Application Note

Environmental

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Fast and Reliable Analysis of Soil and Sediments using ICP-MS with an Innovative Cell

High-throughput and low detection limits with the Agilent 9500 ICP-QQQ and a Dual-Cell System

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Introduction

Ever since ICP-MS instruments were first commercialized, their high sensitivity and multi-elemental capabilities have made them a suitable technique for quantifying trace elements in soil and sediment, particularly in support of environmental monitoring and regulatory compliance. To help laboratories transition to ICP-MS for these important environmental applications, the US Environmental Protection Agency (EPA) first introduced the performance-based test Method 6020 in 2007, and revised it as Method 6020B in 2014.[1] Method 6020B remains widely used worldwide for the quantitative measurement of a broad range of elements, including arsenic (As), cadmium (Cd), mercury (Hg), selenium (Se), and lead (Pb), in solid and aqueous wastes by ICP-MS.

Traditionally, single-quadrupole ICP-MS (SQ ICP-MS) instruments equipped with collision/reaction cells (CRCs) have been used to meet the requirements of Method 6020B for sample analysis. Laboratories have typically used a combination of acquisition strategies to control interferences, including no gas mode for interference-free analytes and low mass analytes, helium (He) collision mode for analytes affected by polyatomic ion interferences, and half-mass mode to correct doubly charged ion (M^{2+}) interferences. This multi-mode approach has enabled laboratories to meet Method 6020B performance and quality control (QC) criteria over many years.

Now, for the first time, the Agilent 9500 Triple Quadrupole ICP-MS featuring the Dual-Cell System (DCS) cell introduces two innovative cell gas modes, Advanced Helium Mode (AHM) and Air cell mode.[2,3] These modes simplify interference removal approaches, enhance analytical performance, and accelerate analysis times.

- AHM
  - Significantly enhances sensitivity for low-mass analytes while providing robust interference removal across the entire mass range.
  - Eliminates the need for multiple gas modes, replacing no gas, conventional He, and high energy (HE) He mode with a single gas mode.
- Air cell mode
  - Uses oxygen (O_2) from ambient air as a reactive cell gas to improve detection limits (DLs) for some analytes.
  - Effectively eliminates interferences, including M^{2+} that are challenging to resolve in He collision mode.
  - Eliminates the need for O_2 gas cylinders, reducing operating costs and simplifying workflows.

In this study, a range of environmentally certified reference materials (CRMs) was selected to represent sample types routinely analyzed in high-throughput environmental testing laboratories. As these laboratories prioritize productivity across all aspects of their operations to remain competitive, the 9500 ICP-QQQ configuration and analytical method were optimized to maximize sample throughput. The performance of the method was evaluated according to criteria set out in Method 6020B.

Experimental

Instrumentation

All measurements were performed using an Agilent 9500 ICP-QQQ that includes the following standard components: Ultra High Matrix Introduction (UHMI), nickel (Ni) interface cones, and a unique DCS cell. The design features and functionality of the 9500 and DCS are explained elsewhere.[2,3]

The 9500 was also equipped with the optional Agilent Advanced Valve System (AVS MS) discrete sampler for improved productivity. The AVS MS, which uses a high-speed piston pump, integrates seamlessly with the Agilent SPS 4 and SPS 6 autosamplers for sample delivery, enabling the high sample throughput required by most environmental testing laboratories. The UHMI guarantees high matrix tolerance of the 9500 using aerosol dilution. The AVS MS further improves robustness for large sample batches of high matrix samples by minimizing the time that the instrument is exposed to the sample during each measurement.

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 quickly create a new batch method. All the plasma parameters listed in Table 1 were automatically loaded by selecting UHMI-4.

Table 1. Agilent 9500 ICP-QQQ operating parameters.

