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EPA 200.8: Demonstration of Analytical Performance for Addressing Requirements from the Proposed Revised Lead and Copper Rule

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Introduction

At the end of 2019, US EPA proposed the first major revision of the Lead and Copper Rule (LCR) since 1991. Although the proposed rule is still under revision, it is essential for environmental laboratories to be ready for responding to the potential increase in analytical demands associated with the revised LCR. In this work, we demonstrated the performance of ICP-MS 2030 for method EPA 200.8 and analyzed tap water samples from various sources to determine the stability and robustness of the system. The poster demonstrates that the method performed well according to EPA Quality Assurance criteria for lead and copper and other 11 elements included in EPA 200.8, providing a robust solution for supporting the LCR monitoring requirements as well as meeting other federal regulations. Outcomes from the evaluation of tap water samples are also presented.

Experimental

Instrumentation

A Shimadzu ICPMS-2030 was used for all analysis in conjunction with a Shimadzu AS-10 Autosampler. In-line addition of internal standards to calibration and unknown samples was accomplished using the Shimadzu Internal Standard Addition Kit. Detailed operating conditions are listed in Table 1.

Table 1. Operating conditions of Shimadzu ICPMS-2030

Parameter	Setting	Parameter	Setting
Radio Freq. Power	1.20 kW	Plasma Gas	8.0 L/min
Sampling Depth	5.0 mm	Auxiliary Gas	1.10 L/min
Chamber Temp.	5 °C	Carrier Gas	0.70 L/min
Total Integration Time	2 sec	Number of Repetition	3

Sample Preparation

Drinking water samples from different locations were preserved in 2% nitric acid before analysis. Calibration standards prepared by diluting and acidifying the Inorganic Ventures multi-elements standards.

A quality control standard sample was prepared by spiking acidified deionized water. Yttrium, scandium, indium, and bismuth were used as internal standard and were added to the calibration standards and samples using the internal standard addition kit. The final concentration of the internal standards upon reaching the plasma was 50 ppb.

Analytical elements, mass, and their corresponding method parameters are shown in Table 2.

The instrument was allowed to warm up for 30 minutes and was tuned to adjust instrument parameters prior to analysis using Shimadzu's ICP-MS tuning solution. Following tuning, the status of the instrument was evaluated using Inorganic Ventures 200.8 Method Tuning Solution. The evaluation procedure consisted of ensuring a resolution of 0.75 amu for several masses including 24Mg, 25Mg, 26Mg, 206Pb, 207Pb, and 208Pb.

Table 2. Analytical elements and their corresponding measurement parameters

Element	Mass	Internal Standard	Calibration Range (ppb)	Calibration R	Scan Time (Sec)	Number of Scans	Spiked Concen. (ppb)
As	75	Y (89)	0 - 100	0.99981	0.2	10	20
Be	9	Sc (45)	0 - 100	0.99994	0.2	10	20
Cd	114	In (115)	0 - 100	0.99998	0.2	10	20
Co	59	Sc (45)	0 - 100	0.99975	0.2	10	20
Cr	52	Sc (45)	0 - 100	1.00000	0.2	10	20
Cu	63	Sc (45)	0 - 100	0.99993	0.2	10	20
Ni	60	Sc (45)	0 - 100	0.99986	0.2	10	20
Pb	208	Bi (209)	0 - 100	1.00000	0.2	10	20
Se	82	Y (89)	0 - 500	0.99994	0.2	10	100
Th	232	Bi (209)	0 - 100	0.99941	0.2	10	20
Tl	205	Bi (209)	0 - 100	0.99995	0.2	10	20
U	238	Bi (209)	0 - 100	0.99932	0.2	10	20
V	51	Sc (45)	0 - 100	0.99996	0.2	10	20

Results and Discussion

Instrument Detection Limits (IDL) are measured by running ten (10) replicates of a blank solution and calculating the three times the standard deviation. Method Detection Limits (MDL) are calculated as 3.14 times the standard deviation of seven (7) replicate analyses of each analyte spiked into reagent water at a concentration of two- to five-times the IDL. The IDL and MDL for Cu and Pb which are focus of this work are shown in table 3.

