Application Note

Environmental

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Analysis of Soil and Sediments by ICP-MS with Advanced Sample Introduction Tools

Simplified sample preparation enabled by the Agilent Mira Mist Nebulizer and an In-Line Particle Filter

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Introduction

ICP-MS is widely employed for monitoring inorganic elements in soil and sediments, with established methods such as US EPA 6020B providing a validated framework for the accurate, multi-element analysis of environmental samples.[1] In a separate study, we demonstrated the outstanding performance capabilities of the Agilent 9500 Triple Quadrupole ICP-MS (ICP-QQQ) in compliance with EPA 6020B requirements.[2] The instrument, which includes the Ultra High Matrix Introduction (UHMI) system for enhanced plasma stability and robustness, employed Advanced Helium Mode (AHM) and Air cell mode to achieve lower limits of detection (LODs) compared to conventional cell gas approaches.[3,4]

The two collision/reaction cell (CRC) modes of operation were enabled by the Agilent Dual-Cell System (DCS) —a unique CRC that provides the following performance advantages when operated in AHM and Air cell mode:

- AHM
  - This innovative He-based gas collision-cell mode significantly enhances sensitivity for low-mass analytes while providing robust interference removal across the entire mass range.
  - This single gas mode eliminates the need for multiple modes, replacing no gas, conventional He, and high energy (HE) He mode.
- Air cell mode
  - Using oxygen (O_2) from air and an integrated air filter for the 9500 ICP-QQQ, Air cell mode leverages reactions between O_2 and analytes to improve detection limits (DLs) for oxygen-reactive species.
  - Air cell mode effectively eliminates interferences, including M^{++} that are challenging to resolve using He-based gas methods.[5]
  - With an unlimited supply of O_2 in air, Air cell mode eliminates the need for O_2 gas cylinders, reducing operating costs and simplifying workflows.

Environmental laboratories typically analyze soils using acid-digested solutions prepared from the original samples. Although the major component of soil is silicon dioxide (SiO_2) and complete decomposition normally requires the use of hydrofluoric acid (HF), the acid is often avoided in environmental labs due to safety concerns. However, when digestion is performed using only nitric acid (HNO_3) and hydrochloric acid (HCl), particulate residues may remain. These particles can clog components of the ICP-MS sample introduction system—particularly the nebulizer and discrete sampling system—making filtration of the digested samples necessary.

To address the challenges caused by particles and high matrix samples that are prone to salt precipitation, optional and advanced sample introduction components have been developed for ICP-MS. These components include an in-line filter that can be installed directly on the sample flow path and a nebulizer, both designed to resist clogging. The use of these accessories allows laboratories to reduce or eliminate the sample filtration step, improving workflow efficiency and avoiding downtime caused by blockages.

The in-line particle filter, which includes a 120 micron filter, is easily inserted in the sample introduction tubing between the autosampler probe and the sample introduction system. At approximately 8 cm in length, the filter does not noticeably increase the sample line volume, sample uptake, or stabilization time.

Of all the ICP-MS nebulizers, the Agilent Mira Mist nebulizer offers the best resistance to concentrated acids and organic solvents. Its parallel path design ensures good tolerance to high levels of particulates and total dissolved solids (TDS), providing excellent resistance to clogging.

In this study, the 9500 ICP-QQQ equipped with an in-line particle filter and Mira Mist nebulizer was used to determine trace and major elements in five environmental certified reference materials (CRMs) and a real sediment sample. The performance of the method was evaluated according to criteria set out in EPA Method 6020B.

Experimental

Instrumentation

All measurements were performed using an Agilent 9500 ICP-QQQ that includes the following standard components: nickel (Ni) interface cones and newly designed DCS cell. The design features and functionality of the 9500 and DCS are explained elsewhere.[3,4] The 9500 was also equipped with the optional Agilent Advanced Valve System (AVS MS) discrete sampling system for improved productivity. The AVS MS system's high-speed piston pump works seamlessly with the Agilent SPS 4 and SPS 6 autosamplers for sample delivery, ensuring the high sample throughput necessary to meet the operational needs of most environmental testing laboratories.

To simplify sample preparation, the 9500 was fitted with an in-line particle filter (Figure 1) and Mira Mist nebulizer (Figure 2). The in-line particle filter removes particles larger than 120 µm, and the Mira Mist nebulizer has a larger inner diameter (id) than the standard MicroMist nebulizer. These advanced tools free operators from having to filter the samples before analysis.

Method parameters (recommended list of elements, preferred isotopes, integration times, internal standards, etc.) were loaded from the "EPA 6020B" preset method in the Agilent OpenLab ICP-MS software, allowing the analyst to create a new batch method quickly.