	Advanced He Mode (AHM)	Air Cell Mode
Plasma Mode	UHMI-4	
RF Power (W)	1600	
Sampling Depth (mm)	12	
Nebulizer Gas (L/min)	0.94	
Dilution Gas (L/min)	0.16	
Extract 1 (V)	−11.2	
Extract 2 (V)	−300	
Omega Bias	−200	
Omega Lens	−5.0	
Cell Gas Flow Rate (mL/min)	14	0.4
Front Energy Discrimination (V)	10	−1

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) 1% nitric acid (HNO_3) and 0.5% hydrochloric acid (HCl) from Kanto Chemical Co., Inc., Japan, was used as the blank, diluent, and rinse solutions. For most elements, calibration standards, spikes, and continuous calibration verification (CCV) solutions were prepared from the Agilent multi-element Environmental Calibration Standard, and initial calibration verification (ICV) solutions were prepared from the Agilent mixed multi-element ICV standard. Calibration standards for Li, Al, Mn, Zn, Sr, and Hg were prepared using 1000 ppm single-element stocks (Kanto Chemical Co., Inc.). A mixed internal standard (ISTD) comprising Sc, Ge, Rh, In, Tb, and Lu was prepared from 1000 ppm single-element stocks (Kanto Chemical Co., Inc.).

Standard and sample preparation

All standards and samples were prepared in an acid matrix of 1% HNO_3 and 0.5% 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 collision-cell mode 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 1. The term "mineral elements" refers to Na, Mg, Al, K, Ca, Mn, Fe, and Zn.

For in-run 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 1% HNO_3 and 0.5% HCl acid matrix. The ISTD solution containing 1 ppm 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 internal diameter.

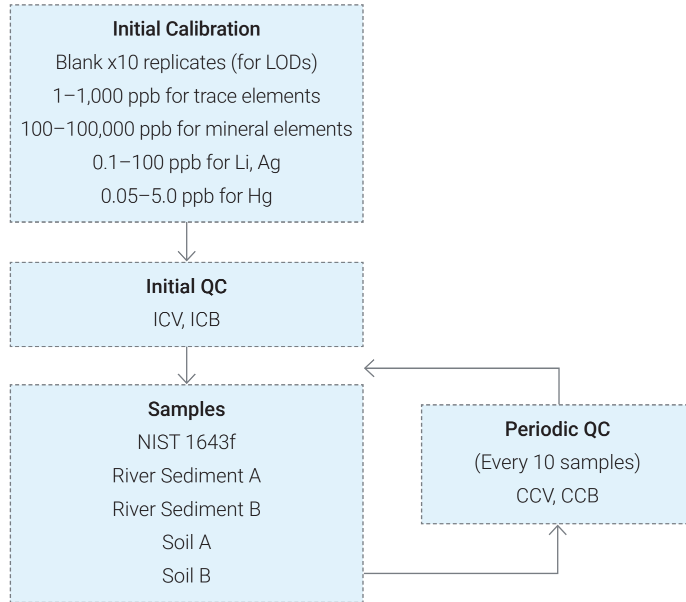

Figure 1. Analytical sequence of calibrants, QC solutions, and samples. The sample block was analyzed repeatedly by the Agilent 9500 ICP-QQQ with automatic insertion of the periodic QC block after every 10 samples.

Results and discussion

Limits of Detection (LODs)

All 28 analytes were measured by the 9500 ICP-QQQ in AHM, while selected elements (K, Ca, V, 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 (Table 3).

Operating the DCS of the 9500 ICP-QQQ in AHM achieved low-level LODs for low-mass elements such as Li and Be, which are traditionally analyzed in no gas mode. Additionally, AHM delivered an LOD for Se approximately one order of magnitude lower than that of the conventional He mode, even with a shorter integration time (reduced from 3 to 1 s).