Table 4. Concentrations of elements in ppb in original samples and spiked samples for three different water samples as well as recovery yields

		As	Be	Cd	Co	Cr	Cu	Ni	Pb	Se	Th	Tl	U	V
Blank	Mean value	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	RSD (n = 3)	----	----	----	----	----	----	----	----	----	----	----	----	----
Spiked Blank (QC Standard)	Mean value	19.3	19.7	19.9	21.7	20.8	20.5	19.2	19.5	99.0	20.7	19.2	20.8	19.7
	RSD (n = 3)	1.34	1.94	3.42	2.33	2.9	2.12	2.95	3.17	2.88	2.93	4.90	2.15	3.16
Recovery (%)		96.5	98.5	99.5	108.5	104	102.5	96.0	97.5	99	103.5	96	104	98.5
Water Sample 1	Mean value	0.209	n.d.	n.d.	0.03	0.653	28.1	0.44	0.163	0.244			0.011	0.309
	RSD (n = 3)	1.3	----	----	3.53	4.82	0.52	3.71	1.77	1.99	----	----	3.68	2.16
Spiked Sample	Mean value	20	18.4	19.3	20.6	20.8	48.3	18.6	19.8	100	21.0	18.8	21.0	19.9
	RSD (n = 3)	2.12	2.61	3.32	1.83	2.73	0.30	0.88	2.42	2.90	0.54	2.52	1.70	1.78
Recovery (%)		98.9	92.0	96.5	102.8	100.7	101	90.8	98.2	99.8	105	94	104.9	98
Water Sample 2	Mean value	0.223	n.d.	n.d.	0.0940	0.853	4.13	0.527	n.d.	0.235	n.d.	n.d.	0.012	0.684
	RSD (n = 3)	2.51	----	----	3.69	1.69	2.45	1.6	----	4.45	----	----	4.18	1.56
Spiked Sample	Mean value	20.3	20	19.8	21.2	21.3	23.3	19.2	19.9	104	21.9	19.5	22.0	20.2
	RSD (n = 3)	2.64	2.43	2.97	2.40	2.28	1.85	1.59	3.64	1.95	2.18	3.68	3.81	3.56
Recovery (%)		100.3	100.0	99	105.5	102.2	95.8	93.4	99.5	103.8	109.5	97.5	109	97.6
Water Sample 3	Mean value	0.220	n.d.	n.d.	0.072	0.850	3.47	1.04	n.d.	n.d	n.d.	n.d.	0.0124	0.234
	RSD (n = 3)	2.62	----	----	2.55	3.69	2.44	4.84	----	----	----	----	0.55	4.89
Spiked Sample	Mean value	19.7	20.4	19.5	20.3	20.6	22.2	19.5	19.4	102	21.2	19.0	21.2	19.6
	RSD (n = 3)	3.25	0.66	2.12	1.44	2.52	4.10	3.5	2.20	0.60	1.10	1.75	2.84	3.3
Recovery (%)		97.4	102.0	97.5	101.1	98.75	93.7	92.3	97	102	106	95.0	105.9	96.8

Table 3. Method 200.8 instrument detection limit (IDL) and method detection limit (MDL)

Element	IDL (ppb)	MDL Spike (ppb)	MDL (ppb)
Cu	0.00324	0.01	0.00799
Pb	0.00382	0.01	0.00097

Table 4 shows the concentrations of Cu, Pb and other 11 elements included in EPA 200.8 in three tap water samples (unspiked and spiked) as well as blank sample.

Evaluation of the recoveries of spiked water sample (QC standard) and 3 types of samples were performed using Shimadzu ICPMS-2030. All the obtained recoveries were within the acceptable range of 90 to 110% of the spiked values as shown in Table 4. All relative standard deviations (RSD) are also below 5%, further demonstrating the methodology and the high precision.

Conclusions

The metal content of drinking water samples can be analyzed with the Shimadzu ICPMS-2030 to ensure meeting all of the analytical requirements established in method EPA 200.8 to support the LCR and its latest revision. The detection limit (IDL and MDL) were calculated for Pb and Cu. Three water samples were analyzed to demonstrate method performance. The obtained recovery were in the acceptable range between 90 to 110%.

The Shimadzu ICPMS-2030 equipped with AS-10 autosampler and Internal Standard Addition Kit provides excellent sensitivity, precision, accuracy, tolerance and fast time response to meet and exceed compliance with regulations on trace elements analysis in water.

References

1. "Analysis of Trace Elements in Water by EPA Method 200.8 using the Shimadzu ICPMS-2030", Shimadzu Application News, No. ICP-005