To optimize signal stability with the Mira Mist nebulizer, the nebulizer pump speed was adjusted and a makeup gas was introduced (Table 1). In the 9500 ICP-QQQ, both the makeup gas and dilution gas can be added, enabling finer control of the plasma gas flow rates to optimize signal %RSD within the target sensitivity zone. The lens parameters were set automatically using the Autotune function in the software. The in-line filter's internal volume is sufficiently small that its presence does not influence AVS MS sample introduction timing or method parameters. As a result, the same settings were used with and without the filter installed (Table 2).

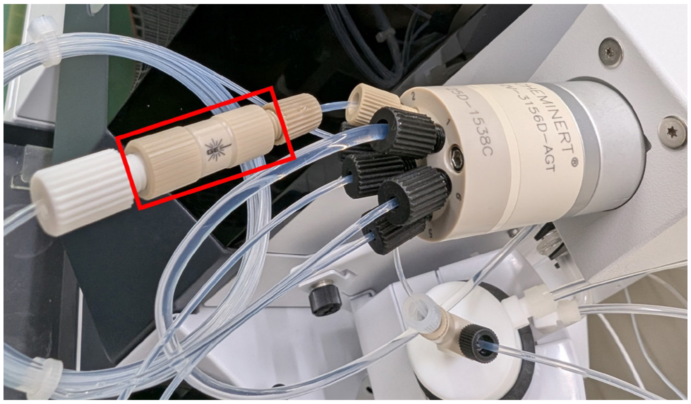

Figure 1. In-line particle filter.

Figure 2. Mira Mist nebulizer.

Table 1. Agilent 9500 ICP-QQQ operating parameters.

	Advanced He Mode (AHM)	Air Cell Mode
RF Power (W)	1600	
Sampling Depth (mm)	12	
Nebulizer Gas (L/min)	0.93	
Nebulizer Pump (rpm)	7.0	
Makeup Gas (L/min)	0.10	
Dilution Gas (L/min)	0.13	
Extract 1 (V)	−3.8	
Extract 2 (V)	−250	
Omega Bias	−200	
Omega Lens	−5.0	
Cell Gas Flow Rate (mL/min)	14	0.4
Front Energy Discrimination (V)	10	−1

The shaded parameters were defined automatically by selecting the EPA 6020 preset method or following autotuning.

Table 2. Agilent AVS MS operating parameters.

	Time (s)	Speed (%)
Sample Load	11	50
Stabilization	18	5
Probe Rinse	15	5
Rinse 1	6	80
Rinse 2	35	5
Optional Loop Probe Wash	10	50
Optional Loop Wash	1	50
Lower Sample Consumption Option	3	–
Sample-to-Sample Analysis Time	122	–

Chemicals and reagents

A solution containing high purity (EL grade) 2% HNO_3 and 1% HCl from Kanto Chemical Co. Inc., Japan was used as the blank, diluent, and rinse solution. For most elements, the calibration standards, spikes, and continuous calibration verification (CCV) solutions were prepared from the Agilent multi-element Environmental Calibration Standard.
The initial calibration verification (ICV) solution was prepared from an Agilent mixed multi-element ICV standard. Calibration standards for Li, Al, Mn, Zn, Sr, and Hg were prepared using 1000 mg/L single-element stocks (Kanto Chemical Co., Inc.). A mixed internal standard (ISTD) solution comprising Sc, Ge, Rh, In, Tb, and Lu was prepared from 1000 mg/L single-element stocks (Kanto Chemical Co., Inc.).

Standard and sample preparation

All standards and samples were prepared in an acid matrix of 2% HNO_3 and 1% HCl. HCl is routinely added to samples for ICP-MS analysis to ensure the stability of elements such as antimony (Sb) and mercury (Hg). The 9500 ICP-QQQ in AHM removes any Cl-based interferences that would be formed from the added chloride matrix.

Six-point calibrations including the calibration blank were prepared at the concentration ranges given in Figure 3. The term "mineral elements" refers to Na, Mg, Al, K, Ca, Mn, Fe, and Zn.

For in-run quality control (QC), the ICV and CCV standards were used.

The CRMs included Trace Elements in Water (NIST 1643f), River Sediment A, River Sediment B, Soil A, and Soil B (High Purity Standards). The CRMs were all supplied as predigested solutions. NIST 1643f was introduced with no dilution, and the other CRMs were diluted 10x using the 2% HNO_3 and 1% HCl acid matrix. The ISTD solution containing 1 mg/L Sc, Ge, Rh, In, Tb, and Lu was automatically added online via the dedicated seventh port of the AVS MS valve. The flow rate of the ISTD solution was about 1/15 of the sample flow rate, achieved using pump tubing with a smaller id. The analytical sequence is shown in Figure 3.

An estuarine sediment sample was digested using a Mars 6 Microwave Digestion System (CEM Corporation, Japan) according to the method outlined in Figure 4. First, approximately 0.25 g of sample was digested in 9 mL HNO_3 and 3 mL HCl. The temperature was ramped to 180 °C over 25 min and held for 5 min. Second, the temperature was ramped to 50 °C over 25 min and held for 15 min to dissolve any precipitated fluorides.