Operating the 9500 ICP-QQQ DCS in Air cell mode reduced the K background, improving the LOD to 0.029 µg/L compared with 0.97 µg/L in AHM. For elements such as V, As, and Se, which exhibit high reactivity with O_2, Air cell mode utilizes oxygen-based mass-shift reactions (for example, $As^+ \rightarrow AsO^+$), resulting in enhanced sensitivity and reduced background compared to conventional He mode. Ca was measured in Air cell mode to demonstrate the M^{2+} removal capability of the DCS (removal of $^{88}Sr^{2+}$ interference on $^{44}Ca^+$).

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

Table 3. Integration times and LODs of analytes in the blank.

Analyte	DCS Cell Mode	Integration Time (s)	LOD (µg/L)
7 Li	AHM	0.3	0.0013
9 Be	AHM	0.3	0.0006
23 Na	AHM	0.1	0.096
24 Mg	AHM	0.1	0.020
27 Al	AHM	0.3	0.024
39 K	AHM	0.1	0.970
39 -> 39 K	Air	0.1	0.029
44 Ca	AHM	0.1	0.260
44 -> 60 Ca	Air	0.1	0.410
51 V	AHM	0.3	0.0014
51 -> 67 V	Air	0.3	0.0004
52 Cr	AHM	0.3	0.0035
55 Mn	AHM	0.1	0.0051
56 Fe	AHM	0.1	0.011
59 Co	AHM	0.3	0.0003
60 Ni	AHM	0.3	0.0025
63 Cu	AHM	0.3	0.0040
66 Zn	AHM	0.1	0.0044
75 As	AHM	1	0.0006
75 -> 91 As	Air	1	0.0004
78 Se	AHM	1	0.0057
78 -> 94 Se	Air	1	0.0068
88 Sr	AHM	0.3	0.0028
95 Mo	AHM	0.3	0.0009
107 Ag	AHM	0.3	0.0002
111 Cd	AHM	0.3	0.0003
121 Sb	AHM	0.3	0.0049
137 Ba	AHM	0.3	0.0004
201 Hg	AHM	1	0.0001
205 Tl	AHM	0.3	0.00004
Pb*	AHM	0.3	0.0005
232 Th	AHM	0.3	0.00004
238 U	AHM	0.3	0.00002

**Reported as the sum of the lead isotopes 206+207+208.*

ICV

The recommended control limits for the ICV in Method 6020B are ±10% of the actual value. All analytes were measured within these control limits (Table 4). The ICV solution contained 1000 µg/L Sr, which caused a Sr^{2+} interference on Ca, resulting in a high recovery in AHM. However, the Sr^{2+} interference was effectively resolved using a mass-shift reaction with O_2 in Air cell mode, resulting in a recovery of 93.9%.

Long-term stability

To demonstrate the robustness of the 9500 ICP-QQQ, 180 samples and 38 QC solutions were analyzed for approximately eight hours.

Figure 2 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 minimal drift. The recovery data confirms that the robust, matrix tolerant plasma of the 9500 ensured that minimal matrix deposition occurred 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 UHMI.

Table 4. ICV recoveries.

Analyte	DCS Cell Mode	ICV		
		Measured Conc (µg/L)	Actual Conc (µg/L)	Recovery (%)
7 Li	AHM	9.84	10	98
9 Be	AHM	102	100	102
23 Na	AHM	10,000	10,000	100
24 Mg	AHM	9,900	10,000	99
27 Al	AHM	9,520	10,000	95
39 K	AHM	10,200	10,000	102
39 -> 39 K	Air	10,100	10,000	101
44 Ca	AHM	12,600	10,000	126
44 -> 60 Ca	Air	9,390	10,000	94
51 V	AHM	96.7	100	97
51 -> 67 V	Air	98.7	100	99
52 Cr	AHM	97.8	100	98
55 Mn	AHM	9,570	10,000	96
56 Fe	AHM	9,940	10,000	99
59 Co	AHM	99.5	100	100
60 Ni	AHM	99.6	100	100
63 Cu	AHM	103	100	103
66 Zn	AHM	9,830	10,000	98
75 As	AHM	98.0	100	98
75 -> 91 As	Air	98.0	100	98
78 Se	AHM	98.7	100	99
78 -> 94 Se	Air	99.2	100	99
88 Sr	AHM	988	1,000	99
95 Mo	AHM	93.7	100	94
107 Ag	AHM	99.4	100	99
111 Cd	AHM	99.7	100	100
121 Sb	AHM	96.4	100	96
137 Ba	AHM	99.1	100	99
201 Hg	AHM	0.524	0.5	105
205 Tl	AHM	106	100	106
*Pb	AHM	104	100	104
232 Th	AHM	99.4	100	99
238 U	AHM	101	100	101

