

Maximizing productivity for high matrix sample analysis using the Agilent 7900 ICP-MS with ISIS 3 discrete sampling system

EPA 6020A compliant analysis in less than 90 seconds per sample

Application note

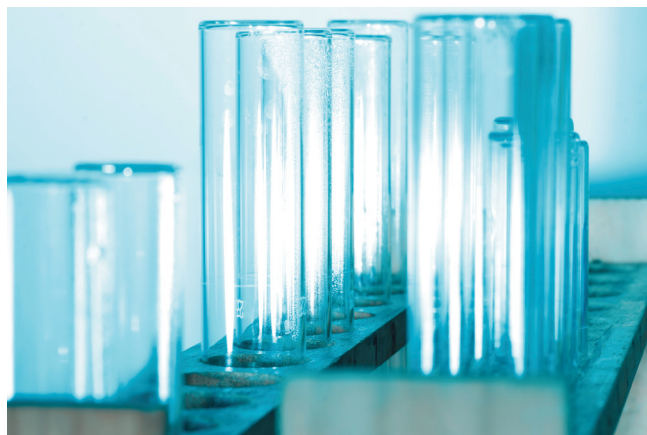
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Environmental



Introduction

With the development of Agilent's helium (He) mode Octopole Reaction System (ORS) collision cell and the introduction of High Matrix Introduction (HMI) aerosol dilution technology, robust and accurate ICP-MS analysis of complex high matrix environmental samples such as soils and sludges has become routine [1]. However, increased competition and financial pressure in contract environmental laboratories has led to a greater focus on the productivity of the analytical instruments and methods used. At the same time, it is essential that data quality and ease-of-use are not compromised as sample throughput is increased. To address these more demanding productivity requirements, Agilent has developed a new version of the Integrated Sample Introduction System, ISIS 3, which allows Agilent



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7900 ICP-MS users to perform high speed discrete sampling analysis while maintaining data quality in full compliance with US Environmental Protection Agency (EPA) requirements for data acquisition.

By combining the unique benefits of the Agilent 7900 ORS⁴ He mode collision cell, Ultra High Matrix Introduction (UHMI) and ISIS 3, a rare synergy has been achieved, resulting in a system that provides much faster analysis while also offering improved ease-of-use and superior matrix tolerance. This work demonstrates the benefits of combining He mode for interference removal, aerosol dilution using UHMI, and discrete sampling to achieve the best possible productivity and data quality.

Contract laboratory success factors

In a contract environmental laboratory, a successful analytical run is measured by:

- Accuracy (is the analytical result correct?)
- Precision (how reproducible is the result?)
- Regulatory compliance (will it hold up to legal scrutiny?)
- Expense (how much did it cost to obtain?)

Given the increasing focus on productivity as a key aspect of the “expense” of the analysis, it is apparent that performing the analysis more quickly while maintaining detection limits that are “fit for purpose” (in other words, meet the regulatory requirements) is a primary goal of routine contract laboratories. The fast cell gas switching capability of the ORS⁴ permits the

use of discrete sampling in combination with multiple gas modes for optimum measurement of both interfered and uninterfered (mostly low mass) elements. The high sensitivity of the 7900 ICP-MS also permits the use of very short integration times without degrading detection limits.

Experimental

Instrumentation

An Agilent 7900 ICP-MS fitted with standard nickel cones, glass concentric nebulizer and UHMI option was used for the analysis. UHMI maximizes the plasma robustness of the 7900 ICP-MS through a combination of aerosol dilution and automated plasma temperature optimization. Compared to the previous generation HMI used on the Agilent 7700 Series ICP-MS, UHMI with the 7900 ICP-MS provides a wider range of aerosol dilution (up to 100x) and improves sample washout performance.

The 7900 ICP-MS was fitted with the optional ISIS 3 discrete sampling (DS) accessory to achieve the required high sample throughput. ISIS-DS also contributes to further improvement in matrix tolerance for large sample batches by minimizing the period that the instrument is exposed to the sample during each measurement. The ISIS 3 switching valve is positioned close to the nebulizer to minimize the tubing length and optimize the discrete sampling analysis cycle. A seventh port on the ISIS 3 switching valve allows the internal standard (ISTD) solution to be added online to the carrier flow in the valve, providing efficient mixing of the solutions and rapid stabilization prior to nebulization.

