From Seawater to Shellfish: Microplastics... Find out what's slowly krilling you, and the best way to stay happy as a clam!



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GLASS EXPANSION Quality By Design

High Efficiency Sample Introduction System (HE-SIS)



HE-SIS Bracket Support

Every HE-SIS is designed to suit a specific instrument model, and includes an instrument-specific mounting bracket support.

Part Number	Description
KT-1155	HE-SIS for Agilent® ICP-MS
KT-1172	HE-SIS for TOFWERK icpTOF
KT-1172	HE-SIS for Thermo Scientific® Q, RQ, TQ ICP-MS
KT-1172	HE-SIS for Thermo Scientific® Neoma MC-ICP-MS
KT-1184	HE-SIS for PerkinElmer® NexION 1000, 1100, 2000, 2200, 5000 ICP-MS
KT-1204	HE-SIS for PerkinElmer® NexION 300, 350 ICP-MS
KT-1205	HE-SIS for NU ATTOM
KT-1213	HE-SIS for Thermo Scientific® X-Series
KT-1215	HE-SIS for Thermo Scientific® Neptune/Element
KT-1219	HE SIS for Nu Vitesse





GLASS EXPANSION Quality By Design

HE-SIS Kit Features

This specially designed concentric glass nebulizer is based on our popular MicroMist[™] design, capable of efficiently nebulizing limited sample volumes at low sample and argon gas flow rates.

Our patent-pending MicroJet[™] gas adapter shapes the nebulizer aerosol plume to reduce sample deposition on the spray chamber walls and enhance transport efficiency.



The Lotis[™] HE spray chamber directly couples to the ICP-MS torch, providing the highest transport efficiency and excellent washout between samples.



GLASS EXPANSION Quality By Design

Optimizing Operating Parameters





• Nebulizer gas flow rate (L/min)

• MicroJet gas flow rate (L/min)

• Nebulizer sample flow rate (µL/min)

*Combined gas flow rate through the injector is typically close to 1.0 L/min





Optimizing Nebulizer Gas Flow Rate



Nebulizer Gas Flow Rate (L/min)

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GLASS EXPANSION Quality By Design

Optimizing MicroJet Gas Flow Rate



MicroJet Gas Flow Rate (L/min)

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7 Li_No Gas 47 Ti_No Gas 55 Mn_No Gas 66 Zn_No Gas 111 Cd_No Gas 208 Pb_No Gas



GLASS EXPANSION Quality By Design

Average Sensitivity Ratio – Comparison Brand X



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Average Sensitivity Ratio (GE/BrandX)



GLASS EXPANSION Quality By Design

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Demountable Torch – Interchangeable Injector



- A demountable torch provides the benefit of interchangeable injectors.
 - 1.5mm and 2.5mm ID quartz studied
 - Other injector ID's and materials available

* D-Torch[™] for NexION 2200/5000 ICP-MS shown, P/N <u>30-808-3927</u>

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GLASS EXPANSION Quality By Design

Sensitivity Comparison – Injector ID





55 Mn_No Gas_1.5mm ID Injector



55 Mn_No Gas_2.5mm ID Injector



GLASS EXPANSION Quality By Design

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HE-SIS Literature

The HE-SIS has been coupled to many different ICP-MS platforms for a wide variety of applications, including single-cell, single-particle, nanoparticle, and low-volume sample studies, such as nanoplastics and microplastics with up to 95% transport efficiency.

- Development of single-cell ICP-TOFMS to measure nanoplastics association with human cells, Environ. Sci.: Nano, 2023, 10, 3439.
- Breaking barriers in Microplastic Detection using Single-Particle ICP-TOFMS, Lyndsey Hendriks, TOFWERK.
- Towards Automated Routine Analysis of the Distribution of Trace Elements in Single Cells using ICP-MS, Current Trends in Mass Spectrometry, March 2020.
- Very low mass isotope data collection with the Nu Vitesse, measurement of microplastic particles, Vitesse Note NT10.
- In addition to many scientific presentations.