Following the two-step digestion, the solutions were made up to 200 mL with ultrapure water (UPW). Final acid concentrations were 4.5% HNO_3 and 1.5% HCl. The concentration of TDS in the final digests was approximately 200 mg/L. Since this procedure does not use HF, it is considered an extraction rather than a complete digestion.

Samples were spiked either before or after the digestion procedure. Each spiked element had a final concentration of 50 µg/L in the 50 mL digests that were analyzed. The sample tubes were loaded on the SPS 4 autosampler ready for analysis.

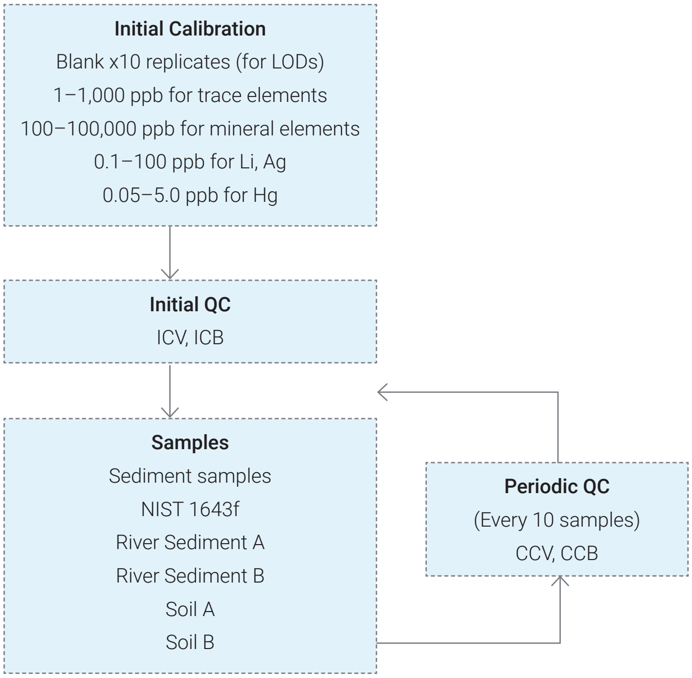

Figure 3. Analytical sequence of calibrants, QC solutions, and samples. The sample block was analyzed repeatedly by the Agilent 9500 ICP-QQQ with automatic insertion of the periodic QC block after every 10 samples.

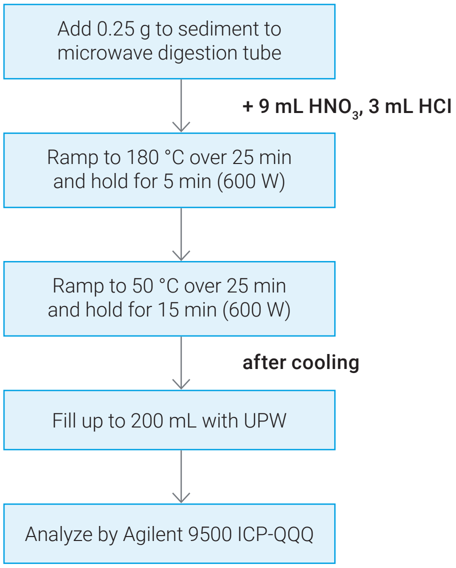

Figure 4. Microwave digestion method used for preparation of the estuarine sediment sample.

Results and discussion

Limits of Detection

All 30 analytes were measured by the 9500 ICP-QQQ in AHM, and selected elements (K, Ca, As, and Se) were also measured in Air cell mode using the acquisition parameters listed in Table 1. Limits of Detection (LODs) were calculated from three times the standard deviation of 10 measurements of the reagent blank solution. While the standard MicroMist nebulizer provides slightly better RSD performance than the Mira Mist due to a small id, the Mira Mist delivers similar sensitivity and comparable LODs.[2]

As shown in Table 3, LODs for the trace analytes were mostly in the low ng/L (ppt) level, confirming the high sensitivity of the 9500 ICP-QQQ for the trace analysis of analytes in complex environmental samples.

Table 3. Agilent 9500 ICP-QQQ operating parameters.

