Effect of Chilled Spray Chamber Design on ICP-MS Performance

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Glass Expansion

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This work focuses on the importance of the spray chamber in determining the quality of results in ICP-MS. Many ICP-MS manufacturers supply chilled spray chambers as standard equipment. The reasoning for this is two-fold. First, stabilizing the spray chamber at any temperature reduces temperature related signal drift. Secondly, many analysts have found that spray chamber temperature along with other parameters, particularly nebulizer gas flow, can be optimized to minimize oxide formation in the plasma. In general, oxides (as measured by the Ce+ to Ce ratio) are recommended to be kept below 2%.

Oxide Reduction in ICP-MS

Isobaric oxide interferences

Element/Isotope	Interference
^{56}Fe	$^{40}Ar^{16}O^{+}$
$^{51}V^{+}$	$^{35}Cl^{16}O^{+}$
$^{44}Ca^{+}$	$^{14}N^{14}N^{16}O^{+}$
$^{48}Ti^{+}$	$^{32}S^{16}O^{+}$
$^{52}Cr^{+}$	$^{34}S^{18}O^{+}$
$^{64}Zn^{+}$	$^{32}S^{16}O^{16}O^{+}$
$^{64}Zn^{+}$	$^{48}Ca^{16}O^{+}$

Robert Thomas, **Practical Guide to ICP-MS**, Marcel Dekker, NY, 2004.

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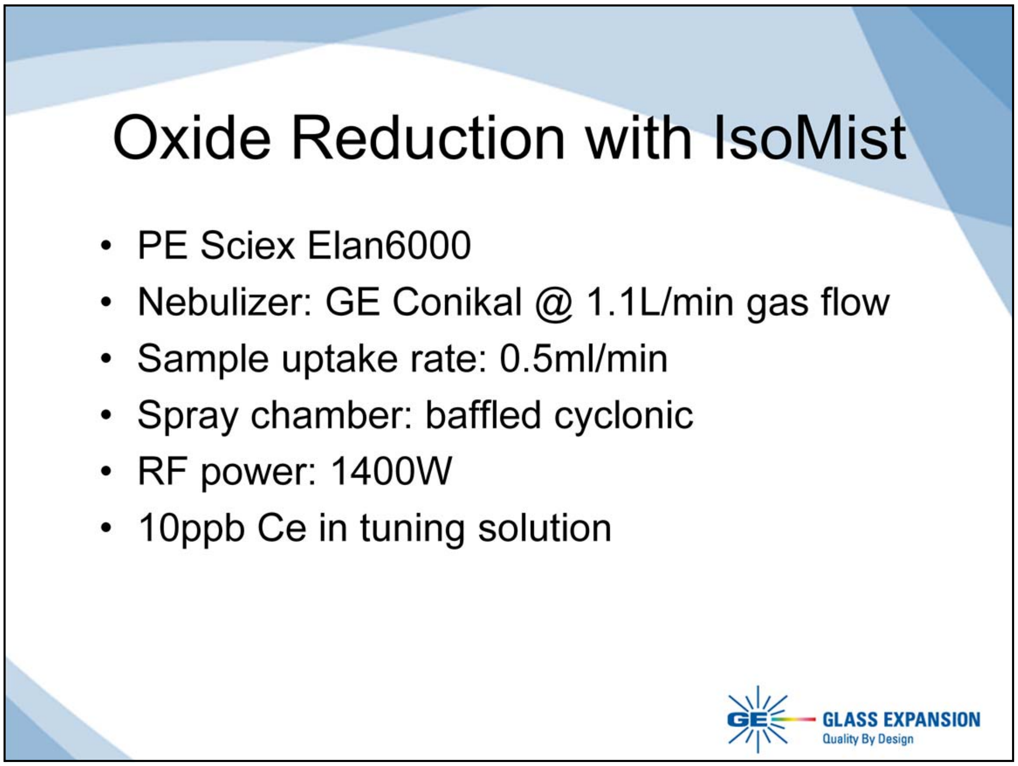
This data taken from Rob Thomas' book lists a number of oxides and the masses on which they interfere isobarically.



Using an Elan 6000 ICP-MS from PerkinElmer, David Jones from ALS Chemex in Brisbane, Australia, found that a nebulizer gas flow of 1.1L/min minimized oxide formation when the spray chamber was held at the appropriate temperature.

An IsoMist Programmable Temperature Spray Chamber (Glass Expansion) was used to vary the spray chamber temperature in the desired range. Shown above, the Peltier heat transfer component is housed to the right behind the company logo, while the nebulizer and spray chamber are to the left and a pumped drain line is configured below.

