Application Note

Environmental

Analysis of Environmental Waters by ICP-QQQ with a Dual-Cell System and Discrete Sampling

Fast, accurate measurements by the Agilent 9500 ICP-QQQ in Advanced Helium Mode

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Introduction

Agilent ICP-MS instrumentation has become standard equipment in many environmental laboratories, recognized for its speed, sensitivity, accuracy, and robustness. Its ability to integrate seamlessly with autosamplers, high-speed discrete sampling systems, and autodilutors has further contributed to its widespread adoption.[1–3] These capabilities are especially important in contract environmental laboratories, where increasing competition and financial pressures have made sample throughput and productivity a top priority. At the same time, laboratories are expected to process more samples in less time, without sacrificing data quality or ease-of-use. To address these demands, the Agilent 9500 Triple Quadrupole ICP-MS (ICP-QQQ) includes a unique Dual-Cell System (DCS) collision/reaction cell (CRC). For routine testing of large numbers of samples, the DCS can be operated in Advanced Helium Mode (AHM), a revolutionary collision-cell mode that delivers excellent-quality data across a wide range of analytes.

AHM delivers high-sensitivity measurements while effectively removing interferences through a dual mechanism of collision-induced dissociation (CID) and kinetic energy discrimination (KED), eliminating the need for multiple cell conditions.

AHM: A revolution in helium-based interference removal

AHM represents a major advancement in He-KED collision-cell technology by dynamically optimizing DCS conditions for both low- and high-mass elements in real time. As a result, AHM provides an approximately 20-fold improvement in sensitivity for low-mass elements and approximately a twofold improvement for mid- to high-mass elements. By enabling interference removal across the entire periodic table in a single gas mode, AHM replaces no gas, conventional He, and high energy (HE) He mode. Eliminating cell-gas switching simplifies the workflow and accelerates sample measurement using the 9500 ICP-QQQ with DCS.

As shown in Figure 1, the DCS incorporates a two-stage ion guide design (front and rear ion guides) and rapid cell voltage control. These features underpin the improved performance of AHM relative to conventional He-KED mode. Further technical details of the DCS and AHM are described elsewhere.[4]

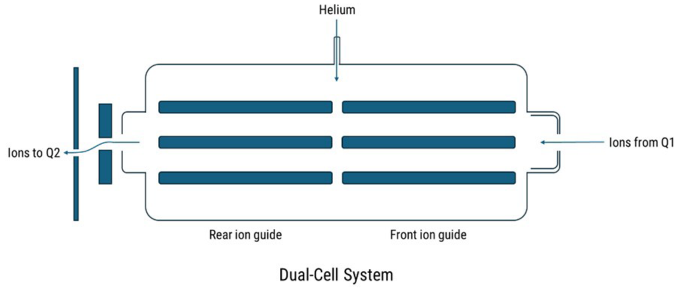

Figure 1. Agilent Dual-Cell System (DCS) collision/reaction cell used in the Agilent 9500 ICP-QQQ.

AVS MS discrete sampling: Maximizing sample throughput and minimizing maintenance

The 9500 ICP-QQQ can also be fitted with the optional Agilent AVS MS discrete sampling accessory, which is ideal for high-throughput applications. The AVS MS minimizes both sample delivery and rinsing times, significantly accelerating the analytical cycle. When combined with AHM, the 9500 with AVS MS reduces both the analysis time per sample and the interface's exposure to the complex sample matrices common in environmental samples.

The AVS MS is an automated multi-port switching valve system that directs sample and rinse solutions through different flow paths.[1,2] It introduces sample to the ICP-MS only during data acquisition, while a clean blank solution is nebulized at all other times. This approach not only increases sample throughput but also reduces the amount of sample that reaches the instrument, resulting in lower cleaning requirements and improved long-term stability.

By combining AVS MS discrete sampling and a single AHM collision cell mode for all elements, laboratories can achieve higher sample throughput and lower maintenance frequency, ultimately reducing operating costs and improving margins.

