

Analysis of Environmental Samples with the Agilent 710-ES Following US EPA Guidelines

Application Note

Inductively Coupled Plasma-Optical Emission Spectrometers

Authors

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Introduction

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 23 target analytes (including mercury) and cyanide in both water and soil/sediment environmental samples [1]. Data from 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 [2].

This application note describes the use of the Agilent 710-ES to carry out the US EPA/CLP compliant analysis of water samples.



Agilent Technologies

Instrumentation

The Agilent 710-ES with megapixel CCD detector was used, providing simultaneous measurement for fast, precise and accurate analysis. The MultiCal feature in the ICP Expert II software extends the linear range, allowing the measurement of elements from microgram per litre to percentage levels from a single plasma view. The optical system is thermally stabilized and contains no moving parts, ensuring excellent long-term stability.

The Agilent SPS3 autosampler was used for sample introduction.

ILM05.3 Exhibit D recommends the use of an ionization buffer if ionization effects are pronounced. The potential for ionization interference caused by easily ionized elements (Na, K and Ca) was addressed by adding a solution containing 1% caesium (ionization buffer) online to the sample stream [3].

Materials and Reagents

Calibration and Quality Control (QC) solutions were prepared from Inorganic Ventures, Inc. custom-grade multi-element solutions including the Superfund CLP ICP Kit for ILM05.2. NIST certified standard reference material 1643e Trace Elements in Water, was measured as a Laboratory Control Sample (LCS). The Superfund CLP ICP Kit for ILM05.2 contained the following solutions:

Table 1. Superfund CLP ICP Kit for ILM05.2

Solution	Conc (mg/L)	Elements	Purpose	Required dilution
CLPP-CAL-1	5000	Ca, Mg, K, Na	Calibration standard	1:100
	2000	Al, Ba		
	1000	Fe		
	500	Co, Mn, Ni, V, Zn		
	250	Ag, Cu		
	200	Cr		
	50	Be		
CLPP-CAL-2	1000	Sb	Calibration standard	1:100
CLPP-CAL-3	1000	As, Pb, Se, Ti	Calibration standard	1:100
	500	Cd		
CL-AES-CRQL	500	Ca, Mg, K	CRQL (CRI)	1:100
	20	Al, Ba		
	10	Fe		
	6	Sb, Zn		
	5	Co, V		
	4	Ni		
	3.5	Se		
	2.5	Cu, Ti		
	1.5	As, Mn		
	1	Cr, Pb, Ag		
	0.5	Be, Cd		
CLPP-ICS-A	5000	Al, Ca, Mg	ICSA	1:20
	2000	Fe		
CLPP-ICS-B4	100	Cd, Ni, Zn	ICSAB	1:100
	60	Sb		
	50	Ba, Be, Co, Cr, Cu		
		Mn, V		
	20	Ag		
	10	As, Ti		
5	Pb, Se			
QCP-CICV-1	2500	Ca, Mg, K, Na	ICV, CCV	1:500 (ICV) 1:100 (CCV)
	1000	Al, Ba		
	500	Fe		
	250	Co, Mn, Ni, Zn		
	125	Ag, Cu		
	100	Cr		
	25	Be		
QCP-CICV-2	500	Sb	ICV, CCV	1:500 (ICV) 1:100 (CCV)
QCP-CICV-3	500	As, Pb, Se, Ti	ICV, CCV	1:500 (ICV) 1:100 (CCV)
	250	Cd		
CLPP-SPK-1	2000	Al, Ba	Matrix spike	1:1000
	1000	Fe		
	500	Co, Mn, Ni, V, Zn		
	250	Cu		
	200	Cr		
	50	Ag, Be		
CLPP-SPK-5	100	Sb	Matrix spike	1:1000
	50	Cd, Se, Ti		
	40	As		
	20	Pb		

Standard and Sample Preparation

The calibration and QC solutions were diluted with > 18 MΩ/cm³ deionized water and stabilized in 1% v/v concentrated nitric acid (HNO₃) and 5% v/v concentrated hydrochloric acid (HCl) (both Merck Ultrapur). The sample solutions were also prepared in 1% v/v HNO₃ and 5% v/v HCl.

