GLASS EXPANSION NEWSLETTER

Quality By Design

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CONTENTS

APPLICATION SPOTLIGHT

SELECTING THE RIGHT ICP SPRAY CHAMBER

The Applications Spotlight in this issue is a follow up to last issue's on nebulizer selection. The sample introduction system of an ICP spectrometer includes all components involved in carrying the sample from the autosampler to the spectrometer, as shown in Figure 1. This paper will focus on the spray chamber component of the system.

The function of the spray chamber is to filter the aerosol produced by the nebulizer so that only the smallest droplets reach the plasma. The most commonly used spray chambers in plasma spectrometry are the cyclonic, barrel (or Scott type), and conical (Figure 2).

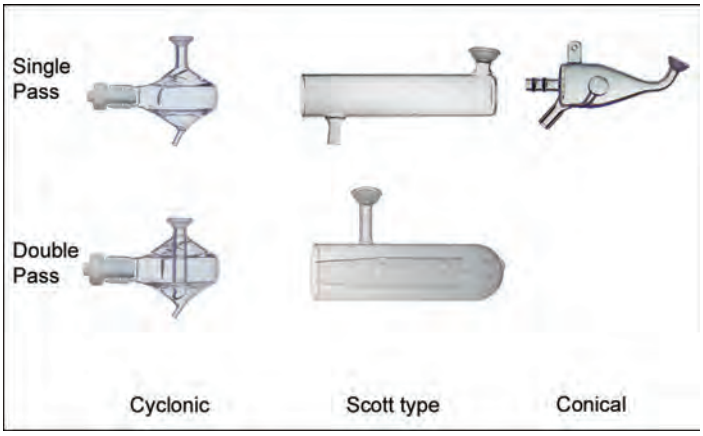
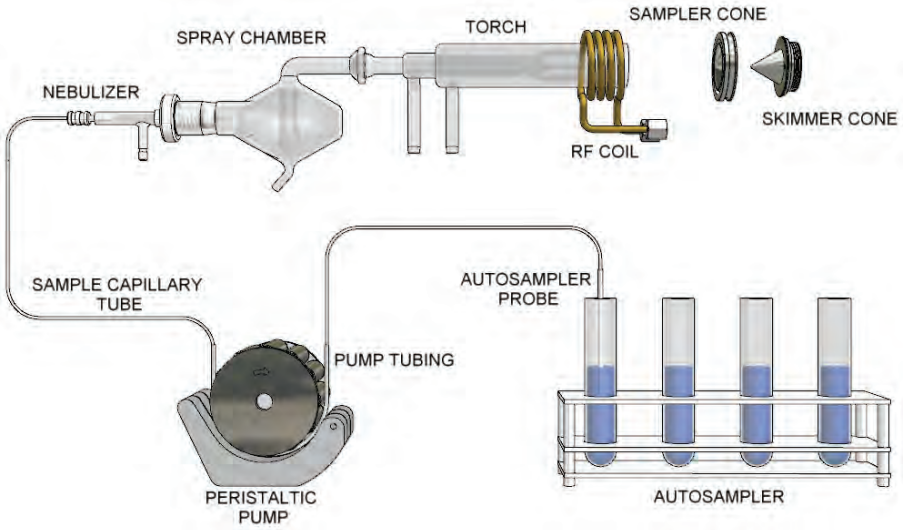


Figure 2: Comparison of spray chamber types

Notice that the cyclonic and Scott style spray chambers are available as both single and double pass (or baffled) configurations. The double pass acts as a secondary filter to further reduce the mean droplet size. The conical spray chamber uses a built-in impact bead to create a finer mist. In all three designs, gravity is used to remove the larger drops and divert them to the drain. Only in the cyclonic

Figure 1: Schematic of an ICP-MS sample introduction system



design is centrifugal force also employed to remove the larger droplets. This provides faster washout and also reduces the possibility of renebulization which can contribute to poor precision. For this reason, the remainder of this discussion will be confined to cyclonic spray chambers.

Cyclonic Spray Chamber Configurations

There are a number of cyclonic spray chamber configurations, as shown in Figure 3 and described below.

