NEWS

Glass Expansion Newsletter • October 2009 • issue 20

APPLICATION SPOTLIGHT

THE BASICS OF SAMPLE TRANSPORT INTO AN ICP SPECTROMETER

Introduction

The form of the sample as it is injected into a torch has a profound effect on the quality of the analytical results for ICP optical emission spectrometry (ICP-OES) and ICP mass spectrometry (ICP-MS). We will examine the criteria for optimum sample transport and discuss the proper selection of sample introduction components and parameters.

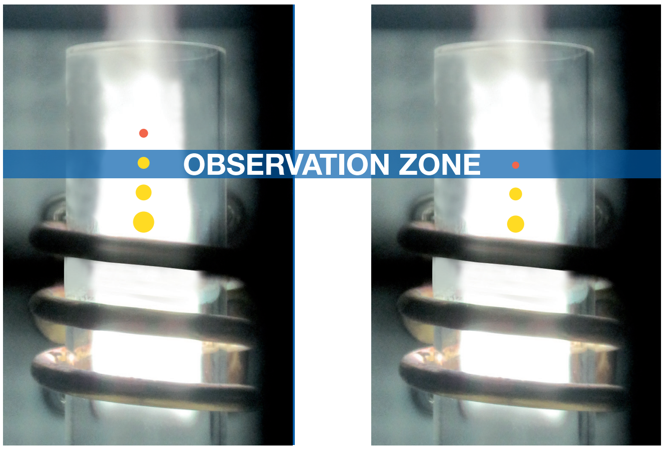

Fig. 1 Illustration of droplet vaporization and atomization in a radial plasma; *left*: large droplet; *right*: small droplet

Droplet Size

The mean droplet size of the aerosol generated by the combination of the nebulizer and spray chamber (conventionally regarded as the tertiary aerosol) is an important parameter. In essence, the smaller the droplets, the better. Once the droplet enters the plasma, it must complete the desolvation process (a process already begun in the spray chamber) followed by vaporization, and atomization/ionization (and emission in the case of OES) in order to deliver signal to the detector. The smaller the droplet, the less energy is required for desolvation and atomization, creating a more efficient emission process. In fact, larger droplets that reach the plasma may pass through the observation zone of the detector prior to reaching the emission or ionization state. Figure 1 demonstrates this effect for a radially viewed plasma. Droplet size also is positively related to the degree of matrix interference since larger droplets contain more solvent and solvent loading degrades the tolerance of the plasma to matrix[1]. The mean droplet size is determined in two stages as described below.

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GE NEWS

WINTER CONFERENCE 2010

A full range of Glass Expansion products will be on display at the 2010 Winter Conference on Plasma Spectrochemistry, Fort Myers, Florida, January 4 – 9, 2010. The display will include nebulizers, spray chambers, torches, RF coils and accessories (including the new TruFlo Sample Monitor). Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP.

NEW ICP SPECIALIST

Dr Jol Desmarchelier has joined our Australian office as ICP Specialist. He has a background in Earth Sciences, particularly using ICP-MS, and he has previously worked at University of Tasmania, Australian National University and Australian Nuclear Science and Technology Organisation. Jol is working with Vesna Dolic in our ICP laboratory and he will enhance our ability to solve problems for ICP users and to help them get the best possible performance with their applications. Jol can be contacted at: **jdesmarchelier@geicp.com**.

Primary Aerosol

The primary aerosol is created by the nebulizer itself. The droplet distribution in the primary aerosol is a function of a number of variables. The Nukiyama and Tanasawa equation [2] gives the relationship between the Sauter mean diameter, d3,2, of droplets generated by a concentric nebulizer and is shown below:

$$d_{3,2} = \frac{585}{V}\left[\frac{\sigma}{\rho}\right]^{0.5} + 597\left[\frac{\eta}{(\sigma\rho)^{0.5}}\right]^{0.45}\left[\frac{10^3 Q_l}{Q_g}\right]^{1.5}$$

$d_{3,2}$ = Sauter mean diameter - (µm)

V = Velocity difference of gas liquid - (m/s)

