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ADVANCES IN ICP SPRAY CHAMBERS

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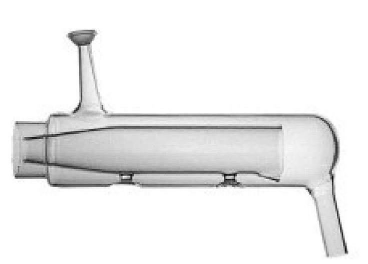
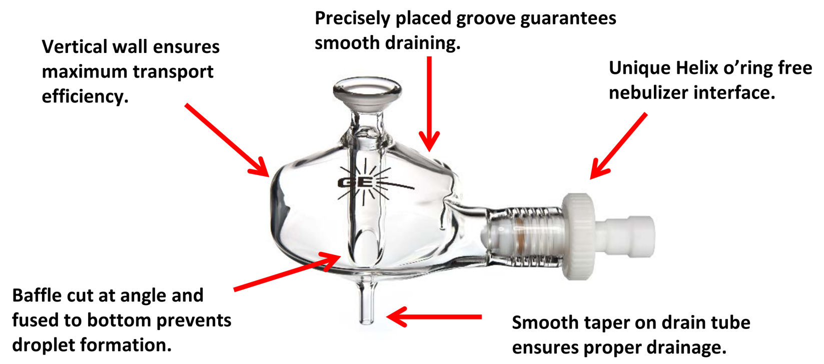
Introduction

The purpose of the spray chamber in ICP optical and mass spectrometry is to filter the aerosol generated by the nebulizer so that only small droplets capable of completing the atomization process enter the plasma. This is generally considered to be droplets with diameters less than about 8 microns. The most commonly used types of spray chambers are Scott and cyclonic (shown below).

Scott Design



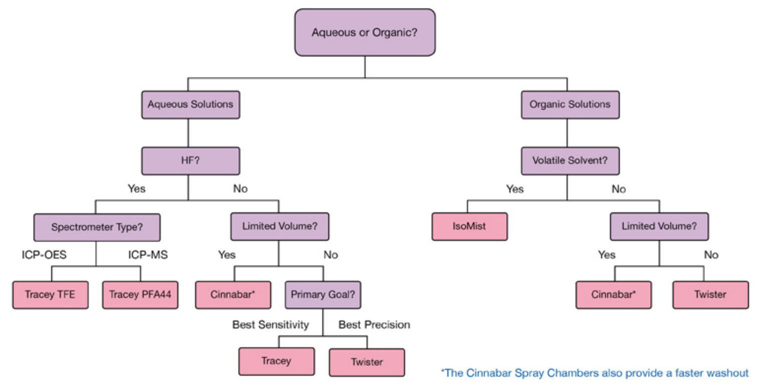
GE Cyclonic (Twister™)



The advantages of the cyclonic spray chamber design are as follows:

- Faster washout. The cyclonic design has dramatically lower surface area and volume from which to remove the previous sample (less carryover).
- More efficient removal of large droplets. Unlike the Scott chamber, the cyclonic chamber uses centrifugal force to impact the larger droplets on the chamber wall.

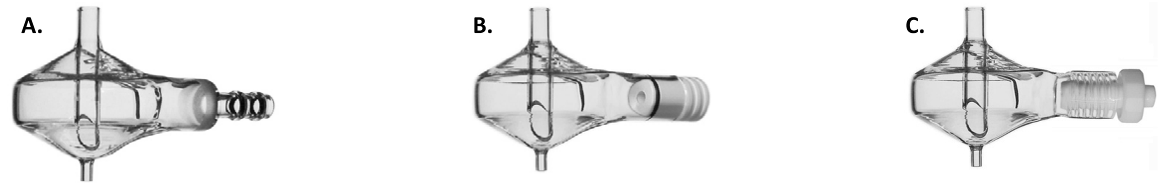
GE Cyclonic Spray Chamber Designs and Selection Guide



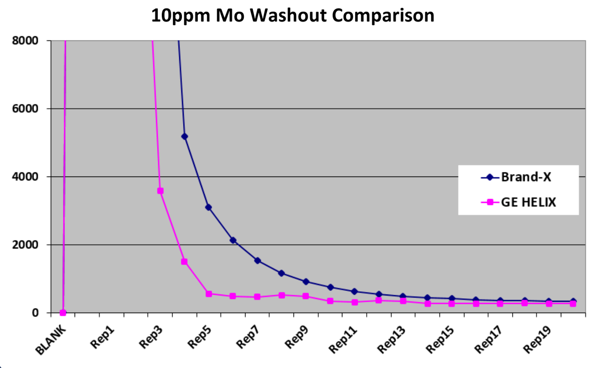
- Glass Tracey – non-baffled cyclonic, 50 ml internal volume
- Glass Twister – baffled cyclonic, 50ml volume
- TFE Tracey – inert 50ml volume with treated internal surface
- Glass Cinnabar – non-baffled glass mini-cyclonic, 20ml volume
- Glass Twinnabar – baffled mini-cyclonic, 20ml volume
- PFA Tracey – inert 40ml volume with treated internal surface