Features of IsoMist

- Powerful Peltier-effect chiller reaches -5C in 15 minutes
- Programmable from -10 to 60C in 1 degree increments
- Maintains temperature to within 0.1 degree
- Compact design (7.5x4x4 inches)
- 100% self-contained (no external lines)
- Incorporates Bluetooth® technology for clean wireless control (USB available)

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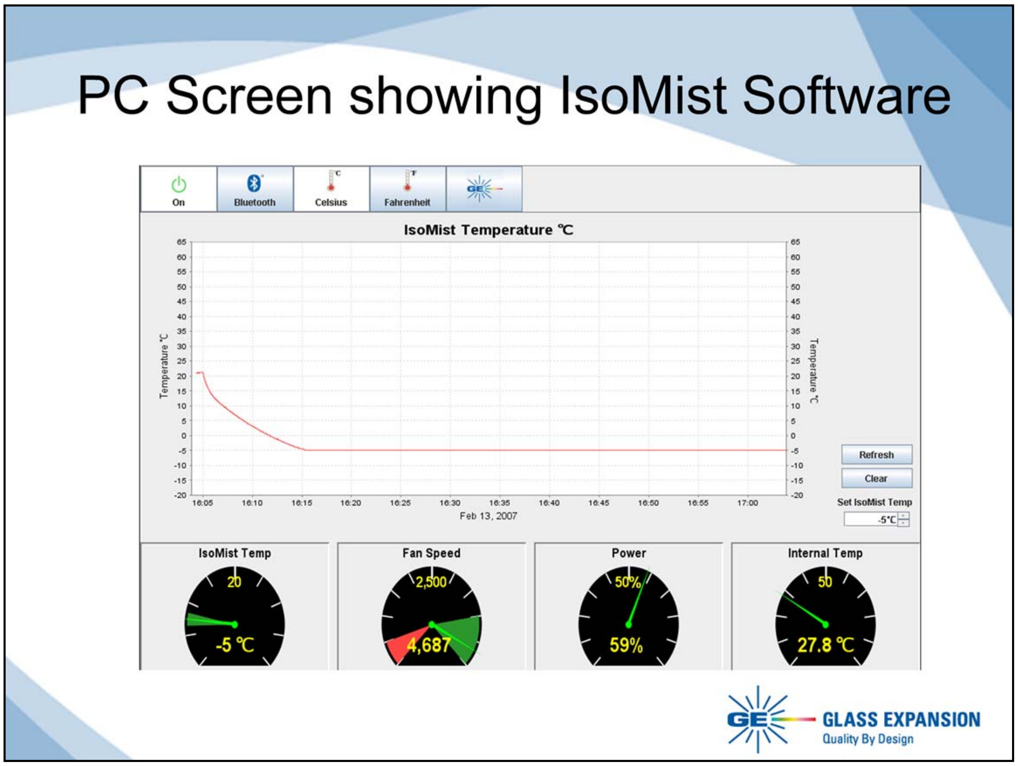
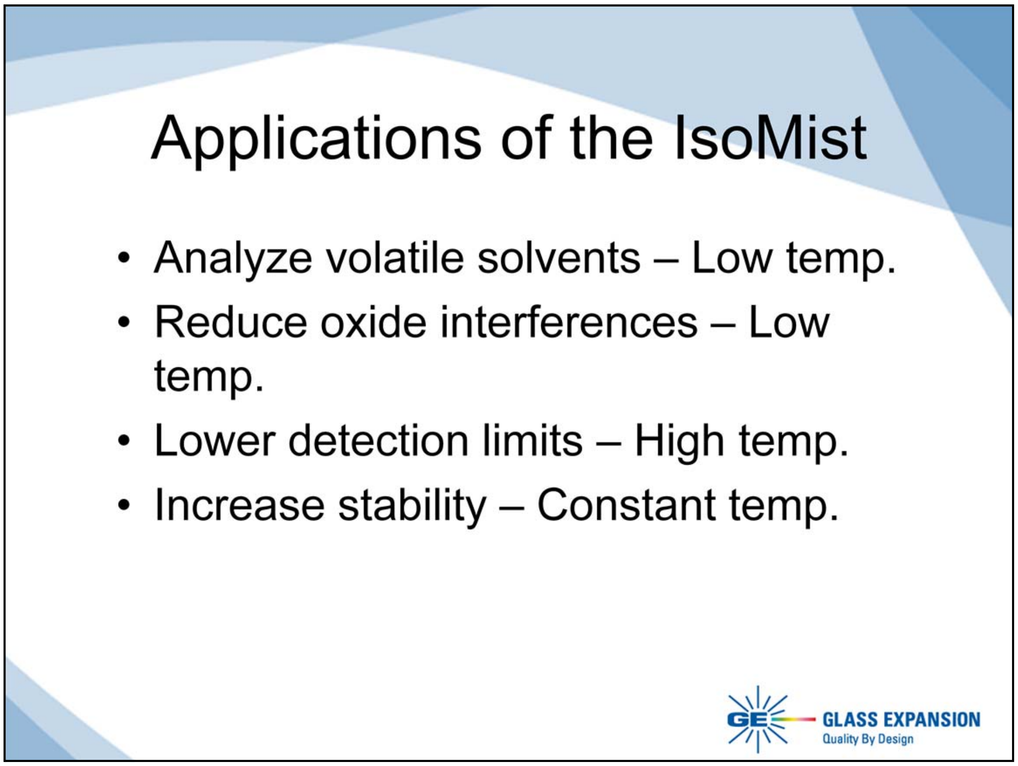
The features of the IsoMist are shown here. It should also be mentioned that the IsoMist “remembers” the last temperature at which it was set. Therefore, if you are using the same temperature daily, there is no need to connect it to a PC once it has been set.