HE-SIS Summary

- In order to achieve optimum performance, it is necessary to optimize all operating conditions for both the instrument and sample introduction system.
- Our example showed the optimum sensitivity was observed at a nebulizer gas flow rate of 0.35 L/min and sample uptake in the range of 20 to 40 μ L/min.
- Glass Expansion's HE-SIS is 2–4x more sensitive than another commercially available system.
- Optimum make-up gas flow was dependent on the ID of the injector:
 - Smaller bore injector (1.5mm ID) provided highest sensitivity at a make-up gas flow of 0.50 L/min, combined gas flow of 0.85 L/min.
 - Larger bore injector (2.5mm ID) provided highest sensitivity at a make-up gas flow of 0.80 L/min, combined gas flow of 1.15 L/min.





Approaches and Strategies for the Detection and Quantification of Microplastics by Single Particle-ICP-MS





Contact Me

Aaron Hineman

Atomic Spectroscopy Product Line Leader, PerkinElmer



Microplastics

Analytical Techniques

Microbeads in toiletries - Method 623.1

• Microbeads in products are extracted and analyzed to determine the composition of the beads. FT-IR is used to determine and provides confirmation of the presence or absence of plastic microbeads.

TGA-IR-GC/MS

- TGA measures both the weight loss and the rate of evolution of products, which provides detailed kinetic information of polymers' decomposition.
- IR quantitation of individual polymers and mixtures, functional group analysis of the volatile products.
- GC/MS quantitation of polymers mixtures and functional group of the volatile products.









Microplastics

Analytical Techniques

Single Particle-ICP-MS

- Detection and counting of carbon-based particles
 - Short dwell times reduce the background
 - Accurate results for particles down to 2 μm
- Linear pass spray chamber is recommended to efficiently transports microplastics to the plasma
- SP-ICP-MS serves as a good screening technique for microplastics
 - Other techniques are required to determine composition



Unlocking Carbon-13 with Single Particle ICP-MS: Feasibility Study for Microplastic Detection

Introduction Carbon is difficult to measure with ICP-MS because of its high ionization potential (11.3 eV) and its presence in both the

argon used to generate the plasma (primarily in the form of CO₂, as an impurity) and in reagents, including acids and water. As a result, extremely high backgrounds exist at both of the naturally occurring isotopes of carbon: C12 (98.94% abundance) and C13 (1.05% abundance). With no easy way to remove these sources of carbon, limits of detection with either isotope are severely affected.

One way to greatly reduce backgrounds is by shortening the measurement times using dwell times in the range of microseconds, as is typically done with single particle ICP-MS (SPI-CP-MS)². Working at these short dwell times in SPI-CP-MS mode, the background signal is reduced whereas the overall signal from the particles remains unaffected, allowing particles to be detected and measured at levels that were previously unstanable ³.

By using SP-ICP-MS, the C13 background is reduced significantly, permitting carbon-containing particles to be detected, counted, and measured. As a result, SP-ICP-MS may be used as a screening tool for the detection of microplastics, as discussed in detail by Laborda *et al.*¹. This work summarizes the principles involved in the detection of microplastics with SP-ICP-MS, and also shows examples.





Approaches and Strategies for Method Development

Platform selection

• ICP-MS vs ICP-MQ

Gas selection

• Std vs NH₃ vs. H₂

Isotope selection

• C₁₂ vs C₁₃

Universal Cell Parameters

- Cell gas flow rate => Affect reaction rate
- Rejection parameter q (RPq) => Control product formation
- Rejection parameter a (RPa) => Attenuate selected element signal



Instrumentation





HE-SIS (P/N KT-1184)







Operating Conditions

Parameter	Value
Neb Gas Flow (L/min)	0.46
AMS/Carrier Gas Flow (L/min)	0.6
Dwell Time (μs)	25
IGM	Extraction
Scan Time (s)	30 - 180
Sample Flow Rate (µL/min)	13
Transport Efficiency (%)	89.23