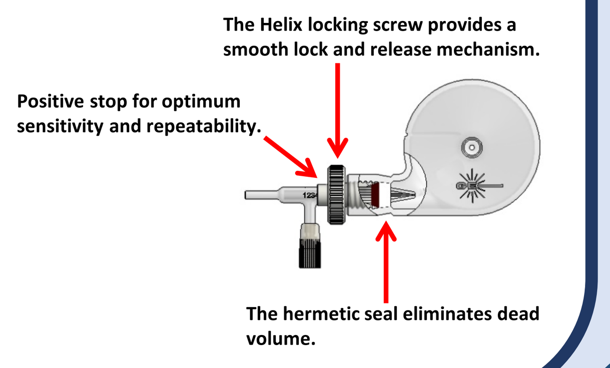
However, not all cyclonic spray chambers are the same. Glass Expansion pays careful attention to all details of the spray chamber design. An example is the evolution of the GE nebulizer interface (below). The original nebulizer interface incorporated dual o'rings to seal the chamber and hold the nebulizer straight (A). Glass Expansion developed the inert nebulizer plug to prevent solution from pooling in the nebulizer port although this version still used o'rings to hold the nebulizer (B). Finally, the Helix seal was developed (C). The Helix eliminated o'rings so that even strong solvents could be used without degradation and contamination from the o'ring material. It also had the least dead volume and hence lowest carryover (see graph comparing a Helix spray chamber with an o'ring-based brand X).

Evolution of the Nebulizer Interface



Advantages of the Helix Design





PCC Kit for Agilent ICP-MS

Agilent Chilled Scott Style Spray Chamber — Glass Expansion PCC Kit

The Glass Expansion PCC Kit is a Peltier cooled cyclonic spray chamber that replaces the Scott Double Pass spray chamber of the Agilent ICP-MS. The PCC Kit uses the same electronics, software, and cooling lines used with the standard Agilent spray chamber for easy installation and operation.

Additional Key Features:

- Interchangeable glass, quartz and PFA cyclonic spray chambers
- Low volume Twinnabar™ option available
- Faster washout and sample throughput than standard Scott style spray chamber
- Peltier based on GE IsoMist design
- Temperature controlled directly from Agilent software (2°C or 5°C)
- Supplied with convenient mounting bracket

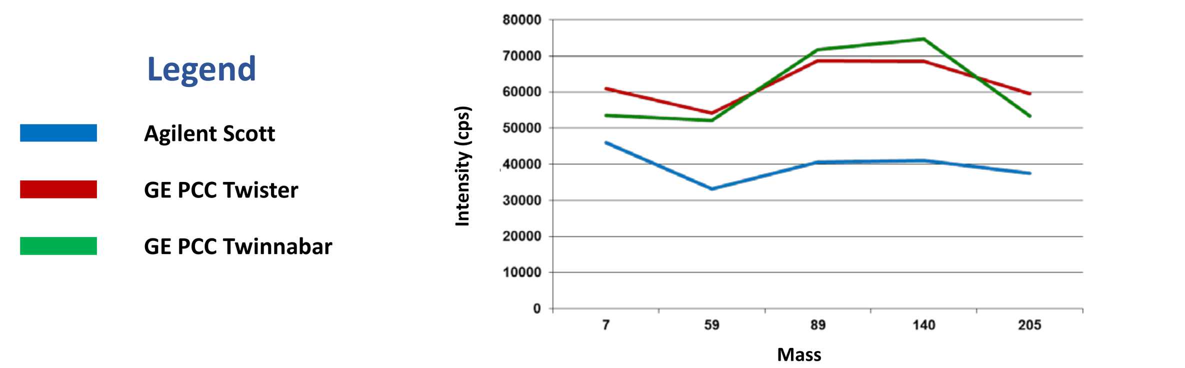
Configuration	Spray Chamber	Temp (C)	Nebulizer Uptake (mL/min)	Injector Bore (mm)
A	Agilent Scott	2	0.4	2.0
B	PCC Twister	2	0.4	2.0
C	PCC Twinnabar	2	0.2	2.0

The performance of the Agilent Scott spray chamber was compared with two different types of cyclonic spray chambers available in the PCC configuration (Table above). The graph below (top right) compares sensitivity using the 3 different designs. The two GE cyclonic designs were roughly equivalent and both gave about 50% higher intensities compared to the standard Agilent setup.

