

NEWS

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APPLICATION SPOTLIGHT

SLURRY ANALYSIS BY ICP SPECTROMETRY

Introduction

Slurry is the name used to define a suspension of particles in a liquid. When a sample is not easily rendered into a liquid state, slurry analysis may be a reasonable alternative. Examples of these types of samples are refractory ceramics, coal, slags, rocks, and silicon carbide. The sample must be finely ground into consistently sized particles and suspended in an appropriate liquid. An example of a commonly analyzed slurry is used engine oil which contains a suspension of small metal particles resulting

from the wear of the engine components. If the particle size and density are optimized, the slurry can be routinely aspirated into an ICP-OES or ICP-MS and its metal content accurately determined. An excellent review by Ebdon et al,¹ provides a thorough review of this topic.

A number of atomic and mass spectrometric techniques have been used for slurry analysis, including those listed in Table 1.

To determine the relative usage of these techniques, we searched the archives of the Journal of Analytical

Atomic Spectroscopy (JAAS) from 1995 to the present and plotted the number of papers on each in Figure 1.

As you can see, the majority of papers utilized electrothermal vaporization either into an AAS, ICP-OES, or ICP-MS. This technique ►

Table 1

ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
GFAAS	Graphite Furnace Atomic Absorption Spectroscopy
ETV-ICP-MS	Electrothermal Vaporization ICP-MS
ETV-ICP-OES	Electrothermal Vaporization ICP-OES

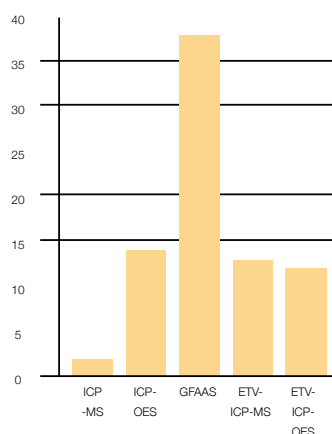


Figure 1: Number of published papers on slurry analysis in JAAS

KEY EVENTS

PITTCON 2009

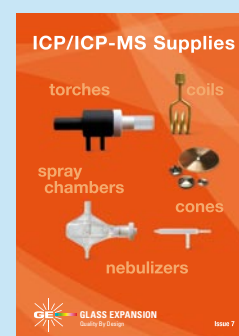
A wide selection of Glass Expansion products will be on display at Pittcon 2009, Chicago, USA, March 9 to 12, 2009. The display will include nebulizers, spray chambers, torches, RF coils, ICP-MS cones and accessories (including the new TruFlo Sample Monitor). Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP. Please visit us at Booth 1473.



PITTCON 2008

New 2009 Catalog

The new 2009 Glass Expansion catalog will be available in the next few weeks. This full color catalog lists nebulizers, spray chambers, torches, RF coils, ICP-MS cones, accessories and consumables for over 50 ICP-OES and ICP-MS models. If you would like your personal copy, please send your mailing address to enquiries@geicp.com.



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◀ allowed the analyst to introduce a discrete mass or volume of sample into the spectrometer. For ICP-MS and ICP-OES, slurry nebulization was utilized. Since these techniques are readily and commercially available, we will focus on these. Note that there were significantly more papers on ICP-OES, due presumably to the more fragile nature of the ICP-MS sampling interface.

Sample Preparation

Sample preparation is of particular importance for a slurry sample. First and foremost, the particle size must be small enough so that the analytes behave the same as they would in a solution. This enables the use of solutions as standards and that is certainly the best and easiest way to proceed as the alternative is to standardize with a known slurry standard. Secondly, the particulates must be dispersed in the proper liquid or dispersant in order to achieve homogeneity. Lastly, the concentration of the slurry must be optimized to achieve ample analyte intensity without resulting in a degradation of the sample introduction system or interface.