**Reported as the sum of the lead isotopes 206+207+208.*

Figure 2. 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.

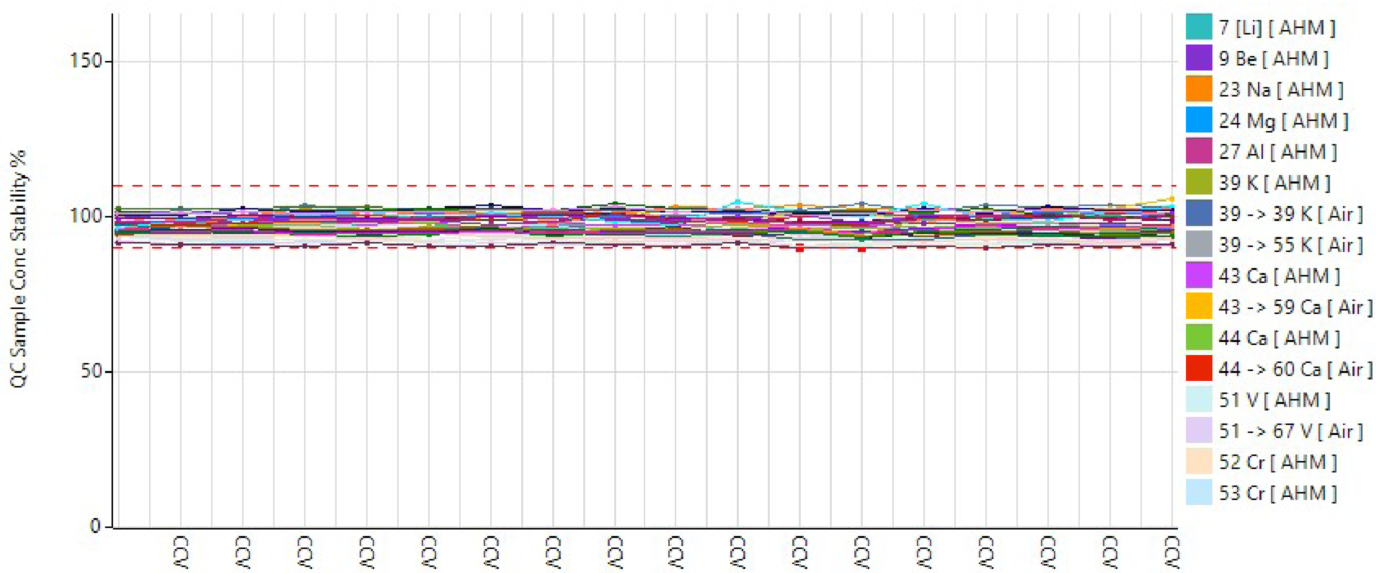

Figure 3. CCV stability of 18 CCVs measured using the Agilent 9500 ICP-MS.

Accurate analysis of CRMs

The five CRMs were each analyzed 18 times during the sequence, and the mean concentration and recovery were calculated for each analyte (Table 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), were recovered within ±10%. Not all analytes have certified values in every reference material, therefore, any blank cells in Table 5 indicate the absence of a certified value. Regarding Co in River Sediment A, the spike recovery test showed a good recovery (94.6% for recovery of a 20 µg/L spike), suggesting that the CRM may be contaminated.

