with all ICP-OES and ICP-MS models. The actual sample flow is shown on the TruFlo's inbuilt digital display and a graph of the flow versus time can also be displayed on your computer. The current TruFlo range is as follows:

Part No.	Description
70-803-0643	TruFlo Sample Monitor 0 – 4mL/min
70-803-0788	TruFlo Sample Monitor 0 – 1mL/min
70-803-0774	TruFlo Sample Monitor 0 – 0.05mL/min
70-803-0890	TruFlo Sample Monitor for HF 0 – 4mL/min
70-803-0891	TruFlo Sample Monitor for HF 0 – 1mL/min
70-803-0892	TruFlo Sample Monitor for HF 0 – 0.05mL/min

Click here for full specifications or contact **enquiries@geicp.com** for more information.



TruFlo

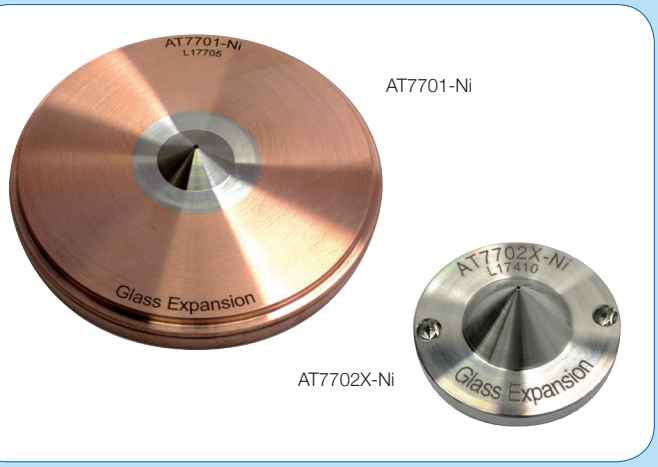


IsoMist

Cones for Agilent 7700 ICP-MS

We have expanded our range of cones for the Agilent 7700 and we now have the complete range available. The Agilent 7700 was released in 2009, replacing the 7500. We can supply a full range of consumables for this model, including nebulizers, torches and cones. The range of cones is as follows:

Part No.	Description
AT7701-Ni	Nickel Sampler Cone for Agilent 7700
AT7701-Ni/Ni	Nickel Plated Sampler Cone for Agilent 7700
AT7706-Pt	Platinum Sampler Cone for Agilent 7700
AT7706A-Pt	Platinum Sampler Cone for Agilent 7700 (18mm insert)
AT7704	Retaining Ring for Agilent 7700 Sampler Cone
AT7703	Graphite Gasket for Agilent 7700 Sampler Cone (PKT 3)
AT7702X-Ni	Nickel Skimmer Cone for Agilent 7700x
AT7702S-Ni	Nickel Skimmer Cone for Agilent 7700s
AT7702S-Cu	Copper Skimmer Cone for Agilent 7700s
AT7708X-Pt	Platinum Skimmer Cone for Agilent 7700x
AT7708X-Pt/Ni	Platinum Skimmer Cone with Nickel Base for Agilent 7700x
AT7708S-Pt	Platinum Skimmer Cone for Agilent 7700s
AT7708S-Pt/Ni	Platinum Skimmer Cone with Nickel Base for Agilent 7700s



AT7701-Ni

AT7702X-Ni

IsoMist and TruFlo for Windows 7

The software for the IsoMist and the TruFlo is now compatible with the Microsoft Windows 7 operating system. The current versions of the IsoMist and TruFlo can be used with Windows 7, Vista or Windows XP. If you already have an IsoMist or a TruFlo and you wish to use Windows 7, please contact **enquiries@geicp.com** and we will send you an upgrade.

INSTRUMENT NEWS

From Agilent Technologies – New MassHunter Software & New 7700 Series ICP-MS Family

Agilent Technologies has released a new easy-to-learn version of its popular ICP-MS MassHunter Workstation software for the 7700 Series ICP-MS. Agilent has also expanded its ICP-MS instrument platform with the Agilent 7700e ICP-MS, designed to deliver high-performance, routine elemental analysis at an affordable price.

A key feature of the new ICP-MS MassHunter software is the use of pre-set methods, which allow users to choose from pre-defined sets of operating and acquisition conditions for the analysis of well-characterized sample types. The result is that many routine applications can be run without any manual tuning or method development, using consistent and optimum conditions regardless of the operator's expertise. The new software release also introduces innovative productivity and ease-of-use features such as a startup scheduler and batch-and-queue sequence control.