In this study, certified reference materials (CRMs) representative of the routine workload in contract environmental laboratories were analyzed by the 9500 ICP-QQQ to assess accuracy, productivity, and long-term stability.

Experimental

Instrumentation

Data was collected using an Agilent 9500 ICP-QQQ fitted with standard nickel cones, MicroMist glass concentric nebulizer, and Ultra High Matrix Introduction (UHMI). The 9500 ICP-QQQ was also equipped with the optional Agilent AVS MS discrete sampling accessory.

UHMI uses aerosol dilution to ensure a high level of matrix tolerance during the analysis of a varied sample load. The AVS MS also contributes to a further improvement in matrix tolerance for large sample batches by minimizing the time the instrument is exposed to the sample during each measurement.

The shaded instrument parameters shown in Table 1 were set automatically by selecting the generic AHM-only preset method and UHMI-4 plasma conditions for relatively high-matrix samples in the Agilent OpenLab ICP-MS software. Under UHMI-4 conditions, samples containing up to 4% total dissolved solids (TDS) can be measured, allowing stable long-term analysis at concentrations up to 500 ppm. The ion lens voltages were also automatically loaded by the software after optimization for sensitivity.

The operating conditions for the 9500 ICP-MS and AVS MS are summarized in Tables 1 and 2, respectively.

Table 1. Agilent 9500 ICP-QQQ operating conditions.

Parameter	Advanced He Mode (AHM)
Plasma Mode	UHMI-4
RF Power (W)	1600
Sampling Depth (mm)	12
Nebulizer Gas (L/min)	0.92
Dilution Gas (L/min)	0.16
Lens Tune	Autotune
Cell Gas Flow Rate (mL/min)	14
Front Energy Discrimination (V)	10
Number of Elements	26 analytes, 5 internal standards
Total Acquisition Time (3 reps) (s)	24.7

Selecting the UHMI-4 preset plasma conditions automatically sets the shaded parameters.

Table 2. Agilent AVS MS operating conditions using a 0.75 mL loop volume.

Parameter	Time (s)	Uptake Pump Speed (%)
Sample Load	8	50
Stabilization	7	5
Probe Rinse	14	5
Probe Rinse 1	5	50
Probe Rinse 2	11	0
Optional Loop Probe Wash	7	50
Optional Loop Wash	1	5

Samples and standards

CRMs for water, soil, and sediment were used to represent typical environmental samples and to verify method accuracy. The materials included NIST 1643e Trace Elements in Natural Water (Gaithersburg, MD, USA), HPS Soil B, and HPS River Sediment A (High Purity Standards, Charleston, SC, USA). Each CRM was diluted 1:10 with de-ionized water, and the soil and sediment CRMs were further diluted 1:50 to ensure that the analyte concentrations fell within the calibration range.

Five unknown environmental water samples were also prepared to simulate real-world lab conditions and assess method performance.

For the determination of Limits of Quantitation (LOQs), a blank sample containing 1% HNO_3 and 0.5% HCl was prepared.

Calibration standards

Calibration standards for most elements were prepared from Agilent multi-element Environmental Calibration Standard, while mercury (Hg) standards were prepared from a 1000 ppm single-element stock solution (Kanto Chemical Co., Inc., Japan). All calibration standards were prepared in a matrix of 1% HNO_3 and 0.5% HCl. The concentration range of the six-point calibration curves for trace and mineral elements is shown in Figure 2.

Quality Control solutions

Quality Control (QC) solutions, including the Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB), were prepared in the same manner as the calibration standards. Each solution was prepared in a matrix of 1% HNO_3 and 0.5% HCl, using the Agilent multi-element Environmental Calibration Standard for all analytes except Hg. Mercury QC solutions were prepared from 1000 ppm single-element stock solutions (Kanto Chemical Co., Inc.).

