Application Note

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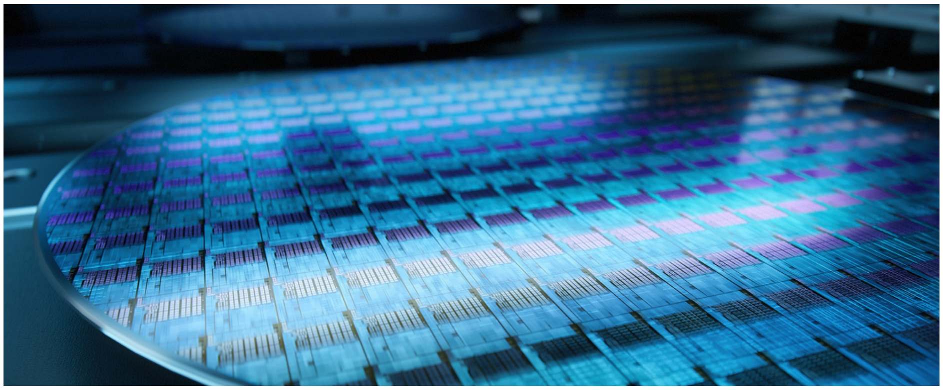
Agilent 9500 ICP-QQQ with m-Lens for Ultratrace Analysis of High-Purity Reagents

Achieving low backgrounds under hot plasma conditions using ICP-QQQ preset methods



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Introduction

Controlling trace-metal contaminants is essential in both semiconductor reagent analysis and metal-based materials characterization. High-purity chemicals such as nitric acid (HNO_3) and sulfuric acid (H_2SO_4), as well as high-purity metals, require analytical workflows capable of achieving sub-ppt detection limits (DLs) while maintaining excellent robustness and reproducibility. To meet these needs, the Agilent 9500 Triple Quadrupole ICP-MS (ICP-QQQ) equipped with a Dual-Cell System (DCS)[1] and the optional m-lens ion optics provides a highly stable platform, enabling low backgrounds and reliable performance under hot plasma conditions.

The m-lens is engineered to suppress background signals for elements including easily ionized elements such as Na and K that typically show elevated background equivalent concentrations (BECs) under hot plasma conditions. The 9500 ICP-QQQ with m-lens is suitable for the analysis of metal digests and other sample types that demand robust performance and low backgrounds.[2]

However, its benefits also extend to low-matrix, high-purity reagents, supporting sub-ppt impurity measurements of simpler matrices.[3]

Agilent's ICP-QQQ portfolio allows users to select instruments based on desired DLs and operational objectives. For laboratories seeking the lowest possible DLs, the Agilent 8900 Semiconductor configuration ICP-QQQ equipped with an s-lens provides exceptional sensitivity, making it suitable for laboratories operating at the extreme limits of ultratrace quantification. The 9500 ICP-QQQ with m-lens provides robust, high sensitivity measurements, ideal for routine trace level applications. This model is appropriate for labs that may not require the lowest DLs for every element but still demand high-quality, reproducible data.

In this study, we evaluated the performance of the 9500 ICP-QQQ using a method designed specifically for low-matrix, high-purity reagents. Using preset methods implemented in the instrument software for ultrapure water (UPW) and for H_2SO_4—the UPW method is also applicable to diluted HNO_3 and H_2O_2— we analyzed 1% HNO_3 and high purity H_2SO_4 separately. Sub-ppt DLs and low backgrounds were achieved for target analytes in both matrices. These results confirm that the 9500 ICP-QQQ with m-lens can be used for ultratrace impurity analysis of semiconductor-grade reagents, as well as demanding metal-matrix applications.

Experimental

Reagents, sample preparation, and calibration

UPW produced by Puric ω II (Organo Corp, Japan), Tamapure-AA-10 grade HNO_3, and Tamapure-AA-100 grade H_2SO_4 from Tama Chemicals (Japan) were used for sample preparation. Calibration standards and spike solutions were prepared using the following multi-element standards: XSTC-7, XSTC-8, and XSTC-331 (SPEX CertiPrep LLC, USA). Unless otherwise specified, all concentrations reported in this study are expressed as w/w%. Before analysis, HNO_3 samples were diluted to 1% and H_2SO_4 samples were diluted to 9.8%.