The calibration blank was prepared from > 18 MΩ/cm³ deionized water in 1% v/v HNO₃ and 5% v/v HCl (both Merck Ultrapur).

A 1% w/v ionization buffer solution was prepared from Merck Tracepur CsNO₃ and stabilized in 1% v/v HNO₃ (Merck Ultrapur). The solution was introduced online to the sample line using a spare channel on the peristaltic pump. A “Y-connector” located between the nebulizer and peristaltic pump was used to add the ionization buffer solution to the sample stream.

Conditions

The instrument operating conditions are shown in Table 2.

Table 2. Instrument Operating Conditions

Parameter	Setting
Power	1.4 kW
Plasma gas flow	15 L/min
Auxiliary gas flow	1.5 L/min
Spraychamber type	Glass cyclonic
Torch	Standard axial torch
Nebulizer type	SeaSpray
Nebulizer gas pressure	220 kPa
Pump tubing	Sample: white-white (1.02 mm id) Waste: blue-blue (1.65 mm id) Buffer/Reference (internal standard) element: black-black (0.76 mm id)
Pump speed	12 rpm
Sample uptake	5 mL
Replicate read time	30 s
Number of replicates	2
Sample delay time	20 s
Stabilization time	15 s
Rinse time	40 s
Fast pump	On
Background correction	Off-peak

Table 3 shows the background correction points used.

Table 3. Off-Peak Background Correction Points Used

Element	BC point left (nm)	BC point right (nm)
Ag 328.068	0.030	n.u.
Al 308.215	n.u.	0.033
As 188.980	0.017	0.016
Ba 585.367	0.070	0.061
Be 234.861	n.u.	0.024
Ca 315.887	n.u.	0.044
Ca 370.602	0.053	n.u.
Cd 226.502	0.025	0.022
Co 228.615	0.026	0.022
Cr 267.716	0.029	0.025
Cu 324.754	0.039	0.033
Cu 327.395	0.034	0.037
Fe 258.588	0.028	0.025
Fe 259.940	0.022	0.025
K 404.721	0.046	n.u.
K 769.897	n.u.	0.099
Mg 279.800	0.030	n.u.
Mg 285.213	0.025	0.028
Mn 257.610	0.032	0.025
Na 330.237	0.026	0.032
Na 568.821	0.062	0.079
Na 589.592	0.065	0.076
Ni 231.604	0.021	0.023
Pb 220.353	0.014	0.015
Sb 206.834	0.020	0.019
Se 196.026	0.014	n.u.
Tl 190.794	0.021	n.u.
V 311.837	n.u.	0.047
Zn 206.200	0.019	0.019

Note: n.u. indicates “not used”

Discussion

Method Detection Limits (MDL) and Contract Required Detection Limits (CRDL)

The MDL of each element was determined following the procedure defined in 40 CFR, part 136, as specified in Section 12.10.1.1 of Exhibit D in ILM05.3 Statement of Work. A standard solution containing analytes at a concentration of 3 to 5 times the instrument manufacturer’s suggested IDL was measured on 3 non-consecutive days. A replicate read time of 30 seconds was found to be sufficient to meet the contract required detection limits (CRDLs) as specified in Section 12.10.1.2, Exhibit D of ILM05.3 Statement of Work. These

CRDLs are reported in Table 4. Note that in this application, the instrument operating parameters were optimized for elements having low-wavelength emission lines.

Table 4. Method Detection Limits

Element	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	0.8	Pass
Al 308.215	200	200	100	5	Pass
As 188.980	5	10	5	2	Pass
Ba 585.367	20	200	100	3	Pass
Be 234.861	1	5	2.5	0.03	Pass
Ca 315.887	5000	5000	2500	2	Pass
Ca 370.602	5000	5000	2500	8	Pass
Cd 226.502	2	5	2.5	0.1	Pass
Co 228.615	5	50	25	0.4	Pass
Cr 267.716	5	10	5	0.4	Pass
Cu 324.754	5	25	12.5	0.9	Pass
Fe 258.588	100	100	50	1	Pass
K 766.491	5000	5000	2500	13	Pass
K 769.897	5000	5000	2500	18	Pass
Mg 279.800	5000	5000	2500	2	Pass
Mn 257.610	10	15	7.5	0.06	Pass
Na 589.592	5000	5000	2500	1	Pass
Ni 231.604	20	40	20	0.5	Pass
Pb 220.353	3	10	5	1	Pass
Sb 206.834	5	60	30	2	Pass
Se 196.026	5	35	17.5	3	Pass
Tl 190.794	5	25	12.5	1	Pass
V 311.837	10	50	25	0.8	Pass
Zn 206.200	10	60	30	0.3	Pass