σ = Surface tension - (dyn/cm)

ρ = Liquid density - (g/cm³)

η = Liquid viscosity - (Poise or dyn.s/cm²)

Q_l = Volume flowrate, liquid - (cm³/s)

Q_g = Volume flowrate, gas - (cm³/s)

For water or aqueous matrices, the surface tension, liquid density, and viscosity remain relatively constant.The droplet size then is inversely proportional to the velocity difference of the gas and liquid, i.e. droplet size lowers as the gas velocity increases and the liquid velocity decreases. Droplet size is also determined by the ratio of liquid flow rate to gas flow rate; the smaller the ratio, the smaller the mean droplet size.

In practical terms then, to achieve a very small droplet size in the primary aerosol would involve using a high flow and pressure for the argon gas and a low flow and pressure for the liquid.This is for the most part how our MicroMist low-flow nebulizers are designed to function.

Tertiary Aerosol

The primary aerosol is filtered by the spray chamber to produce a tertiary aerosol. In general, droplets larger than 10 microns in diameter do not contribute significantly to plasma emission[3]. The task of the spray chamber is to remove droplets larger than 10 microns and divert them to the drain and allow as many of the sub 10

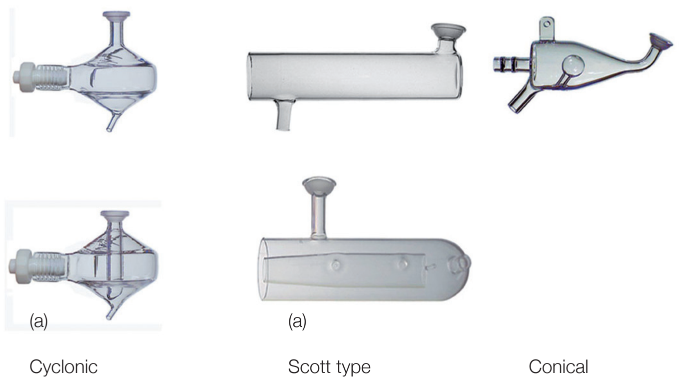


Fig. 2 Common spray chamber types; a: double pass designs

micron drops to pass into the plasma as possible. Almagro, et al, described a novel flow focusing pneumatic nebulizer (FFPN) which generated a primary aerosol with significantly lower mean droplets than a Glass Expansion Conikal nebulizer[4]. However, the tertiary aerosols generated for the two nebulizers were all but indistinguishable due to the filtering capacity of the spray chamber. Others have observed similar behavior[5]. The major advantage then of a nebulizer that produces an aerosol with a small mean drop size is greater transport efficiency. Yet mass transport to the plasma is limited by the characteristics of the plasma itself, with a plasma load of 40 to 60mg/min generally believed to be the upper limit.

Cyclonic spray chambers are the most commonly used spray chambers in plasma spectroscopy. Unlike other types of spray chambers such as the conical and Scott style (see Figure 2), cyclonic spray chambers incorporate two means to filter large droplets. All spray chambers use gravity to drop out the large droplets, but the cyclonic chambers also use centrifugal force to remove them. Fast and efficient removal of large droplets reduces the likelihood of recombination, which can result in a reduction in the population of small droplets. The cyclonic spray chamber serves as an effective cutoff filter for the aerosol and has been shown to limit the droplet size to less than 15 microns [4].

Droplet Velocity

The speed at which a droplet passes through the plasma plays an important role in determining the emission/ ionization intensity. Ideally, a slow moving droplet is desired to give it ample time to go though all of the processes required prior to producing a signal. This means that a low nebulizer gas flow rate is beneficial with respect to droplet velocity. However, this is contrary to our findings for droplet size. The spray chamber serves as an intermediate, reducing the primary droplet speed. This has been shown to be one of the primary disadvantages of a direct injection nebulizer (DIN) in which the nebulizer and injector tube are one and the same[6]. However, this approach has some advantages over a spray chamber-nebulizer combination, including higher transport efficiency, low memory and dead volume.