Table 1. Agilent 7900 ICP-MS and ISIS 3 operating conditions

ICP-MS Parameters	No gas mode	He mode
RF power (W)	1600	
Carrier gas flow (L/min)	0.77	
Dilution gas flow (L/min)	0.28	
Lens tune	Autotune	Autotune
Cell gas flow (mL/min)	0.0	4.3
Energy discrimination (V)	5.0	
Number of elements	1 analyte, 1 ISTD	25 analytes, 6 ISTDs
Total acquisition time (3 reps) (seconds)	41	

ISIS-DS Parameters	1000	
Loop volume (µL)	1000	
	Time	Uptake Pump Speed
Sample load	12 sec	28%
Stabilize	7 sec	5%
Probe rinse	23 sec	5%
Probe rinse 1	6 sec	80%
Probe rinse 2	10 sec	5%
Optional loop probe wash	9 sec	50%
Optional loop wash	1 sec	5%

The fundamentals of ISIS-DS operation are described in Agilent Technologies publication 5990-3678EN [2]. A new feature of ISIS 3 is the addition of a computer controlled three-way valve for switching between the internal standard solution and the tuning solution. This valve permits fully automatic instrument optimization and sample analysis without the need to manually switch tubing. In this study, autotune with fixed plasma parameters was used for instrument optimization. Instrument conditions are shown in Table 1.

Figure 1 shows the basic operation of the ISIS-DS system: Step 1 – Load (upper left). The sample is rapidly drawn into the sample loop using the high speed ISIS 3 piston pump while the blank carrier with online internal standards is constantly pumped to the nebulizer. Step 2 – Inject (upper right). Rotation of the 7 port valve then diverts the carrier through the loop, pushing the sample ahead of it to the nebulizer. At the same time, the autosampler probe moves to the rinse port to rinse the sample introduction tubing before the next sample is loaded.