Linear Range Analysis (LRA)

The LRA represents the upper concentration limit for each analyte of the ICP-OES linear range beyond which results cannot be reported without dilution of the sample. The maximum error for each calibration standard within the linear range cannot exceed 5% [4]. Table 5 shows the results for the LRA.

Using the MultiCal feature, a second less sensitive wavelength was added for the elements Ca, Fe, K, Mg and Na, as shown in Table 5. To extend the linear dynamic range further during the analysis, MultiCal automatically assigns sample results to the appropriate wavelength. In the same way, QCP tests and actions are applied only to those wavelengths for which the results fall within the specified LDR.

It should be noted also, the LDR can be extended even further by using the online over-range dilution capability of the SPS3 autosampler and diluter accessory in conjunction with MultiCal.

Table 5. Linear Range Analysis

Element	Curve type	Minimum conc (mg/L)	Maximum conc (mg/L)
Ag 328.068	Linear	0	50
Al 308.215	Linear	0	40
As 188.980	Linear	0	50
Ba 585.367	Linear	0	200
Be 234.861	Linear	0	10
Ca 315.887	Linear	0	100
Ca 370.602	Linear	0	600
Cd 226.502	Linear	0	10
Co 228.615	Linear	0	60
Cr 267.716	Linear	0	50
Cu 324.754	Linear	0	50
Cu 327.395	Linear	0	100
Fe 258.588	Linear	0	600
Fe 259.940	Linear	0	200
K 404.721	Linear	100	600
K 766.491	Linear	0	100
K 769.897	Linear	0	100
Mg 279.800	Linear	0	600
Mg 285.213	Linear	0	50
Mn 257.610	Linear	0	50
Na 330.237	Linear	0	600
Na 568.821	Linear	0	600
Na 589.592	Linear	0	50
Ni 231.604	Linear	0	60
Pb 220.353	Linear	0	100
Sb 206.834	Linear	0	10
Se 196.026	Linear	0	100
Tl 190.794	Linear	0	100
V 311.837	Linear	0	60
Zn 206.200	Linear	0	100

Note: The US EPA recommends that the maximum silver concentration is limited to 2 mg/L.

Inter-Element Correction (IEC) and Interference Check Samples (ICSA and ICSAB)

Inter-Element Correction factors are commonly used in ICP-OES to compensate for spectral interferences that may arise on analyte lines. Interference Check Samples (ICS) are used to confirm that interfering elements likely to be encoun-

tered in environmental samples do not cause inaccurate determination of analyte concentrations.

To verify that inter-element and background correction factors are accurate, the interference check samples (ICS) must be analyzed and reported for all elements and interferents. ILM05.3 SOW states that the determined concentration of an analyte in the ICSA must be within $\pm 2 \times$ CRQL. All analytes in the ICSAB must be recovered to within $\pm 20\%$ of their true value. Tables 6 and 7 show the results for all target elements in ICSA and ICSAB without applying IEC factors. Since the measured concentrations for both solutions meet the specified requirements, inter-element corrections for the application were not needed on the Agilent 710-ES.

