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Since high productivity analysis is at issue, only automated operation will be considered, i.e. an unattended autosampler run. Figure 1 illustrates the stages of analysis for a typical autosampler run on an ICP-AES or ICP-MS spectrometer. Most of the stages can be shortened by one means or another.

APPLICATION SPOTLIGHT

Increasing the Productivity of ICP Analyses

INTRODUCTION

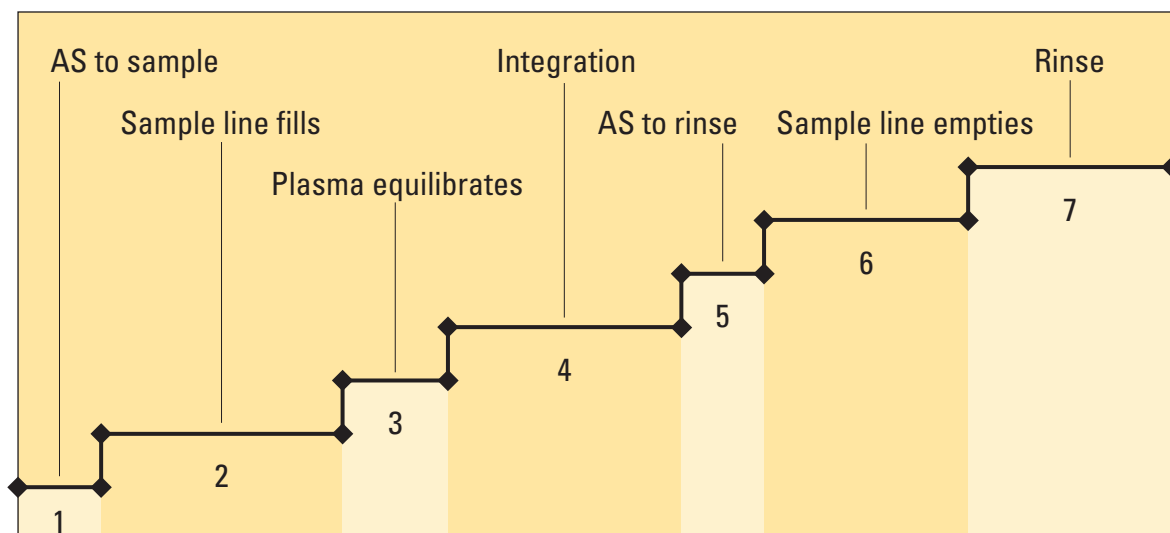
This application note is based upon a poster paper that was presented at the 2005 FACSS Conference in Quebec City, Canada. The paper was presented by Jerry Dulude of our US office and was entitled, **Techniques for Increasing Productivity in ICP Spectrometry**.

As both ICP-AES and ICP-MS spectrometers become more and more an integral part of the production laboratory, the emphasis on productivity is increasing. This article will examine a number of ways in which the length of the analytical cycle can be reduced without jeopardizing the quality of the results. These methods include the selection of the ICP-AES configuration, choice of autosampler and sample introduction components, and software and hardware approaches for shortening or eliminating the rinse step.

EFFECT OF SPECTROMETER & AUTOSAMPLER SELECTION

The choice of an ICP-AES spectrometer in particular can have a significant effect on the measurement time (Stage 4). This discussion will be confined to chip-based array detectors since they are now the most common ICP-AES detectors. One concern is the number of exposures which need to be taken to cover the analytical range. Systems which cover the entire analytical range in one exposure are the fastest. Some systems need to divide the spectrum into two exposures in order to achieve sufficient resolution. On top of this, there is the choice of axial, radial, and dual view systems. Dual view systems take one exposure in the axial mode and another in the radial mode. Therefore a worst case scenario with respect to speed would be a dual view system with two spectral regions for a total of four exposures. Another consideration is the extra time needed for data processing, which can be significant. The only true comparison is to time competing systems using the same number of sample measurements and the same integration and rinse times.

Figure 1. Sample cycle for unattended ICP analysis



Similarly, autosamplers vary in the speed at which the arm moves. A very fast moving system can shave 10 or more seconds off the sample cycle time (Stages 1 and 5).