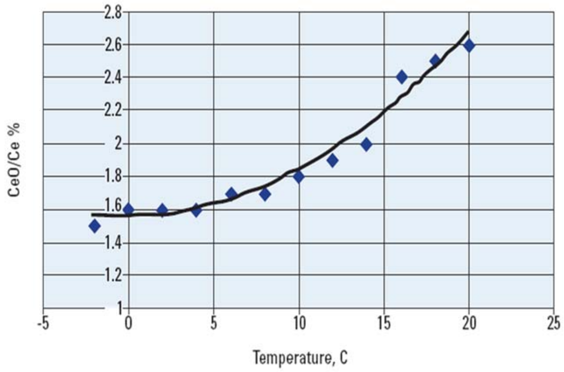
This is the one page proprietary application that controls the IsoMist. The temperature vs. time plots can be saved over the long term for regulatory compliance.



For volatile organic solvents, a low temperature in the region of -5 to -10 C is typically used to reduce plasma loading (especially for ICP-OES), while somewhat higher temperatures (above zero) are used to reduce oxides for ICP-MS. Elevated temperatures are useful in situations where sample volume is severely limited and the analyst is forced to employ micro nebulizers. The elevated temperature increases sample transport thus recovering much of the intensities forfeited by using very low sample uptake rates. For both OES and MS, increased temperature stability reduces signal drift thereby improves accuracy.

Reducing Oxides for ICP-MS

Effect of IsoMist Temperature



Data courtesy of David Jones, ALS Chemex, Brisbane, Australia

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It is generally regarded that an oxide ratio of less than 2% is desirable to provide a more robust system. Even instruments which use collision/reaction cells to reduce oxide and other molecular isobaric interferences benefit from a reduced oxide ratio. In this experiment, it was found that using an IsoMist temperature of 2ºC was optimum.

IsoMist Customization

Models

- PE ICP & ICP-MS
- Varian (Agilent) ICP
- Agilent ICP-MS
- ThermoFisher ICP & ICP-MS
- Horiba J-Y ICP
- Teledyne Leeman ICP
- GBC ICP
- Spectro ICP
- Bruker ICP-MS