Particle size

To achieve close to 100% recovery using solution standardization, a consistent particle size of 3 microns or less is generally necessary.^{2,3} Goodall, et al. wisely included the density of the particle into the determination such that the higher the density the smaller the particle size needed to be.⁴ A variety of grinding methods have been used to achieve the optimum particle size and some of the more common ones are listed below ¹:

- Bottle and bead method
- Mixing mill
- Micronizing mill
- Pack-type grinder
- Pack-type grinder followed by rotary grinder
- Grinding mill
- Vibration pot mill

Dispersants

The proper dispersant prevents conglomeration and sedimentation of the particles. Particles which are lyophilic have a high affinity for the liquid in which they are dispersed and are easy to put into a homogeneous slurry. Lyophobic particles, on the other hand, have low affinity for the surrounding liquid and therefore must be modified with stabilizing agents to create a more lyophilic surface ⁵. A listing of the various dispersants used and the samples to which they were applied is given by Ebdon et al. ¹

Slurry concentration

For ICP-OES, Ebdon and Wilkinson found that the optimum concentration (maximum signal to noise ratio) was achieved at 20% m/v ⁶ for coal slurries. For ICP-MS, cone problems resulted from slurry concentrations as low as 1% m/v ⁷, but were eliminated by a 20X dilution. This means that the ICP-OES is able to handle slurries 400 times as concentrated as the ICP-MS, which in most cases eliminates the sensitivity advantage of the ICP-MS.

Mixing

Once the particles have been successfully dispersed in the proper liquid, the slurry in many cases will require mixing while it is being pumped to the nebulizer. Magnetic stirring is a good choice as long as the particles do not contain iron. Alternatively, vortex stirring can be applied.

Sample introduction system

When choosing the components of the sample introduction system for the analysis of slurry samples, there are certain considerations that should be made in order to improve both the performance and ruggedness of the ICP spectrometer.

Nebulizers

Much of the literature describes a modified Babington style nebulizer such as the quartz V-groove nebulizer for slurry nebulization ^{6,8,9}. In this design, the sample is pumped through the nebulizer body and allowed to pass through the V-shaped groove in the nebulizer end over the gas orifice which breaks up the liquid into an aerosol. Our ceramic VeeSpray nebulizer meets these criteria and is designed to accommodate all solvents including hydrofluoric acid (HF). In addition, the outlet end of the nebulizer is contoured to produce smooth and consistent flow of liquid through the groove (Figure 2).

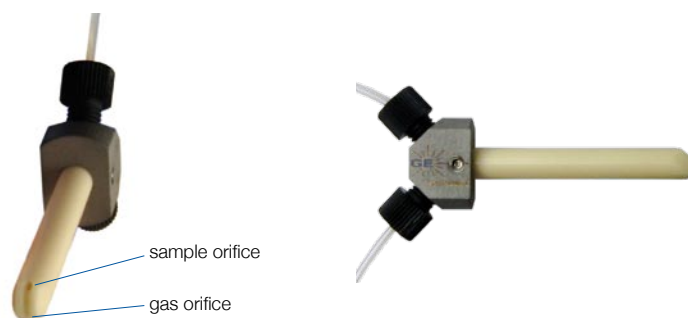


Figure 2: VeeSpray modified Babington nebulizer

We also offer a concentric glass nebulizer that is designed specifically for slurries, not so surprisingly called the Slurry nebulizer (Figure 3). The Slurry nebulizer can accommodate particles up to 150 microns in diameter, well above the preferred maximum size of 3 microns. Because it is a self-aspirating nebulizer (unlike the VeeSpray or V-Groove), it is less sensitive to the pulsations of the pump and therefore provides better precision in general. However, since it is made of glass, it cannot be used with samples containing HF.



Figure 3: Slurry nebulizer (concentric glass)

Spray chambers

To minimize the mean particle size reaching the plasma, some kind of double pass spray chamber is recommended. The Twister spray chamber shown in Figure 4 not only contains a baffle but also minimizes carryover, as it has a much smaller internal volume than a standard Scott style chamber. ►



Figure 4: Twister baffled cyclonic spray chamber with Helix fitting

◀ Work has been reported that shows that cooling the spray chamber results in improved analyte signal.¹⁰ The IsoMist Programmable Temperature Spray Chamber (Figure 5) has a range of -10 to +60°C and incorporates a Twister spray chamber, making it an ideal system for the analysis of slurries.

