Analysis of Environmental Samples by ICP-OES Following US EPA Guidelines

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The United States Environmental Protection Agency (US EPA) Contract Laboratory Program (CLP) defines the analytical methods accepted for the isolation, detection, and quantitative measurement of 22 target analytes in water and soil/sediment environmental samples. The Statement of Work (SOW) for Multi Media, Multi Concentration Inorganic Analysis (ILM05.3) is used to define the nature and extent of contamination and determine appropriate cleanup actions, emergency response actions, and enforcement/litigation activities (1).

Instrumentation, Experimental Conditions, and Results

The Varian 730-ES simultaneous CCD ICP-OES was utilized in this study (2). The 730-ES has an axially viewed plasma, featuring a high efficiency 40 MHz RF generator and low gas consumption. A Cooled-Cone Interface (CCI) displaces the cooler plasma tail, reducing interferences and increasing linear dynamic range. The system includes the SVS1 Switching Valve System as standard for maximum productivity.

The instrument operating parameters were optimized for low wavelength elements. Two 30-s replicate readings were sufficient to meet the contract required detection limits (CRDLs) as specified in ILM05.3.

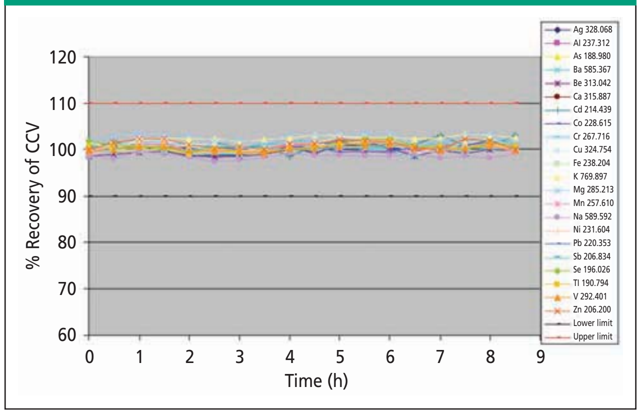

Figure 1: Long-term stability.

NIST SRM 1643e Trace Elements in Water and Melbourne drinking (tap) water were analyzed. All sample and standard solutions were made up in 1% v/v HNO_3 and 5% v/v HCl. $CsNO_3$ ionization buffer was added on-line to all solutions (1% w/v).

All required method confirmation and quality control tests were performed. These included Linear Range Analysis (LRA), Inter-Element Correction (IEC) and Interference Check Sam-

Table I: Method detection limits

Element Wavelength	CRDL (µg/L)	CRQL ILM05.3 (µg/L)	MDL required ILM05.3 (µg/L)	MDL obtained (µg/L)	Result
Ag 328.068	5	10	5	0.5	Pass
Al 237.312	200	200	100	5	Pass
As 188.980	5	10	5	1	Pass
Ba 585.367	20	200	100	0.6	Pass
Be 313.042	1	5	2.5	0.009	Pass
Ca 315.887	5000	5000	2500	1	Pass
Cd 214.439	2	5	2.5	0.09	Pass
Co 228.615	5	50	25	0.4	Pass
Cr 267.716	5	10	5	0.2	Pass
Cu 324.754	5	25	12.5	0.7	Pass
Fe 238.204	100	100	50	0.3	Pass
K 769.897	5000	5000	2500	2	Pass
Mg 285.213	5000	5000	2500	0.4	Pass
Mn 257.610	10	15	7.5	0.06	Pass
Na 589.592	5000	5000	2500	0.6	Pass
Ni 231.604	20	40	20	0.7	Pass
Pb 220.353	3	10	5	0.8	Pass
Sb 206.834	5	60	30	1	Pass
Se 196.026	5	35	17.5	1	Pass
Tl 190.794	5	25	12.5	1	Pass
V 292.401	10	50	25	0.3	Pass
Zn 206.200	10	60	30	0.5	Pass

Table II: Melbourne water analysis

Element Wavelength	Melbourne tap water measured (mg/L)	Duplicate measured (mg/L)	Control limit	% RPD or difference (mg/L)	Sample spike measured (mg/L)	Added QC spike conc. (mg/L)	% spike recovery
Ag 328.068	<CRQL	<CRQL	-	-	0.0484	0.0491	98.6
Al 237.312	0.0934	0.0944	CRQL	0.001	2.11	1.96	103
As 188.980	<CRQL	<CRQL	-	-	0.0395	0.0361	109
Ba 585.367	0.0180	0.0172	CRQL	0.00078	2.05	1.96	104
Be 313.042	<CRQL	<CRQL	-	-	0.0513	0.0491	104
Ca 315.887	3.64	3.63	CRQL	0.01	-	-	-
Cd 214.439	<CRQL	<CRQL	-	-	0.0486	0.0451	108
Co 228.615	<CRQL	<CRQL	-	-	0.510	0.491	104
Cr 267.716	<CRQL	<CRQL	-	-	0.206	0.196	105
Cu 324.754	0.162	0.161	20% RPD	0.40%	0.412	0.246	102
Fe 238.204	0.0935	0.0912	CRQL	0.0023	1.10	0.982	102
K 769.897	0.598	0.596	CRQL	0.002	-	-	-
Mg 285.213	1.115	1.112	CRQL	0.003	-	-	-
Mn 257.610	0.00617	0.00611	CRQL	0.00006	0.524	0.491	105
Na 589.592	4.075	4.073	CRQL	0.002	-	-	-
Ni 231.604	<CRQL	<CRQL	-	-	0.516	0.491	105
Pb 220.353	<CRQL	<CRQL	-	-	0.0201	0.0180	112
Sb 206.834	<CRQL	<CRQL	-	-	0.101	0.0901	112
Se 196.026	<CRQL	<CRQL	-	-	0.0493	0.0451	109
Tl 190.794	<CRQL	<CRQL	-	-	0.0474	0.0451	105
V 292.401	<CRQL	<CRQL	-	-	0.503	0.491	102
Zn 206.200	0.00589	0.00685	CRQL	0.00096	0.530	0.491	107

ples (ICSA and ICSAB), Laboratory Control Sample (LCS), Duplicate Sample Analysis, Spike Sample Analysis (Matrix Spike) and Quality Control (QC) tests - Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Contract Required Quantitation Limit (CRQL) Check Standard (CRI), Initial and Continuing Calibration Blank (ICB/CCB). In all cases ILM05.3 requirements were met.

NIST 1643e Trace Elements in Water was used as the lab control sample (LCS). Analyte, duplicate, and spike recoveries for all target elements fell within US EPA requirements. Duplicate results and matrix spike recoveries for the Melbourne tap water sample also met US EPA requirements.

The CCV solution was measured over an 8-h period to test stability. Repeatability of 0.75% RSD was found for most elements (maximum 0.98% for cadmium). All CCV recoveries were easily within specified limits.

Due to the requirements set by the US EPA, a large number of QC solutions must be measured to ensure compliance, making these types of analyses very time-consuming. The Varian 730-ES Simultaneous ICP-OES can meet all US EPA requirements in a total analysis time of 2 min 25 sec per solution, providing high sample throughput and low cost per analysis.

Conclusion

The Varian 730-ES simultaneous ICP-OES with axially viewed plasma meets the stringent requirements of ILM05.3 methodology and environmental regulations set by the US EPA for waters and wastewaters.

References

(1) ILM05.3, EPA Publication 540-F-04-001 (2004).
(2) ICP-OES Application Note, Varian, Inc., Number 38 (2006).

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