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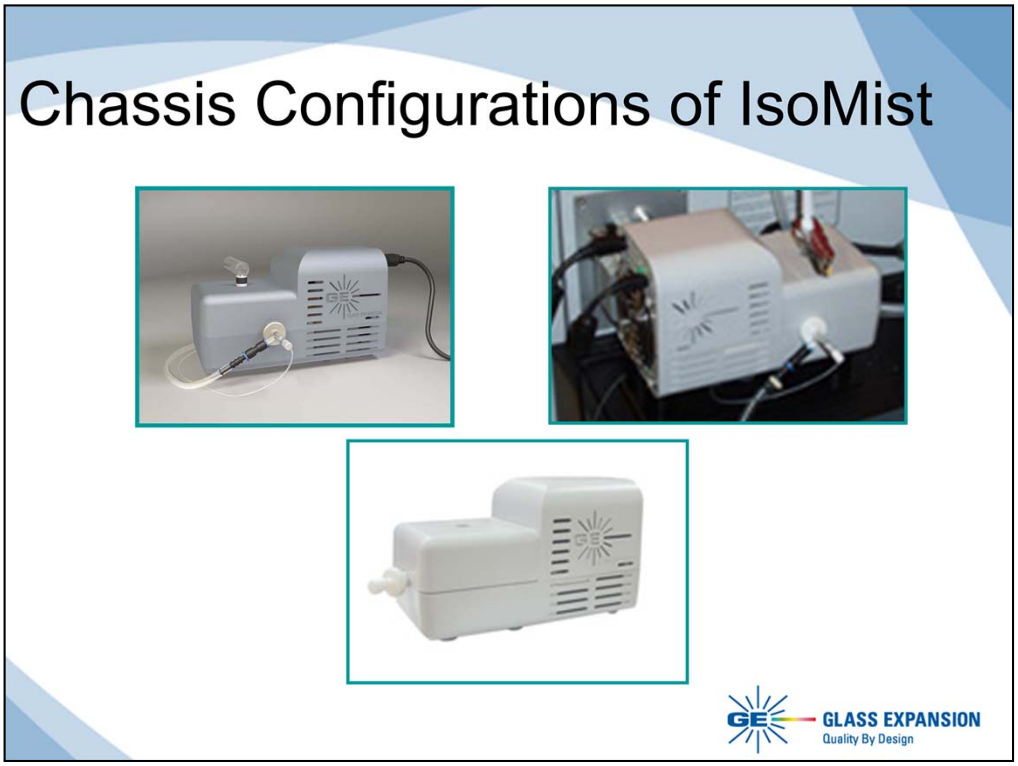
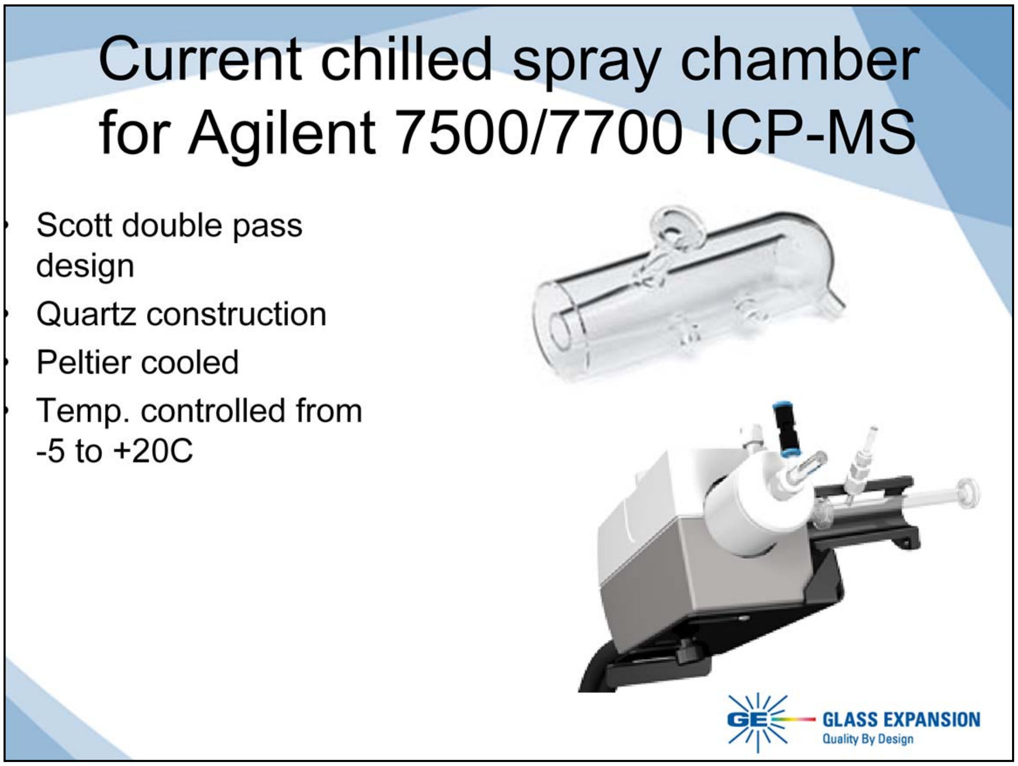
Configurations of the IsoMist are available which are customized for all of the models of ICP-OES and ICP-MS shown here. Furthermore, each one can be configured with a spray chamber made of borosilicate glass, quartz, or inert PFA.



The basic assembly of the IsoMist is customized in several configurations as shown here. The nebulizer and chamber can be configured on the left or right, or on the left end or right end depending upon the space available inside the sample compartment and the orientation of the torch and peristaltic pump.



Agilent supplies a chilled Scott style double pass spray chamber with their ICP-MS instruments. The ChemStation instrument software controls the spray chamber temperature in the range of -5 to +20.

We designed a cyclonic spray chamber accessory that would be a plug-and-play replacement for the Agilent supplied chamber. It uses the same electrical and water connections (unlike the IsoMist, Agilent uses water cooling instead of air cooling) and runs off the same ChemStation software.