Table 5. Recovery of certified values for CRMs. Concentration units µg/L.

n=18		NIST 1643f			HPS River Sediment A		
Analyte	**DCS Cell Mode**	**Mean Conc. (µg/L)**	**Expected Conc. (µg/L)**	**Recovery (%)**	**Mean Conc. (µg/L)**	**Expected Conc. (µg/L)**	**Recovery (%)**
7 Li	AHM	15.4 ± 0.178	16.6	93	0.267 ± 0.172	-	-
9 Be	AHM	12.9 ± 0.159	14	94	0.346 ± 0.235	-	-
23 Na	AHM	18,900 ± 198	18,830	100	51,500 ± 753	50,000	103
24 Mg	AHM	7,320 ± 70.9	7,454	98	70,600 ± 881	70,000	101
27 Al	AHM	121 ± 16.5	134	90	257,000 ± 2,700	250,000	103
39 K	AHM	1,840 ± 12.9	1,933	95	143,000 ± 1,100	150,000	96
39 -> 39 K	Air	2,000 ± 25.0	1,933	104	151,000 ± 2,770	150,000	101
44 Ca	AHM	29,200 ± 219	29,430	99	282,000 ± 3,030	300,000	94
44 -> 60 Ca	Air	29,100 ± 473	29,430	99	311,000 ± 6,680	300,000	104
51 V	AHM	33.7 ± 0.274	36	93	232 ± 2.04	250	93
51 -> 67 V	Air	33.7 ± 0.435	36	93	226 ± 3.86	250	90
52 Cr	AHM	17.4 ± 1.41	19	94	286,000 ± 2,440	300,000	95
55 Mn	AHM	34.3 ± 1.70	37	92	7,440 ± 72.7	8,000	93
56 Fe	AHM	80.3 ± 12.6	93	86	1,180,000 ± 10,900	1,200,000	98
59 Co	AHM	24.0 ± 0.259	25	95	118 ± 1.61	100	118
60 Ni	AHM	55.5 ± 0.667	60	93	497 ± 7.97	500	99
63 Cu	AHM	20.7 ± 0.336	22	95	964 ± 14.9	1,000	96
66 Zn	AHM	70.3 ± 1.22	74	94	14,100 ± 135	15,000	94
75 As	AHM	52.8 ± 0.417	57	92	536 ± 6.80	600	89
75 -> 91 As	Air	53.5 ± 0.625	57	93	543 ± 6.28	600	91
78 Se	AHM	10.8 ± 0.146	12	92	18.3 ± 0.568	20	92
78 -> 94 Se	Air	10.9 ± 0.182	12	93	18.3 ± 1.37	20	92
88 Sr	AHM	304 ± 1.94	314	97	2.77 ± 1.65	-	-
95 Mo	AHM	104 ± 0.532	115	90	1.87 ± 0.707	-	-
107 Ag	AHM	0.913 ± 0.062	1.0	94	0.668 ± 0.448	-	-
111 Cd	AHM	5.42 ± 0.081	6.0	92	95.0 ± 1.37	100	91
121 Sb	AHM	50.0 ± 0.475	55	90	448 ± 4.17	0	-
137 Ba	AHM	477 ± 3.14	518	92	457 ± 4.27	500	91
201 Hg	AHM	0.004 ± 0.001	-	-	0.0371 ± 0.0094	-	-
205 Tl	AHM	6.71 ± 0.079	-	-	9.51 ± 0.0720	10	95
*Pb	AHM	18.6 ± 0.327	18	101	6,950 ± 39.5	7,000	99
232 Th	AHM	0.006 ± 0.006	-	-	18.6 ± 0.113	20	93
238 U	AHM	0.010 ± 0.008	-	-	9.50 ± 0.0972	10	95

**Reported as the sum of the lead isotopes 206+207+208.*

Table 5 continued. Recovery of certified values for CRMs. Concentration units µg/L.

