

Should You Cool (or Heat) Your Spray Chamber?

There is a large body of research which shows that a modern argon plasma can handle a mass load of about 40 - 60 mg/min. If you do the math, and assume the nebulizer-spray chamber is 5% efficient, at 1 mL/min you are putting about 50 mg/min into the plasma. Solvent loading is the term generally used to describe changes in the plasma when too much solvent reaches it. Essentially, more plasma energy must be expended for vaporization of the solvent, leaving less energy for decomposition, excitation, and ionization of the analyte. In ICP-AES sensitivity and detection limits will be degraded; additionally, in ICP-MS oxide levels will increase if the solvent is water. This solvent loading is a major reason why "more efficient" nebulizers really don't deliver better instrument detection limits. Short answer: cooling is a good idea, heating isn't, and here is why.

The spray chamber has a number of roles. The foremost role is to reduce the aerosol size to something less than 10 um on average (<5 um is most desirable). It achieves this through gravitational settling and through impaction on surfaces. In a cyclonic spray chamber, inertial impaction occurs as the aerosol undergoes a cyclonic swirl before it leaves the spray chamber. Larger droplets hit the walls and, for the most part, collect and travel to the drain. This ordinarily accounts for about 95% of the sample that goes into the nebulizer. But some droplets will impact the walls and splatter, putting material that was on the walls back into the aerosol stream. In a cyclonic spray chamber, or a conical with impact bead, this is a cause of memory or carryover which is manifested as a longer washout time.



PC3 - a Peltier Cooled Cyclonic
Spray Chamber from Elemental Scientific

The second role is to very rapidly and smoothly remove the collected liquid from the walls to minimize this effect. The current cyclonic and conical spray chamber designs utilize improved geometry and aerodynamics as a result of research. But if the spray chamber is "dirty" and does not drain well, you can expect an increase in washout time.

Third, it should be understood that desolvation of the aerosol begins in the spray chamber. If a droplet moves through an atmosphere of dry argon, it will rapidly lose solvent as vapor to the argon. (Continued, Page 2)

Conference Notes

FACSS 2007, Memphis, October 14 – 18, Booth 67

Once again, Meinhard will provide **two student poster awards** at FACSS – the premier international conference on analytical chemistry and spectroscopy. The outstanding student posters will be selected by the FACSS Program Committee. Each winner will receive a membership in the Society for Applied Spectroscopy and an honorarium.

Be sure to stop by Booth 67 to talk about advances in sample introduction.

Winter Conference on Plasma Spectrochemistry, January 7 – 12, 2008, Booth 18

Temecula, California is host to the 2008 conference which focuses on advances in ICP, ICPMS, and related plasma techniques for elemental analysis. Symposia range from fundamental studies to laser ablation and chemical speciation.

Look for Meinhard in Booth 18 to see and discuss our sample introduction components as well as Spetec peristaltic pumps and laminar flow boxes.

Pittcon 2008, March 2 – 7, New Orleans

We will be there and hope you stop to see us as Pittcon returns to New Orleans.

Should You Cool (or Heat) Your Spray Chamber? *(Continued from Page 1)*

How rapidly it desolvates depends on the nature of the solvent, solvent-solute interactions, the size (surface area) of the droplet, the surface area of the spray chamber, and the temperature of the spray chamber (among other things). Cooling the spray chamber will reduce the solvent load by condensing solvent vapor on the walls, and by removing the condensed liquid along with the "excess" aerosol. Clearly, heating the spray chamber will enhance desolvation and will put more vapor into the plasma unless there is a downstream mechanism for solvent removal such as a cold condenser or membrane desolvator as, for example, in the Apex from Elemental Scientific.