Variety of Cyclonic Spray Chambers

- Borosilicate glass: most aqueous and organic applications
  - Twister baffled cyclonic (50ml)
  - Twinnabar mini-baffled cyclonic (20ml)
- Quartz baffled: Trace boron determinations
- PFA: HF and ultra trace applications

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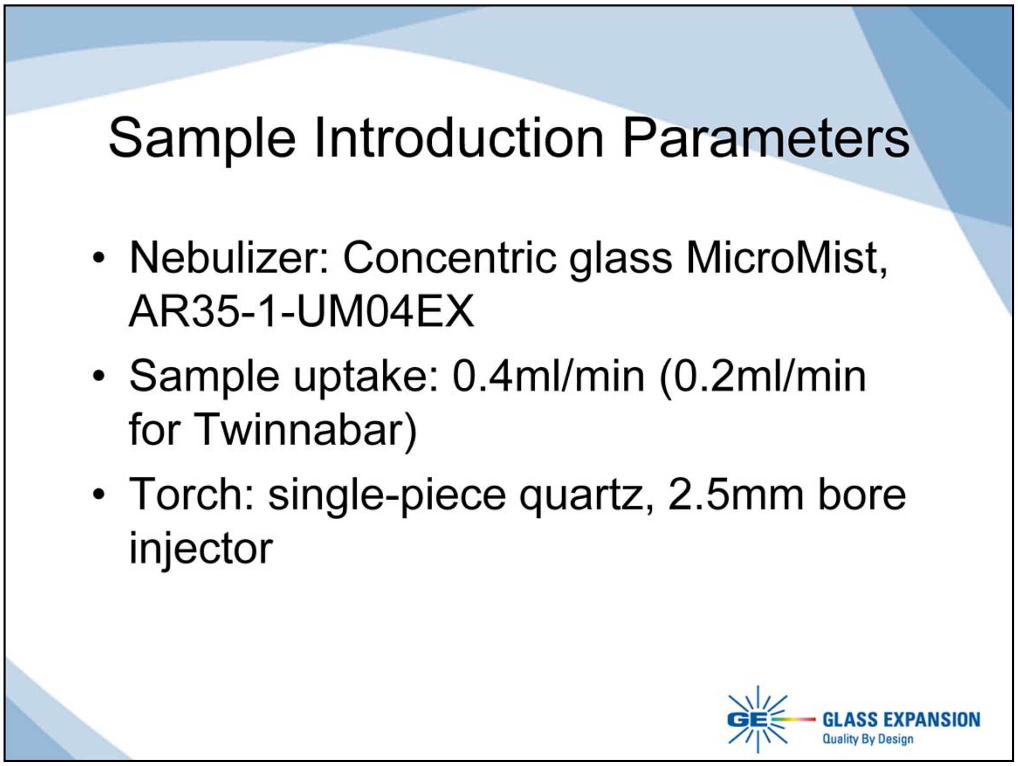
The glass and quartz chambers are encapsulated in a heat conductive resin. This serves two purposes; it facilitates heat transfer in and out of the chamber and it forms a tight seal with the heat transfer block eliminating any air which could otherwise result in condensation and freezing. The spray chamber is also available in HF resistant high-purity PFA.



The standard Agilent MicroMist nebulizer was used at an uptake rate of 0.4ml/min. For the Twinnabar study, a lower uptake rate of 0.2ml/min was employed to emulate the normal conditions of a low volume spray chamber.

Parameters

Parameter	Original Agilent spray chamber	Peltier cooled Twister cyclonic spray chamber (50mL)	Peltier cooled Twinnabar cyclonic spray chamber (20mL)
RF power [W]	1400	1400	1430
RF matching [V]	1.63	1.63	1.66
Sample depth [mm]	5.6	5.3	5
Torch-H [mm]	0.4	0.5	-0.1
Torch-V [mm]	0.3	0	0
Carrier gas [L/min]	0.87	0.85	0.84
Makeup gas [L/min]	0.21	0.23	0.22
Nebulizer pump [RPS]	0.2	0.2	0.1
Spray chamber temperature [ºC]	2	2	2
Extract 1 [V]	-189.5	-189.5	-161.1
Extract 2 [V]	-65	-71	-56
Einzel 2 [V]	14	15	10
Omega Bias [V]	-55	-55	-44
Omega + [V]	6.7	6.7	5.4
QP Focus [V]	6.1	6.1	4.1
Plate Bias [V]	0.9	0.9	0.2