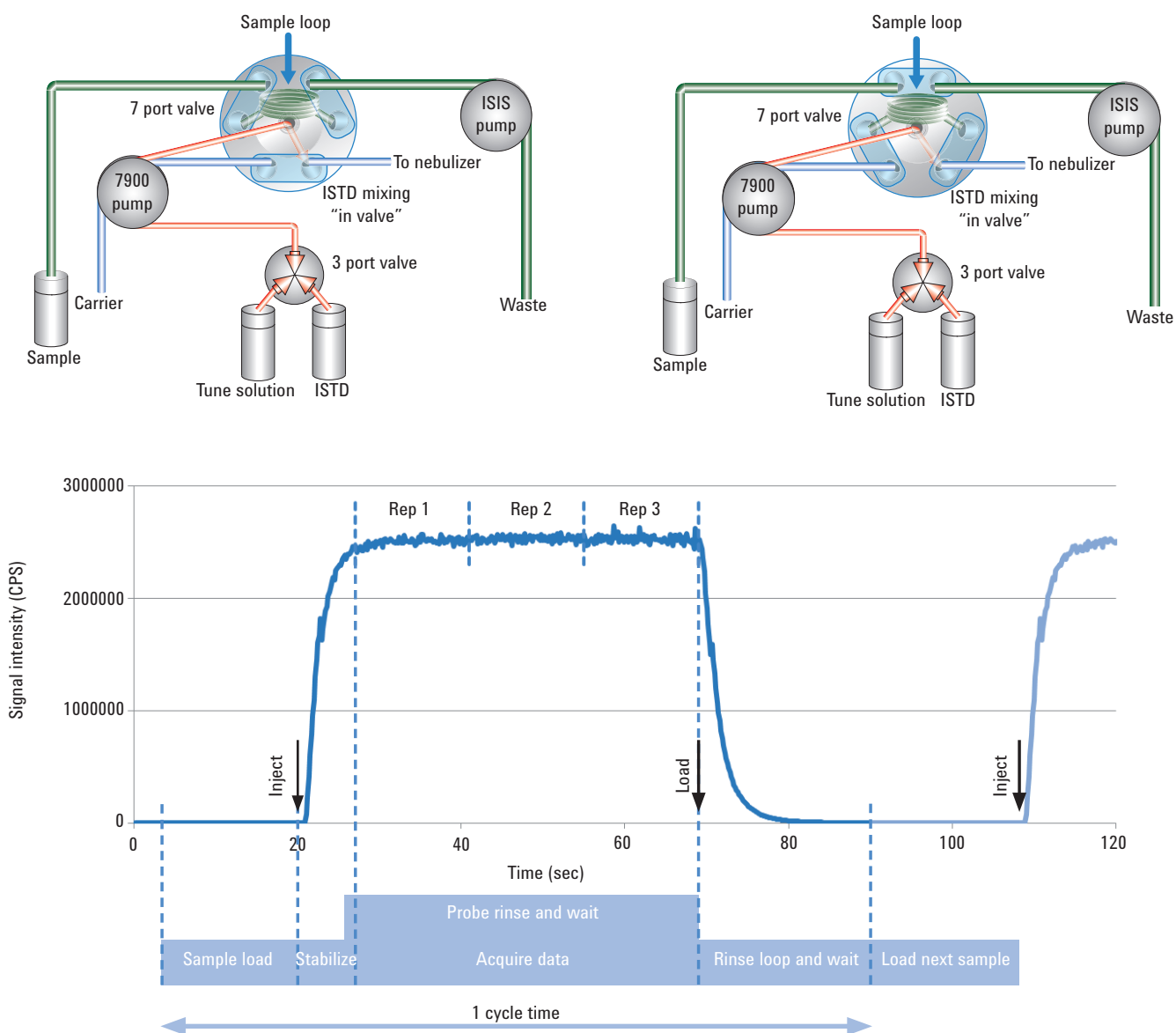


Figure 1. Overview of ISIS-DS operation. Valve in load position (upper left) and inject position (upper right). Actual analyte and internal standard signals during ISIS 3 operation are shown in lower plot with annotation.

The loop volume of 1000 μL provides a steady-state signal long enough for three replicate measurements in two different cell modes to be acquired. This highlights one of the important practical benefits of effective interference removal in He mode, where the same cell gas and conditions can be used to remove all polyatomic interferences so only a single cell mode is required for all interfered analytes. The Agilent ORS⁴ on the 7900 ICP-MS also includes a fast cell gas switching capability, so two cell modes (He mode and no gas mode) can be acquired in triplicate during each injection. In contrast, when discrete sampling is used on ICP-MS systems that utilize reactive cell gases, significantly longer time is required to switch between the different reaction gases and cell settings required

for multi-element analysis, so cell settings must be compromised, the sample must be run several times, or alternative means of interference control must be implemented.

Accuracy, productivity and long term stability were tested by analyzing a sequence of typical environmental samples for an extended period, representing the routine workload performed in contract environmental laboratories. The samples consisted of Certified Reference Materials (CRMs) for water, soil, and sediment, spiked samples and Quality Control (QC) samples as specified by EPA method 6020A (Figure 2). Calibration standards were prepared in a 1% HNO_3 and 0.5% HCl matrix. No matrix matching was required and no recalibrations were performed throughout the 9.5 hour sequence.

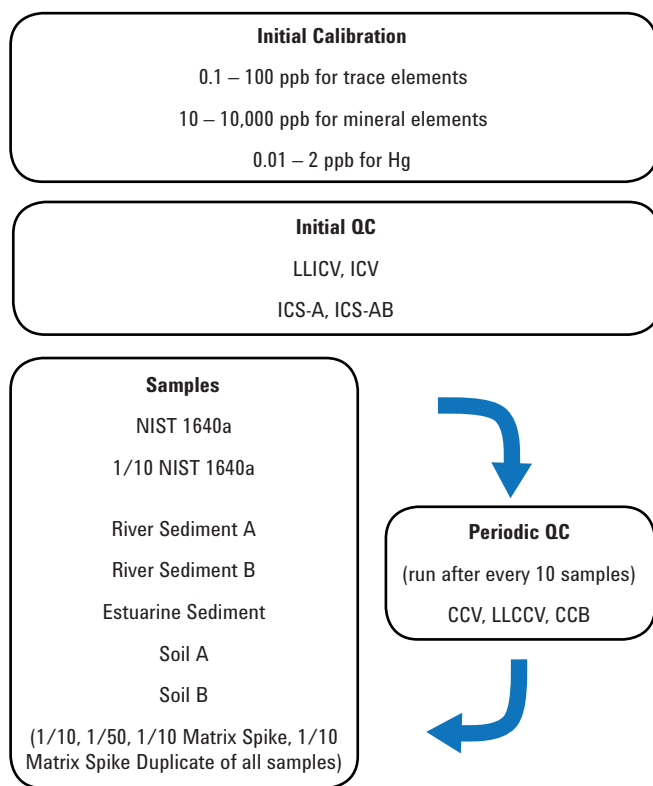


Figure 2. Sequence of samples, calibrants and QC solutions analyzed in a single 9.5 hour sequence. Sample Block was repeated continuously with automatic insertion of Periodic QC Block after every 10 sample runs. Number of analysis in 9 hours 35 minutes totaled 383.

Acronyms: Low Level Initial Calibration Verification (LLICV), Initial Calibration Verification (ICV), Interference Check Sample (ICS), Continuing Calibration Verification (CCV), Low Level Continuing Calibration Verification (LLCCV), Continuing Calibration Blank (CCB)

Results

This study used He cell mode for most elements and no gas mode for low mass elements. In all, 383 samples were measured in 9 hours and 35 minutes with a run-to-run time of 90 seconds per sample. The throughput was increased by ~ 30% compared to a similar study carried out using a 7700x ICP-MS with ISIS 2 [3].