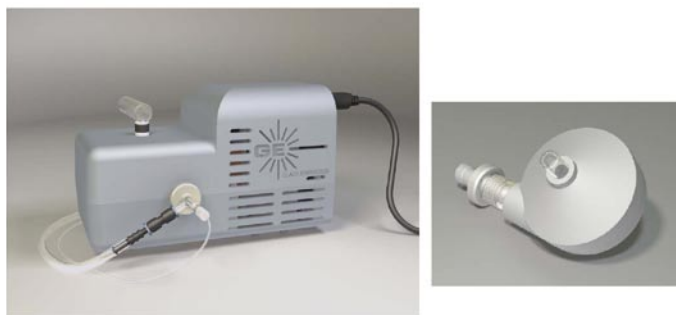


Figure 5: IsoMist Programmable Temperature Spray Chamber with encapsulated Twister cyclonic

Torches

The torch injector is the critical component with respect to analytical performance for slurries.¹² The injector orifice needs to be as large as possible and still cause adequate piercing of the plasma. The larger the orifice the lower the particle velocity in the plasma and the more complete the atomization, excitation, and ionization processes. A 3mm bore injector was found to be optimum in two studies.^{11, 12} Since most single piece quartz torches are equipped with injectors of smaller bores than this, a semi or fully demountable torch may be an easier way of assembling the proper torch for slurries (an example of a semi-demountable torch is shown in Figure 6).



Figure 6: Semi-demountable quartz torch with interchangeable injector

Cones

For ICP-MS, it is more important to have all particles below a specific size. In ICP-OES, the larger particles that do not completely atomize are carried up the exhaust, while in ICP-MS, they are carried into the cones and may result in clogging. Cones with enlarged bores are available for many instrument models to accommodate high dissolved solids and these would be preferred for slurries as well.

Internal standardization

Use of an internal standard in solution is a good way to compensate for transport efficiency changes. An in-line reagent addition kit such as the Trident™ accessory provides both automation and modularity. In order to compensate for atomization differences of the particles, one may be able to use a major constituent of the particle as the internal standard. For example, silicon could be used as an internal standard for trace analysis of silicon carbide.

Mixed gas plasmas

For some very refractory samples, it may be difficult to efficiently atomize even very small particles. It has been found that the addition of an auxiliary gas (O₂ or N₂) increases the temperature of the plasma thereby enhancing the atomization of these refractory samples.¹³

Summary

Slurry analysis is a viable alternative to sample digestion, particularly when the sample is refractory. A complex and lengthy digestion can often lead to contamination as well as the presence of acids which can cause matrix interference and/or present personal injury potential. As long as the slurry sample is properly prepared and suspended in solution, and the appropriate sample introduction system is employed, a simple solution standardization should provide accurate results. ■

References

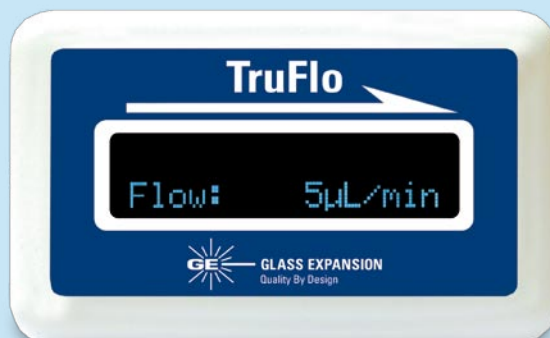
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NEW PRODUCTS

TruFlo Sample Monitor

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. Please contact **enquiries@geicp.com** for more information.