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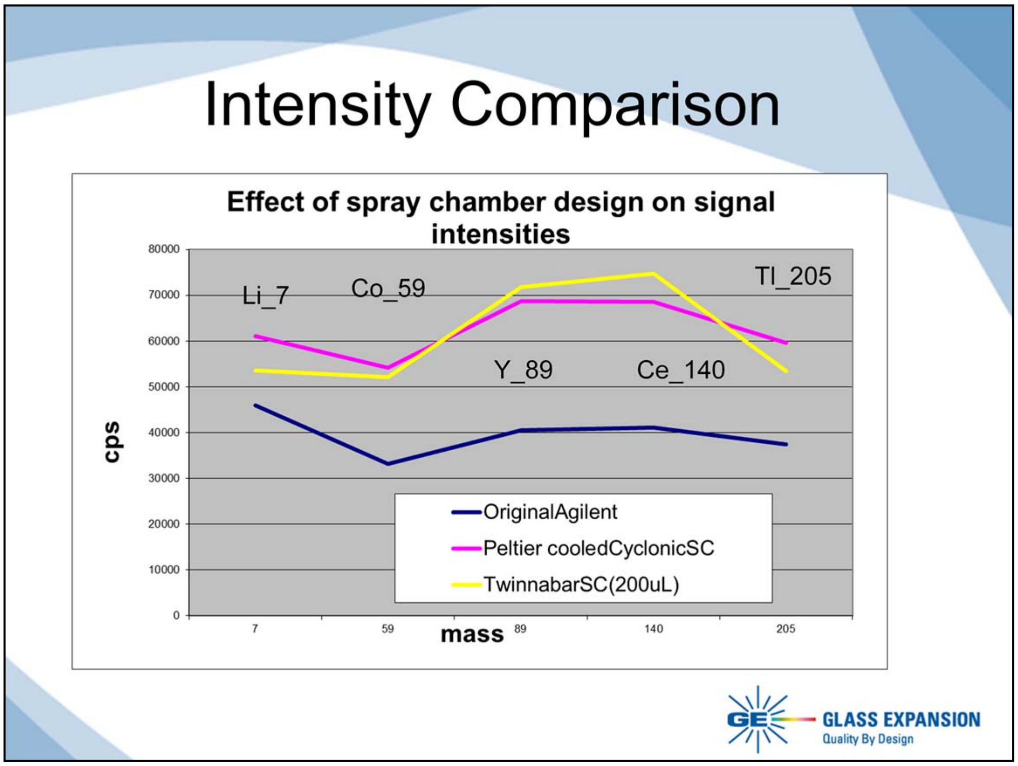
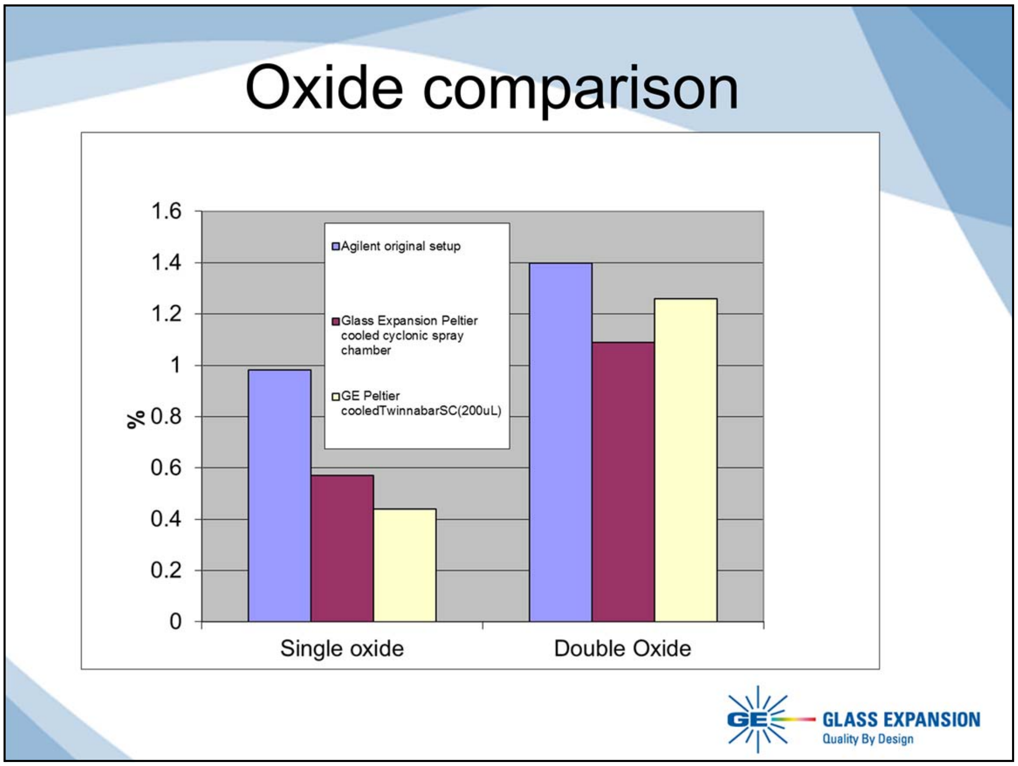
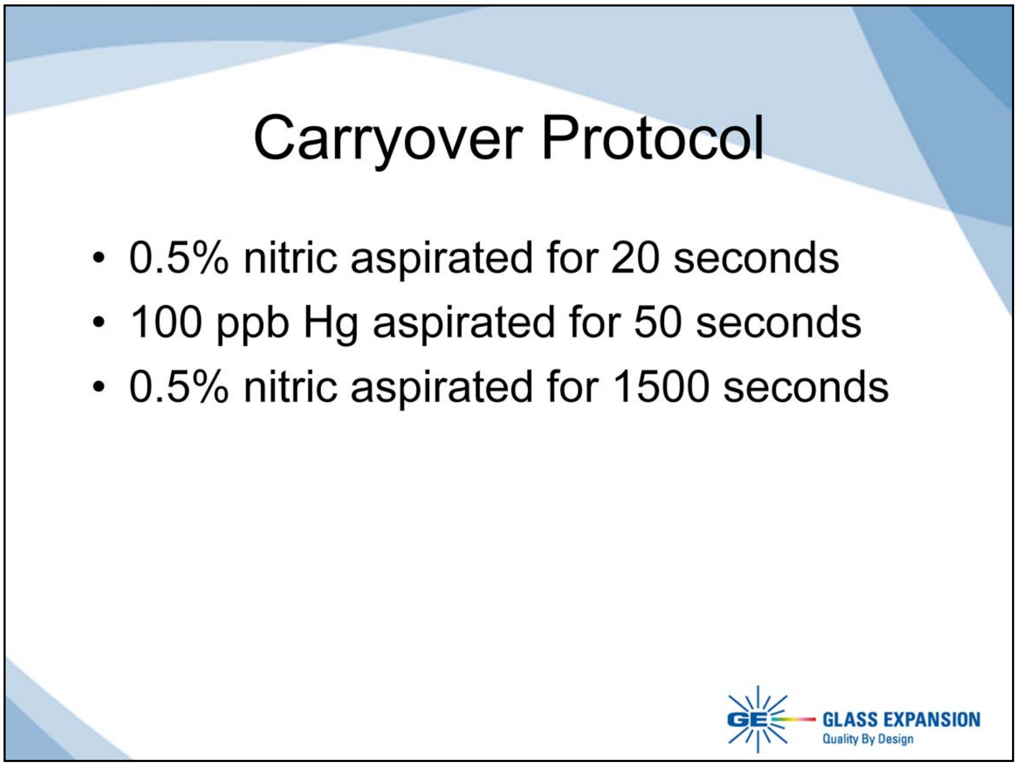
Rather than use identical parameters for all three chamber configurations, we felt it was a better test to have the automated optimization protocol of the instrument determine the best conditions for each chamber. For this reason, there are slightly different conditions for each.