Method Detection Limits

Three sigma Method Detection Limits (MDLs) were calculated from ten replicates of the low standard (trace elements: 0.1 ppb, mineral elements: 10 ppb, Hg: 0.01 ppb) (Table 2). Beryllium was acquired in no gas mode and the other elements were acquired in He mode. Integration times were kept as short as possible in order to minimize the total acquisition time. These are not “best possible” detection limits but are more than sufficient for the method requirements. Lower detection limits could be achieved with a longer analysis time and significant investment in higher purity reagents and clean working practices to reduce blank contamination levels, but this would come with a commensurate increase in the cost of analysis.

Meeting EPA QA/QC requirements

EPA method 6020A is a performance-based method. Consequently, unlike method 200.8 for drinking water analysis, the EPA has not restricted the use of collision/reaction cell (CRC) technology for method 6020 applications.

Table 2. Three sigma method detection limits calculated from 10 replicates of the low calibration standard

Isotope/ Element	Cell Mode	Integration Time (s)	3 sigma MDL (ppb)
9 Be	No gas	0.5	0.015
23 Na	He	0.1	2.1
24 Mg	He	0.1	0.87
27 Al	He	0.5	0.22
39 K	He	0.1	3.9
44 Ca	He	0.2	5.4
51 V	He	0.3	0.020
52 Cr	He	0.3	0.020
55 Mn	He	0.3	0.033
56 Fe	He	0.3	0.23
59 Co	He	0.3	0.014
60 Ni	He	0.3	0.022
63 Cu	He	0.3	0.009
66 Zn	He	0.3	0.055
75 As	He	1.0	0.022
78 Se	He	2.0	0.20
95 Mo	He	0.3	0.016
107 Ag	He	0.3	0.010
111 Cd	He	0.3	0.027
121 Sb	He	0.3	0.023
137 Ba	He	0.3	0.031
201 Hg	He	2.0	0.007
205 Tl	He	0.3	0.006
208 Pb	He	0.3 (0.1x3)*	0.016
232 Th	He	0.1	0.007
238 U	He	0.1	0.008

* Pb is measured as the sum of the three most abundant isotopes, 206, 207 and 208

The most recent version of EPA 6020A (Feb 2007) has added the requirement for a Low Level Initial Calibration Verification (LLICV) check at approximately the method reporting limit. The recommended control limits are $\pm 30\%$ of the actual value. The results for the LLICV and Initial Calibration Verification (ICV) check solution are shown in Table 3. LLICV results confirm sub ppb reporting limits for all trace elements and reporting limits in the 10–50 ppb range for the mineral elements. Reducing laboratory and reagent contamination for the mineral elements would allow lower reporting limits if required.

In addition to calculating and verifying MDLs and reporting limits, method 6020A requires that internal standard (ISTD) recoveries for all samples exceed 70% of the ISTD response in the calibration blank. Continuing Calibration Verification (CCV) samples at the midpoint concentration of the calibration must be analyzed after every 10 real samples and must be recovered within $\pm 10\%$ of the true value. If either of these criteria is not met, the affected samples must be re-analyzed, either after dilution in the case of ISTD failure, or after recalibration in the case of CCV failure. CCV recoveries for all 28 CCV replicates run over the course of the sequence are shown in Figure 3. Only sodium, aluminum and magnesium exceeded the +10% limit as a result of cross contamination from high concentration sample analysis. Internal standard recoveries are shown in Figure 4. The near horizontal slope of the ISTD plot indicates that there was no loss in sensitivity due to matrix accumulation on the interface or lenses for the entire sequence. In addition, there were no internal standard failures in any of the samples, which demonstrates the exceptional matrix tolerance delivered by the 7900 ICP-MS system with UHMI.

