

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 $\mu\text{g/L}$ 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



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/>

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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 SnCl₂ into the nebulizer aerosol plume. The aerosol/liquid interaction ensures a rapid mixing of the aerosolized Hg in the nebulizer plume with the acidified SnCl₂, 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.

Table 1. 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.

SnCl₂ Preparation

As SnCl₂ can form an insoluble salt when dissolved in water, it is usually dissolved in concentrated HCl acid first and then diluted with H₂O to form a 5% SnCl₂ solution in 5% HCl.

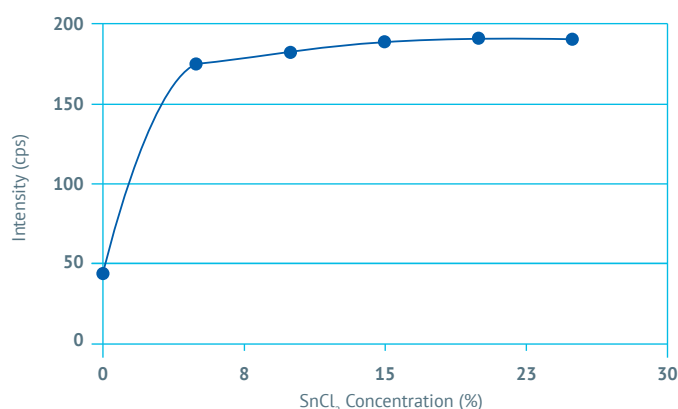
Sample Preparation

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

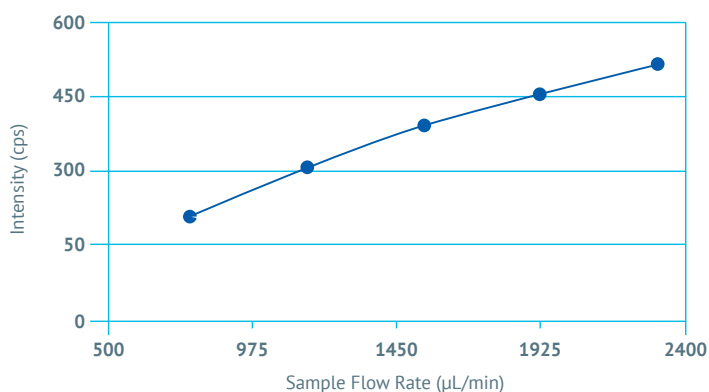
As can be seen in Figure 2, the optimum concentration of SnCl₂ reaches a maximum at about 5% and plateaus after that.

Figure 2. Sensitivity of Hg 194 with SnCl₂ 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)

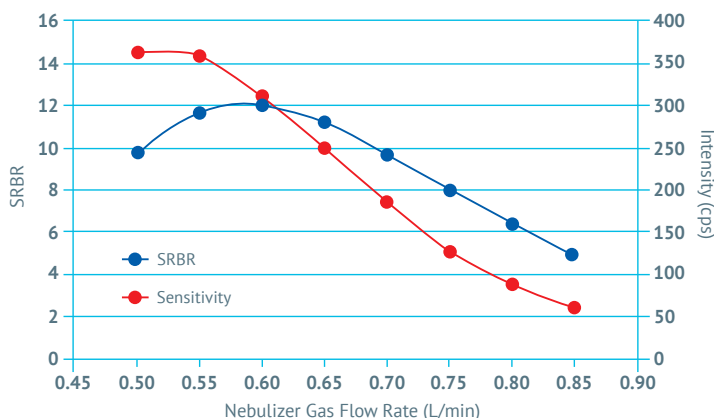


Figure 5 shows a comparison of blank intensity, and 5ppb and 10ppb Hg intensities with and without SnCl₂ showing Hg sensitivity enhancements due to cold vapour generation using this SnCl₂ - HydraMist method.

Figure 5. Mercury sensitivity with and without SnCl₂

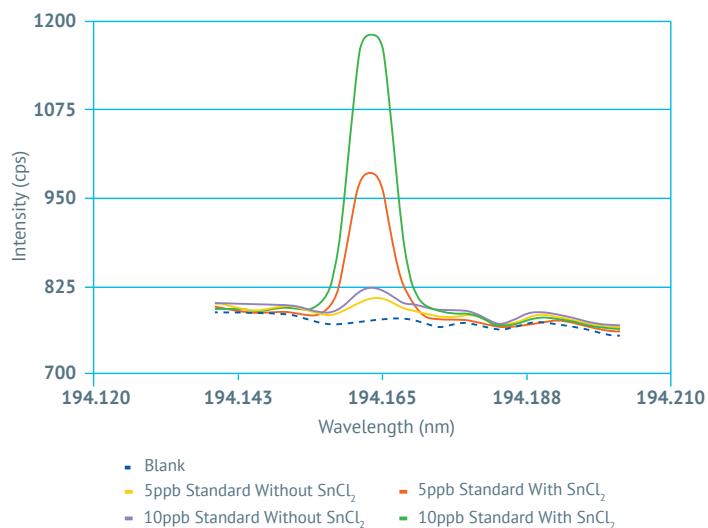


Table 2 presents 3σ Detection Limits (ppb) comparison between HydraMist and Tracey spray chambers.

Table 2. 3σ method detection limits using a HydraMist spray chamber with SeaSpray nebulizer simultaneously determined using pneumatic 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 |

Application Spotlight

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 $\mu\text{g/L}$ compared to the 4.2 $\mu\text{g/L}$ detection limits with a conventional Tracey single-pass spray chamber. The measured results for the “naturally occurring” elements and the 5 $\mu\text{g/L}$ 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 iCAP Duo 6000/7200/7400 |
| KT-1156 | Thermo iCAP 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:

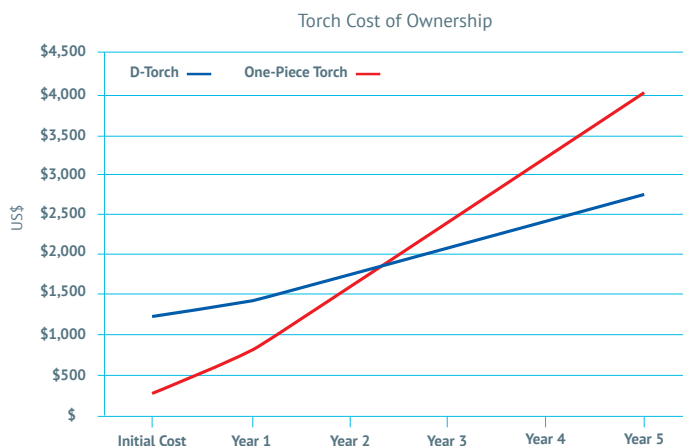
- Flexibility with a choice of interchangeable quartz or alumina injectors, and a range of internal diameters for optimum analysis for sample containing organic solvents, HF acid or high dissolved salts
- Low cost, replaceable, quartz outer tube reduces running costs
- Only require a single torch for 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

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



| Ordering Information for the D-Torch for the Shimadzu ICPE-9800 | |
|---|-----------------------------------|
| 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 |

Laser Ablation Connectors

Standard cup size of S13

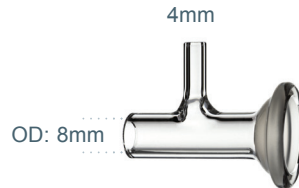
P/N: [21-809-4309](#)

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.