Table 6. Interference Check Sample A

Element	CRQL ILM05.3 (µg/L)	ILM05.3 ± limit (µg/L)	ICSA (µg/L)	Result
Ag 328.068	10	20	-12	Pass
As 188.980	10	20	-10	Pass
Ba 585.367	200	400	0.8	Pass
Be 234.861	5	10	7	Pass
Cd 226.502	5	10	7	Pass
Co 228.615	50	100	0.05	Pass
Cr 267.716	10	20	0.4	Pass
Cu 324.754	25	50	-21	Pass
Mn 257.610	15	30	-2	Pass
Ni 231.604	40	80	3	Pass
Pb 220.353	10	20	3	Pass
Sb 206.834	60	120	1	Pass
Se 196.026	35	70	8	Pass
Tl 190.794	25	50	2	Pass
V 311.837	50	100	-3	Pass
Zn 206.200	60	120	3	Pass

Table 7. Interference Check Sample AB

Element	Expected ICSAB (mg/L)	Found ICSAB (mg/L)	% Recovery ICSAB	Result
Ag 328.068	0.20	0.18	90	Pass
As 188.980	0.10	0.084	83	Pass
Ba 585.367	0.60	0.52	85	Pass
Be 234.861	0.60	0.52	86	Pass
Cd 226.502	1.01	0.97	96	Pass
Co 228.615	0.60	0.50	83	Pass
Cr 267.716	0.60	0.51	84	Pass
Cu 324.754	0.60	0.51	84	Pass
Mn 257.610	0.50	0.51	101	Pass
Ni 231.604	1.01	0.99	98	Pass
Pb 220.353	0.05	0.042	84	Pass
Sb 206.834	0.60	0.63	104	Pass
Se 196.026	0.05	0.052	103	Pass
Tl 190.794	0.10	0.087	86	Pass
V 311.837	0.50	0.49	98	Pass
Zn 206.200	1.01	1.00	99	Pass

Laboratory Control Sample (LCS)

An LCS obtained from the US EPA must be analyzed to demonstrate that sample preparation procedures are appropriate for the sample type. With the exception of silver and antimony, the percentage recovery for the LCS must not fall outside the control limits of 80–120%. If an LCS cannot be obtained from the US EPA, the Initial Calibration Verification (ICV) solution may be used [4].

For this application, NIST 1643e Trace Elements in Water was used as the LCS. Table 8 shows the percentage recovery for all target elements falling within the US EPA requirements.

Duplicate Sample Analysis

The reproducibility of sample preparation was determined by performing a duplicate sample analysis. A duplicate pair was created by processing 2 aliquots of the same sample through the sample preparation procedure. A control limit of 20% for the Relative Percentage Difference (RPD) applies for samples with a concentration greater than or equal to $5 \times$ CRQL. For samples with a concentration less than $5 \times$ CRQL, but greater than the CRQL, an absolute difference in concentration of \pm CRQL applies. If the sample has concentrations less than the CRQL, the difference is not reported. Table 8 shows the RPDs for all target analytes meeting the required US EPA specifications.

Spike Sample Analysis (Matrix Spike)

The spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology. Exhibit D (ICP-OES) of the ILM05.3 document states the spiking levels required for each of the analyzed elements. The spike recoveries must fall

within the limits of 75–125%. A post-digestion spike may need to be performed if the recoveries do not fall within these limits [4].

Table 8 shows the matrix spike recoveries for all target elements falling within the US EPA requirements.