Analyte	DCS Cell Mode	Integration Time (s)	LOD (µg/L)
7 Li	AHM	0.3	0.010
9 Be	AHM	0.3	0.002
23 Na	AHM	0.1	0.313
24 Mg	AHM	0.1	0.831
27 Al	AHM	0.3	0.080
39 -> 39 K	Air	0.1	1.02
44 -> 60 Ca	Air	0.1	0.706
51 V	AHM	0.3	0.003
52 Cr	AHM	0.3	0.007
55 Mn	AHM	0.1	0.135
56 Fe	AHM	0.1	0.021
59 Co	AHM	0.3	0.007
60 Ni	AHM	0.3	0.012
63 Cu	AHM	0.3	0.006
66 Zn	AHM	0.1	0.039
75 As	AHM	1	0.002
75 -> 91 As	Air	1	0.003
78 Se	AHM	1	0.010
80 -> 96 Se	Air	1	0.013
88 Sr	AHM	0.3	0.002
95 Mo	AHM	0.3	0.002
107 Ag	AHM	0.3	0.001
111 Cd	AHM	0.3	0.0004
121 Sb	AHM	0.3	0.001
137 Ba	AHM	0.3	0.001
201 Hg	AHM	1	0.002
205 Tl	AHM	0.1	0.0004
Pb*	AHM	0.3 (0.1 x 3)	0.001
232 Th	AHM	0.1	0.0002
238 U	AHM	0.1	0.0002

**Pb = ^{206}Pb + ^{207}Pb + ^{208}Pb*

Washout test

Washout performance was evaluated using the in-line filter configuration, focusing on elements known to exhibit memory effects—As, Se, Sb, and Hg (Figure 5). For As, Se, and Hg, the washout efficiency was equivalent with and without the in-line filter across the first, second, and third rinse cycles.

Sb showed a small difference during the first rinse, with washout efficiencies of 0.005% (with filter) and 0.002% (without filter). Even in a scenario where 100 µg/L Sb is analyzed, this corresponds to only 0.005 µg/L of residual memory—well below levels that would impact typical soil measurements. By the second rinse, values converged to near identical levels. These results indicate that the presence of the in-line filter does not have a significant impact on Sb and other washout behavior for soil applications.

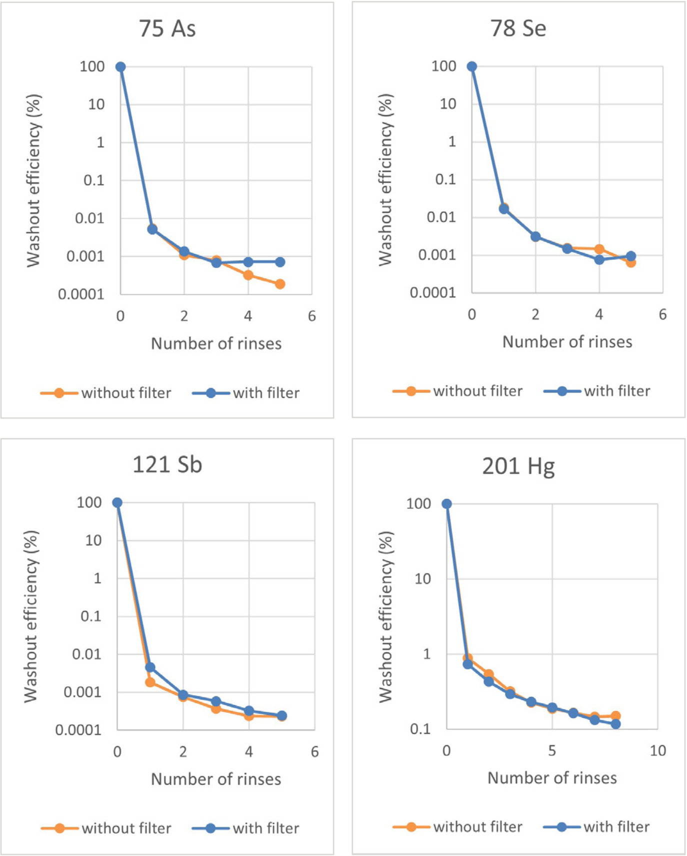

Figure 5. Washout efficiency of the Agilent 9500 ICP-QQQ with and without the in-line filter.

Long-term stability

To demonstrate the robustness of the 9500 ICP-QQQ with the Mira Mist nebulizer and in-line filter, 180 samples and 38 QC samples were analyzed for approximately eight hours.

Figure 6 shows the percent recovery of the raw ISTD responses for the entire eight-hour sequence normalized to the calibration blanks. All samples passed the >70% test, with no mass-dependent drift. The recovery data confirms that the 9500's robust, matrix tolerant plasma minimized matrix deposition over eight hours. The ISTD plot also shows that the 9500 provides excellent control of signal suppression, with ISTD signals being consistent for elements covering a range of masses and ionization potentials.

The results demonstrate the long-term robustness and high matrix tolerance of the 9500 ICP-QQQ with Mira Mist nebulizer.

Accurate analysis of CRMs and real sediment sample

The five CRMs were each analyzed nine times during the sequence, and the mean concentration and recovery were calculated for each analyte (Tables 4 and 5). Most of the certified elements, which included major elements at 100s of mg/L (ppm) and trace elements at levels as low as 10 µg/L (ppb), recovered within ±10%. Not all the analytes have certified values in every reference material, so blank cells in the tables indicate the absence of a certified value. Regarding Co in River Sediment A, a spike recovery test showed a good recovery (94% of 100 µg/L spike, data not shown), suggesting that the CRM may be contaminated.