EFFECTS OF THE SAMPLE INTRODUCTION SYSTEM

Some of the components of the sample introduction system can have a profound effect upon the speed of analysis. These components include the spray chamber, nebulizer, capillary tubing and any junction between components where dead volume may be an issue (stages 2, 6, and 7).

Nebulizer

All nebulizers must be linked to the sample line via a connector of some type. Glass Expansion has for many years manufactured its nebulizers to accommodate the EzyFit connector (Figure 2). The EzyFit consists of a Teflon plug that fits snugly inside the sample port of the nebulizer. When properly installed, the plug will rest against the back wall of the port resulting in zero dead volume. The presence of dead volume within the sample introduction system requires increased rinse time to prevent carryover to the next sample.

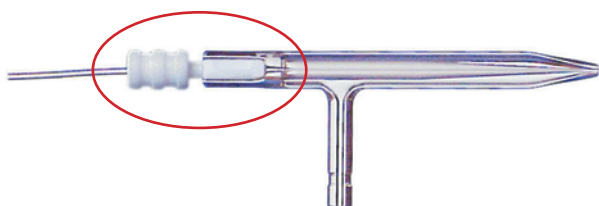


Figure 2. EzyFit Sample Line Connector on Conikal Nebulizer

Spray Chamber

There are a number of considerations to be made when selecting a spray chamber for high productivity. The required rinse time is dependent upon both the internal volume and the area of internal surface. A typical cyclonic spray chamber has a 50 ml internal volume while a Scott style chamber has 2 to 3 times the volume and therefore requires a much longer washout time. Also, surface area is minimized in a spherical chamber and the cyclonic chamber closely approaches this geometry. Another potential source of carryover is the nebulizer port of the spray chamber. If not designed properly, this can be a source of sample build-up, requiring a lengthy rinse stage to remove. All Glass Expansion spray chambers are designed with a wide bore nebulizer port so that any solution will drain quickly. Another advance in spray chamber design has been the Helix™. The Helix is designed so that a Teflon ferrule seals the spray chamber and minimizes dead volume (Figure 3).

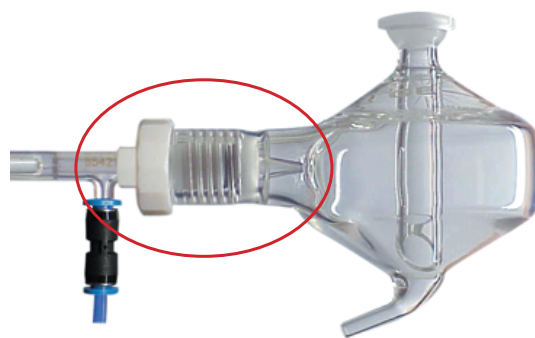


Figure 3. Twister Cyclonic Spray Chamber with Helix Nebulizer Fitting.

The Sample Line

The sample line consists of all components between the sample itself and the nebulizer, and includes the autosampler probe and attached capillary tubing, peristaltic pump tubing and capillary tubing to the nebulizer. The sample flow can be increased by reducing the diameter of the tubing. Capillary tubing with 0.75mm ID is commonly used but this can be reduced. For fast sample flow, it is recommended to use 0.25mm ID capillary tubing throughout the system both before and after the pump. In many cases the length of the tubing can also be reduced to further shorten both the rinse and sample load steps (Stages 2 and 6).

SOFTWARE APPROACHES TO HIGH PRODUCTIVITY

Several software approaches have been used to shorten the rinse time. Although other approaches may have been implemented, three representative approaches will be described here.

Fast Pump During Rinse (Stages 2, 6, and 7)

Most spectrometer software systems can change the peristaltic pump speed at various stages of the analytical cycle. To speed the rinsing and refilling of the sample introduction system, the pump can be programmed to run at faster speed during these stages. Although a significant time saving can result from the use of a faster pump speed during rinse, an added equilibration time of 10 to 20 seconds is typically required to achieve plasma stability prior to analyte measurement.