Analytical sequence

The analytical sequence shown in Figure 2 was designed to represent matrices commonly encountered in contract environmental laboratories. This sequence was then analyzed using the 9500 ICP-QQQ. The samples consisted of the water, soil, and sediment CRMs, real-world environmental water samples, and QC samples. Calibration was performed without matrix matching, and no recalibrations were performed throughout the two-hour sequence.

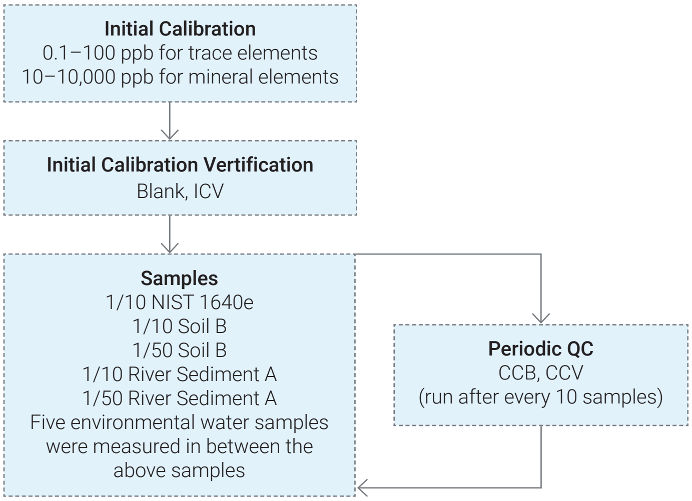

Figure 2. Sequence of calibration standards, QC solutions, and samples analyzed in a two-hour sequence. The “Samples” block was repeated continuously with automatic insertion of the “Periodic QC” block after every 10 sample runs. A total of 141 solutions were analyzed in 138 minutes, averaging less than 1 minute per sample.

Results and discussion

Sample throughput

The 9500 ICP-QQQ with an integrated AVS MS was used to measure all elements using the DCS in AHM. A total of 141 samples was analyzed in 138 minutes, with each sample requiring less than 1 minute of measurement time. In comparison, a similar sample sequence analyzed using an Agilent single-quadrupole ICP-MS required approximately 1 minute 30 seconds per sample.[1] This workflow therefore delivers a 50% increase in sample throughput relative to the single quad ICP-MS method.

Detection limits

Limits of Quantitation (LOQs) based on 10σ were calculated from 10 measurements of the blank (Table 3). Although the LOQs do not represent the lowest possible detection limits (DLs), they are sufficient for the application. Lower DLs could be achieved by extending the integration time and investing in higher purity reagents and cleaner working practices to reduce blank contamination. However, these improvements could increase the costs of the analysis.

Table 3. Ten sigma LOQs calculated from 10 measurements of the blank.

Element	Integration Time (s)	LOD (µg/L)
9 Be	0.3	0.023
23 Na	0.1	2.2
24 Mg	0.1	0.54
27 Al	0.3	0.66
39 K	0.1	5.4
44 Ca	0.2	2.4
51 V	0.2	0.072
52 Cr	0.2	0.054
55 Mn	0.2	0.072
56 Fe	0.1	0.22
59 Co	0.2	0.004
60 Ni	0.2	0.071
63 Cu	0.2	0.017
66 Zn	0.2	0.095
75 As	0.3	0.015
78 Se	0.5	0.12
95 Mo	0.2	0.007
107 Ag	0.3	0.002
111 Cd	0.3	0.005
121 Sb	0.2	0.004
137 Ba	0.2	0.016
201 Hg	1.0	0.015
205 Tl	0.1	0.002
Pb‡	0.1	0.008
232 Th	0.1	0.004
238 U	0.1	0.002

‡ Reported as the sum of the lead isotopes 206+207+208

Analysis of water, soil, and sediment CRMs

The three CRMs were analyzed multiple times throughout the analytical sequence. To simulate a low concentration natural water sample, NIST 1643e was analyzed after a 10x dilution, while the soil and sediments were analyzed following 10x and 50x dilutions. The mean concentrations, percent relative standard deviations (%RSDs), and mean recoveries were calculated for each analyte, as shown in Table 4. The results for all elements across the CRM samples showed excellent agreement with the certified reference values. The only exception was Co in River Sediment A, which recovered outside the 100 ± 10% range. Because both the 10x and 50x dilutions were out of range, the results suggest potential contamination of the CRM.