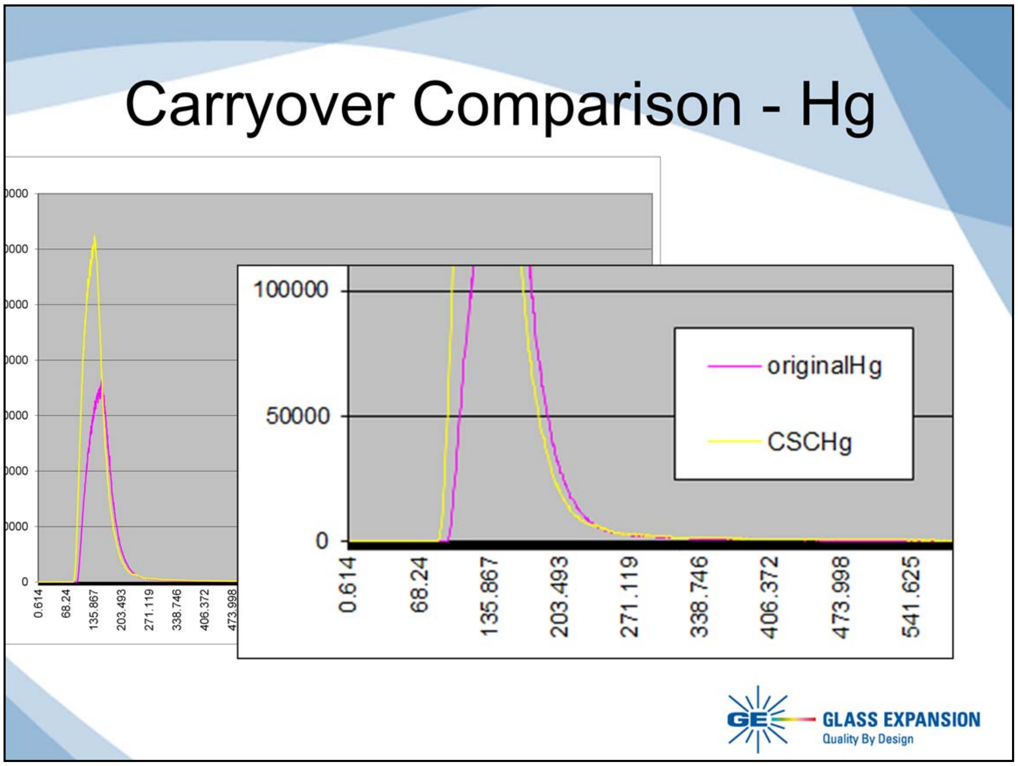
The intensity experiment showed large improvements when using either the Twister or Twinnabar. The Twinnabar experiment utilized half the sample uptake rate but also yielded a higher transport efficiency due to the smaller sample uptake rate.