Predictive Rinse (Stages 1, 2, 5, 6, and 7)

Some spectrometer software allows the autosampler probe to move on from the current sample prior to its measurement. This allows the analyte measurement to occur while the sample remains in the line even though the autosampler has moved to the rinse or the next sample. This requires careful timing but can result in a large improvement in productivity. For the following reasons, this approach is usually reserved for trend determination assays that do not require accurate or precise measurements, such as the determination of wear metals

in engine oils or agricultural assays:

- It is difficult to use QC checking programs with this approach since the measurement is not made until after the sampler has moved on.
- Precision may suffer due to changes in back pressure within the sample line. This is particularly significant once the air gap from autosampler movement goes past the peristaltic pump.

Intelligent Rinse (variable savings)

This is a more complex program but has been used to minimize rinse time without adversely affecting the quality of results. The program uses a form of QC checking on each sample and determines whether each analyte concentration exceeds each of 2 trigger concentrations (set point 1 and set point 2). The following protocol is implemented:

- If all element concentrations are below set point 1, the rinse step is eliminated.
- If any element concentration is greater than set point 1 and less than set point 2, the normally programmed rinse is performed.
- If any element concentration is greater than set point 2, the rinse is monitored until each element is less than set point 1.

This is an excellent program to use for high accuracy analyses and can be used with other QC protocols. The disadvantage is that for samples which are high in at least one element or when concentrations vary widely between elements, there may be little time saving over a standard automated run.

AN ACCESSORY TO ACHIEVE HIGH PRODUCTIVITY

The Niagara™ Rapid Rinse Accessory (Figure 5) significantly shortens the analysis cycle time and as a result achieves higher productivity and faster sample turnaround times. The Niagara begins the rinsing of the nebulizer and spray chamber the instant the sample measurement is completed and continues to rinse until the next sample is ready. Thus the rinse is carried out in the time that is usually wasted waiting for the sample and the rinse solutions to flow from the autosampler to the nebulizer. The data in Figure 6 demonstrates the capability of the Niagara in comparison to a standard system for removing boron which is considered a “sticky” element. In this experiment, an ICP-MS system was used in the normal mode with a 30 second rinse and then with the Niagara with a 10 second rinse. A time saving of 20% resulted from the use of the Niagara.



Figure 5. Niagara Rapid Rinse Accessory

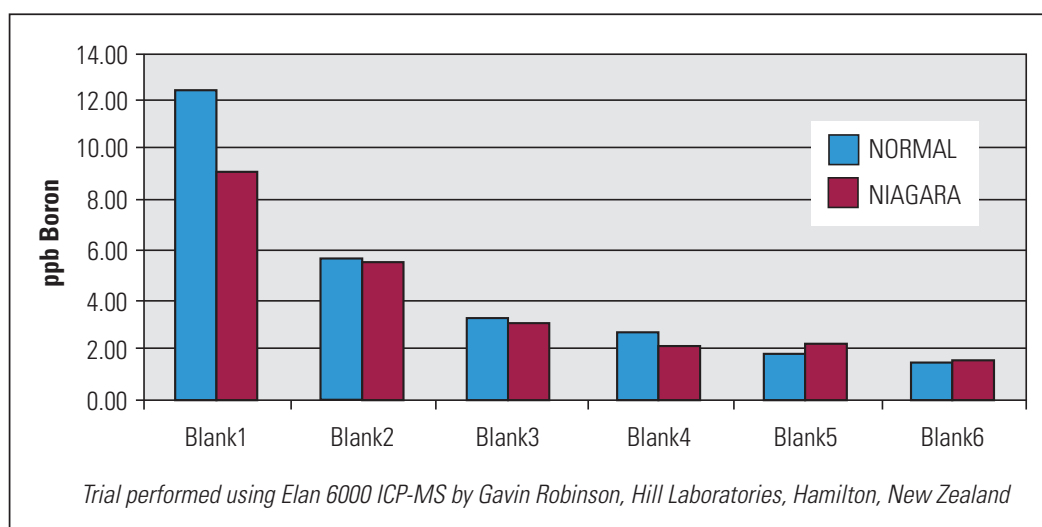
Figure 6. Boron Carryover from 5000ppb

Normal 30s wash

Uptake = 15s
Read delay = 8s
Data acq approx = 40s
Wash = 30s
Total estimated time = 1m33s
Total actual time = 1m40s
(additional time = autosampler moving etc)

Niagara 10s wash

Uptake = 15s
Read delay = 8s
Data acq approx = 40s
Wash = 10s
Niagara delay = 22s
Total estimated time = 1m13s
Total actual time = 1m20s
(additional time = autosampler moving etc)