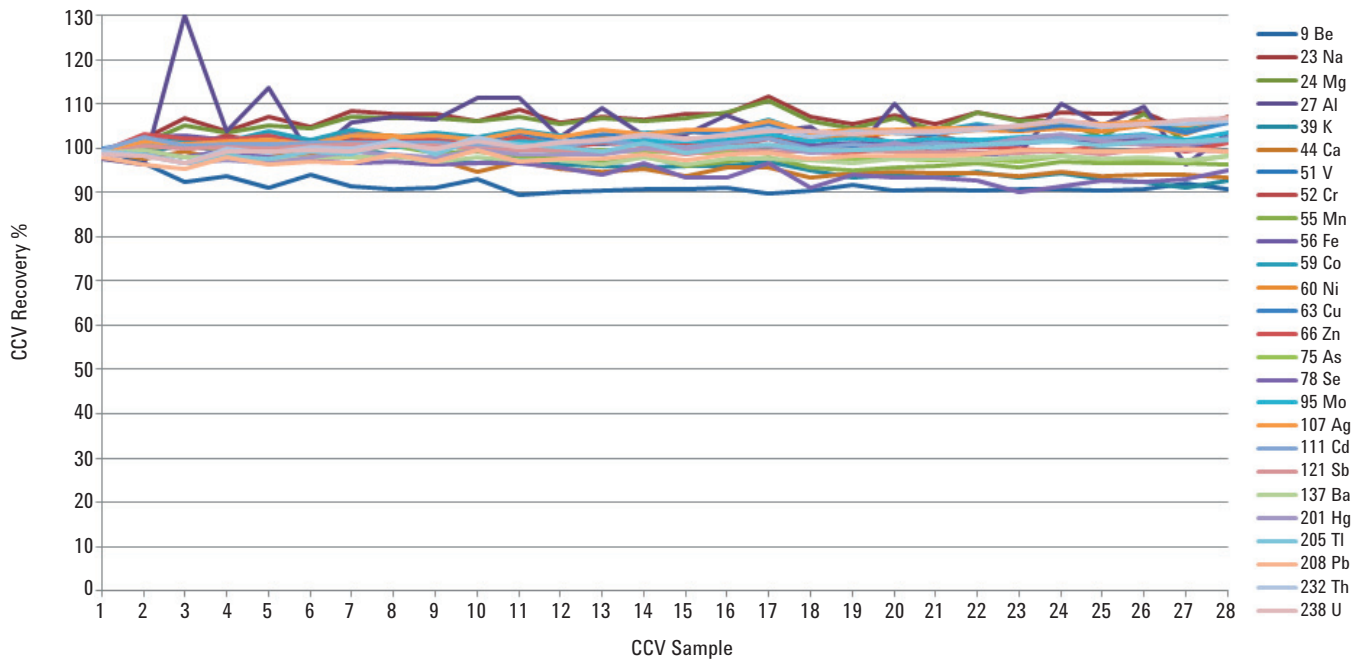


Figure 3. Continuing calibration verification (CCV) recoveries over the course of the sequence. Note: some sodium, aluminum and magnesium plots exceeded the +10% limit as a result of cross contamination from high concentration sample analysis.

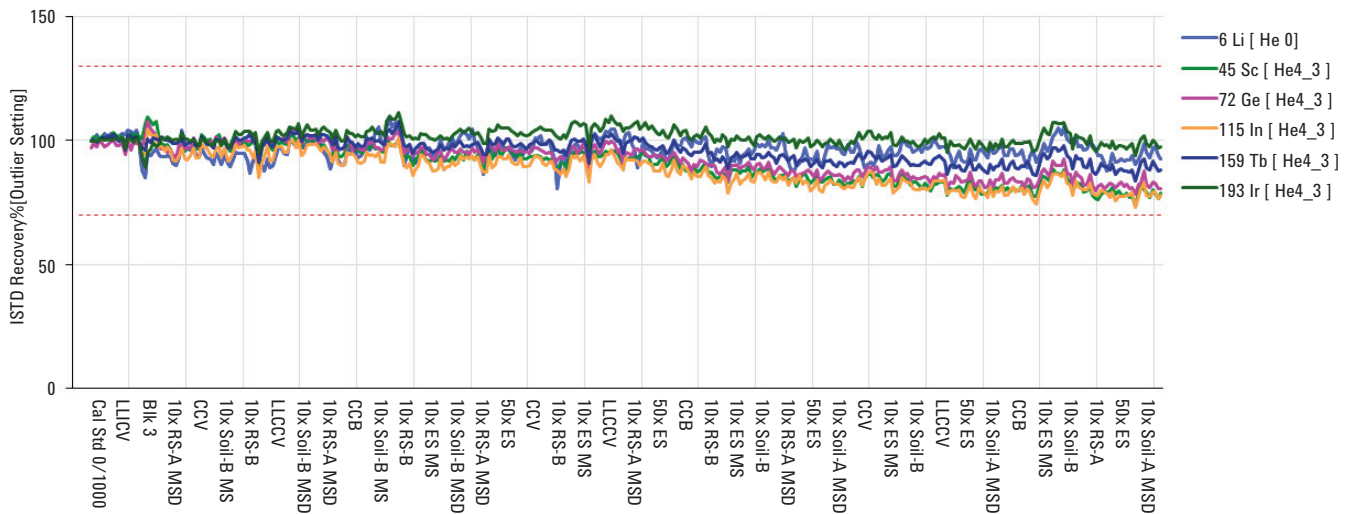


Figure 4. Internal standard recoveries normalized to the calibration blank for all samples. Due to limited space, not all sample names are shown. No internal standard failures occurred.