Parameters that affect droplet velocity are nebulizer gas flow rate and bore size of the injector. There is a specific limiting gas speed under which the sample will not pierce the plasma and of

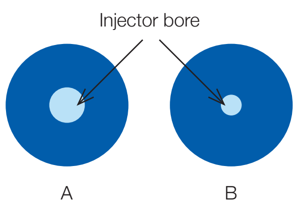


Fig. 3 Illustration of top down view of an axially viewed plasma showing the observation zone as determined by injector bore; A: large bore; B: small bore

course that speed must be exceeded, but ideally by as little as possible. Since speed is decreased by lowering the gas flow rate and by enlarging the injector orifice, these two parameters must be properly balanced to achieve the optimum droplet speed. For an axially viewed OES plasma, the injector bore size has an additional effect. It determines the size of the viewing zone of the optics and therefore a larger orifice typically enhances intensity (see Figure 3).

Transport Efficiency

Transport efficiency is defined as the percentage of the sample taken up into the nebulizer that is transported to the plasma. As discussed above, transport efficiency is a function of both the nebulizer and the spray chamber and the conditions under which they are operated.

Spray chamber effects

We measured the transport efficiency for three different designs of cyclonic spray chamber keeping the nebulizer conditions the same.

- Tracey glass spray chamber, 50mL internal volume, unbaffled
- Twister glass spray chamber, 50mL internal volume, baffled
- Cinnabar glass spray chamber, 20mL internal volume, unbaffled

As shown in Figure 4, the Tracey spray chamber yielded the highest transport efficiency. The Cinnabar was next with efficiency reduced by 8%. We attribute this to the fact that the nebulizer tip is that much closer to the chamber wall so that droplets hit the wall at a higher velocity (droplet velocity goes down as distance from the tip increases). The Twister spray chamber gave the lowest efficiency, 21% lower than the Tracey due to the baffle filtering out the larger

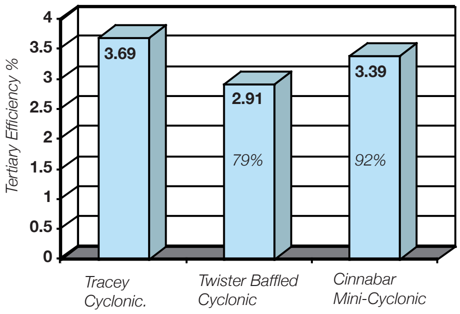


Fig. 4 Tertiary transport efficiency for various spray chambers; percentage is normalized to cyclonic.

droplets. Although one might expect a commensurate decrease in intensity, it is not the case since many of the droplets produced by the Tracey chamber are too large to contribute appreciably to the signal.

Sample uptake effects

We looked at the effect of varying sample uptake for a given spray chamber design, in this case a Twister cyclonic (Figure 5). At 2.1mL/min, the transport efficiency is a little better than 2% and this increases to more than 50% down at 0.03mL/min

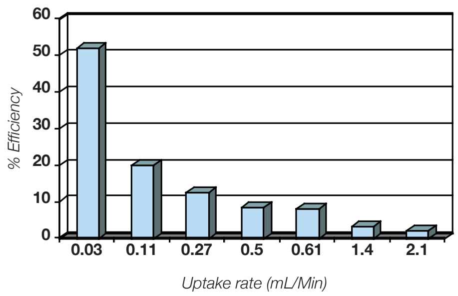


Fig. 5 Effect of sample uptake rate on tertiary transport efficiency using a glass concentric nebulizer and Twister cyclonic spray chamber.

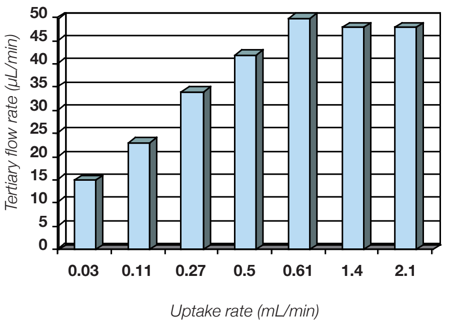


Fig. 6 Effect of sample uptake rate on tertary flow rate (same data as used in Fig. 5).

uptake. Figure 6 gives a bit more insight on the same data, plotting tertiary flow rate instead of transport efficiency vs. uptake rate. Note that the tertiary flow rate does not begin to decrease until the uptake rate goes below 0.6mL/min. This means that an uptake rate higher than 0.6mL/min gives no additional signal and simply increases the amount of waste that goes down the drain.