Matrix Spike (MS) results for Soil A are given in Table 5. Recoveries are within ±10% of the spike level, demonstrating the method's accuracy.

The real sediment sample was digested in three vessels which were analyzed nine times each during the sequence. The mean concentration and recoveries were calculated for each analyte. All spike recoveries were within ±10%, as shown in Table 6.

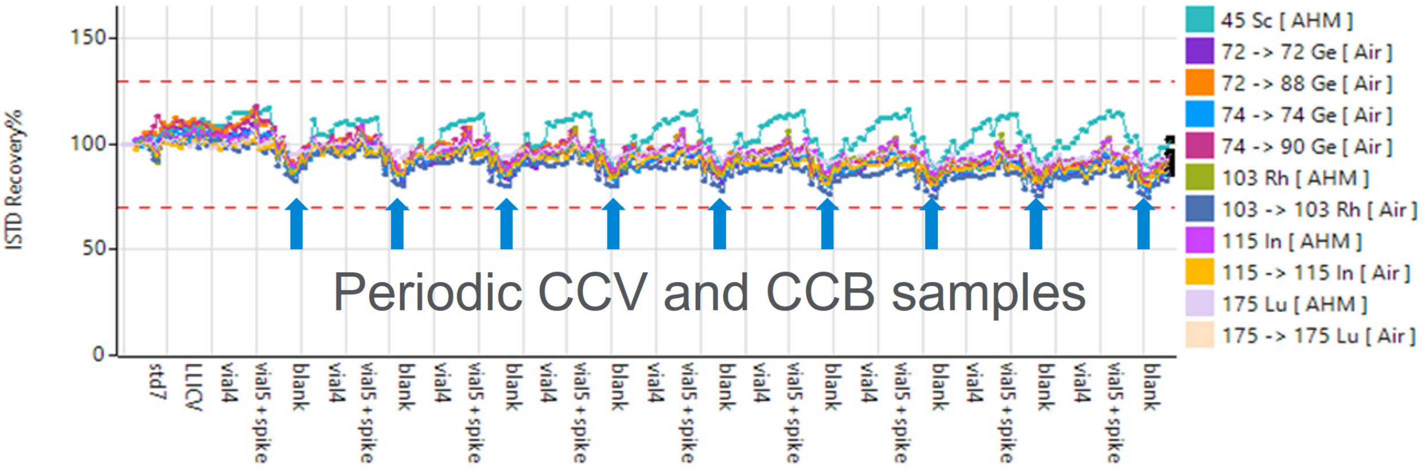

Figure 6. ISTD stability during the analysis of 180 samples over approximately eight hours using the Agilent 9500 ICP-QQQ. ISTD recoveries normalized to the calibration blank for all samples. Due to limited space, not all sample names are shown.

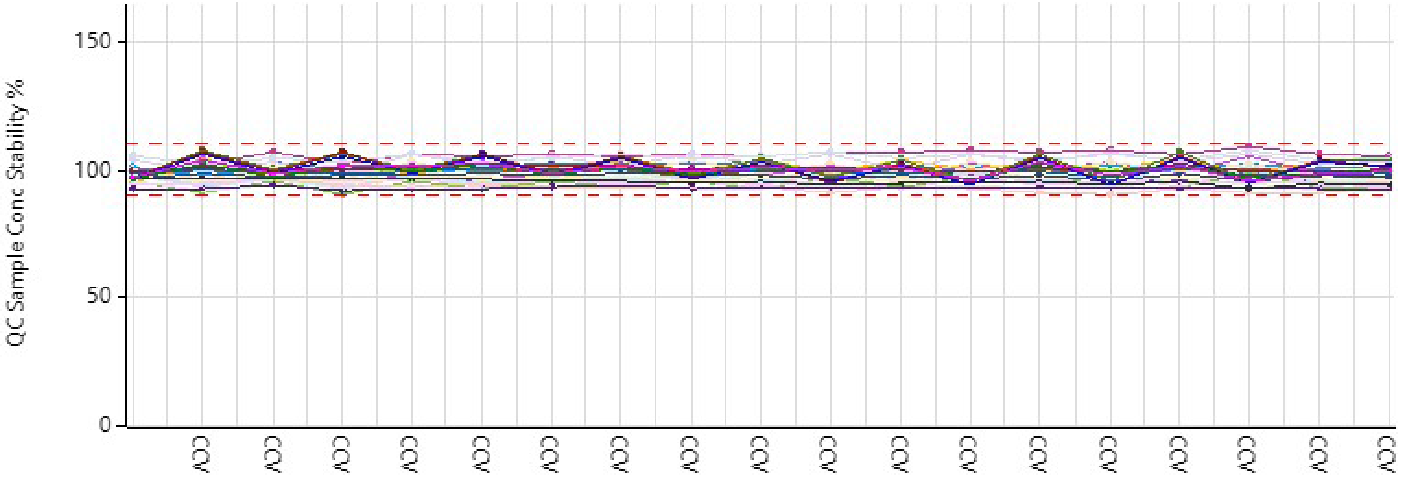

Figure 7. CCV stability of 18 CCVs measured using the Agilent 9500 ICP-QQQ.