n=18		HPS River Sediment B			HPS Soil A			HPS Soil B		
Analyte	DCS Cell Mode	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	1.11 ± 0.196	-	-	0.608 ± 0.135	-	-	0.617 ± 0.146	-	-
9 Be	AHM	0.270 ± 0.172	-	-	0.304 ± 0.188	-	-	0.256 ± 0.161	-	-
23 Na	AHM	50,600 ± 510	5,000	101	70,500 ± 658	70,000	101	100,000 ± 855	100,000	100
24 Mg	AHM	121,000 ± 737	12,000	101	70,900 ± 508	70,000	101	81,200 ± 506	80,000	101
27 Al	AHM	633,000 ± 3,610	60,000	105	526,000 ± 3,360	500,000	105	731,000 ± 4,720	700,000	104
39 K	AHM	189,000 ± 1,170	20,000	94	186,000 ± 1,670	200,000	93	195,000 ± 2,010	210,000	93
39 -> 39 K	Air	199,000 ± 3,360	20,000	100	202,000 ± 1,810	200,000	101	207,000 ± 2,370	210,000	99
44 Ca	AHM	286,000 ± 2,730	30,000	95	336,000 ± 1,750	350,000	96	115,000 ± 471	125,000	92
44 -> 60 Ca	Air	298,000 ± 5,070	30,000	99	353,000 ± 8,550	350,000	101	128,000 ± 3,110	125,000	103
51 V	AHM	997 ± 6.85	100	100	91.9 ± 0.710	100	92	726 ± 5.37	800	91
51 -> 67 V	Air	966 ± 15.3	100	100	91.6 ± 1.06	100	92	720 ± 11.2	800	90
52 Cr	AHM	13,800 ± 170	1,500	92	1.04 ± 0.814	-	-	364 ± 6.78	400	91
55 Mn	AHM	5,470 ± 55.0	600	91	103 ± 42.3	100	103	90,100 ± 661	100,000	90
56 Fe	AHM	373,000 ± 2,960	40,000	93	187,000 ± 1,540	200,000	93	326,000 ± 2,990	350,000	93
59 Co	AHM	141 ± 2.22	15	94	2.54 ± 0.149	-	-	95.5 ± 1.35	100	95
60 Ni	AHM	480 ± 7.65	50	96	273 ± 3.17	300	91	180 ± 2.98	200	90
63 Cu	AHM	925 ± 15.4	100	92	284 ± 4.34	300	94	2,840 ± 31.8	3,000	94
66 Zn	AHM	4,530 ± 77.2	500	91	962 ± 38.3	1,000	96	63,800 ± 662	70,000	91
75 As	AHM	196 ± 2.56	20	98	182 ± 2.28	200	91	5,300 ± 75.2	6,000	88
75 -> 91 As	Air	194 ± 1.64	20	98	184 ± 1.42	200	92	5,420 ± 57.7	6,000	90
78 Se	AHM	10.1 ± 0.418	1.0	101	9.18 ± 0.355	10	92	1.49 ± 0.166	-	-
78 -> 94 Se	Air	9.50 ± 0.583	1.0	95	9.06 ± 0.557	10	91	1.50 ± 0.300	-	-
88 Sr	AHM	3.30 ± 0.117	-	-	3.37 ± 0.0832	-	-	1.35 ± 0.0555	-	-
95 Mo	AHM	4.10 ± 0.158	-	-	0.379 ± 0.100	-	-	1.10 ± 0.105	-	-
107 Ag	AHM	0.300 ± 0.115	-	-	0.551 ± 0.225	-	-	0.266 ± 0.156	-	-
111 Cd	AHM	28.4 ± 0.323	3.0	91	3.17 ± 0.0855	3	96	191 ± 2.61	200	92
121 Sb	AHM	36.7 ± 0.377	4.0	92	29.5 ± 0.209	30	98	362 ± 3.02	400	91
137 Ba	AHM	3,690 ± 27.8	400	92	4,630 ± 37.2	5,000	93	6,440 ± 52.1	7,000	92
201 Hg	AHM	0.033 ± 0.008	-	-	0.0280 ± 0.00870	-	-	0.0470 ± 0.0148	-	-
205 Tl	AHM	9.50 ± 0.103	1.0	95	0.0640 ± 0.0204	-	-	0.491 ± 0.0432	-	-
*Pb	AHM	1,930 ± 14.9	200	97	389 ± 3.65	400	97	58,600 ± 269	60,000	98
232 Th	AHM	92.8 ± 0.636	10	93	94.0 ± 0.666	100	94	95.3 ± 0.543	100	95
238 U	AHM	27.7 ± 0.231	3.0	92	9.43 ± 0.124	10	94	235 ± 3.33	250	94