Long-term stability

To demonstrate the method's stability, 141 samples and QC solutions were analyzed continuously for over two hours with the 9500 ICP-QQQ system's DCS operating in AHM.

Figure 3 shows that the internal standard (ISTD) recoveries remained within 80–110% throughout the run, demonstrating excellent stability. The graph also includes Total Matrix Solid (TMS) values for each sample, obtained using the IntelliQuant feature of the OpenLab ICP-MS software. IntelliQuant performs a full-mass scan in approximately two seconds, providing a rapid semiquantitative profile of the sample.[5]

Since TDS is estimated as twice the TMS value, several samples with a TDS content exceeding 1000 ppm were repeatedly introduced to the 9500 ICP-QQQ during the sequence. Despite the elevated TDS levels, ISTD signals remained stable, demonstrating the excellent robustness of the 9500 ICP-QQQ with UHMI under challenging matrix conditions.

Table 4. Mean measured values, mean recoveries, %RSDs for all certified elements in the three CRMs, n=10. Blank cells indicate the absence of a certified value.

Element	NIST 1643e (1/10)*			Soil B (1/10)*			Soil B (1/50)*			River Sediment A (1/10)*			River Sediment A (1/50)*		
	Mean Conc (ppb)	%RSD	Mean Recovery (%)	Mean Conc (ppb)	%RSD	Mean Recovery (%)	Mean Conc (ppb)	%RSD	Mean Recovery (%)	Mean Conc (ppb)	%RSD	Mean Recovery (%)	Mean Conc (ppb)	%RSD	Mean Recovery (%)
9 Be	1.42	4.2	102	0.01	44.4		0.00	78.1		0.01	56.1		0.01	43.6	
23 Na	2010	0.9	97	10100	0.9	101	2120	1.0	106	5240	0.8		1070	1.3	
24 Mg	783	0.8	97	8170	1.0	102	1670	0.8	105	7330	0.9	105	1490	1.4	106
27 Al	15.3	5.3	108	70900	0.7	101	14900	1.1	106	25800	0.5	103	5310	1.5	106
39 K	190	2.0	93	20200	0.8	96	4290	0.7	102	15000	0.6	100	3100	1.5	103
44 Ca	3060	0.8	95	12200	0.7	98	2560	0.9	102	29500	0.6	98	6170	1.7	103
51 V	3.62	1.2	96	78.4	0.7	98	16.3	0.8	102	24.8	0.7	99	5.09	1.7	102
52 Cr	2.03	2.9	99	39.1	0.7	98	8.32	1.0	104	30300	0.6	101	6270	1.4	104
55 Mn	4.00	1.2	103	9650	0.8	96	2100	1.3	105	783	0.8	98	165	1.4	103
56 Fe	10.3	4.8	105	32000	1.2	91	6680	1.0	95	110001	1.8	92	22500	1.9	94
59 Co	2.56	1.3	95	9.75	0.5		2.12	1.2		12.3	1.2	123	2.61	1.8	130
60 Ni	5.89	2.4	94	19.0	1.0	95	3.95	1.4	9	52.7	0.9	105	10.9	2.1	109
63 Cu	2.22	1.4	98	285	0.4	95	61.6	1.0	103	96.2	1.1	96	20.6	1.6	103
66 Zn	7.94	1.3	101	6830	0.6	98	1470	1.0	105	1480	0.9	99	309	1.5	103
75 As	5.86	1.0	97	573	0.6	95	121	0.9	101	58.7	1.0	98	12.2	1.8	102
78 Se	1.18	5.8	98	0.16	16.7		0.05	27.0		2.09	5.3	104	0.44	11.9	109
95 Mo	11.8	1.0	97	0.11	5.8		0.02	13.5		0.13	4.3		0.03	9.6	
107 Ag	0.10	3.7	95	0.01	8.9		0.00	27.7		0.06	5.0		0.01	7.8	
111 Cd	0.61	1.2	93	19.9	0.9	99	4.27	1.7	107	10.0	1.1	100	2.15	2.4	107
121 Sb	5.57	1.0	95	40.4	0.7		8.20	1.0		50.0	0.3	100	10.3	1.5	103
137 Ba	51.9	0.8	95	701	0.9	100	142	0.8	101	49.6	0.6	99	10.2	1.9	102
201 Hg	0.02	31.6		0.03	14.7		0.03	20.4		0.03	22.3		0.03	27.1	
205 Tl	0.72	0.9	96	0.05	4.1		0.01	7.9		0.99	1.0	99	0.21	2.8	103
Pb‡	1.83	0.6	93	5980	0.6	100	1240	0.7	103	708	0.3	101	144	1.4	103
232 Th	0.00	63.0		10.2	1.0	102	2.04	0.5	102	2.00	1.0	100	0.40	2.1	101
238 U	0.00	42.0		25.3	0.8	101	5.08	0.7	102	1.02	1.3	102	0.20	3.0	102