This spray chamber desolvation process is the source of a couple of interferences that lead to bias in the analytical result. In an elegant set of experiments, Stewart and Olesik (*J. Anal. At. Spectrom.* 1998, 13, 843) showed that the difference in analytical response between a 2% HNO₃ solution and a 10% HNO₃ solution of Mn results from the time it takes the newly-formed aerosol to achieve vapor-phase equilibrium with the liquid that remains on the walls. If a spray chamber is washed with 10% HNO₃ then the sample is introduced in 2% HNO₃, there will be an initial surge in signal of perhaps 15%, which might take several minutes to stabilize at some level that is lower than the peak, while HNO₃ on the walls and the vapor in the spray chamber slowly decreases from 10% to 2%.

Practically, what this means is that the rinse matrix as well as the calibration standard matrix should match the sample matrix as closely as possible.

Desolvation and equilibration in the spray chamber depend on temperature. If the temperature changes, signal will drift. In fact changes in spray chamber temperature account for a large fraction of the long-term drift in modern ICP spectrometers because the amount of analyte and the amount of solvent change with time. Some instruments have been designed with a heater in the sample intro area in order to stabilize the spray chamber temperature and improve long-term stability. Generally, this will only be a few degrees above the normal high ambient temperature that the instrument is expected to encounter. This approach is less desirable in ICP-MS since, for aqueous solutions, the oxide level will increase. But in ICP-AES, the improved stability comes at a very small price.

For aqueous solutions, the ideal temperature is just above 0 C to remove the most solvent vapor. Usually chillers are set to +2 C; you do not want solvent to freeze in the spray chamber; you do not want to induce crystallization or precipitation so that solids collect and block the drain.

Organic solvents are generally far more volatile than water, and lead to a more significant solvent loading problem. Moreover, in ICP-AES there is hydrocarbon band structure

below 300 nm that can overwhelm analyte emission, again because so much solvent is reaching the plasma relative to analyte. Band structure that is not stable is highly problematic. Toluene is more of a problem than kerosene, and gasoline is much more of a problem than toluene. Cooling the spray chamber to less than 0 C dramatically reduces vapor transport and plasma loading. If there is no water present (kerosene can carry several percent water), one might surmise – the colder the better. And one might be wrong.

There are practical limits to how well the spray chamber can be protected. If condensation will form on the outside and freezes between the spray chamber and its mount, it could crack a glass spray chamber or move it enough to diminish the integrity of the spray chamber – injector connection. With organic solvents, viscosity can be nearly as sensitive to temperature as is vapor pressure. For the analysis of lubricating oils, even at 1:10 dilution, a very cold spray chamber will not drain very well, and will leave a lot of material on the walls of the spray chamber. Usually, very little is gained by cooling to less than -5 C unless the solvents are highly volatile, like methanol, acetone, or methylene chloride.

Finally, in the analysis of organic solvent solutions, it is imperative to let nebulization control the rate of analyte transport to the plasma. At room temperature, slightly volatile organometallic compounds will be transported both as vapor and as aerosol. If the material used to calibrate is not chemically identical to the analyte in the sample, there can be significant bias in the analytical data. Among the more obvious examples would be the determination of sulfur – anyone who has ever handled an organo-sulfur compound has smelled it. That vapor would be sample transported to the plasma with essentially no control, and no hope of analytically useful data.

Jacketed spray chambers are available from Meinhard in several common varieties – double pass, cyclonic, conical, etc., for many instruments, as a direct replacement. Simply attach a recirculating chiller with a suitable coolant, set the temperature, let it stabilize (with the plasma on), and off you go. However, the PC3 from Elemental Scientific has a Peltier-cooled cyclonic spray chamber (with or without baffle, in quartz, Teflon PFA, or polypropylene) which, starting at about \$2200, offers tremendous value – no external chiller required.

The PC3 has two "on" settings, +2 C and -5 C, to control the spray chamber temperature. (A new low temperature version has a -20 °C setpoint, as well). It operates at 110 – 240 VAC and requires less than 1.9 A. It will improve sample introduction system stability, reduce solvent loading, and improve washout for all ICP instruments. It will reduce polyatomic interferences and the frequency of cone cleaning in ICP-MS. Contact Meinhard for more information.