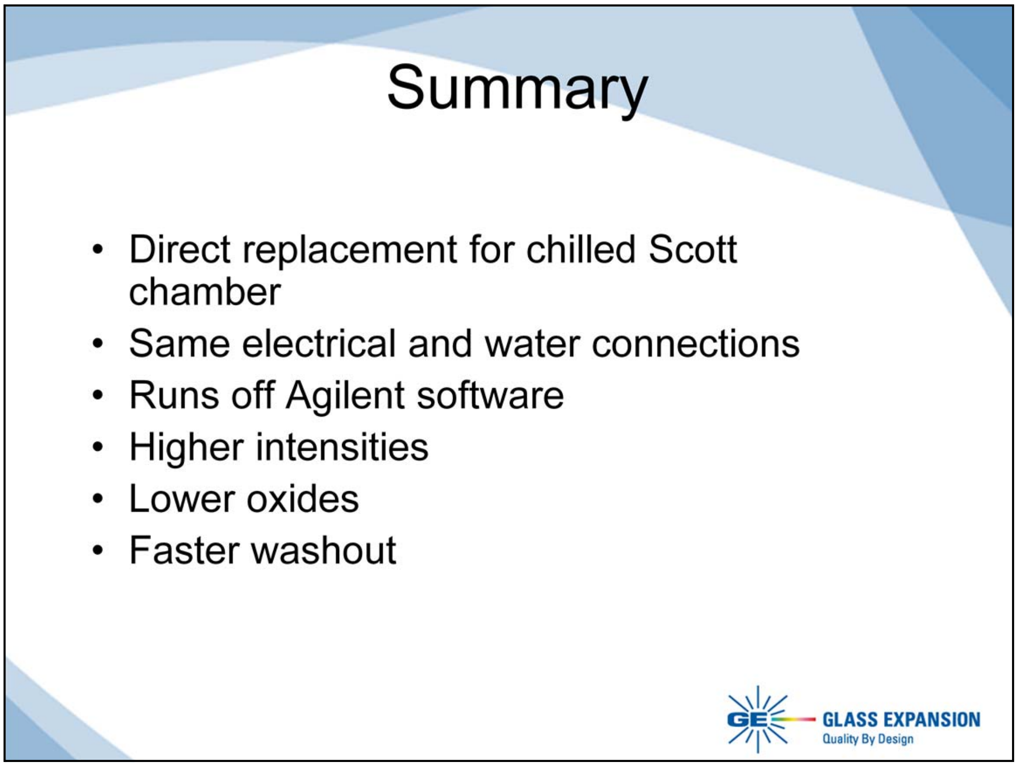
Comparing the oxide ratios, we found that the cyclonic spray chambers resulted in lower single charged and double charged oxide ratios than the Scott style design. For single charged species, the Twinnabar chamber was the lowest while the Twister had the lowest oxides for the double charged species.



We also compared the carryover of the Scott style with the Twister. This was done by monitoring the Hg signal after a 50 second aspiration and monitoring it for 1500 seconds.



The graph on the left shows the full scale peaks and the difference in intensity is clear. The graph on the right shows a zoomed in view of the baseline. Despite the higher intensity, the Hg comes down to baseline at least as quickly as it does with the Scott style chamber.



In summary, a chilled cyclonic spray chamber is now available for the Agilent 7500 and 7700 that is a plug-and-play replacement. It's advantages are higher intensities, lower oxides and faster washout.