‡ Reported as the sum of the lead isotopes 206+207+208

**1/10: Concentration of 1/10 diluted solution; 1/50: Concentration of 1/50 diluted solution*

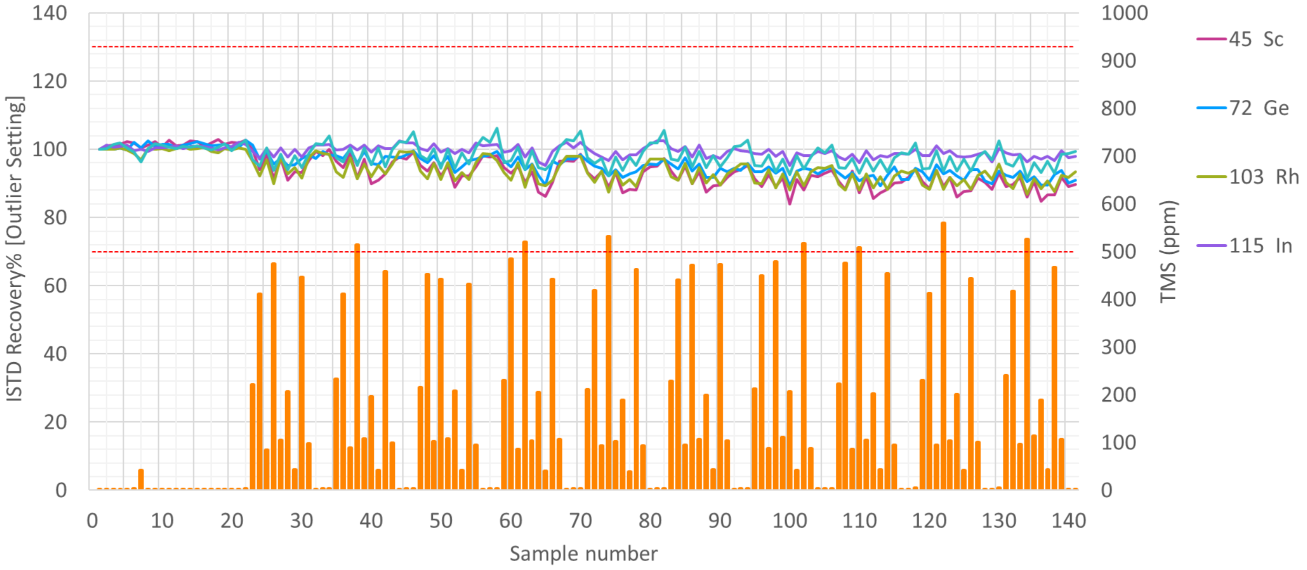

Figure 3. Internal standard recoveries normalized to the calibration blank for all samples, including samples with a high TMS content. No internal standard failures occurred over the 138-minute run.