Table 4. Recovery of certified values for CRMs, *n*=9.

Analyte	DCS Cell Mode	NIST 1643f			HPS River Sediment A			HPS River Sediment B		
		Mean Conc. (µg/L)	Expected Conc. (µg/L)	Recovery (%)	Mean Conc. (µg/L)	Expected Conc. (µg/L)	Recovery (%)	Mean Conc. (µg/L)	Expected Conc. (µg/L)	Recovery (%)
7 Li	AHM	15.0 ± 0.313	16.6	90	0.659 ± 0.288	-	-	0.941 ± 0.343	-	-
9 Be	AHM	12.7 ± 0.251	13.7	93	< LOD	-	-	< LOD	-	-
23 Na	AHM	18700 ± 174	18800	99	49,100 ± 482	50,000	98	52,400 ± 714	50,000	105
24 Mg	AHM	7360 ± 81.3	7450	99	68,700 ± 716	70,000	98	126,000 ± 1,690	120,000	105
27 Al	AHM	134 ± 1.95	134	100	241,000 ± 2,750	250,000	96	613,000 ± 11,400	600,000	102
39 -> 39 K	Air	1950 ± 22.7	1930	101	147,000 ± 3,450	150,000	98	205,000 ± 2,230	200,000	102
44 -> 60 Ca	Air	28600 ± 1480	29400	97	301,000 ± 4,780	300,000	100	319,000 ± 3,130	300,000	106
51 V	AHM	33.7 ± 0.489	36.1	94	234 ± 3.81	250	94	953 ± 14.6	1,000	95
52 Cr	AHM	17.3 ± 0.296	18.5	94	287,000 ± 3,980	300,000	96	14,300 ± 266	15,000	95
55 Mn	AHM	36.3 ± 0.492	37.1	98	8,060 ± 196	8,000	101	6,040 ± 143	6,000	101
56 Fe	AHM	90.4 ± 1.05	93.4	97	1,230,000 ±	1,200,000	103	397,000 ± 7,640	400,000	99
59 Co	AHM	23.9 ± 0.345	25.3	94	126 ± 1.49	100	126	154 ± 2.28	150	102
60 Ni	AHM	55.7 ± 0.752	59.8	93	538 ± 5.82	500	108	496 ± 6.78	500	99
63 Cu	AHM	20.5 ± 0.332	21.7	95	982 ± 10.1	1,000	98	976 ± 12.7	1,000	98
66 Zn	AHM	74.6 ± 0.671	74.4	100	15,000 ± 217	15,000	100	5,170 ± 78.3	5,000	103
75 As	AHM	52.6 ± 0.652	57.4	92	565 ± 5.73	600	94	191 ± 1.77	200	96
75 -> 91 As	Air	53.9 ± 0.889	57.4	94	586 ± 5.62	600	98	195 ± 1.72	200	98
78 Se	AHM	10.9 ± 0.172	11.7	93	19.4 ± 0.362	20	97	9.96 ± 0.340	10	100
80 -> 96 Se	Air	10.7 ± 0.345	11.7	91	18.3 ± 0.760	20	91	9.40 ± 0.452	10	94
88 Sr	AHM	314 ± 3.18	314	100	2.21 ± 0.027	-	-	1.73 ± 0.034	-	-
95 Mo	AHM	105 ± 0.76	115	91	1.26 ± 0.025	-	-	0.957 ± 0.034	-	-
107 Ag	AHM	0.966 ± 0.035	0.97	100	0.659 ± 0.019	-	-	0.509 ± 0.011	-	-
111 Cd	AHM	5.64 ± 0.039	5.89	96	98.3 ± 0.888	100	98	30.3 ± 0.440	30	101
121 Sb	AHM	51.3 ± 0.635	55.5	92	474 ± 3.82	-	-	41.2 ± 0.328	40	103
137 Ba	AHM	476 ± 7.25	518	92	466 ± 3.93	500	93	3,820 ± 41.7	4,000	96
201 Hg	AHM	0.004 ± 0.001	-	-	0.050 ± 0.012	-	-	0.270 ± 0.016	-	-
205 Tl	AHM	6.62 ± 0.092	-	-	9.54 ± 0.215	10	95	10.0 ± 0.268	10	100
Pb*	AHM	17.7 ± 0.268	18.5	96	6,740 ± 95.3	7,000	96	2,030 ± 51.6	2,000	102
232 Th	AHM	0.003 ± 0.000	-	-	19.0 ± 0.334	20	95	99.0 ± 2.03	100	99
238 U	AHM	0.005 ± 0.000	-	-	9.73 ± 0.196	10	97	29.9 ± 0.719	30	100

**Pb = ^{206}Pb + ^{207}Pb + ^{208}Pb*

Table 5. Recovery of certified values for CRMs, *n*=9.