Table 3. Low Level Initial Calibration Verification (LLICV) and Initial Calibration Verification (ICV) concentrations and recoveries. The EPA limits for LLICV recoveries are ± 30 percent of actual; limits for ICV recoveries are ± 10 percent of actual

Isotope/ Element	LLICV (ppb)	LLICV Recovery (%)	ICV (ppb)	ICV Recovery (%)
9 Be	0.1	96	50	101
23 Na	50	95	5,000	100
24 Mg	10	103	5,000	101
27 Al	10	94	50	101
39 K	50	102	5,000	101
44 Ca	50	119	5,000	103
51 V	0.1	99	50	100
52 Cr	0.5	100	50	102
55 Mn	0.5	104	50	101
56 Fe	10	97	5,000	100
59 Co	0.1	98	50	102
60 Ni	0.1	87	50	102
63 Cu	0.1	100	50	102
66 Zn	0.5	105	50	101
75 As	0.1	103	50	102
78 Se	1.0	115	50	100
95 Mo	0.1	92	50	101
107 Ag	0.1	105	50	103
111 Cd	0.1	103	50	101
121 Sb	0.1	110	50	100
137 Ba	0.1	99	50	100
201 Hg	0.1	126	1	105
205 Tl	0.1	108	50	102
208 Pb	0.5	102	50	100
232 Th	0.1	106	50	102
238 U	0.1	107	50	101

Recovery of certified reference values

Six certified reference samples were analyzed repeatedly. They were NIST 1640a Natural water (NIST, Gaithersburg MD), CRM River Sediment A, CRM River Sediment B, CRM Estuarine Sediment, CRM Soil A and CRM Soil B (High Purity Standards – Charleston SC, USA). NIST 1640a was analyzed undiluted, while the other reference materials were analyzed after both 10x and 50x dilutions. Values shown in Table 4 were taken from the 10x dilutions. Each sample was measured multiple times over the sequence and the mean concentration, percent relative standard deviation (% RSD), and mean recovery were calculated for each analyte (Table 4). Not all reference materials are certified for all analytes – blank cells indicate the absence of a certified value.

Matrix spike and matrix spike duplicates

Along with the above CRMs, Matrix Spike (MS) CRMs and Matrix Spike Duplicates (MSD) CRMs spiked at the calibration midpoint (50 ppb for trace elements, 5000 ppb for mineral elements (Na, Mg, K, Ca and Fe), and 1 ppb for Hg) were analyzed periodically, interspersed with the other samples throughout the sequence. The number of repeats was 14 for each of these sample types except for 7 repeats of NIST 1640a. No matrix matching of the calibration standards and blanks to these samples was required, as UHMI effectively eliminates matrix suppression by ensuring very high temperature robust plasma conditions (around 0.2% CeO/Ce ratio in He mode). Results are shown in Table 5.

All elements met the EPA limits of 75% to 125% recovery for matrix spikes with excellent precision. Relative Percent Difference (RPD) was calculated between the MS and MSD samples. EPA 6020A requires the RPD to be less than $\pm 20\%$ for analytes whose concentration is greater than 100x the MDL. All elements passed the MS/MSD test with RPD below 2% for all elements further highlighting the excellent precision capabilities of the 7900 ICP-MS.

Table 4. Mean measured values, percent Relative Standard Deviations (%RSDs), mean recoveries for all certified elements in the six CRMs analyzed. Blank cells indicate no certified value.