Conclusion

The study showed that the Agilent 9500 ICP-QQQ with Dual-Cell System (DCS) operating in Advanced Helium Mode (AHM) and equipped with an integrated AVS MS discrete sampler delivered exceptional performance for routine environmental analyses.

Over 140 samples, including real-world environmental water samples, were analyzed in under 140 minutes, with no need for matrix matching the calibration standards or recalibration. The 9500 ICP-QQQ workflow delivered a 50% increase in sample throughput compared with a comparable single-quad ICP-MS method, substantially improving overall productivity.

The method's accuracy was confirmed through multiple measurements of water, soil, and sediment CRMs. Each sample was analyzed in under one minute, highlighting the method's capacity for high-throughput analysis while maintaining accuracy, as demonstrated by recoveries within 100 ± 10%. The long-term stability test results confirmed the robust performance of the 9500 ICP-QQQ with UHMI and AVS MS for the analysis of high-TDS samples, with no internal standard failures over more than two hours.

The results confirm fast analysis, effective interference removal, and robust, stable performance during the analysis of high-matrix samples over long runs—all critical performance criteria for demanding environmental workflows.

In an increasingly competitive landscape, where environmental laboratories are under pressure to improve productivity without compromising data quality, the 9500 ICP-QQQ controlled by Agilent OpenLab ICP-MS software operating in a single gas cell mode offers a powerful solution. AHM streamlines analytical workflows, reduces instrument maintenance demands, and delivers robust, reproducible results, enhancing overall operational efficiency.

References

1. Maximizing productivity for high matrix sample analysis using Agilent 7900 ICP-MS with ISIS 3 Discrete Sampling system, Agilent publication, 5991-5208EN
2. Simple Reliable Analysis of High matrix Samples according to US EPA 6020A ICP-MS, Agilent publication, 5990-5514EN
3. Zou, A.; Yamanaka, M. Intelligent Analysis of Wastewaters using an Agilent ICP-MS with Integrated Autodilutor, Agilent publication, 5994-7113EN
4. Dual-Cell System (DCS) and Advanced Helium Mode (AHM), Agilent publication, 5994-8985EN
5. Agilent ICP-MS IntelliQuant Software, Agilent publication, 5994-1677EN

Products used in this application

Agilent products

Product Type	Description	Part Number
Sample Introduction System	Quartz sample introduction system for 9500 ICP-MS	M5150-67107
	MicroMist nebulizer for 9500 ICP-MS	M5150-67024
Interface	ICP-MS sampler cone for 9500 ICP-MS, Ni tip with Cu base	M5150-67000
	Nickel skimmer cone for 9500 ICP-MS with u-lens	M5150-67005
	Extraction-Omega lens assembly, u-lens, stainless steel base	M5150-67022
Tubing Kits	Easy-fit peristaltic-pump tubing, PVC, white/white, 1.02 mm id, for sample	5005-0020
	Easy-fit peristaltic-pump tubing, PVC, blue/orange, 0.25 mm id, for internal standard	5005-0021
	Easy-fit peristaltic-pump tubing, beige thermoplastic, yellow/blue, 1.52 mm id, for drain	5005-0022
	Sample loop for ADS 2/AVS MS, 0.75 mL 1.00 mm id	5005-0422
	AVS MS preconfigured tubing kit for 9500 ICP-MS	M5171-67001
Bottle Kits	Diluent/carrier 6 L bottle kit, includes a 6 L can, GL45 StaySafe cap, fittings, and venting valve	5005-0435
	Diluent 2 L PFA bottle kit for ICP-MS, includes 2 L PFA bottle, GL45 StaySafe cap, fittings, and venting valve	5005-0436
	Waste container kit, includes a 10 L waste can, S60 StaySafe cap, fittings, and acid vapor filter	5005-0437
Chemical Standards	Initial calibration verification standard	5183-4682
	Environmental calibration standard	5183-4688

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