Analyte	DCS Cell Mode	1/10 HPS Soil A						1/10 HPS Soil B		
		Mean Conc. (µg/L)	Expected Conc. (µg/L)	Recovery (%)	Soil A Spiked Measured Conc (µg/L)	Spike Conc. (µg/L)	Spike Recovery (%)	Mean Conc. (µg/L)	Expected Conc. (µg/L)	Recovery (%)
7 Li	AHM	0.010 ± 0.011	-	-	10.2	10	102	0.077 ± 0.024	-	-
9 Be	AHM	<LOD	-	-	92.0	100	92	0.0127 ± 0.004	-	-
23 Na	AHM	7280 ± 121	7000	104	17200	10000	99	10,000 ± 171	10000	100
24 Mg	AHM	7290 ± 97.7	7000	104	17300	10000	100	8,110 ± 108	8000	101
27 Al	AHM	50900 ± 812	50000	102	60200	10000	92	68,900 ± 1,220	70000	98
39 -> 39 K	Air	20800 ± 118	20000	104	30400	10000	96	21,400 ± 136	21000	102
44 -> 60 Ca	Air	37600 ± 245	35000	108	47700	10000	101	13,500 ± 133	12500	108
51 V	AHM	9.4 ± 0.149	10	94	102	100	93	72.3 ± 0.970	80.0	90
52 Cr	AHM	0.204 ± 0.012	-	-	93.5	100	93	36.8 ± 0.479	40.0	92
55 Mn	AHM	10.7 ± 0.146	10	107	9980	10000	99	9,650 ± 180	10000	96
56 Fe	AHM	19600 ± 339	20000	98	28600	10000	90	33,500 ± 490	35000	96
59 Co	AHM	0.522 ± 0.007	-	-	93.7	100	93	9.75 ± 0.105	10.0	98
60 Ni	AHM	28.9 ± 0.184	30	96	119	100	90	18.4 ± 0.199	20.0	92
63 Cu	AHM	28.5 ± 0.21	30	95	121	100	93	279 ± 2.58	300	93
66 Zn	AHM	101 ± 1.04	100	101	10400	100	102	6,870 ± 49.6	7000	98
75 As	AHM	18.8 ± 0.094	20	94	112	100	93	546 ± 4.80	600	91
75 -> 91 As	Air	19.1 ± 0.247	20	96	114	100	94	558 ± 6.77	600	93
78 Se	AHM	1.27 ± 0.031	1	127	97.3	100	96	0.153 ± 0.016	-	-
80 -> 96 Se	Air	1.28 ± 0.031	1	128	96.2	100	95	0.169 ± 0.019	-	-
88 Sr	AHM	0.222 ± 0.004	-	-	10.0	10	98	0.164 ± 0.003	-	-
95 Mo	AHM	0.077 ± 0.002	-	-	94.1	100	94	0.125 ± 0.004	-	-
107 Ag	AHM	0.038 ± 0.001	-	-	98.8	100	99	0.021 ± 0.002	-	-
111 Cd	AHM	0.308 ± 0.006	0.3	103	98.7	100	98	19.8 ± 0.181	20	99
121 Sb	AHM	3.04 ± 0.034	3	101	102	100	99	38.8 ± 0.317	20	97
137 Ba	AHM	481 ± 4.86	500	96	574	100	93	668 ± 7.43	700	95
201 Hg	AHM	0.039 ± 0.003	-	-	0.542	0.5	101	0.00588 ± 0.001	-	-
205 Tl	AHM	0.002 ± 0.000	-	-	105	100	105	0.0628 ± 0.003	-	-
Pb*	AHM	40.8 ± 1.02	40	102	145	100	104	6,230 ± 111	6000	104
232 Th	AHM	9.81 ± 0.167	10	98	111	100	101	10.1 ± 0.126	10	101
238 U	AHM	1.05 ± 0.021	1	105	102	100	101	25.6 ± 0.332	25	102

**Pb = ^{206}Pb + ^{207}Pb + ^{208}Pb*

Table 6. Recovery of certified values for real sediment samples, (*n*=3 x 9 replicates). Concentration units µg/L.