- •Glass Tracey: 50ml internal volume, borosilicate glass
- •Glass Twister: 50ml internal volume with central baffle, borosilicate glass
- •Glass Cinnabar: 20ml internal volume, borosilicate glass
- •Glass Twinnabar: 20ml internal volume with central baffle, borosilicate glass
- •Tracey TFE: 50ml internal volume, inert polymer
- •Tracey PFA44: 44ml internal volume, inert polymer

The Cinnabar and Twinnabar chambers have a smaller internal volume (20ml as opposed to 50ml) and are recommended for applications which utilize low sample uptake. With these applications, they provide better sensitivity and faster washout times. The baffled Twister and Twinnabar spray chambers act as droplet size cutoff filters that yield a smaller mean droplet size for the tertiary aerosol. This means that the Twister will produce a quieter more robust plasma (less noise) and the Tracey in general will produce greater overall intensities (more signal). The net effect on detection limits (signal/noise) is small and varies with application. The design and construction of the cyclonic spray chambers are critical if optimum performance of the spectrometer is to be attained. For example, Figure 4 describes some of the important design characteristics which must be precisely controlled for a glass baffled cyclonic spray chamber.

Materials for Cyclonic Spray Chambers

Borosilicate glass is by far the most widely used material for spray chambers because of its relatively low cost and excellent wetting characteristics. Quartz (silicon dioxide) is more difficult to work with (requires higher temperatures to soften) but is sometimes preferred when low level boron work is attempted. Polymer-based spray chamber materials include polypropylene (PP), perfluoroalkoxy (PFA) and polytetrafluoroethylene (PTFE). These are often preferred when ultra trace levels of analytes are measured with an ICP-MS or when hydrofluoric (HF) acid is present in the samples. Work performed by Dr. Canals, et al at the University of Alicante in Spain in collaboration with Glass Expansion demonstrated that the PFA and PTFE materials gave performance closest to glass, but only if the interior surface was chemically modified to alter the hydrophobic nature of the material (Figure 5). Table 1 defines the abbreviations used in Figure 5. As a result of these tests, all of Glass Expansion's ICP-OES polymer spray chambers are made of PTFE and all ICP-MS polymer spray chambers are made of PFA (due to the higher purity required).