INSTRUMENT NEWS

From Agilent Technologies – “New “MassHunter” Software Revision

Agilent Technologies has introduced a new revision of the ICP-MS MassHunter software for the 7500 Series ICP-MS. Major improvements in revision A.02.01 include expanded chromatographic data analysis functions, such as more flexible manual integration, together with improved isotope ratio operation, now incorporated in the main Batch View data table. This greatly simplifies isotope ratio data analysis by allowing easy access to the electronic data, including all replicate measurements. All users of ICP-MS MassHunter revision A.01.01 are entitled to a free upgrade to the new revision.

From PerkinElmer – Web Site Tailored To Meet Needs Of Environmental Customers

PerkinElmer Life and Analytical Sciences has introduced its enhanced environmental market Web site. The site provides improved functionality and searching capabilities to enable environmental scientists to quickly obtain information on regulatory compliance, methods or standards, applications and product information.

“The development and launch of the new environmental Web site is an integral part of our EcoAnalytix™ initiative and highlights our continued commitment to support the needs of our customers in the environmental market,” said Richard Begley, Ph.D., president, Analytical Sciences, PerkinElmer. Begley noted that extensive customer input from usability tests, focus groups, surveys and demonstrations was a key driver in the enhancements, which include an intuitive tab structure that filters information relevant to customer needs such as air, water, soil and hazardous waste, industrial hygiene or biomonitoring testing and analysis.

Upon reaching the site's home page, users will find information sorted by method, technology or environmental application. The added search functionality will guide visitors to useful information in less time. The site also features a streamlined left-side navigation and enables straightforward transitions from one portion of the site to another, along with direct links to compliance information on regulatory bodies' Web sites.

For more information, visit: **www.perkinelmer.com/environmental**.

From Spectro - ARCOS Generator Is Unflappable

A picture says more than a thousand words and a three minute film maybe more than a photo album. That's why we decided to try an experiment: Instead of describing how robust the SPECTRO ARCOS generator is in piles of white papers and articles, we shot a short film for you. In the film, our Product Expert Dr. Dirk Ardelt, proves, using a pretty drastic example, that the generator in our flagship SEPCTRO ARCOS can't be shaken even under extreme experimental conditions. Do you have three minutes? Then come visit us at:

www.spectro.com/arcos/generator-video.

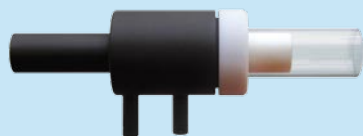
If, after watching the film, you want to know more about the SPECTRO ARCOS and its generator, our technical experts are available to answer your questions at: **spectro.info@ametec.com**. ►

NEW PRODUCTS

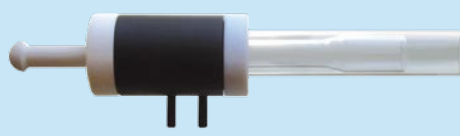
D-Torch for PerkinElmer Elan and Spectro SOP

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 Elan, and the Spectro SOP (Radial) models, as well as the PerkinElmer Optima 4/5/7300V and Thermo iCAP 6000 announced previously. 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.



Elan D-Torch



Spectro D-Torch

From Teledyne Leeman Labs - New Application Note on the Analysis of Lead-Based Paint

◀ Teledyne Leeman Labs, a leading manufacturer of analytical instrumentation for elemental analysis, announces the publication of a new application note on the analysis of lead-based paint using the Prodigy High Dispersion ICP. In 1978, the U.S. Consumer Product Safety Commission (CPSC) set the maximum allowable lead content in paint for residential use to 0.06% (600 ppm) as outlined in 16 C.F.R. §1303.1. As of August 14, 2009, the maximum allowable lead content will drop to 0.009% (90 ppm). While lead has been banned from all paint used for domestic purposes, the use of lead in plastics is still permitted.

This application note demonstrates the ability of the Teledyne Leeman Labs Prodigy High Dispersion ICP to analyze lead-based paints. The sensitivity and large linear dynamic range of the instrument in the axial view mode enable it to determine a wide range of lead concentrations in several different paint samples.