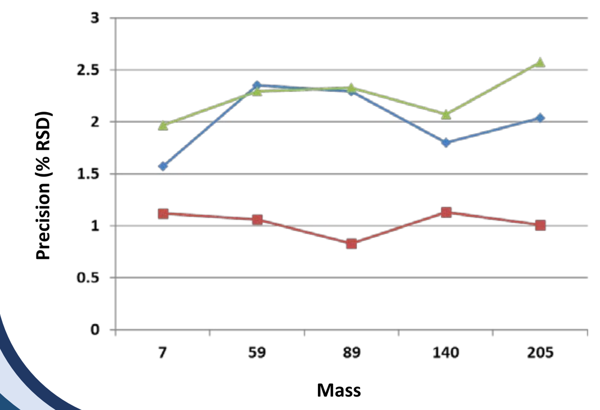
The graph bottom left compares the precision using the 3 different spray chambers. In this case the PCC with the Twister spray chamber clearly gave the best precision while the small volume (20mL) Twinnabar and the Agilent Scott spray chamber produced RSD's about twice as high. The graph on the bottom right compares single and double oxide ratios. The low volume PCC Twinnabar gave the best single oxide ratio while the PCC Twister gave the best double oxide ratio.

The PCC brings the cyclonic design and its advantages to the Agilent controlled chilled spray chamber system.

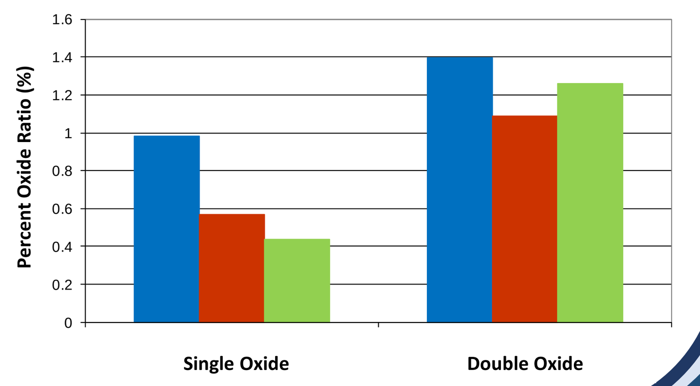
Sensitivity Comparison



Precision Comparison



Oxide Ratio Comparison

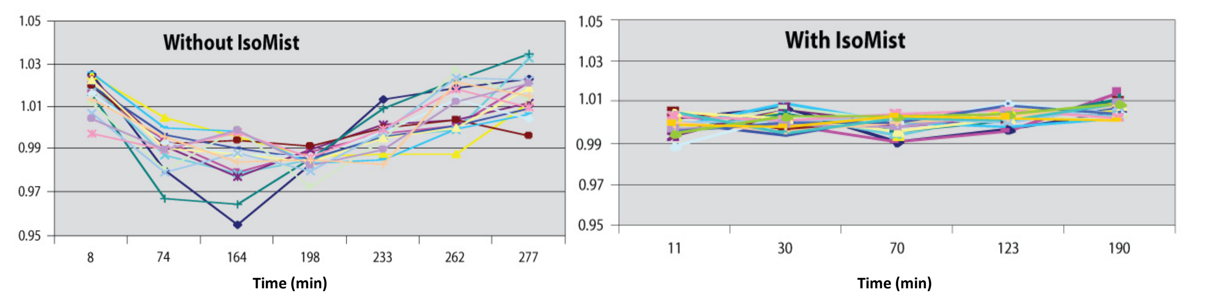


IsoMist Spray Chamber

The IsoMist is a stand alone Peltier based programmable temperature spray chamber with the characteristics listed below. As shown above, the IsoMist can be configured with a glass, quartz, or PFA spray chamber (all of which are interchangeable).