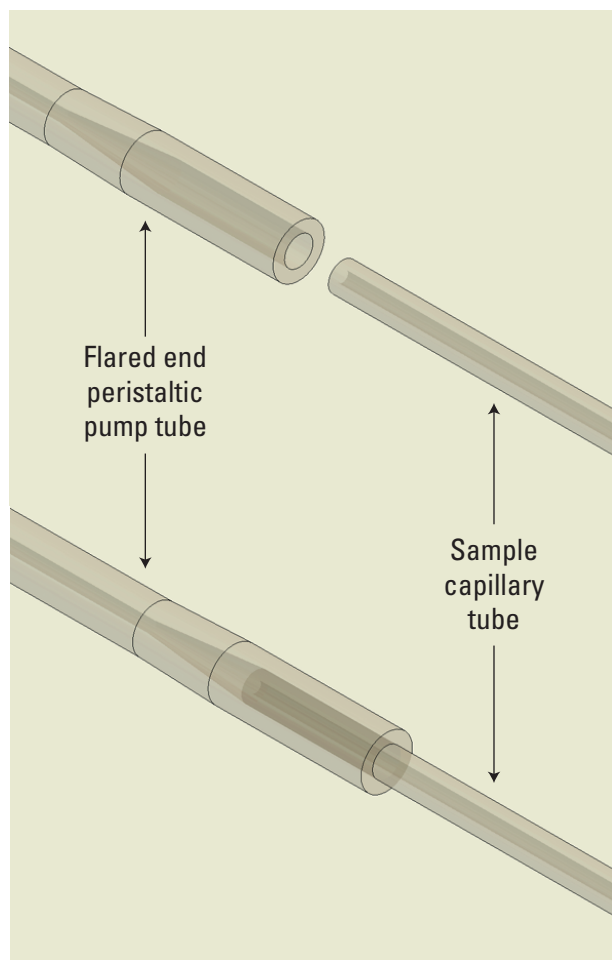
CONCLUSION

Through careful attention to the sample introduction system, the analytical cycle can be significantly shortened. For accurate analyses, even faster runs can be achieved using a fast pump software approach. Still faster analyses can be achieved with the Niagara Rapid Rinse Accessory.

NEW PRODUCTS

FLARED END PERISTALTIC PUMP TUBING

It can often be quite difficult to insert the sample tube into the peristaltic pump tube. This is particularly so if the peristaltic pump tube has a small internal diameter (ID). An example is the pump tubing used for the internal standard which usually has an ID of 0.2 to 0.4mm and is incompatible with the sample tubing OD of 1.3mm. To facilitate this connection, Glass Expansion now supplies flared-end pump tubing. The flared end of this pump tubing allows the larger sample capillary tubing to be inserted.



Any of the pump tubing that we normally supply can be supplied with flared end. This includes PVC 2-tag and 3-tag, Tygon MH 2-tag and 3-tag, Solva 2-tag and Viton 2-tag. The flared-end tubing is supplied in packs of 6 and the full range can be seen on our website at www.geicp.com. Simply click on "PRODUCTS" then "Pump Tubing".

INSTRUMENT NEWS

FROM AGILENT

Agilent has introduced new Options for the 7500 ICP-MS ORS Systems - 3rd Cell Gas Line. The 7500 Octopole Reaction System (ORS) systems are shipped as standard with two cell gas lines, which are used for helium and hydrogen cell gases. Agilent now offers the option to add a 3rd cell gas line, either at instrument purchase or as an upgrade. The ORS 3rd cell gas line option is available in 2 types – Low Flow (max. flow: 1mL/min – designed for Xe) and High Flow (max. flow: 10mL/min – designed for 10% NH₃ in He mix). More information about this option is found in the Agilent ICP-MS Journal #24 linked from www.agilent.com/chem/icpms.