Isotope/ Element	NIST 1640a (n=7)			River Sediment A (1/10, n=14)*			River Sediment B (1/10, n=14)*		
	Mean conc. (ppb)	%RSD	Mean recovery (%)	Mean conc. (ppb)	%RSD	Mean recovery (%)	Mean conc. (ppb)	%RSD	Mean recovery (%)
9 Be	2.80	2.4	93	0.026	9.8		ND		
23 Na	3,112	2.0	107	5,326	1.8	107	5,610	1.6	112
24 Mg	1,062	1.7	101	7,375	1.8	105	13,130	1.3	109
27 Al	57.8	4.4	109	25,180	1.3	101	61,460	0.6	102
39 K	561	5.4	97	14,730	2.4	98	20,220	2.3	101
44 Ca	5,373	3.2	97	29,050	1.8	97	28,960	1.6	97
51 V	15.4	1.5	103	25.5	1.1	102	104	0.7	104
52 Cr	40.5	1.6	101	30,950	1.1	103	1,592	0.9	106
55 Mn	40.0	1.7	100	786	2.2	98	587	1.1	98
56 Fe	44.2	5.7	121	122,000	0.9	102	41,610	0.6	104
59 Co	20.7	2.1	103	12.1	1.5	121	16.3	1.3	109
60 Ni	25.7	1.8	103	52.2	1.4	104	52.8	1.4	106
63 Cu	87.9	2.0	103	101.9	1.7	102	104	1.4	104
66 Zn	56.1	1.3	102	1,454	0.9	97	493	0.8	99
75 As	7.99	1.4	100	60.2	1.3	100	20.4	1.4	102
78 Se	18.5	5.1	93	2.28	5.7	114	0.95	10.5	95
95 Mo	45.8	2.0	101	0.074	7.0		0.17	4.8	
107 Ag	8.25	2.8	103	ND			0.17	2.3	
111 Cd	3.97	1.8	100	10.2	1.1	102	3.18	2.4	106
121 Sb	5.07	1.8	100	50.7	0.7	102	4.22	0.9	106
137 Ba	147	1.1	98	49.3	0.7	99	392	0.5	98
201 Hg	ND			ND			0.023	10.9	
205 Tl	1.58	3.0	98	0.99	1.4	99	1.15	1.6	115
208 Pb	12.5	4.4	104	742	1.8	106	212	1.7	106
232 Th	0.002	40		2.04	2.6	102	9.93	2.3	99
238 U	25.2	3.8	100	1.02	3.1	102	3.02	2.9	101

* Concentration of 1/10 diluted solution.

ND: less than detection limit

Table 4 continued. Mean measured values, percent Relative Standard Deviations (%RSDs), mean recoveries for all certified elements in the six CRMs analyzed. Blank cells indicate no certified value.

Isotope/ Element	Estuarine Sediment (1/10, n=14)*			Soil A (1/10, n=14)*			Soil B (1/10, n=14)*		
	Mean conc. (ppb)	%RSD	Mean recovery (%)	Mean conc. (ppb)	%RSD	Mean recovery (%)	Mean conc. (ppb)	%RSD	Mean recovery (%)
9 Be	1.78	1.7	89	ND			ND		
23 Na	22,170	1.2	111	7,659	1.2	109	10,980	1.7	110
24 Mg	10,950	1.2	110	7,583	1.1	108	8,720	2.0	109
27 Al	70,960	0.7	101	50,750	1.1	101	71,180	1.7	102
39 K	15,120	2.6	101	19,870	3.0	99	20,920	3.1	100
44 Ca	7,821	2.2	98	33,620	2.1	96	12,140	2.2	97
51 V	104	0.6	104	10.4	0.7	104	82.7	1.6	103
52 Cr	82.8	0.5	104	0.36	32.4		41.6	1.6	104
55 Mn	393	1.5	98	10.4	1.4	104	10,055	1.8	101
56 Fe	36,250	0.7	104	20,540	0.5	103	36,110	1.6	103
59 Co	11.2	1.5	112	0.34	1.1		11.0	2.1	110
60 Ni	31.6	1.3	105	31.0	1.2	103	21.3	1.7	106
63 Cu	20.9	1.5	104	30.8	1.3	103	326	2.3	109
66 Zn	150	0.8	100	99.6	0.9	100	7,050	1.7	101
75 As	10.4	1.6	104	20.2	1.4	101	607	2.1	101
78 Se	4.63	5.0	93	1.00	13.9	100	ND		
95 Mo	0.04	11.4		0.044	10.7		0.16	5.2	
107 Ag	0.016	7.0		0.020	6.3		0.080	4.7	
111 Cd	0.11	9.1		0.37	3.3		20.5	1.7	103
121 Sb	0.56	1.6		3.13	1.0	104	40.9	1.7	102
137 Ba	1.33	2.6		493	0.5	99	697	1.6	100
201 Hg	0.016	12.8		0.048	5.3		0.007	29	
205 Tl	0.010	8.3		0.016	4.1		0.15	2.6	
208 Pb	29.3	1.5	98	39.0	1.7	98	6,352	2.6	106
232 Th	10.1	2.4	101	10.0	2.5	100	10.2	3.3	102
238 U	0.003	17.4		1.01	3.2	101	25.6	3.6	103

* Concentration of 1/10 diluted solution.