Table 8. LCS Analysis

Element	NIST 1643e certified (mg/L)	NIST 1643e measured LCS (mg/L)	LCS % recovery	NIST 1643e sample measured (mg/L)	NIST 1643e 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	0.001062	< CRQL	–	< CRQL	< CRQL	–	–	0.0516	0.0485	106%
Al 308.215	0.1418	0.153	108%	0.153	0.157	CRQL	0.004	2.29	1.94	110%
As 188.980	0.06045	0.0571	95%	0.0632	0.0586	20% RPD	7.5%	0.106	0.0389	110%
Ba 585.367	0.5442	0.558	103%	0.558	0.555	CRQL	0.003	2.68	1.94	110%
Be 234.861	0.01398	0.0136	97%	0.0138	0.0140	CRQL	0.0002	0.0668	0.0485	109%
Ca 370.602	32.3	32.3	100%	32.5	32.6	20% RPD	0.5%	–	–	–
Cd 226.502	0.006568	0.00650	99%	0.00680	0.00679	CRQL	0.00001	0.0617	0.0487	113%
Co 228.615	0.02706	0.0284	105%	0.0288	0.0295	CRQL	0.0007	0.576	0.485	113%
Cr 267.716	0.0204	0.0209	103%	0.0216	0.0213	CRQL	0.0003	0.240	0.194	112%
Cu 324.754	0.02276	0.0217	95%	0.0222	0.0217	CRQL	0.0005	0.288	0.242	110%
Fe 258.588	0.0981	0.104	106%	0.1059	0.1063	CRQL	0.0004	1.19	0.969	111%
K 769.897	2.034	2.13	105%	2.19	2.23	CRQL	0.04	–	–	–
Mg 279.800	8.037	7.85	98%	8.04	8.31	CRQL	0.27	–	–	–
Mn 257.610	0.03897	0.0409	105%	0.0420	0.0415	CRQL	0.0005	0.590	0.485	113%
Na 589.592	20.74	21.0	101%	21.3	21.8	CRQL	0.5	–	–	–
Ni 231.604	0.06241	0.0632	101%	0.0652	0.0641	CRQL	0.0011	0.613	0.485	113%
Pb 220.353	0.01963	0.0192	98%	0.0201	0.0191	CRQL	0.0010	0.0393	0.0195	99%
Sb 206.834	0.0583	0.0591	101%	0.0619	0.0613	CRQL	0.0006	0.169	0.0974	110%
Se 196.026	0.01197	< CRQL	–	< CRQL	< CRQL	–	–	0.0519	0.0487	107%
Tl 190.794	0.007445	< CRQL	–	< CRQL	< CRQL	–	–	0.0443	0.0487	91%
V 311.837	0.03786	0.0361	95%	0.0374	0.0367	CRQL	0.0007	0.568	0.485	109%
Zn 206.200	0.0785	0.0802	102%	0.0836	0.0864	CRQL	0.0028	0.644	0.485	116%

Quality Control (QC) Tests

The QC tests outlined in ILM05.3 are designed to ensure accuracy and precision the results obtained. Refer to Exhibit D - Part A “Analytical Methods for Inductively Coupled Plasma - Atomic Emission Spectroscopy” for detailed explanations of the QC tests. The following QC solutions were used in this work.

Initial Calibration Verification (ICV)

The ICV test is run directly after the initial calibration to check the validity of the calibration standards. The ICV solution is obtained from the EPA or a secondary source. It must not be prepared from the same solution as the calibration standards. The recoveries of all target elements must be within the control limits of 90–110%.

Continuing Calibration Verification (CCV)

The Continuing Calibration Verification test is used to ensure the validity of the calibration throughout the analysis and is carried out at a frequency of 10% (every 10 analytical samples) or every 2 hours, whichever is more frequent. The CCV is also measured at the beginning, but not before the ICV and at the end of the analysis. The recoveries of all target elements must be within the control limits of 90–110%.

Contract Required Quantitation Limit (CRQL) Check Standard (CRI)

A standard at the CRQL (CRI) must be analyzed to verify the instrument calibration at low analyte concentrations. The CRQL is the minimum level of quantification acceptable under the contract Statement of Work (SOW). The CRI is measured

immediately after the ICV/ICB followed by the ICS. The CRI must be run for every wavelength used for the analysis, except Al, Ba, Ca, Fe, Mg, Na and K. The recoveries of all CRI target elements must not fall outside the control limits of 70–130% and 50–150% for antimony, lead and thallium.

Initial and Continuing Calibration Blank (ICB/CCB)

The ICB/CCB must be measured to ensure that adequate washout is being achieved and sample is not being carried over. The absolute value of the calibration blanks (ICB/CCB) must not exceed the CRQL. The ICB is measured directly after the ICV and the CCB is measured directly after the CCV.

Table 9 shows the results for the QC tests. All tests meet the required US EPA specifications.