**Reported as the sum of the lead isotopes 206+207+208.*

Matrix spike recoveries

Matrix Spike (MS), Matrix Spike Duplicate (MSD), and relative percent difference (RPD) results for 10x diluted Soil A are given in Table 6. Soil A contains Na, Mg, Al, K, Ca, Fe, and Ba at a higher concentration than the 100 µg/L spike amount, so no spike recoveries are given for these elements. However, the CRM recovery results for Soil A in Table 5 show that all these major elements can be analyzed with good accuracy. Recoveries for the other elements were generally within ±10% of the spike level, and the RPD between MS and MSD was less than 3.5%, demonstrating the method's accuracy.

Table 6. Matrix Spike (MS)/Matrix Spike Duplicate (MSD) spike concentration recoveries and relative percent difference (RPD) for 10x diluted Soil A CRM.

Analyte	DCS Mode	Soil A Measured Conc (µg/L)	Soil A MS Measured Conc (µg/L)	Soil A MSD Measured Conc (µg/L)	Soil A MSD Recovery (%)	RPD MS/MSD (%)
7 Li	AHM	0.061	9.650	9.710	96	0.6
9 Be	AHM	0.030	18.900	19.000	95	0.6
51 V	AHM	9.19	28.8	28.7	98	0.1
51 -> 67 V	Air	9.16	29.1	29.2	100	0.6
52 Cr	AHM	0.104	19.3	19.3	96	0.3
55 Mn	AHM	13.3	9270	9440	94	1.8
59 Co	AHM	0.254	19.4	19.3	95	0.9
60 Ni	AHM	27.3	47.8	47.5	101	0.6
63 Cu	AHM	28.4	49.7	49.6	106	0.3
66 Zn	AHM	96.2	9590	9560	95	0.3
75 As	AHM	18.2	38.6	38.3	100	0.9
75 -> 91 As	Air	18.4	38.7	38.5	100	0.4
78 Se	AHM	0.918	20.0	19.8	94	1.1
78 -> 94 Se	Air	0.906	20.1	19.5	93	3.1
88 Sr	AHM	0.337	10.1	9.92	96	2.2
95 Mo	AHM	0.038	18.7	18.6	93	0.8
107 Ag	AHM	0.055	19.1	19.0	95	0.3
111 Cd	AHM	0.289	19.5	19.5	96	0.1
121 Sb	AHM	2.95	22.3	22.4	97	0.6
201 Hg	AHM	0.003	0.483	0.481	96	0.3
205 Tl	AHM	0.006	19.8	19.8	99	0.1
*Pb	AHM	38.9	60.5	60.7	109	0.4
232 Th	AHM	9.40	29.4	29.9	103	1.7
238 U	AHM	0.943	20.3	20.4	97	0.5