New Products

New Fit Kit 3T Goes Both Ways



Developed primarily for liquid input to our nebulizers, the Fit Kit 3T can easily be used as a drain connection on spray chambers having a 4 mm o.d. drain fitting.



Today, very few spray chambers have something other than a 4 mm drain. While the Fit Kit 3T is included with every standard Meinhard nebulizer, additional pieces are available at \$17 each.

Thermo iCAP 6000 Sample Introduction New from Meinhard

Meinhard has high quality nebulizers, torches, injectors, spray chambers, and adapters for the new ICP spectrometer from Thermo. We can also supply superior HF-resistant components in Teflon PFA – at fair prices.

New Flared-end Peripump Tubing

Tired of burning your fingers while trying to assemble internal standard mixing kits? Frustrated with fitting nebulizer tubing to peripump tubing? Flared-end peripump tubing really simplifies the assembly process, saving you time and fingers. Meinhard has it in common sizes from 0.010" (orange/blue) to 0.045" (red/red) at an introductory price of \$35/pack of 12 pieces.



Solid Sample Analysis by DC Arc and ElectroThermal Vaporization (ETV)

In about six months, a new spectrometer will be available that will incorporate ICP, ETV and DC Arc sample excitation or any combination of these excitation sources, including all three.

The traditional liquid analysis ICP applications plus the micro-sampling solid (and liquid) sample capability of ETV, and DC Arc will serve a very wide field of applications. Direct analysis of solids has some distinct advantages: no costly, time-consuming digestion and very low detection limits.

This may be just the right choice for your laboratory. Please contact us for further information about this remarkable instrument system.

Sale – Spectro Components - until December 31, 2007

Spectro Part No.	Meinhard Part No.	Description	Regular \$US	Sale \$US
48105001	ML175026	Cyclonic Spray Chamber, Top Socket, Pump Drain	180	153
48105003	ML175012	Spray Chamber, Upward Conical Open	98	83
48105008	ML175021	Quartz Injector, 1.5 mm	97	82
48105009	ML175023	Quartz Injector, 2.5 mm	97	82
48105010	ML175022	Quartz Injector, 1.8 mm	97	82
48105011	ML175031	Adapter, 12/5 socket, 45 mm for ICP	54	46
48105051	ML175019	Spray Chamber, EOP, Large Double Pass	178	151
48105052	ML175040	Quartz Torch, EOP, Fixed Flared End, 2.5 mm Injector (75160526)	300	255
48105052A	ML175042	Quartz Torch, EOP, Fixed Flared End, 1.8 mm Injector (75160592)	300	255
48105061	ML175026EOP	Cyclonic Spray Chamber, EOP, Pump Dr	180	153
48205001	ML175007	Quartz Torch, Spectroflame, Fixed, 1.0 mm Injector, Oils	282	240
48206002	ML175043	Quartz Torch, EOP, Demountable, No Flare, 100 mm Length	214	182
48206007	ML175044	Quartz Torch, EOP, Demountable, No Flare, 80 mm Length	172	146
75060545	ML175006	Quartz Torch, Radial, Demountable, 80 mm Length	168	143
75060596	ML175005	Quartz Torch, Spectroflame, Fixed, 1.8 mm Injector	277	235
75160537	ML175048	Quartz Torch, EOP, w/Sheath Gas, 2.5 mm Injector	427	363
75160549	ML175049	Quartz Torch, EOP, w/Sheath Gas, 1.8 mm Injector	427	363
75260533	ML175047	Quartz Torch, EOP, w/Sheath Gas, 3.2 mm Injector	427	363
76060510	TR-30-C1	Type C nebulizer, glass, 1 mL/min and 1 L/min @ 30 psi	425	361
	MM175055	Tubing and gas connectors 44302072, 44302077	25	21
	MM175100	Liquid inlet fitting and argon tubing with quick disconnect for TR-30-C1	45	38

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Please indicate your needs. When finished please FAX this form back to us at (303) 216-2649

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