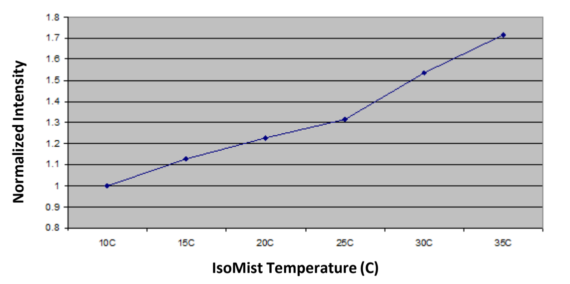
- Range of -10 to +60C in 1 degree increments
- Stable to 0.1 degree C
- 100% self contained
- Bluetooth and USB compatible
- Graphic display of Temp vs. Time
- Customized for each ICP or ICP-MS model

Constant Temperature Benefits



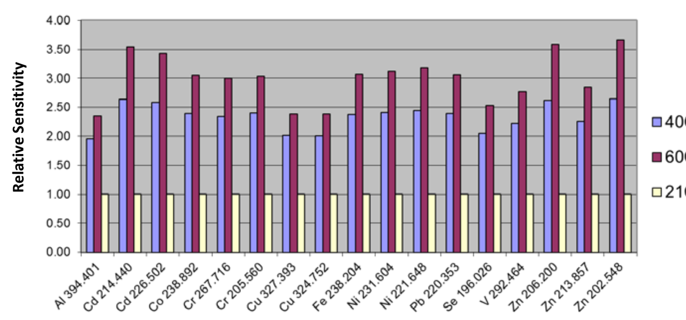
Benefits of Elevated Temperature

Average Intensity vs. Temperature (1mL/min)

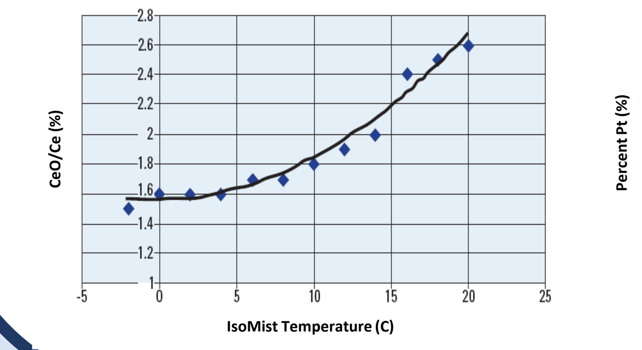


- 31% increase from 10 to 25C or 2% per degree C
- 31% increase from 25 to 35C or 3% per degree C

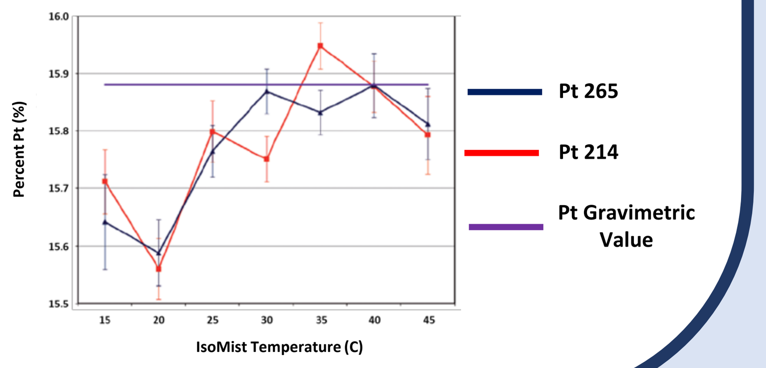
Average Intensity vs. Temperature (20uL/min)



Reduced Oxides for ICP-MS



Improved Accuracy for Precious Metals



The data above summarizes the advantages of controlling the spray chamber temperature. The top graphs display the advantage of maintaining a constant temperature. Without temperature control, the ICP signal varies 4 to 5%, however, with the IsoMist maintaining a temperature of 24C, the signal varies no more than 1%.

The middle graphs describe the effect of spray chamber temperature on intensity for a normal (1ml/min) uptake rate and for a very low (20ul/min) uptake rate. At 1ml/min, 35C is the highest temperature that does not overload the plasma. From 10 to 25C a 2% signal increase is observed per degree, and from 25 to 35 C a 3% increase per degree. Overall a 31 % signal increase in intensity is observed over 25C. At 20ul/min, a temperature of 60C is tolerated and results in 200% intensity increase over 25C.

The bottom graphs show the effect of temperature on oxides for ICP-MS and accuracy for precious metal assays.

IsoMist XR Spray Chamber

For very volatile solvents we recommend the new IsoMist XR with the ability to reach -25C. The IsoMist XR is based on the standard IsoMist design with the same features, but packaged with a more powerful Peltier. The new Peltier provides an unmatched temperature range of -25C to +80C, in increments of 1C and accuracy of +/- 0.1C.