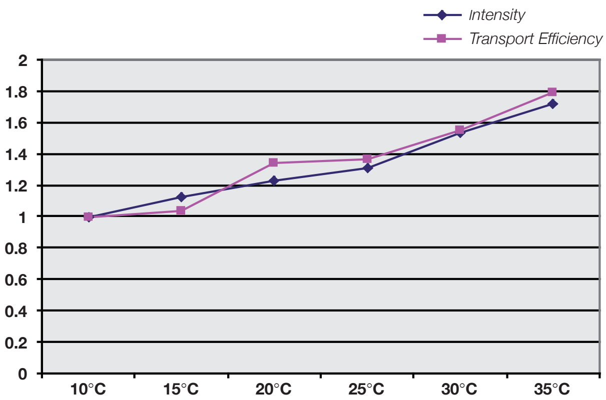


Fig. 7 Overlay of transport efficiency and intensity versus spray chamber temperature; 1mL/min uptake with glass concentric nebulizer and cyclonic spray chamber.

Temperature effects

The temperature of the sample introduction system has a significant effect on the transport efficiency of the sample[7]. It has been useful to lower the spray chamber temperature for volatile organic solvents which would otherwise overload the plasma[8]. With a programmable temperature spray chamber such as the IsoMist, which can heat as well as cool, the operator can precisely control the transport efficiency of the system. Figure 7 shows the effect of spray chamber temperature on the average intensity of 18 analyte lines overlaid with the transport efficiency in an aqueous matrix. Note that the average intensity closely tracks the increase in transport efficiency due to temperature. Also note that a 45% increase in signal was attained at 1mL/min uptake between 20°C and 35°C. This indicates that at room temperature, the mass transport was significantly below the maximum for the plasma being used. This work was performed on a Perkin Elmer Optima 2100DV ICP-OES. Interestingly, at very low sample uptake rates, sensitivity enhancements surpass that which is attributed solely to transport efficiency. Figure 8 shows the effect of temperature on 20µL/min uptake compared to transport efficiency. We believe that part of this enhanced sensitivity is due to partial desolvation in the spray chamber at elevated temperatures which in turn increases atomization and emission efficiency.

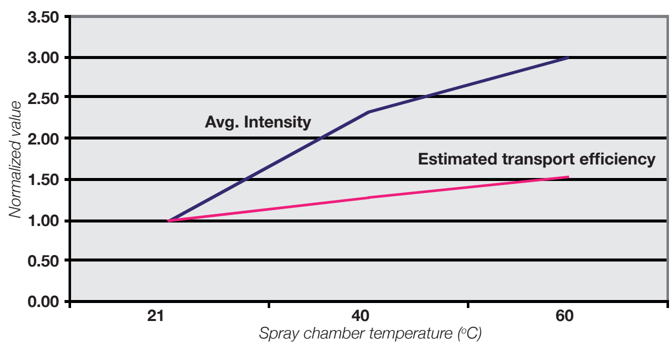


Fig. 8 Overlay of transport efficiency and intensity versus spray chamber temperature for 20µL/min sample uptake.

Summary

Sample transport into an ICP is one of the most critical aspects of both ICP-OES and ICP-MS. It is determined by the precise dimensions and parameters of the nebulizer, spray chamber, and injector tube. If properly configured, this system will deliver to the plasma an aerosol consisting of similar sized small droplets traveling at low velocities.