ND: less than detection limit

Table 5. Spike recoveries and Matrix Spike (MS)/Matrix Spike Duplicate (MSD) relative percent differences (RPD) for 1/10 River Sediment-A (n=13)

Isotope/Element	Matrix Spike Mean (ppb)	%RSD	Spike (ppb)	Mean Recovery (%)	RPD MS/MSD (%)
9 Be	9.42	1.2	10	94	0.5
23 Na	6,374	1.9	1,000	105	0.4
24 Mg	8,360	1.8	1,000	98	0.1
27 Al	24,890	1.0	10	n/a	
39 K	15,700	2.2	1,000	96	1.0
44 Ca	29,840	1.4	1,000	n/a	
51 V	35.7	0.9	10	101	0.4
52 Cr	31,150	1.1	10	n/a	
55 Mn	795	2.0	10	n/a	
56 Fe	123,600	0.7	1,000	n/a	
59 Co	22.6	1.4	10	105	0.3
60 Ni	62.8	1.3	10	106	0.5
63 Cu	113	1.6	10	107	0.5
66 Zn	1,466	0.7	10	n/a	
75 As	70.2	1.5	10	100	0.5
78 Se	11.6	3.5	10	94	0.7
95 Mo	10.4	1.0	10	104	0.6
107 Ag	10.4	1.6	10	104	0.5
111 Cd	20.3	0.9	10	101	0.8
121 Sb	60.9	0.7	10	101	0.3
137 Ba	59.4	1.0	10	100	0.2
201 Hg	0.108	3.1	0.1	105	0.4
205 Tl	10.9	1.6	10	99	0.1
208 Pb	750	1.7	10	n/a	
232 Th	12.1	2.3	10	101	0.3
238 U	11.1	2.6	10	101	0.6

Table 5 continued. Spike recoveries and Matrix Spike (MS)/Matrix Spike Duplicate (MSD) relative percent differences (RPD) for 1/10 River Soil-B (n =14)

Isotope/Element	Matrix Spike Mean (ppb)	%RSD	Spike (ppb)	Mean Recovery (%)	RPD MS/MSD (%)
9 Be	8.87	0.8	10	89	0.4
23 Na	12,020	1.0	1,000	103	0.1
24 Mg	9,768	0.9	1,000	105	0.2
27 Al	70,970	1.2	10	n/a	
39 K	21,790	3.4	1,000	87	0.0
44 Ca	13,060	2.5	1,000	92	0.1
51 V	92.6	1.0	10	99	0.2
52 Cr	51.6	1.1	10	100	0.0
55 Mn	10,030	1.7	10	n/a	
56 Fe	36,970	1.1	1,000	86	0.2
59 Co	21.5	1.1	10	104	0.1
60 Ni	31.5	1.0	10	103	0.0
63 Cu	334	1.5	10	86	0.2
66 Zn	7,007	1.2	10	n/a	
75 As	615	2.1	10	n/a	
78 Se	9.15	4.1	10	91	1.9
95 Mo	10.4	1.4	10	103	0.6
107 Ag	10.6	1.7	10	105	0.4
111 Cd	30.5	1.0	10	100	0.8
121 Sb	50.8	1.3	10	99	0.1
137 Ba	705	1.1	10	80	0.2
201 Hg	0.117	2.9	0.1	110	0.7
205 Tl	10.1	1.6	10	99	0.2
208 Pb	6,329	1.7	10	n/a	
232 Th	20.4	2.4	10	101	0.1
238 U	35.9	2.7	10	103	1.4

Conclusions

This study shows that long sequences of high Total Dissolved Solids (TDS) samples can be analyzed with high accuracy, precision, and long term stability using the Agilent 7900 ICP-MS. This was shown by combining the benefits of He mode for removing polyatomic interferences with the UHMI for highly robust plasma conditions, and the ISIS 3 discrete sampling system. The advantages are as follows:

- ISIS 3 uses constant flow nebulization to eliminate the need for stabilization after high speed uptake, reducing the run time by as much as a minute per sample.
- ISIS 3 separates sample uptake and analysis so the system can begin rinsing before the previous analysis is finished, reducing the run time by as much as an additional minute.
- ISIS 3 introduces sample to the mass spectrometer only during actual data acquisition; at all other times, a clean blank solution is being nebulized. This results in a 3–5x reduction in the amount of sample reaching the instrument with a resulting decreased requirement for cleaning and maintenance.
- ISIS 3 enables continuous workflow from system optimization to sample measurement and data analysis without interruption by manual handling of tubing.
- Faster washout is achieved by close coupling of the ISIS 3 valve, nebulizer and UHMI, reducing required rinse time.
- UHMI reduces the total sample load introduced to the plasma by accurately and reproducibly controlling aerosol dilution, eliminating costly reruns and reducing system maintenance.
- Fast cell gas switching supports the use of universal He mode and high sensitivity no gas mode in a single discrete sampling analysis (three replicate measurements per sample), optimizing acquisition conditions regardless of the sample matrix composition, without significant impact on acquisition time.

The net benefit is simpler operation, higher throughput and improved long term stability compared to conventional ICP-MS systems.

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Published October 15, 2014

Publication number: 5991-5208EN



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