To receive a copy of technical note 1056, "The Analysis of Lead-Based Paint using the Prodigy High Dispersion ICP", contact Teledyne Leeman Labs, 6 Wentworth Drive, Hudson, NH 03051. Telephone (603) 886-8400, Fax: (603) 886-9141 or email: **LeemanLabsinfo@Teledyne.com** or visit our website at: **www.LeemanLabs.com**.

From Thermo Fisher Scientific - An ICP Solution to Facilitate Rapid Analysis of Impurities in Solid Metallic Samples

Thermo Fisher Scientific Inc., the world leader in serving science, has developed a unique solution to provide fast and efficient impurity analysis of solid metals, including steels, platinum group metals, gold and other conductive materials. Combining the Thermo Scientific iCAP 6500 Duo ICP emission spectrometer with a Separate Sampling and Excitation Accessory (SSEA), the solution enables close monitoring of all stages of the silver refinery process. The iCAP 6000 Series enables the sensitive, multi-element determination of impurities at ppb and sub-ppb levels in liquid phase matrices and by adding the SSEA accessory, solid phase metallic matrices can also be analyzed with only minimal sample preparation.

The analysis of silver for purity and characterization of impurities is an important example of the application of the combined power offered by the iCAP 6000 Series and SSEA accessory. The capabilities of this unique system are detailed in an application note entitled "Rapid Analysis of Impurities in Silver using a Separate Sampling and Excitation Accessory (SSEA) and Thermo Scientific iCAP 6500 Duo" which is available to download free-of-charge via: **www.thermo.com/icap**

For more information on the Thermo Scientific iCAP 6500 Duo ICP emission spectrometer, please email: **analyze@thermofisher.com** or visit **www.thermo.com/trace**

From Varian - The Varian Experience

Varian, Inc. is hosting an engaging and informative online experience covering the full range of customers and applications for which Varian offers a solution. ICP users can participate in a few ways. In addition to Blogs where you can ask questions, you can also contribute a photograph, video or written description of your work. We are looking for people who want to talk about the outcome of their work and what it means to them. Are you developing new solar cells or biofuels, solving crimes, contributing to food safety, etc.?

We are proud of the fact that ICP users have a positive impact on the lives of people around the world every day. In appreciation, we want to offer you the chance to tell the world what it is that you do and why it is so important to you. We are not asking for an endorsement or a testimonial. Just tell us your story. You are welcome to talk about an application or a challenging problem, but we are equally, if not more interested in hearing about the bigger picture – the outcome of the work that you do. You can join us by visiting: **www.thevarianexperience.com**. ■

NEW PRODUCTS

Niagara II Rapid Rinse Accessory

The second generation Niagara II Rapid Rinse Accessory reduces the time for a typical analysis by approximately 30%. It provides all of the benefits of the original Niagara in a more compact easier-to-use design. It 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 time that is usually wasted waiting for the sample solution to flow from the autosampler to the nebulizer. This significantly reduces the cost per sample for busy laboratories such as those analyzing environmental, geochemical and oil samples.

The time that is saved is around 30% for a typical analysis. This will vary depending upon the specific conditions under which each instrument is operated. To estimate the Niagara Benefit for your laboratory, check the Niagara II Time Saving Calculator on our website at:

www.geicp.com/cgi-bin/site/wrapper.pl?c1=Updates_newproducts_niagara.



The Niagara II is compatible with most ICP-OES and ICP-MS spectrometers and autosamplers. Please contact **enquiries@geicp.com** to check the compatibility with your equipment.

Niagara II Plus

The performance of your ICP can be further enhanced by the new Niagara II Plus system. The Niagara II Plus utilises flow injection technology to further reduce the analysis time. It uses a positive displacement pump to rapidly fill a sample loop and it incorporates a proprietary valve with extra low sweep volume to provide the ultimate productivity.

The benefits of the Niagara II Plus are:

- Analysis time typically reduced by 50%
- Internal standard added automatically
- Very low carryover - the sample does not contact any peristaltic pump tubing

Please contact **enquiries@geicp.com** for full details.



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