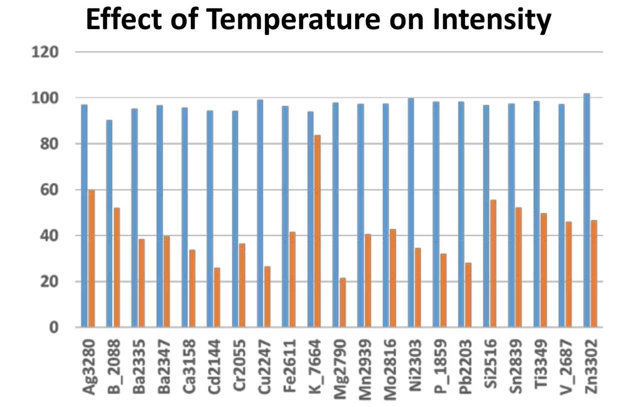
iCAP 6500 Duo Conditions	
Plasma gas flow rate (L/min)	16
Auxiliary gas flow rate (L/min)	2
Forward power (watts)	1350
Nebulizer gas flow rate (L/min)	0.28
Sample uptake rate (mL/min)	0.5
Spray chamber temperature (C)	-10 & -25

The analysis of naphtha by ICP-OES is complicated by the high volatility of the sample, which can overload the plasma. The ability of the IsoMist XR to achieve -25C was evaluated as a means of improving the analysis of naphtha over previous attempts at -10C. The data below was achieved using a Thermo iCAP 6500 Duo ICP-OES, operating parameters listed in the table above.

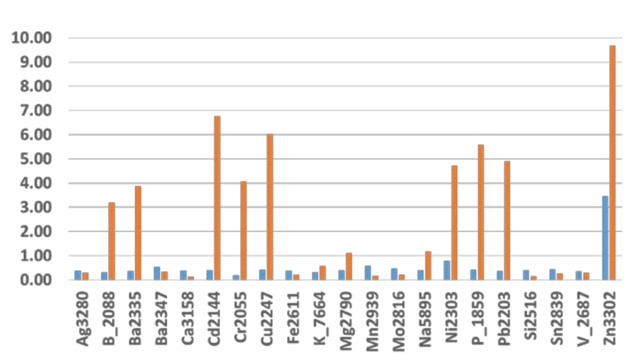
Analysis of Naphtha for Trace Metals

IsoMist XR at -25C — IsoMist XR at -10C

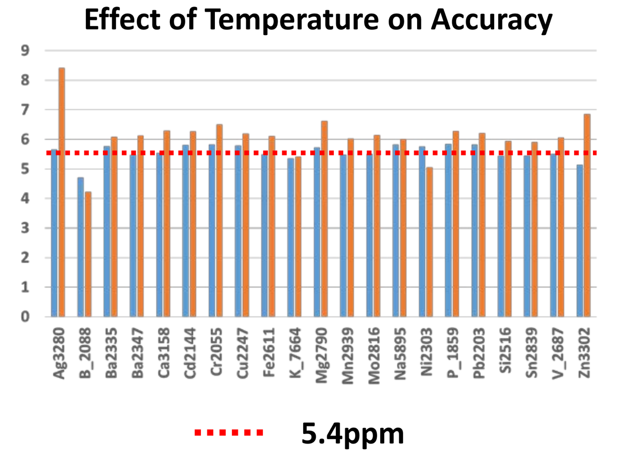
Effect of Temperature on Intensity



Effect of Temperature on Precision



Effect of Temperature on Accuracy



Detection Limits at -25C

Line	Predicted MDL (ppm)	Predicted MQL (ppm)
Ag328.0	0.0013	0.0045
Ba233.5	0.0004	0.0014
Ca315.8	0.0034	0.0114
Cu224.7	0.0031	0.0102
Fe261.1	0.0021	0.0072
Mg279.0	0.0147	0.0489
Mn293.9	0.0017	0.0055
Pb220.3	0.0045	0.0149
Ni230.0	0.0075	0.0249
Si251.6	0.00194	0.0064
V_268.7	0.0022	0.0073
Zn330.2	0.151	0.5035

For the intensity graph, it is clear that a factor of over two is achieved at -25C on average. With respect to precision, a number of lines give rather poor precision at -10C, including Ba, Cd, Cu, and Pb, while all lines except Zn are below 1% RSD at -25C. With the improved precision, the results are also more accurate at -25C. The table provides the minimum detectable level (MDL) and the minimum quantifiable level (MQL) for select lines at -25C. Using the IsoMist XR at -25C allows for direct analysis of naphtha without dilution while providing accurate, precise, and reproducible results not possible at -10C.

The IsoMist and IsoMist XR have been shown to provide important benefits at both elevated and very low ends of the temperature range.