References

1. J. Borkowska-Burnecka, A. Lesniewicz and W. Zymicki, *Spectrochim*. *Acta*,, 61B, 579 (2006)

2. S Nukiyama and Y Tanasawa, *Trans Soc Mech Eng.*, (1938-1940)

3. SE Hobbs and JW Olesik, *Anal Chem*. 64, 274 (1992)

4. B Almagro, AM Ganan-Calvo and A Canals, *J Anal At Spectrom.* 19, 1340 (2004)

5. C Carre, K Lebas, M Marichy, M Mermet, E Poussel, and JM Mermet, *Spectrochim Acta*, 50B, 271 (1995)

6. JA McLean, H Zhang, and A Montaser, *Anal Chem.* 70, 1012, (1998)

7. G Zhu and RF Browner, J *Anal At Spectrom*. 3, 781 (1988)

8. SJ. Hill, J Hartley, and L Ebdon, *J Anal At Spectrom*. 7, 23 (1992)

INSTRUMENT NEWS

From Agilent Technologies - 7700 Series ICP-MS, the New Face of ICP-MS

Replacing the 7500 Series, Agilent Technologies introduced the 7700 Series ICP-MS, comprising the 7700x workhorse and 7700s semicon-configured mainframe. The new 7700 Series features new, fast frequency-matching RF generator, HMI capability, new ion lens and ORS, new MassHunter software on a 30% smaller mainframe than the 7500 Series.

Visit www.agilent.com/chem/icpms and check out the 7700 Series with the interactive animation. A large number of 7700 launch seminars are coming up in Oct/Nov. Be sure to check with your local Agilent office or web site for details of events in your area.

From From Horiba Jobin Yvon - Too many lines, not enough time? CLIP can help you!

In ICP-AES, line-rich spectra are often observed and it is difficult to find analyte lines free of interference from all the matrix or concomitant element lines. This is especially true with matrices such as steel, tungsten, zirconium, precious metals and other complex matrices.

Usually the analyst prepares known solutions, makes profiles for each wavelength at different concentrations of the analyte and concomitant elements and finally selects a line free of interference with adequate sensitivity. This crucial step in method development is long and tedious. It must be conducted with great care because it can greatly influence the accuracy of the results.

HORIBA Scientific simplifies this step with CLIP (Collection of Line Intensity Profiles), which assists the analyst in the development of methods for high resolution sequential ICP-AES: no more solutions to prepare and no more profiles to acquire. The profile of each line is calculated according to the instrument's configuration: focal length, slit combination, diffraction grating and order used. Only a few minutes are needed to select lines for every element.

Every user of an ULTIMA family instrument can have the benefit of CLIP to save time and enhance the quality of their results.

From PerkinElmer - PerkinElmer Expands Distribution Channel for Spectroscopy and Chromatography Instrumentation Consumables and Minor Accessories with VWR International, LLC

PerkinElmer, Inc., a global leader focused on the health and safety of people and the environment, and VWR International, LLC, a global laboratory supply and distribution company, have signed an expanded distribution agreement. Working with VWR through this new agreement provides broader and more streamlined ordering options for PerkinElmer's analytical consumables and minor accessories to the Company's clients in the academic, pharmaceutical, and analytical testing markets in North America.

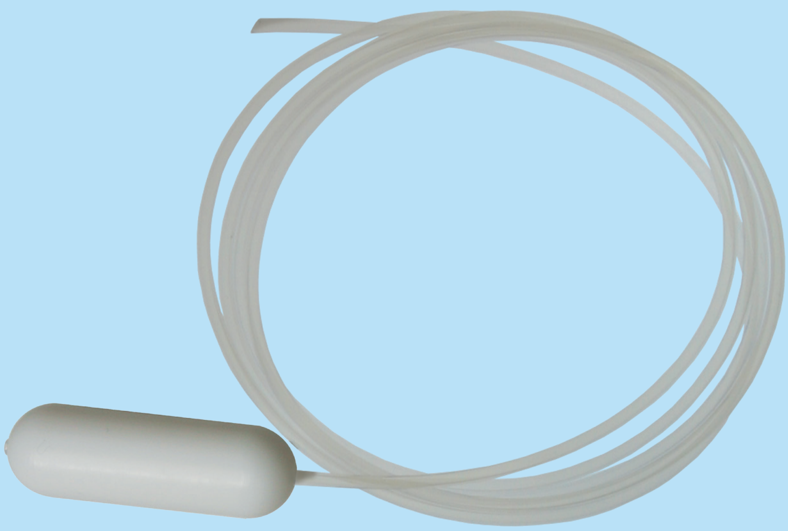
For scientists and lab technicians that require the performance and reliability of PerkinElmer-approved consumables and accessories for their PerkinElmer instruments, the new agreement brings greater convenience and efficiency to the ordering process. The agreement provides laboratory professionals with increased access to thousands of PerkinElmer consumable products and accessories, including PerkinElmer's analytical instrumentation portfolio of Spectroscopy and Chromatography solutions.