Table 9. ICV, CCV, CRI Test Solution Results

Element	ICV conc. (mg/L)	Measured ICV conc. (mg/L)	% R	CCV conc. (mg/L)	Measured CCV conc. (mg/L)	% R	ICV conc. (mg/L)	Measured ICV conc. (mg/L)	% R
Ag 328.068	0.249	0.257	103	1.26	1.29	103	0.0101	0.00946	94
Al 308.215	1.99	2.12	107	10.0	10.5	104	0.202	0.212	105
As 188.980	1.00	1.05	105	5.02	5.08	101	0.0151	0.0145	96
Ba 585.367	1.99	2.15	108	10.0	10.5	104	0.202	0.209	103
Be 234.861	0.0498	0.0521	105	0.251	0.258	103	0.00504	0.00518	103
Ca 370.602	4.98	5.36	108	25.1	26.2	104	5.04	5.25	104
Cd 226.502	0.500	0.535	107	2.51	2.58	103	0.00504	0.00522	104
Co 228.615	0.498	0.542	109	2.51	2.65	105	0.0504	0.0534	106
Cr 267.716	0.199	0.216	108	1.00	1.05	105	0.0101	0.0100	99
Cu 324.754	0.249	0.267	107	1.26	1.31	104	0.0252	0.0254	101
Fe 258.588	0.995	1.07	108	5.02	5.22	104	0.101	0.106	105
K 769.897	4.98	5.43	109	25.1	27.1	108	5.04	5.39	107
Mg 279.800	4.98	5.13	103	25.1	25.8	103	5.04	5.14	102
Mn 257.610	0.498	0.543	109	2.51	2.67	106	0.0151	0.0164	108
Na 589.592	4.98	5.28	106	25.1	26.7	106	5.04	5.15	102
Ni 231.604	0.498	0.544	109	2.51	2.65	106	0.0404	0.0425	105
Pb 220.353	1.00	1.04	104	5.02	5.10	102	0.0101	0.00981	97
Sb 206.834	1.00	1.06	106	5.02	5.25	104	0.0605	0.0642	106
Se 196.026	1.00	1.08	107	5.02	5.29	105	0.0353	0.0375	106
Tl 190.794	1.00	1.03	103	5.02	5.02	100	0.0252	0.0262	104
V 311.837	0.498	0.535	107	2.51	2.61	104	0.0504	0.0497	99
Zn 206.200	0.498	0.538	108	2.51	2.64	105	0.0605	0.0641	106

Long-Term Stability

Long-term stability over 8 hours was measured with a repeatability of 1.23% RSD found for most elements up to a maximum of 1.85% for selenium. As shown in Figure 1, the CCV recoveries over the 8 hour period were all within specified limits.

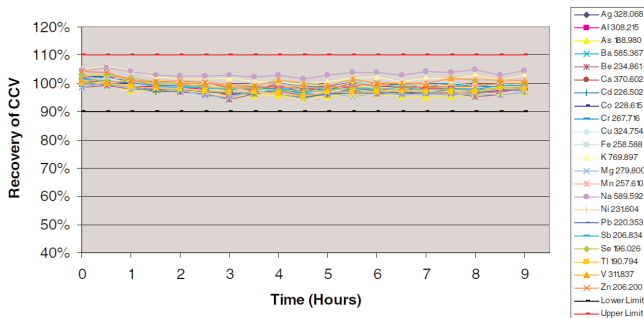


Figure 1. Long-term stability of the 710-ES over 8 hours.

Conclusion

This work demonstrates the ability of the Agilent 710-ES simultaneous ICP-OES with axially viewed plasma, to meet the environmental regulations set by the US EPA for waters and wastewaters.

The Agilent 710-ES provides the advantage of being able to achieve this requirement from a single viewed plasma system. This reduces time delays and costs related to repeating analyses with other techniques or multiple measurements when using dual viewed ICP-OES systems. The developed method allows a sample to be measured for 22 elements in 3 minutes and 34 seconds. An analysis that adheres strictly to US EPA protocols is inherently time-consuming due to the large number of quality control solutions that must be measured.

MultiCal was used to extend the linear dynamic range of Ca, Fe, K, Mg and Na, without requiring the sample to be diluted or measured separately with radial viewing. The suitability of this approach was proven with the CRI and Linear Dynamic Range tests. Using standard US EPA conditions, it was also shown that successful compliance with the Interference Correction Standards tests could be achieved without Inter-Element Correction factors.

The ICP Expert II software provides complete automation of all US EPA protocols and the capability to customize QC protocols to meet the requirements of other regulatory bodies. The Agilent 710-ES has been shown to meet all regulatory requirements of USEPA ILM05.3 in a single, fast and fully automated analysis.

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Printed in the USA
November 1, 2010
10-039



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