The 7700e is a new addition to the Agilent 7700 Series ICP-MS family, developed with the emphasis on streamlined and simplified operation for common applications. With commonly required software functionality and a standardized hardware configuration, the 7700e offers users access to high-performance ICP-MS capabilities at an affordable price. This is especially useful where budgets are limited and ultimate flexibility is not required.

For more information, please visit **www.agilent.com/chem/icpms**

From HORIBA Scientific – New Generation ICP-OES to match customer's expectations

HORIBA Scientific has introduced a New Generation of ULTIMA 2 and ACTIVA ICP spectrometers. This New Generation maintains the unique performance of HORIBA Scientific ICP-OES instruments and offers enhanced ease of use for the analyst.

The instrument was fully redesigned to match customer's expectations. The sample and the plasma compartments provide fast installation of torch and introduction system as well as reduced sample uptake delays. Digital flow-meters are used to monitor all gas flows and the nebulizer is controlled through a sensor based flow-meter for continuous flow and pressure monitoring. Accessibility was also improved for easier maintenance and all connections were located to facilitate the installation of fluids and accessories. For improved speed of data transfer, a new electronic acquisition board is used with Ethernet connection.

All analytical performances of the instruments were maintained with the best resolution of the market for the ULTIMA 2, allowing the highest performance in the most difficult applications and the unique assistance tools for the ACTIVA M to bring the expertise in ICP-OES in laboratories. The radial viewing mode with Total Plasma View feature offers low detection limits and reduced matrix effects for improved performance.

From Spectro – Arcos 165 Completes Spectro's ICP-OES Portfolio

SPECTRO has released the new SPECTRO ARCOS 165. The ICP-OES records the elemental spectrum between 165 and 770 nanometer for every measurement; making it especially suited to challenging tasks in environmental analysis. SPECTRO ARCOS 165 rounds out the ICP-OES analyzer product series between the flagship SPECTRO ARCOS and the entry-level SPECTRO GENESIS.

"The engineering of the SPECTRO ARCOS 165 is based completely on the high-end components utilized in our flagship ARCOS system and achieves exactly the same detection limits, the same precision and equally reproducible results," explains Olaf Schulz, Product Manager for ICP-OES spectrometers at SPECTRO. The optical system is the only difference between the models: While the ARCOS records the entire spectrum starting at 130 nanometers for every measurement, the trimmed down ARCOS 165 measures the wavelength range beginning at 165 nanometers. Schulz: "In practice, this is only noticeable for halogen and metal matrix analysis applications – two applications that don't play a major role in many laboratories. Customers that can do without this wavelength range of 130-165 are rewarded with the exceptional ARCOS performance at a very attractive price."

The SPECTRO ARCOS 165 technical highlights include its proven optic design with an excellent resolution of 8.5 picometer in the wavelength range between 165 and 340 nanometer, and 15 picometer in the range from 340 to 770 nanometer.

From Thermo Fisher Scientific – Webinar Prepares ICP Users for New United States Pharmacopeia Chapters Proposal

Thermo Fisher Scientific Inc. has announced an informative webinar to help users of ICP technologies prepare for the introduction of the proposed United States Pharmacopeia (USP) chapters 232 and 233 on trace elemental impurities in pharmaceutical products. The educational presentation provides the pharmaceutical industry with valuable information about how ICP techniques can facilitate compliance with these emerging legislative requirements. The webinar, entitled "Preparing for the Proposed United States Pharmacopeia Chapters on Trace Elemental Impurities in Pharmaceuticals with ICP Techniques," is now available to view on-demand at **www.spectroscopynow.com/webinars**.

Presented by Matthew Cassap, senior ICP applications specialist for Thermo Fisher Scientific, the comprehensive webinar discusses the limitations of the current USP method governing the analysis of heavy metals in pharmaceutical products. In addition, the webinar provides a historical background and detailed overview of the proposed new chapters.

The presentation features the Thermo Scientific iCAP 6000 Series ICP spectrometer, demonstrating the efficiency of ICP techniques for the reproducible analysis of trace elemental impurities in pharmaceuticals in compliance with the upcoming regulatory requirements. In addition, Thermo Scientific iTEVA Security Software plays a crucial role in legislative compliance efforts as it allows for optimization of instrument parameters and elimination of interferences, delivering maximum accuracy and reliability of results.

For more information about Thermo Scientific trace elemental analyzers for the pharmaceutical industry, please email **analyze@thermofisher.com** or visit **www.thermoscientific.com/trace**.