FROM SPECTRO

SPECTRO, a global leader in Optical Emission and XRF Spectroscopy, has announced the launch of SPECTRO GENESIS - its low cost charged coupled device (CCD) inductively coupled plasma (ICP) offering. For the first time it offers laboratories, predominantly in the environmental market, a simultaneous CCD ICP system at the price of a sequential system to provide a higher sample throughput at a low price. The unit comes factory calibrated and optimized for ease of using SPECTRO's patented ICAL (Intelligent Calibration Logic) system. SPECTRO GENESIS sets the benchmark for SPECTRO's "It can be this simple" campaign as it is the only ICP system on the market that does not require any fine adjustment, offering true "plug in and analyze" functionality. The sample introduction system does not require any adjustment or optimization therefore saving time in both analysis and training while ensuring optimum results regardless of who is operating the system.

The new SPECTRO Genesis spectrometer delivers a comprehensive range of benefits, including:

- Speed and performance - Simultaneous CCD ICP speed with AA ease-of-use.
- Low price - Simultaneous CCD ICP system at the price of a sequential system plus minimal installation and training outlay.
- Fully US-EPA, ISO, EN and DIN Compliant.
- Seamless laboratory integration - Smallest bench top footprint currently available on the market with state of the art XML (Extended Markup Language) LIMS interface.
- ICAL powered for enhanced ease of use - ICAL (Intelligent Calibration Logic) is a completely novel system. The software-based system monitors and controls the state of the instrument independent from external influences. Thus, the time-consuming standardization of the spectrometer can be eliminated.
- Requires no method development – Unit can be ordered with factory calibrated method packages for environmental applications.

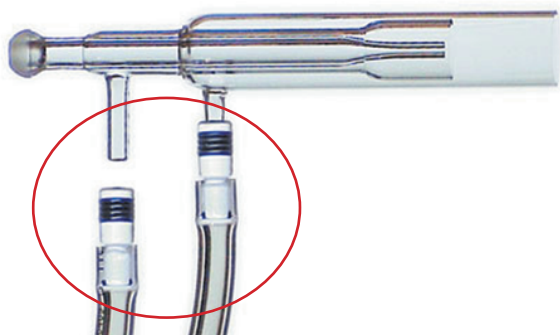
HINTS FOR THE OPERATOR

Making the right connection

The typical ICP sample introduction system comprises a number of components and several different types of tubing. It is important to most operators that individual tubes and components can be quickly and easily connected and disconnected for replacement without the whole system needing to be dismantled. In the past, disconnections were often difficult (and sometimes hazardous) with tubing needing to be cut away with a blade. In recent years, Glass Expansion has released a range of products with the objective of ensuring that all connections can be made quickly, easily and safely. Some of these products are:

GAZFIT

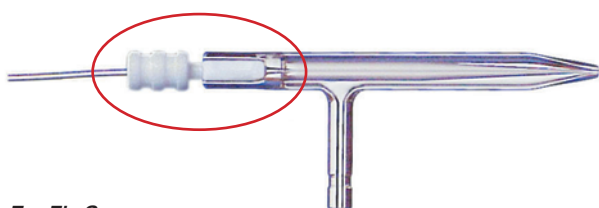
The most common method of connecting the argon gas supply to the torch used to be to slide the gas tubing over the side arm of the torch. Unfortunately, the tubing often adhered to the quartz side arm of the torch and needed to be cut away when the torch needed changing. The GazFit connectors slide smoothly and easily over the torch side arms, provide a gas-tight seal and never adhere to the arms. They are available to fit 4, 5, 6 and 8mm torch side arms.



The GazFit Connector

EZYFIT

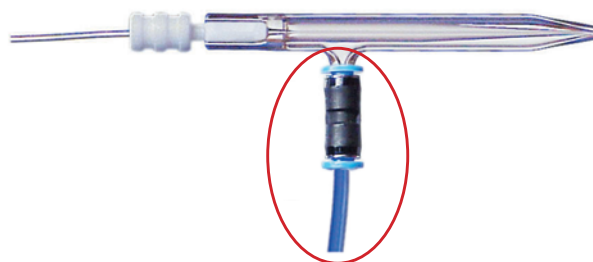
The EzyFit is the preferred method of connecting the sample tubing to the nebulizer. It has minimal dead volume in order to facilitate wash-out and enable rapid sample throughput. It also enables the sample tubing to be changed quickly and easily. EzyFit connectors are available with tubing internal diameters from 0.07 to 0.75mm to suit all necessary nebulizer sample uptake rates.