Analyte	DCS Cell Mode	Measured Conc.	Spike Conc.	Measured Conc of Spiked Samples	Recovery (%)
7 Li	AHM	20.5 ± 1.03	-	20.3 ± 1.02	-
9 Be	AHM	0.688 ± 0.035	50	52.9 ± 0.685	104
23 Na	AHM	6760 ± 343	5000	12200 ± 374	109
24 Mg	AHM	5400 ± 268	5000	10800 ± 287	109
27 Al	AHM	18400 ± 818	50	18400 ± 939	**133
39 -> 39 K	Air	5540 ± 253	5000	10800 ± 216	105
44 -> 60 Ca	Air	6190 ± 290	5000	11600 ± 287	108
51 V	AHM	45.5 ± 1.93	50	98.6 ± 1.76	106
52 Cr	AHM	41.3 ± 1.9	50	95 ± 1.62	107
55 Mn	AHM	276 ± 13.4	50	334 ± 13.3	**115
56 Fe	AHM	27900 ± 1530	5000	33700 ± 1440	**117
59 Co	AHM	6.06 ± 0.323	50	59.6 ± 0.713	107
60 Ni	AHM	30.3 ± 1.76	50	83.6 ± 1.81	106
63 Cu	AHM	14.2 ± 0.86	50	67.6 ± 1.00	107
66 Zn	AHM	59.5 ± 3.19	50	112 ± 3.3	106
75 As	AHM	8.69 ± 0.45	50	58.5 ± 0.735	100
75 -> 91 As	Air	8.25 ± 0.426	50	57.2 ± 0.66	98
78 Se	AHM	0.828 ± 0.043	50	51.9 ± 0.685	102
80 -> 96 Se	Air	0.297 ± 0.011	50	48.7 ± 0.66	97
88 Sr	AHM	39.9 ± 1.69	-	39.9 ± 1.85	-
95 Mo	AHM	2.19 ± 0.143	50	53 ± 0.772	102
107 Ag	AHM	0.070 ± 0.005	50	53.7 ± 0.806	107
111 Cd	AHM	0.205 ± 0.013	50	52.6 ± 0.826	105
121 Sb	AHM	0.298 ± 0.03	50	51.7 ± 0.826	103
137 Ba	AHM	61.6 ± 4.03	50	112 ± 3.56	101
201 Hg	AHM	0.062 ± 0.004	-	0.056 ± 0.005	-
205 Tl	AHM	0.204 ± 0.005	50	52.7 ± 1.04	105
Pb*	AHM	12.6 ± 0.613	50	64.4 ± 1.07	103
232 Th	AHM	6.76 ± 0.422	50	57 ± 0.909	100
238 U	AHM	1.87 ± 0.098	50	55.2 ± 0.92	107

**Pb = ^{206}Pb + ^{207}Pb + ^{208}Pb*

***The sample concentration was much greater than the spike level, compromising the reliability of the recovery result.*

Conclusion

In this study, sediment samples were analyzed in accordance with EPA Method 6020B using the Agilent 9500 ICP-QQQ with UHMI and the optional AVS MS. The system was equipped with advanced sample introduction components, including the Mira Mist nebulizer and an in-line particle filter.

The combination of the Mira Mist nebulizer and in-line filter enabled the analysis of sediment digests without a dedicated filtration step, reducing sample preparation time and consumable costs. At the same time, the configuration minimized the risk of sample introduction blockages, supporting robust and reliable long-term ICP-QQQ operation for complex environmental matrices.

Overall, the 9500 ICP-QQQ with the Mira Mist nebulizer and the in-line particle filter achieved excellent analytical data:

- Low detection limits in the low ng/L (ppt) range for most analytes
- The same or similar washout efficiency of sticky elements as a standard sample introduction configuration
- The same sample introduction timing, regardless of whether the filter was installed or not
- Method robustness and reproducibility over more than eight hours of continuous measurements, as demonstrated by the recoveries of ISTDs and CCVs within the limits specified in EPA Method 6020B
- Recoveries of all certified elements in the five environmental CRMs, matrix spike sample, and spiked real sediment sample within ±10%, confirming the effective control of interferences using AHM and Air cell mode

The study has highlighted how the 9500 ICP-QQQ with the Mira Mist nebulizer and in-line particle filter enhances sample preparation efficiency and analytical performance for the analysis of complex sediment digests.

References

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2. Tetsuo, K. Fast and Reliable Analysis of Soil and Sediments using ICP-MS with an Innovative Cell, Agilent publication, 5994-9128EN
3. Dual-Cell System (DCS) and Advanced Helium Mode (AHM), Agilent publication, 5994-8985EN
4. Air Cell Mode of Agilent 9500 ICP-QQQ with Dual-Cell System, Agilent publication, 5994-8987EN
5. Sugiyama, N. Solving Doubly Charged Ion Interferences using an Agilent 8900 ICP-QQQ, Agilent publication, 5994-1155EN

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
	Mira Mist nebulizer (PEEK) for Agilent ICP-MS	G3161-80001
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, 1.50 mL, 1.00 mm id	5005-0425
	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

**Including Glass Expansion Guardian In-Line Particle Filter, part number 70-803-1108*

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