The 9500 ICP-QQQ was calibrated using a multi-point external calibration curve prepared from matrix-matched standards. The standards were prepared at 0, 5, 10, 20, and 30 ng/kg (ppt) in 1% HNO_3 and 9.8% H_2SO_4, respectively. DLs were calculated as three times the standard deviation of the blank signals, and BECs were obtained by dividing the mean blank signal by the calibration slope. A single blank solution was measured during five replicate runs, and these replicate measurements were used to calculate the DLs and BECs.

Instrumentation

The Agilent 9500 ICP-QQQ fitted with the optional m-lens and the Agilent I-AS autosampler were fully controlled using Agilent OpenLab ICP-MS software version 1.1. Designed for ultratrace metal analysis, the sample introduction system comprised a MicroFlow PFA nebulizer with I-AS probe (operated in self-aspiration mode), a temperature-controlled quartz spray chamber, and a quartz torch with a 2.5 mm inner diameter (id) injector. A platinum-tipped sampler cone with copper base and a platinum-tipped skimmer cone with nickel base for m-lens were used.

The 9500 ICP-QQQ was operated using five tune modes: no gas, Advanced Helium Mode (AHM), O_2, NH_3, and H_2. For the H_2SO_4 samples, additional specialized tunes were prepared for the determination of platinum (Pt) and zinc (Zn) to ensure optimal performance (Table 1). The use of reaction and collision gases enabled effective removal of spectral interferences originating from argon plasma species as well as matrix-related polyatomic ions. The makeup gas flow rate and the Omega lens voltage parameters were automatically optimized by the preset methods for m-lens, resulting in a stable CeO/Ce ratio that was maintained between approximately 1 and 3%.

Table 1. Agilent 9500 ICP-QQQ operating parameters.

Parameter	No Gas	No Gas (Pt)*	AHM	AHM (Zn)*	O_2	NH_3	H_2
Scan Mode	Single Quad				MS/MS		
RF Power (W)	1500						
Nebulizer Gas (L/min)	0.7						
Makeup Gas (L/min)	Autotuned						
Extract 1 (V)	0						
Extract 2 (V)	-80						
Omega Lens (V)	Autotuned						
Q1 Bias (V)	−5	6	−5		0		
He Gas Flow (mL/min)	–	–	14		–	1	–
H_2 Gas Flow (mL/min)	–	–	–	–	–	–	6
O_2 Gas Flow (mL/min)	–	–	–	–	0.4	–	–
NH_3 Gas Flow** (mL/min)	–	–	–	–	–	3 (30%)	–
KED (V)	5		−4.2	−3	−7		−2

** Applies to H_2SO_4 samples only. **10% NH_3 balanced with 90% He.*

Results and discussion

For both acid matrices (1% HNO_3 and 9.8% H_2SO_4), all target elements exhibited excellent calibration linearity with correlation coefficients $r > 0.99$ across the respective calibration ranges. Sub-ppt DLs and BECs were achieved for most elements in both matrices, confirming the method's sensitivity for ultratrace impurity control in high-purity reagents (Table 2).

Particularly noteworthy is the performance for Zn in H_2SO_4, a measurement often considered challenging due to matrix-related backgrounds such as $^{32}S^{32}S$, $^{32}S^{34}S$, and $^{34}S^{34}S$ at *m/z* 64, 66, and 68. Using the DCS in AHM and Zn-specific tune settings, the method delivered a DL of 0.64 ppt and BEC of 0.89 ppt, demonstrating effective background suppression and robust interference control even under hot plasma conditions.

Conclusion

This study evaluated the Agilent 9500 ICP-QQQ with m-lens for ultratrace analysis of high-purity reagents using preset methods for 1% HNO_3 and 9.8% H_2SO_4. Sub- or single-figure ppt detection limits and low background equivalent concentrations were achieved for all elements, demonstrating that ultratrace measurements can be easily performed under hot-plasma conditions. The Dual-Cell System enabled effective removal of matrix-related interferences, with Advanced Helium Mode providing an effective approach for the measurement of challenging elements such as Zn in H_2SO_4. The results show that the 9500 ICP-QQQ with intuitive preset methods can deliver sensitive, low-background data, offering an accessible entry point for laboratories that are new to ultratrace analysis.