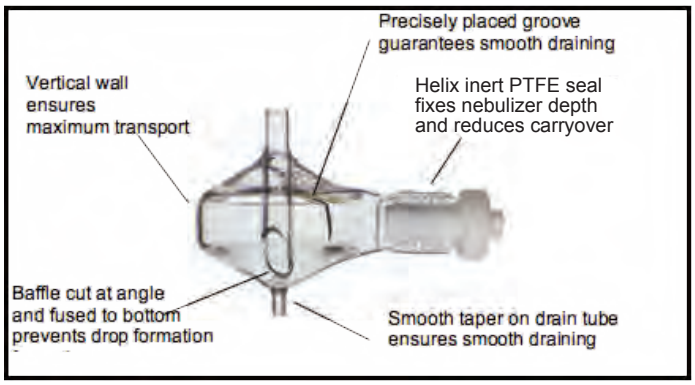


Figure 4: Design of a baffled cyclonic spray chamber

Figure 3: Cyclonic spray chamber configurations

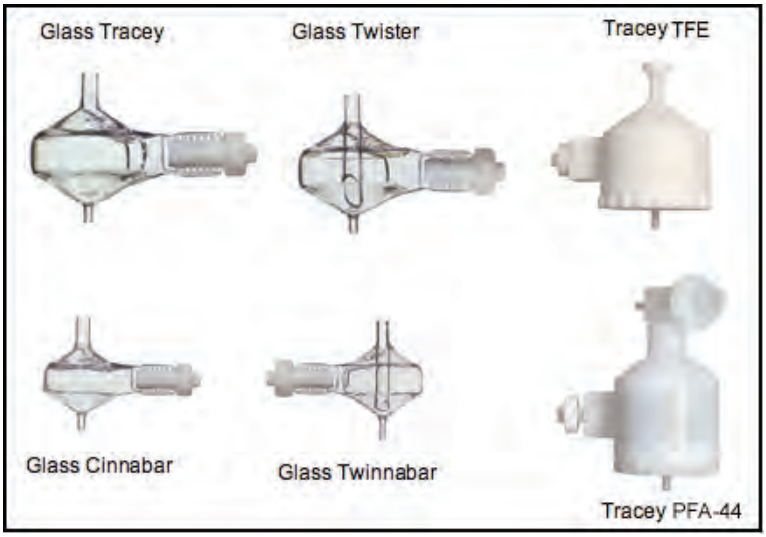


Table 1: Definitions for Figure 5

Polypropylene, 50ml	Tra PP 50
PTFE, 50ml	Tra TF 50
PTFE, 44ml	Tra TF 44
PEI, 44ml	Tra PEI 44
PFA, 44ml	Tra PFA44
PPS, 44ml	Tra TC 44
PEEK, 44ml	TraPEE 44

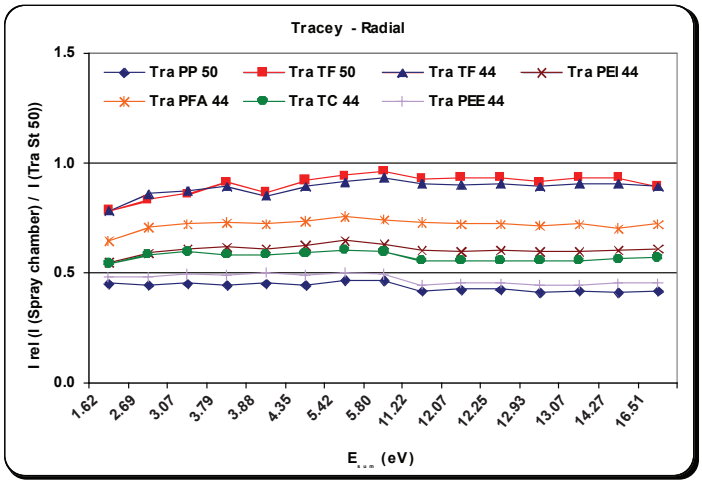
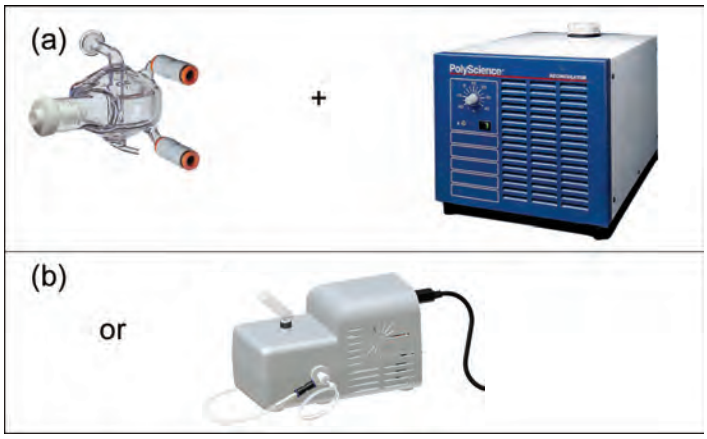


Figure 5: Comparison of various spray chamber materials and volumes relative to 50ml glass Tracey chamber

Controlling Spray Chamber Temperature

For some applications, it is desirable to heat or chill the spray chamber or simply to prevent the temperature from varying. For these applications, glass and quartz spray chambers in jacketed configurations have been used with an external supply to pump temperature controlled liquid through the chamber housing. A more elegant solution is a stand alone Peltier driven device which replaces the existing chamber. Examples of both of these are shown in Figure 6.

Figure 6: Temperature-controlled spray chambers; (a) jacketed spray chamber plus external chiller; (b) IsoMist™ Programmable Temperature Spray Chamber (Glass Expansion)



Because of the bulkiness of the external chillers and the complexity of setting up coolant tubing, these have traditionally been employed only when absolutely necessary, typically for the analysis of volatile organic solvents that would otherwise extinguish the plasma. It is interesting that now that convenient and elegant solutions to this challenge are available that more applications have become evident (see Table 2).

The IsoMist is now available with a choice of spray chamber material - glass, quartz or PFA, each of which is interchangeable with the others.

Summary

Cyclonic spray chambers are available to meet the demands of any application. The specific configuration chosen depends upon the sample type, stabilizing acids, and the goals of the analysis. Table 3 below summarizes the applications of each of the spray chambers described above.