The EzyFit Connector

EZYLOK

The EzyLok is a quick-connect fitting to connect the argon gas supply to the nebulizer. As with the connection of the argon supply to the torch, the old method was to clamp the argon tubing over the side arm, often resulting in the tubing becoming bonded to the nebulizer arm and needing to be cut free. The EzyLok provides a simple one-click connection or disconnection and gives a reliable gas-tight seal. Nevertheless, we recommend that any argon connection to the nebulizer be regularly checked with a leak detector solution such as Snoop.



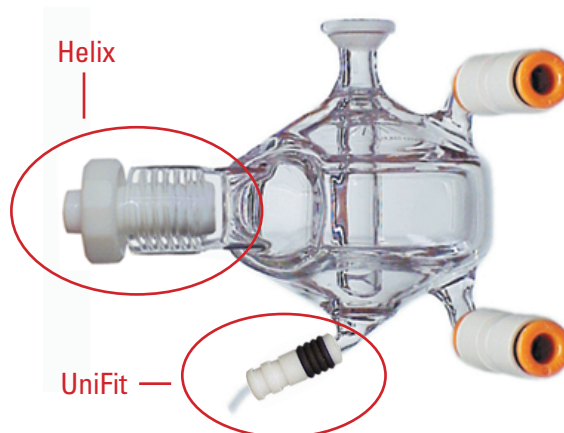
The EzyLok Connector

UNIFIT

The UniFit connector is the most convenient method of connecting the drain tubing to the spray chamber for spray chambers designed for a pumped drain. It fits any spray chamber with a standard 4mm drain and slides easily over the drain outlet. The tubing can also be easily inserted into the peristaltic pump tubing.

HELIX

The Helix is the recommended interface between the nebulizer and the spray chamber. It reduces dead volume to give faster wash-out, is totally inert to strong acids and organic solvents, has no o-rings to replace, seals the spray chamber, locks the nebulizer in place, and allows the nebulizer to be quickly and easily inserted and reproducibly positioned.



The UniFit Connector and Helix Fitting

FLARED END PUMP TUBING

The sample tubing is usually connected to the peristaltic

pump tubing by simply sliding one tube into the other. This is fine if the OD of the sample tubing is close to the ID of the pump tubing. However it can be quite difficult if the pump tubing has a small ID, as is usually the case with tubing used for an internal standard. To facilitate this connection, Glass Expansion is now offering peristaltic pump tubing with flared ends.

For more information on any of the connection systems discussed in this article, please contact enquiries@geicp.com.

torches, RF coils and accessories (including the new Niagara Rapid Rinse Accessory). Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP.

GLASS EXPANSION NEWS

FREE DELIVERY

Glass Expansion is now offering FREE DELIVERY for orders placed through our online shop with a value of more than US\$1,000 (EURO 840, GBP 570, A\$1,500 or JPY 120,000). We hold most products in stock ready for immediate dispatch and delivery to most countries takes 2-4 days. If you are not sure which part you need, click on PRODUCTS, then "Products to suit your ICP". If you know the part number you need, you can go straight to SHOPPING CART to place your order or view the Ordering Instructions for full details. Even if you do not wish to order online you will be able to access the current prices.

CHOICE OF LANGUAGE

Most of the Glass Expansion website is now conveniently available in English, French, German, Spanish, Chinese, Japanese and Russian and will shortly also be available in Italian. Navigation is simple and you can switch languages at any time using the language buttons at the top of each page.

FREE MOUSE MAT

The Glass Expansion Nebulizer and Spray Chamber selection guides have proved very popular with ICP users, enabling them to select the most appropriate components for their application. We have now produced a computer mouse mat with the Nebulizer Selection Guide on the front and the Spray Chamber Selection guide on the back. This mouse mat has an easy-to-use ergonomic shape, is ultra-thin so it does not clutter the desk, has a non-slip backing so it does not move around and an embossed top for unequalled mouseball traction. Please forward your mailing address to enquiries@geicp.com if you would like to have one sent to you free-of-charge.

WINTER CONFERENCE

A wide selection of Glass Expansion products will be on display at the 2006 Winter Conference on Plasma Spectrochemistry, Tucson, Arizona, USA, January 8 – 14, 2006. The display will include nebulizers, spray chambers,