With 150 years of industry experience, VWR offers a well-established distribution network that reaches most of the world's top pharmaceutical and biotech companies, as well as industrial, educational and governmental organizations. For more information, or to order PerkinElmer products, contact VWR by visiting **www.vwr.com**.

HINTS FOR THE OPERATOR

How do you get the reagent tube to sit on the bottom of the flask?

Most operators will be familiar with tubing that seems to have a mind of its own. It can be very annoying when the tubing curls up and you find that the rinse or standard or sample is no longer being delivered. A simple way to overcome this problem is with a weight on the end of the tubing. A reagent tube with a PTFE sinker on the end is now available from Glass Expansion. You can order a pack of three under part number 70-803-0752.



From Spectro - Engineered efficiency that saves you money

Did you know that the SPECTRO ARCOS can shave off up to 30% of the conventional costs associated with ICP-OES analysis, while delivering unparalleled analytical performance for challenging and changing elemental analysis requirements?

ARCOS' unique and proprietary optical system allows unparalleled access into the deep UV without the need for expensive high purity purge gas. The patented UV-PLUS system is a closed loop system that recirculates and cleans the gas, maintaining a stable environment. It is always ready to analyze and can save you thousands a year in purge gas costs. Deployed by SPECTRO for over 20 years and installed in several thousand instruments around the world, UV-PLUS is a proven money saver.

The savings continue with SPECTRO ARCOS' Smart Move / Smart Rinse ultra-efficient autosampler logic that reduces sample analysis time by up to an astonishing 25%!

ARCOS is the only ICP that captures the complete spectrum with each and every measurement, saving you time now by not having to employ a special measure mode or measure again, should you need to investigate the results in more detail long after the sample has been measured.

To find out more about how you can save time and money with SPECTRO ARCOS, please visit: **www.spectro.com/arcos**.

From Teledyne Leeman Labs - New Application Note on the Analysis of Edible Oils using the Prodigy ICP

Edible oils are derived from a wide variety of plants and plant seeds and are used in many aspects of domestic and world-wide food production. Once the oil has been extracted from a plant seed, it is refined as needed for use in foods such as salad dressings, margarine, shortenings, snack foods and frying oils.

The elements of concern for the analysis of edible oils are: phosphorus, calcium, magnesium, iron, copper and nickel.

Significant concentrations of these metals can affect the flavor, color and stability of edible oils. To improve product quality, these substances are removed during the refining process.

This application note demonstrates the ability of the Teledyne Leeman Labs' Prodigy High Dispersion ICP to determine trace elements in edible oils. The Prodigy ICP provides high sensitivity and dispersion which, combined with appropriately chosen wavelengths and background correction points, can be used to provide accurate and reliable results for a large suite of elements in edible oils.

To receive a copy of technical note 1059, "The Analysis of Edible Oils using the Prodigy High Dispersion ICP", email: **LeemanLabsinfo@Teledyne.com** or visit our website at **www.LeemanLabs.com**.

