NEWS

Glass Expansion Newsletter | November 2019 | Issue 48

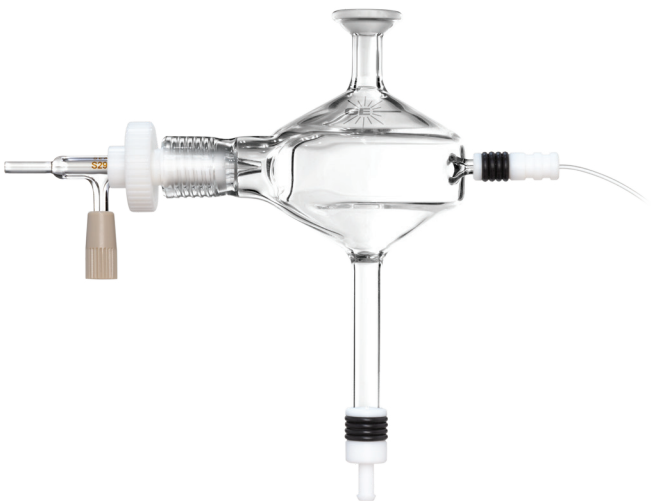
Application Spotlight

Simultaneous Multi-Element Analysis by Pneumatic Nebulization and Hg by Cold Vapor with the Glass Expansion HydraMist Spray Chamber

Introduction

While Hg can be determined by Inductively Coupled Plasma Optical Emission Spectrometry, (ICP-OES) with conventional pneumatic nebulization, the low µg/L detection limits typically achieved are not adequate for most environmental applications. The Cold Vapor technique, using an acidified stannous chloride reductant ($SnCl_2$) solution, is a widely-used technique to improve the sensitivity of Hg in atomic spectroscopy. The Cold Vapor process traditionally uses a dedicated instrument, or a Cold Vapor/Hydride Generation accessory connected to the ICP. However, for laboratories that routinely determine Hg and other elements in the same sample, switching between a cold vapor and conventional nebulization modes adds complexity, increases argon (Ar) consumption and reduces laboratory productivity.

Figure 1. HydraMist - Simultaneous Cold Vapor/Pneumatic Nebulization Spray Chamber



GE News

Annual Vacation Closure

Our factory and office in Australia will be closed for annual vacation from Monday December 23rd, re-opening on Monday, January 6th 2020.

Careers - Product Specialist

Glass Expansion is world leader in the design and manufacture of sample introduction systems for ICP-OES and ICP-MS.

To support our growing business, Glass Expansion has a new position for a Product Specialist based in the Weilburg, Germany office to provide field marketing and technical support to the Glass Expansion distributors and customers across Europe, Middle East and Africa. The successful applicant will be degree qualified with at least 5 years experience developing methods for ICP-MS and/or ICP-OES .

Glass Expansion provides a competitive remuneration package with a supportive, friendly team environment. To be involved with the dynamic, fast-growing successful organization send your resume to the HR manager, Suzie Sharry at ssharry@geicp.com.

2020 Winter Conference

On Plasma Spectrochemistry
Tucson, Arizona
January 13 - 18th, 2020
Please visit us at Booth #16
http://icpinformation.org/

In this issue:

GLASS EXPANSION
Quality By Design

Glass Expansion, world leaders in sample introduction systems for ICP-OES and ICP-MS have made a single-pass cyclonic spray chamber with conventional pneumatic nebulization capabilities and a secondary inlet port to inject a stream of $SnCl_2$ into the nebulizer aerosol plume. The aerosol/liquid interaction ensures a rapid mixing of the aerosolized Hg in the nebulizer plume with the acidified $SnCl_2$, efficiently converting Hg into a volatile form to improve Hg sensitivity. In this simple arrangement, the HydraMist gave a 20-fold improvement in Hg detection limits compared to conventional pneumatic nebulization setup, without degrading the precision or sensitivity of the other elements being conventionally nebulized through the SeaSpray nebulizer.