Table 2: Applications of Temperature Controlled Spray Chambers

Low temperature	Volatile organic solvents
Low temperature	Oxide reduction in ICP-MS
Elevated temperature	Sensitivity enhancement for micro samples
Elevated temperature	Viscosity reduction
Constant temperature	Holding calibration in environmental labs
Constant temperature	Higher accuracy for precious metal assays
Constant temperature	Higher productivity due to fewer recalibrations

Table 3: Applications of Cyclonic Spray Chambers

Aqueous non-HF applications requiring highest signal	Glass Tracey
Same as above with sample uptake of 0.2ml/min or less	Glass Cinnabar
Aqueous non-HF applications requiring best precision	Glass Twister
Same as above with sample uptake of 0.2ml/min or less	Glass Twinnabar
All organic solvent applications	Glass Twister
ICP-OES applications involving HF	TFE Tracey
ICP-MS applications requiring highest purity or involving HF	PFA Tracey

INSTRUMENT NEWS

From Agilent Technologies – New "MassHunter" Software

Agilent Technologies has introduced the new "MassHunter Software" Platform for the 7500 Series ICP-MS. The MassHunter Workstation software is based on the highly successful MassHunter LC/MS software and will become common to all Agilent MS products. There are many new features, but key features of MassHunter Workstation (A.01.01) are "Batch-at-a-glance data review", "Outlier flagging" and "Reporting via Excel 2007". From this new Batch-at-a-glance data review window, all necessary information can be easily reviewed – e.g. analytical results, spectra, calibration plots, ISTD stability chart, allowing the user to review analytical results more easily. Outlier values, such as count RSD, ISTD recovery, calibration linearity, calibration range, etc. are automatically flagged and displayed in the batch table. MassHunter Workstation ships with Microsoft Excel 2007. Export menu directly generates an Excel file and it saves data analysis time. The MassHunter Workstation for the 7500 Series is part of the Agilent MassHunter Workstation family – currently used on Agilent TOF, QTOF and triple quadrupole LC/MS systems. LC/MS users will find the same familiar interface on ICP-MS.

From PerkinElmer - Janus Automated Workstation For Wear Metals Analysis Preparation

PerkinElmer Life and Analytical Sciences, a global leader in application-focused measurement and analysis solutions, has announced a new workstation specifically designed to automate sample preparation of lubricants for wear metals analysis. The 8-tip high throughput JANUS® Oil Prep Workstation enables laboratories to replace manual or single-tip sample preparation methods with an automated system to accelerate testing for wear in large capital equipment including heavy machinery, mining and farming.

The JANUS Oil Prep Workstation automatically dilutes oil samples with kerosene in preparation for wear metals analysis by inductively coupled plasma (ICP), such as with PerkinElmer's Optima™ ICP-OES. Utilizing proprietary liquid level sensing technology and 8-tip pipetting, the system significantly improves productivity and efficiency versus manual or single-tip systems. Use of disposable tips with JANUS eliminates time consuming tipwash steps required with fixed-tip systems, accelerating throughput and significantly reducing the volume of waste solvent. Additionally, use of disposable pipette tips minimizes cross contamination, reducing false-positive results that may require significant time and expense to re-test.

For more information visit
www.perkinelmer.com/labautomation

From SPECTRO - Japanese Office and Demonstration Facility

SPECTRO Analytical Instruments has opened an instrument demonstration and application laboratory along with a direct sales, service and marketing office in Tokyo near Shinagawa station to better serve its growing Japanese customer base. SPECTRO has offered arc/spark, ICP optical emission and X-ray fluorescence spectrometers in Japan since the 1980s with more than 700 systems installed countrywide.

SPECTRO operates the facility jointly with EDAX. Both SPECTRO, based in Germany, and EDAX, based in the United States, are units of AMETEK Materials Analysis Division, which is a global leader in applying analytical instrumentation to satisfy the materials analysis needs of its customers. The products and technologies available include arc/spark and ICP optical emission spectrometers, energy dispersive and EBSD microanalysis systems, and X-ray fluorescence spectrometers for the fast accurate analysis of solids, liquids, and powders. SPECTRO and EDAX provide instruments for applications in the metals, environmental, R&D, petrochemical, pharmaceutical and electronics manufacture.