References

1. Sugiyama, N. Dual-Cell System (DCS) and Advanced Helium Mode (AHM), Agilent publication, 5994-8985EN
2. Yamashita, R. Analysis of High Purity Titanium Using an Agilent 9500 ICP-QQQ, Agilent publication, 5994-9024EN
3. Sakai, K.; Shimamura, Y. Ultrapure Process Chemicals Analysis by ICP-QQQ with Hot Plasma Conditions, Agilent publication, 5994-4025EN

Table 2. Tune modes, measured *m/z*, and calculated DLs and BECs for each analyte. DLs and BECs were derived from five replicate measurements of the same blank solution (*n* = 5). Analytes with no detectable signal are reported as ND (not detected).

Analyte	**Tune**	**Q1**	**Q2**	**1% HNO_3**		**9.8% H_2SO_4**	
				DL (ppt)	**BEC (ppt)**	**DL (ppt)**	**BEC (ppt)**
Li	No gas	–	7	0.05	< DL	0.05	< DL
B	No gas	–	11	0.98	2.08	2.04	6.08
Na	NH_3	23	23	0.16	0.17	0.19	1.42
Mg	NH_3	24	24	0.02	< DL	0.08	< DL
Al	NH_3	27	27	0.06	< DL	0.17	< DL
K	NH_3	39	39	0.25	0.71	0.41	2.04
Ca	H_2	40	40	0.04	0.06	0.16	0.20
Ti	O_2	47	63	–	–	1.30	< DL
Ti	O_2	48	64	0.02	< DL	–	–
V	AHM	–	51	ND	ND	–	–
V	NH_3	51	51	–	–	0.08	< DL
Cr	NH_3	52	52	0.06	0.06	0.14	0.24
Mn	AHM	–	55	0.04	< DL	0.12	< DL
Fe	NH_3	56	56	0.33	0.49	0.41	1.13
Co	AHM	–	59	0.02	< DL	0.02	< DL
Ni	AHM	–	60	0.20	< DL	0.51	< DL
Cu	AHM	–	63	0.14	0.21	0.22	0.25
Zn	AHM	–	66	0.10	< DL	–	–
Zn	AHM (Zn)	–	68	–	–	0.64	0.89
As	O_2	75	91	0.06	< DL	0.05	< DL
Sr	AHM	–	88	ND	ND	ND	ND
Mo	AHM	–	95	0.02	< DL	0.06	< DL
Cd	AHM	–	111	0.03	< DL	ND	ND
Sn	AHM	–	118	0.21	< DL	0.25	0.48
Sb	AHM	–	121	0.05	< DL	0.06	< DL
Ba	AHM	–	137	0.06	< DL	0.14	< DL
W	AHM	–	182	ND	ND	0.01	< DL
Pt	No gas	–	195	0.15	2.52	–	–
Pt	No gas (Pt)	–	195	–	–	0.13	0.34
Pb	AHM	–	208	0.03	0.03	0.10	0.26

Products used in this application

Agilent products

Product Type	Description	Part Number
Sample Introduction System	9500 ICP-MS quartz torch, 2.5 mm id for aqueous samples	M5150-67011
	9500 ICP-MS quartz connector tube, straight	M5150-67014
	9500 ICP-MS quartz spray chamber with straight exit port	M5150-67017
	MicroFlow PFA nebulizer with I-AS probe, self-aspirates at 200 µL/min	G3139-65102
Interface	ICP-MS sampler cone for 9500 ICP-MS, Pt tip with Cu base	M5150-67002
	Skimmer cone, Pt tip with Ni base for m-lens	G8400-67073
	Extraction-Omega lens assembly, m-lens, brass base	M5150-67023
Tubing Kits	Easy-fit peristaltic pump tubing, beige thermoplastic, yellow/blue, 1.52 mm id for drain	5005-0022
Bottle Kits	Waste container kit, includes a 10 L waste can, S60 StaySafe cap, fittings, and acid vapor filter	5005-0437

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