Instrument

The ICP-OES used in this work was an Agilent Technologies® 5100 SVDV ICP-OES, with HydraMist spray chamber and Glass Expansion SeaSpray nebulizer with operating conditions indicated in Table 1 below. To compare the performance of the system without the Cold Vapor process the analysis was repeated using a standard Tracey single-pass spray chamber from Glass Expansion instead of the HydraMist spray chamber.

Table1. Instrumental conditions

Experimental Parameter	Setting
RF power	1.4 kW
Nebulizer gas flow rate	0.60 L/min
Plasma gas flow rate	12 L/min
Auxiliary gas flow rate	1.0 L/min
Read time	30 sec
Number of replicates	3
Peristaltic pump speed	20 rpm
Stabilization time	30 sec
Sample line pump tubing	White/white
$SnCl_2$ line pump tubing	Black/black
Drain tubing	Black/white
Nebulizer	2 mL/min Direct Connection SeaSpray

The instrument was operated at a constant sample uptake with the option of the fast rinse between samples disabled.

$SnCl_2$ Preparation

As $SnCl_2$ can form an insoluble salt when dissolved in water, it is usually dissolved in concentrated HCl acid first and then diluted with H_20 to form a 5% $SnCl_2$ solution in 5% HCl.

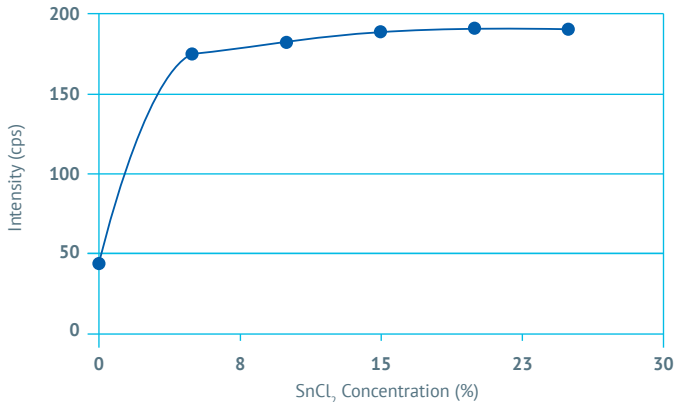
Sample Preparation

The sample was Standard Reference Material 1643f Trace Elements in Water from the National Institute of Standard Technology (NIST) Gaithersburg, MD. SRM 1643f does not naturally have detectable Hg so it was spiked with 5ppb for this work.

Results & Discussion

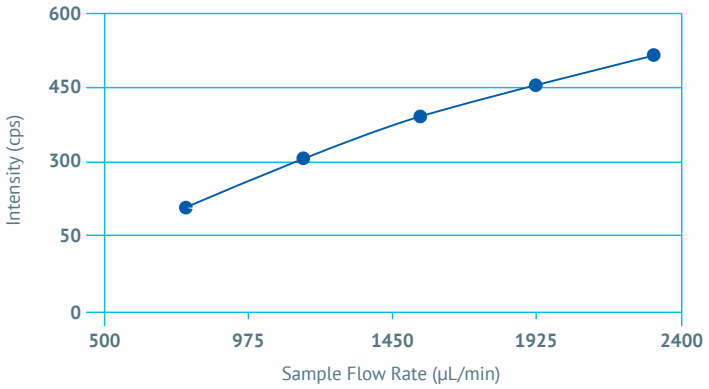
As can be seen in Figure 2, the optimum concentration of $SnCl_2$ reaches a maximum at about 5% and plateaus after that.

Figure 2. Sensitivity of Hg 194 with $SnCl_2$ concentration



In Figure 3, the intensity of Hg at 194nm is measured as a function of sample uptake rate. The sample uptake rate is adjusted by varying the instrument peristaltic pump speed. The intensity of Hg increases as the volume of sample aspirated is increased, indicating the Cold Vapor process is sample limited, not reductant limited.

Figure 3. Hg 194nm intensity versus sample uptake rate.



In Figure 4, the Signal to Root Background Ratio (SRBR) and Net Intensity of Hg at 194nm were evaluated as a function of nebulizer flow. The nebulizer gas flow not only aspirates the analyte solution, but it is also used to transport the sample aerosol and Hg Cold Vapor into the plasma. SRBR is used to optimize nebulizer flow, as it is a good proxy for detection limit performance in a solid-state detector based ICP-OES. As can be seen in Figure 4, optimum nebulizer flow rate is around the 0.6 L/min Ar.