The new SPECTRO Japan facility is located at Shinagawa NSS Bldg, 13-31 Kohnan 2-chome Minato-ku, Tokyo 108-0075, Tel: +81.3.37405172, Fax: +81.3.37405307
In addition, SPECTRO and EDAX operate a joint sales and marketing office in Osaka, Japan. Its address is Shin-Osaka Yachiyo Bldg., 1-45, Miyahara 4-chome, Yodogawa-ku, Osaka 532-003, Tel: +81.6.63503815, Fax: +81.6.63503825

From Teledyne Leeman Labs - New Application Note on Biodiesel

With the high price of crude oil and a growing desire to utilize local resources, bio-diesel products are becoming an attractive alternative to traditional petroleum diesel fuels. Biodiesel is a renewable fuel produced from feedstock such as soy, canola, mustard, sunflower, coconut, palm and cottonseed oils. Accurate elemental analysis of biodiesel requires analytical methodology that is both sensitive and selective.

The Prodigy High Dispersion Inductively Coupled Plasma (ICP) is ideally suited for the analysis of biodiesel fuels. The Prodigy can determine up to 70 elements in a sample simultaneously in under 30 seconds. With the Prodigy, elemental content can be rapidly monitored throughout the entire processing cycle, starting with the raw oil and continuing through to the finished product. Prodigy's detection limits easily satisfy the requirements of the

ASTM standard for biodiesel.

To receive a copy of application note 1039, *"The Determination of biodiesel with the Prodigy ICP"*, visit our website at www.leemanlabs.com/resources/applications or email: LeemanLabsinfo@teledyne.com

From Thermo Fisher Scientific - Reliable Biodiesel Analysis Using the Radial Plasma View iCAP 6000 Series ICP Emission Spectrometers

Thermo Fisher Scientific Inc. has incorporated unique capabilities in the iCAP 6000 Series of ICP emission spectrometers to achieve dependable monitoring of elemental contaminants in biodiesel. The dedicated radial plasma view configuration of the system provides enhanced analytical capabilities for important elements such as sulfur and phosphorus while the enhanced matrix tolerance (EMT) torch and swing frequency RF generator easily handle organic matrix samples and ensure improved stability.

The vast majority of biodiesel production plants currently use plant oils as a starting material for production. However, these plants usually have relatively high phosphorous content which is undesirable in fuels as it can lead to the corrosion of mechanical components of engines. Sulfur also affects engine wear if present in excess concentrations in the starting materials and causes environmentally harmful sulfur dioxide emissions. EN 14214 and ASTM D6751 standards have been introduced specifying the requirements for biodiesel and its analysis. These documents require that the concentrations of elemental contaminants in biodiesel are regularly monitored and specify the method for its analysis, to ensure optimum engine performance and reduce environmental impact. This is especially important in the aviation industry- one of the predicted growth areas of biodiesel, where the first commercial airline flight using biofuel took place in February 2008.

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

HINTS FOR THE OPERATOR

Extend the life of your pump tubing

It is well known that peristaltic pump tubing wears out over time. As it deteriorates, the sample uptake changes, causing the signal to drift and necessitating more frequent recalibrations. Did you know that you can slow the rate at which the pump tubing wears by lubricating the pump rollers with the EzyGlide cloth (part number 70-EZG-CLOTH)? This improves precision, reduces drift and extends the period between recalibrations.

Thanks to Carl Garrison, Profiners Inc. for this helpful hint. If you have any hints that may help your fellow ICP users, we would love to hear from you.

Ensure proper spray chamber drainage

We sometimes hear of situations where the spray chamber fills with sample. This is usually due to an insufficient pump rate on the drain line. To ensure that there is no build up of sample in the spray chamber, the rate of pumping the waste solution should exceed the sample pump rate. Assuming the same peristaltic pump is used for the sample and drain, this is simply achieved by making sure the diameter of the pump tubing on the waste line is greater than that on the sample line. A helpful visual aid is to make sure that there are slugs of argon between the slugs of waste in the drain line. If the drain line has a continuous flow of waste, then the pump rate is insufficient. If the right tubing is being used and there is still a problem, check that the tension on the roller clamp is correct.