Operating Conditions

Mode	Mass (amu)	Mode	Gas	Gas Flow	RPa	RPq	AFT	LOD (nm)
Standard	C13	MS/MS	-	-	0.017	0.25	150	1819
Standard	C12	MS/MS	-	-	0.02	0.25	150	990
DRC H ₂	C13	MS/MS	H ₂	1	-	0.45	225	672
DRC H ₂	C12	MS/MS	H ₂	1	0.043	0.45	200	722
DRC NH ₃	C13	MS/MS	NH ₃	0.1		0.45	125	1559
DRC NH ₃	C12	MS/MS	NH ₃	0.1	0.045	0.45	125	824

Experimental

Standards

- 100 nm Au NP N8151036
- Carbon 1000 ppm Inorganic ventures

Samples

- Negative control UPW
- 8 um polystyrene beads positive control
- Tea bag Sample
- Tap water Sample blank





Method Validation

Mode	lsotope	4	μm	8 µm		
		Size (nm)	Particle/mL	Size (nm)	Particle/mL	
CRM Certified Values		4043	130000	7989	40000	
Standard	C13	3426.8	226420*	9000.3	32579	
Standard	C12	4035.3	123494	8723.1	31025	
DRC H ₂	C13	3726.4	130561	8761.0	36371	
DRC H ₂	C12	3515.3	119638	7636.9	35829	
DRC NH ₃	C13	3135.6	123580	7834.5	44296	
DRC NH ₃	C12	4125.9	133319	9095.2	42637	

* Inaccurate counting due to low signal / high background



Results – 4 μ m – C12 MS/MS DRC H₂





Average = $3.5 \mu m$

Results – 8 μ m – C12 MS/MS DRC H₂



Average = $7.6 \mu m$



Mixed Particle Standards: $4 + 8 \mu m - C12 MS/MS DRC H_2$





Mixed Particle Standards: $4 + 8 \mu m - C12 MS/MS DRC H_2$

Mode	lsotope	4	um	8 µm		
		Size (nm)	Particle/mL	Size (nm)	Particle/mL	
CRM Certified Values		4043	56500	7989	22500	
Standard	C13	4234.2	44210	9046.1	24647	
Standard	C12	4134.2	56189	8769.4	22407	
DRC H ₂	C13	4080.2	75406	9171.8	24992	
DRC H ₂	C12	3852.3	63543	8262.3	24072	
DRC NH ₃	C13	3968.6	44899	8271.0	24820	
DRC NH ₃	C12	4304.2	68943	9601.6	26543	

Tea Bag Analysis

• Brand 1



• Brand 2



Tea Bag Analysis

• Brand 1



• Brand 2

PerkinElmer Science with Purpose

Tea Bag Analysis

Sample	Mode	amu	Start (nm)	End (nm)	Most Freq. Size (nm)	Mean Size (nm)	Part. Conc. (parts/mL)
Matrix Blk	DRC H ₂	C12	722	-	-	-	-
Tea Bag 1	DRC H,	C12	921	8671	3021	3568	3081
Tea Bag 2	DRC H,	C12	796	11146	3896	4549	10212





Conclusions

- Carbon quantification by ICP-MS is feasible, various reaction gases, cell conditions, can be used to bring down the background to a measurable level.
 - Validated using DRC technology using 4 um and 8um standards to sub-micron detection limits
- Single particle can be used to screen for microplastics in a variety of matrices
- Improvement in detection limits is needed to achieve nanoplastics analysis
- Improvement in detection limits can be achieved with carbon free reagents and labware
- Microplastics counting can be achieved by SP-ICP-MS, but sizing is a challenge with unknown density
- Sample analysis time 2-3 min SP-ICP-MS vs 40-45 min using imaging FTIR.



More Information Online



Thank you, questions?

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