Figure 4. SRBR and Net Intensity of Hg 194nm versus Nebulizer gas flow rate (L/min)

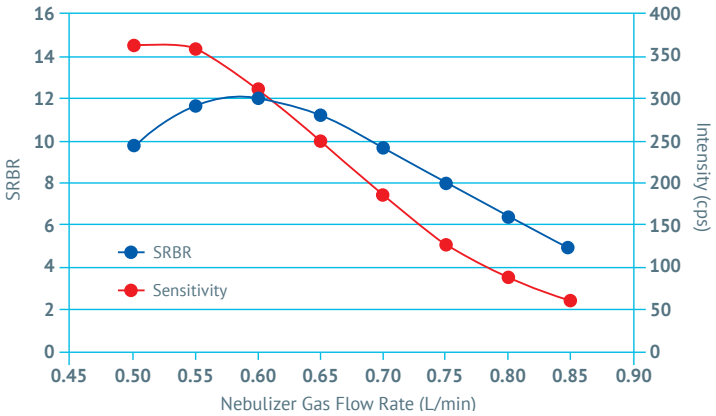


Figure 5 shows a comparison of blank intensity, and 5ppb and 10ppb Hg intensities with and without $SnCl_2$ showing Hg sensitivity enhancements due to cold vapour generation using this $SnCl_2$ - HydraMist method.

Figure 5. Mercury sensitivity with and without $SnCl_2$

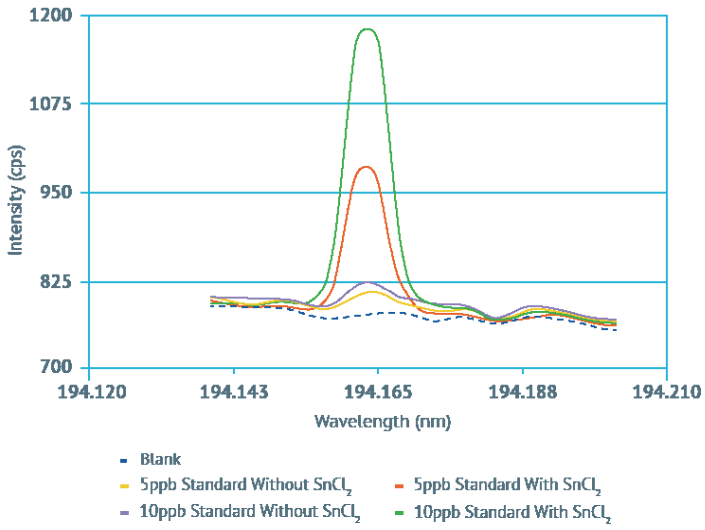


Table 2 presents 3σ Detection Limits (ppb) comparison between HydraMist and Tracey spray chambers.

Table 2. 3σ method detection limits using a HydraMist spray chamber with SeaSpray nebulizer simultaneously determined using pnuematic nebulization and Cold Vapor analysis. The results are an average of 3 separate runs, measured using 3 replicates per sample of 10 blank samples.

Analyte λ (nm)	HydraMist 3σ Detection Limits (ppb)	Tracey 3σ Detection Limits (ppb)
As 188	2.3	2.5
Be 313	0.01	0.01
Cd 214	0.1	0.1
Co 233	0.6	0.6
Cr 268	0.3	0.3
Cu 327	0.7	0.5
Hg 194	0.2	4.2
Mn 257	0.03	0.03
Mo 202	0.5	0.6
Ni 232	0.6	0.8
Pb 220	2.1	2.4
Sb 217	2.8	2.6
Se 196	3.6	3.5
Tl 191	2.4	2.4
V 292	0.4	0.4
Zn 231	0.2	0.2

The measured concentration of 16 elements in SRM 1643f "Trace Elements in Water" using the HydraMist spray chamber and SeaSpray nebulizer under the conditions listed in Table 1 are shown in Table 3. As the SRM does not contain measurable concentrations of Hg, a 50 mL aliquot of the SRM was spiked with 25 µL of 10 ppm Hg, for a 5 ppb spike concentration.*

Table 3. Measured concentration of 16 elements in SRM 1643f spiked with 5 ppb Hg using the HydraMist spray chamber and SeaSpray nebulizer.