**Reported as the sum of the lead isotopes 206+207+208.*

Measurement of Ca, As, and Se in an M^{2+} matrix

If present at high enough concentrations, elements with low second ionization energies can form M^{2+} ions that can interfere with analytes at half their mass (for example, $^{88}Sr^{2+}$ on $^{44}Ca^{+}$, $^{150}Nd^{2+}$ and $^{150}Sm^{2+}$ on $^{75}As^{+}$, $^{156}Gd^{2+}$ and $^{156}Dy^{2+}$ on $^{78}Se^{+}$). However, operating the DCS in Air cell mode minimizes the M^{2+} background on the analyte ions via an O_2 mass-shift reaction.[4] Table 7 shows the recovery results for Ca, As, and Se with no added M^{2+} interferents and with added interferents. High recoveries for all three analytes were observed when operating the DCS in AHM due to M^{2+} interferences, whereas excellent recoveries of 100 ± 10% were achieved in Air cell mode.

Table 7. Recoveries of Ca, As, and Se in 10x diluted Soil A with and without M^{2+} interferents

Analyte [DCS Mode]	Soil A Certified Value (µg/L)	Soil A (No Interferent) Recovery (%)	M^{2+} Interferent	M^{2+} Interferent Conc (µg/L)	Soil A (With Interferent) Recovery (%)
44 Ca [AHM]	35,000	94	Sr	10,000	198
44 -> 60 Ca [Air]		98			107
75 As [AHM]	20	95	Nd & Sm	400 (each)	132
75 -> 91 As [Air]		94			94
78 Se [AHM]	1.0	92	Gd & Dy	20 (each)	288
78 -> 94 Se [Air]		105			100

Conclusion

The study has shown that the Agilent 9500 ICP-QQQ, equipped with the unique Dual-Cell System (DCS) and the optional AVS MS discrete sampling system, is ideal for high-throughput, multi-element analysis of soil and sediment samples.

The use of the EPA 6020B Preset Method and selection of UHMI-4 plasma conditions in the Agilent OpenLab ICP-MS software enabled rapid setup of the instrument's operating parameters, ensuring consistent performance across operators.

The instrument's matrix-tolerant plasma (low CeO/Ce ratio), UHMI, and DCS effectively addressed the challenges of analyzing high-matrix samples, including signal drift, ion suppression, and spectral interferences, thereby delivering an optimized analytical workflow.

The method's productivity was enhanced by operating the DCS in Advanced Helium Mode (AHM) and Air cell mode and by using short integration times, saving 5 to 10 s per sample. AHM replaces conventional no gas, helium (He) collision, and high energy He (HEHe) in a single mode, while Air cell mode uses oxygen from the laboratory, alleviating the need for gas cylinders. The 9500 maintains exceptionally high sensitivity in AHM and Air cell mode, minimizing background signals.

Overall, the 9500 ICP-QQQ with DCS achieved excellent analytical data:

– Low detection limits in the low ng/L (ppt) range for most analytes.

– Removal of M^{2+} interferences on Ca, As, and Se generated by Sr and rare earth elements (REEs) in the soil samples using Air cell mode.

– Recoveries of all certified elements in the five environmental CRMs or matrix spike samples within 100 ± 10%, confirming the effective control of interferences using AHM and Air cell mode.

– 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 6020.

The 9500 ICP-QQQ with DCS and an integrated AVS MS enables high-throughput, routine analysis of complex environmental matrices. The EPA Method 6020-compliant method provided accurate data by effectively handling polyatomic ion and M^{2+} interferences using AHM and Air cell mode.

References

1. U.S. EPA. 2014. "Method 6020B (SW-846): Inductively Coupled Plasma-Mass Spectrometry," Revision 2. Washington, DC, https://www.epa.gov/esam/epa-method-6020b-sw-846-inductively-coupled-plasma-mass-spectrometry (accessed April 2026)
2. Dual-Cell System (DCS) and Advanced Helium Mode (AHM), Agilent publication, 5994-8985EN
3. Air Cell Mode of the Agilent 9500 ICP-QQQ with Dual-Cell System, Agilent publication, 5994-8987EN
4. 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
	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, 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

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