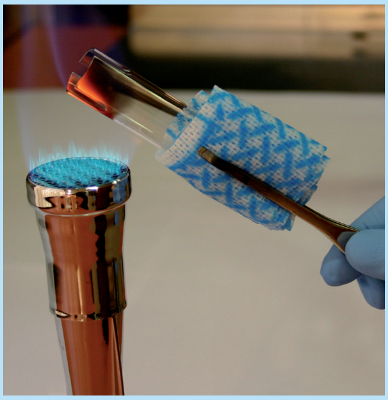
From Thermo Fisher Scientific - Thermo Fisher Scientific Expands Range of Powerful and Cost-Efficient ICP Emission Spectrometers

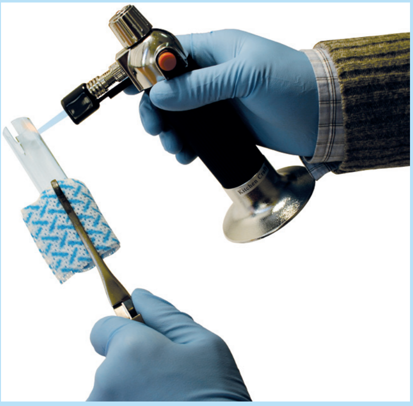
Thermo Fisher Scientific Inc., the world leader in serving science, has announced the launch of the latest addition to the award-winning Thermo Scientific iCAP 6000 Series of high-performance ICP emission spectrometers. The new iCAP 6200 ICP spectrometer is an affordable simultaneous, dual view ICP instrument specifically designed for routine analytical laboratories. The iCAP 6200 enables powerful analyses using a range of pre-optimized sample introduction settings and provides the capability to handle low to moderate sample numbers in a

HINTS FOR THE OPERATOR

What is the best way to remove carbon from a torch?

Organic samples such as oils can cause a deposit of carbon to build up on the outer tube or the injector. Traditionally, the most common method of removing carbon deposits due to organic samples has been to bake the torch in a muffle furnace at around 500°C. However, this is quite time-consuming and is not feasible for laboratories which do not have a muffle furnace. A fast and simple alternative is to burn off the carbon using a flame. This method can be





used for a one-piece torch or for the outer tube and injector of a demountable torch. It can also be used for the outer tube of the D-Torch. Note that the D-Torch outer tube cannot be heated in a muffle furnace since it has a polymer ferrule attached to it. A wet cloth should be wrapped around the ferrule to prevent it from over-heating when the end of the outer tube is heated in the flame.

wide variety of application areas, including environmental, food and beverage, toy safety and WEEE/RoHS testing. The new instrument harnesses the power of the Thermo Scientific innovative ICP technology, to deliver a reliable and cost efficient trace elemental analysis solution.

The iCAP 6200 is supplied with a range of pre-loaded method templates, developed to enable protocol compliant sample analysis in the Environmental, Food Safety, Toy Safety and WEEE/RoHS market sectors. These templates combine with the instruments pre-optimized sample introduction specifications to enable simple, consistent out of box operation and enable both novice and experienced ICP users to achieve powerful analytical performance with minimal method development requirements.

For more information about the Thermo Scientific iCAP 6000 series, please e-mail **analyze@thermofisher.com** or visit **www.thermo.com/icap**.

From Varian - Improved Productivity and Efficiency with Entry Level ICP-OES

Varian's entry level 710-ES axial and 715-ES radial ICP-OES spectrometers now offer improved productivity and optional mass flow control of the nebulizer gas flow. This option, available for both new instruments and existing instruments (with installation of an upgrade kit) ensures simple set-up, enhanced reproducibility and improved stability. The high efficiency signal processing in Varian's ICP Expert™ II version 1.1.3 software improves sample analysis times by up to 50%. Any laboratory performing routine determinations can benefit from the extra sample handling capacity, reduced gas consumption and lower consumables usage; or users can achieve better detection limits without any time penalties. Existing 710-ES Series ICP-OES owners: simply upgrade your software to the latest version to benefit from higher sample throughput and reduced running costs.

You can even combine this with new accessory options for the 710/715-ES to further enhance productivity. Varian's fully automated Switching Valve System (SVS) provides a further improvement in productivity of up to 33% and a 25% reduction in argon usage. When used with the SVS, the peristaltic pump on the 710/715-ES provides instant rinse solution to the SVS for fast washout, increasing sample throughput and reducing carryover.

To find out more about these productivity enhancements for Varian's 710/715-ES ICP-OES, please visit:

http://www.varianinc.com/products/promotions/us_710speedup.html

INTERNATIONAL

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