Analyte λ (nm)	SRM 1643f Found (ppb)	SRM 1643f Certified (ppb)	Recovery %
As 188	60.5	57.4	105
Be 313	13.6	13.7	100
Cd 214	5.8	5.9	99
Co 233	25.5	25.3	101
Cr 268	18.5	18.5	100
Cu 327	22.9	21.7	106
Hg 194	5.3*	5.0	106
Mn 257	37.1	37.1	100
Mo 202	121.9	115.3	106
Ni 232	65.1	59.8	109
Pb 220	19.0	18.5	103
Sb 217	56.3	55.5	102
Se 196	11.9	11.7	103
Tl 191	6.3	6.9	91
V 292	37.3	36.1	103
Zn 231	74.8	74.4	101

The 5.3 ppb measured value for the 5 ppb Hg spike, represents a 106% recovery using the cold vapor mode of the HydraMist spray chamber. The measured values of all 15 naturally occurring elements were within 10% of the certified values, indicating the HydraMist spray chamber is not only sensitive for Hg by cold vapor, but also suitable for measuring trace elements in waters using conventional pneumatic nebulization mode.

Conclusion

When using the Agilent Technologies® 5100 SVDV ICP-OES with HydraMist spray chamber in cold vapor mode, the 3σ detection limits of Hg were found to be 0.2 µg/L compared to the 4.2 µg/L detection limits with a conventional Tracey single-pass spray chamber. The measured results for the "naturally occurring" elements and the 5 µg/L Hg spike in ***SRM 1643f Trace Elements in Water*** were all found to be within 10% of the expected values, demonstrating the HydraMist spray chamber is a simple and sensitive sample introduction system suitable for the simultaneous detection of Hg using Cold Vapor and other trace elements by conventional pneumatic nebulization.

Ordering Information for the HydraMist Spray Chamber

Part Number	Instrument Compatibility
KT-1157	Agilent® 5100/5110, 5800/5900
KT-1168	Agilent® 7700/7800/7900/8900
KT-1157	Analytik Jena® PQ 9000
KT-1157	PerkinElmer® Avio 200/500
KT-1160	Spectro™ Arcos II SOP/EOP & Blue EOP/SOP
KT-1179	Thermo® Duo 6000/7200/7400
KT-1156	Thermo® Duo 7600

NEW D-Torch for Shimadzu® ICPE-9800 ICP-ES

The Shimadzu® ICPE-9800 ICP-ES uses a traditional single-piece torch design that is made up of a fused outer tube, intermediate tube and injector. One-piece torches are an expensive consumable item requiring frequent replacement. Also, as the torch is a one-piece construction, you may need several different versions to cover different sample matrix such as waters, brines, organics and samples containing HF.

Glass Expansion has released the demountable D-Torch for the Shimadzu® ICPE-9800 ICP-ES. The D-Torch is a direct replacement for the standard Shimadzu® torch. The D-Torch provides the benefits of a demountable torch design such as:

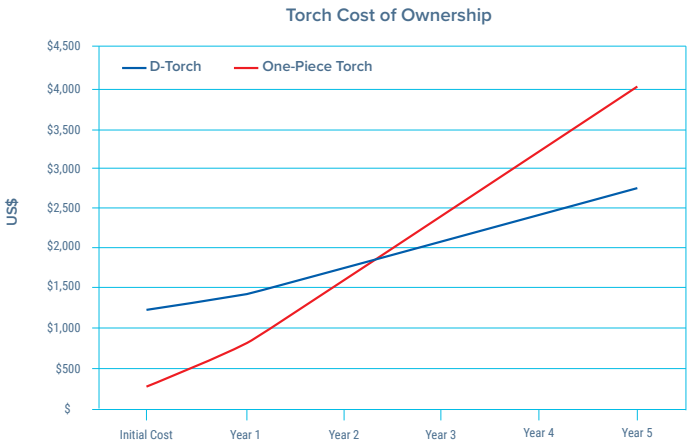
- Flexibility with a choice of interchangeable quartz or alumina injectors, and a range of internal diameters for optimum analysis for sample containing organic solvents, HF acid or high dissolved salts
- Low cost, replaceable, quartz outer tube reduces running costs
- Only require a single torch for various sample matrix
- Easy to clean as the torch can be disassembled allowing the outer tube to be soaked, rinsed and dried easily
- Improved short term precision from the tapered injector design

Ordering Information	
Part Number	**Description**
30-808-3910	D-Torch for Shimadzu® ICPE-9800
31-808-3912	Quartz Outer Tube
31-808-3964	Tapered Alumina Injector 1.5mm ID
31-808-3965	Tapered Quartz Injector 1.0mm ID
31-808-3966	Tapered Quartz Injector 1.5mm ID
31-808-3967	Tapered Quartz Injector 1.8mm ID

The D-Torch is a cost-effective alternative to the standard one-piece or semi-demountable torch designs. It will save money for any laboratory in as little as 2 years as with the D-Torch only the outer tube is replaced, not the entire torch. Also, if your laboratory analyses different sample matrix and need more than one torch configuration, the cost benefits of the D-Torch are even more significant.

Glass Expansion has a wide range of D-Torches to suit most ICP-OES and ICP-MS models. Please go to our web page to find the what other instruments the D-Torch is compatible with: http://www.geicp.com/intro/d-torch

Comparison of Torch costs over a 5 year period using 3 torches per year



Laser Ablation Connectors

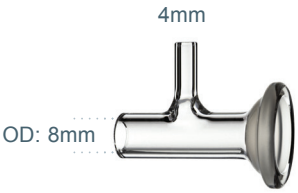
Standard cup size of S13

P/N: 21-809-4309

S13 Ball and Cup 'T'

P/N: 21-809-4140

Connect to 4mm and 8mm tubing by way of GAZ-04U and GAZ-08U



P/N: 31-808-3863

(Compatible with iCAP Q/RQ/TQ)

OD: 4mm
ID: 2mm

P/N: 31-808-3045

For 4mm plastic tubing (Push-in Press Fit)

OD: 4mm
ID: 2mm

P/N: 21-809-0965C

Connect to 6mm tubing by way of GAZ-06U or plastic compression union

OD: 6mm
ID: 4mm

P/N: 31-800-1007

Connect to 4mm tubing by way of GAZ-04U

ID: 4mm

P/N: 31-808-4034

GazFit Connectors

P/N: GAZ-04U

P/N: GAZ-06U

P/N: GAZ-08U

PerkinElmer® Syngistix Automated Method Validation Software Module

Validation of analytical methods is a requirement for many types of labs and is a critical step as these labs seek to obtain national or international accreditation. The challenge is in satisfying regulatory compliance while significantly improving data traceability. An example of such regulatory need for method validation is in USP Chapter <233>, which specifies the various tests that a lab should conduct to validate an ICP-MS method for the analysis of elemental impurities in pharmaceutical products.

Manual method validation is time-consuming and prone to errors, negatively impacting productivity. PerkinElmer's® Syngistix™ Automated Method Validation module, an extension of Syngistix for ICP-MS software and NexION® ICP-MS instruments, is designed to eliminate inefficiencies by streamlining the workflow and avoiding potential human error of traditional method validation.

The Syngistix Automated Method Validation module replaces traditional paper-based method validation where technical data resides in protocols, notebooks, forms, unprotected spreadsheets, and technical reports. By centralizing all instrument validation data, this improves data traceability and integrity. If installed along with Syngistix for ICP-MS Enhanced Security™ software, the user access control, audit trail, and electronic signatures functions are provided, helping the lab comply with 21 CFR Part 11.

The Syngistix Automated Method Validation module streamlines your method validation and improves compliance by eliminating transcription errors, delivering data traceability and reducing process inefficiencies.