

# US EPA Method 200.8 using the XSERIES 2 ICP-MS

## Key Words

- Environmental Analysis
- EPA Method 200.8
- Productivity Pack
- US EPA
- Water Analysis

## Introduction

This Application Note describes the use of the Thermo Scientific XSERIES 2 ICP-MS for the analysis of drinking water, natural water and waste water using the US EPA method 200.8. It gives data showing compliance with each of the requirements and highlights the integrated system tools specifically designed to aid compliance. The data was generated using the procedures and solutions supplied in the Thermo Scientific EPA Method 200.8 Productivity Pack (Part Number 4600542).

## Background

### EPA History

In 1970, the United States government established the Environmental Protection Agency (EPA) in response to growing public demand for cleaner water, air and land. Prior to this, the national government was not structured to deal with pollution that caused harm to human health and degraded the environment. The EPA was tasked with repairing the damage already done and moving towards a cleaner environment. Its mission is to protect human health and to safeguard the natural environment. The Agency consists of 18,000 people in Headquarters, program offices, 10 regional offices and 17 labs across the US. The EPA provides leadership in the nation's environmental science, research, education and assessment efforts and works closely with other federal agencies and local government to develop and enforce regulations under existing environmental law. The Agency is responsible for researching and setting national standards for a variety of environmental programs and delegates the responsibility for issuing permits, and monitoring and enforcing compliance, to local government. Where national standards are not met, the EPA can issue sanctions and take other steps to assist local government in reaching the desired levels of environmental quality. The Agency also works with industries and all levels of government in a wide variety of voluntary pollution prevention programs and energy conservation efforts.

### The Development and Application of Method 200.8

The Safe Drinking Water Act (SDWA) of 1996 gives the US EPA power to set and regulate national standards for the quality of supplied drinking water and drinking water sources, such as ground waters. The EPA's Office of Ground Water and Drinking Water (OGWDW) administer control under the Federal Regulation 40 CFR part 141.

This regulation states that all supplied waters must comply with the Maximum Contaminant Levels (MCLs) for the contaminants specified in the National Primary Drinking

Water Regulations (NPDWRs). Further contaminants are given target maximum values in the National Secondary Drinking Water Regulations (NSDWRs) (40 CFR part 143). Tables 1 and 2 show the contaminants appropriate to ICP-MS measurement and their levels.

CONTAMINANT	MCL (µg/l)
Barium	200
Cadmium	5
Chromium	100
Mercury	2
Selenium	50
Antimony	6
Beryllium	4
Thallium	2
Arsenic	10

Table 1: Metals MCLs from Primary Drinking Water Standard (40CFR141.51)

CONTAMINANT	LEVEL (µg/l)
Aluminum	50 to 200
Copper	1000
Iron	300
Manganese	50
Silver	100
Zinc	5000

Table 2: Metals Levels from Secondary Drinking Water Standard (40CFR143.3)

The approved ICP-MS method for the determination of metallic contaminants for compliance measurements is the EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma Mass Spectrometry". Optical ICP is also frequently employed for such measurements using method 200.7, although the required detection limit for some analytes is problematic with this technique, e.g. antimony, arsenic, mercury and thallium. In fact, under the Arsenic Rule (part of 66 FR 6976, 2001) the EPA state that as of January 2006 optical ICP methods will be withdrawn from approval for the determination of arsenic.

This leaves graphite furnace atomic absorption spectrophotometry (GF-AAS), hydride generation atomic absorption spectrophotometry (HG-AAS) and ICP-MS as the only available techniques for this analysis. Most of these problematic analytes have frequently been analyzed by GF-AAS. Mercury is often determined using cold vapour generation atomic absorption spectrophotometry (CV-AAS). These techniques yield lower detection limits than optical ICP, but have the disadvantage of allowing the determination of only one analyte at a time and having a somewhat slower analysis time per sample. Consequently, many labs have moved away from atomic absorption techniques and are adopting ICP-MS as the preferred technique for all analytes.

ICP-MS method 200.8 is also increasing in its useful scope for regulatory analysis since the EPA recently proposed its use for regulatory measurements on wastewater samples for compliance with the National Pollutant Discharge Elimination System (NPDES) under the Clean Water Act (CWA) (40 CFR part 136). Method 200.2 is proposed as a general sample preparative method and method 200.8 is proposed for ICP-MS determinations (see FR/Vol.69/Tuesday April 6, 2004).

Large numbers of water samples are analyzed using this method in the USA. These include supplied waters and private waters. Some States also require well waters within properties to be analyzed prior to purchase of real estate. Method 200.8 has also been used as the basis of water analysis methods by ICP-MS across the world, especially in regions whose environmental monitoring industries developed later than that of the US.

## **Method 200.8 Summary**

### **Method Detection Limit**

Method 200.8 describes a protocol for determining the Method Detection Limit (MDL) as well as the linear dynamic range (LDR). For the MDL, the analyst sets up the instrument hardware and method as intended for the analysis, and then analyses a reagent blank solution spiked at 2-5 times the estimated detection limit. Seven repeats are required over three non-consecutive days. The Standard Deviation (SD) is determined for each of the sets and multiplied by 3.14 to arrive at the MDL. The data for MDL calculation must have all the required calculations included, e.g. interference correction equations, as these can have a substantial influence on the MDL. In general, the MDL is a function of sensitivity, background signal or contamination, and signal stability. The ICP-MS technique generally produces a high signal-to-background ratio and, when properly optimized, signal stability which allows for MDLs that easily meet most environmental analysis requirements with sufficient integration time. It is important that contamination is kept under control, especially for environmentally abundant elements such as Al, Zn, etc., since any contamination will degrade the MDL. Some elements are limited in their MDL by spectral background features. Interference equations also affect the MDL, since they employ the monitoring of additional peaks and, accordingly, propagate their errors.

### **Linear Dynamic Range**

Whilst MDL values are important, it is often at higher concentration ranges where ICP-MS technology is challenged to meet everyday sample needs. It would be desirable to employ an analytical technique capable of measuring tens to hundreds of mg/L in environmental samples, as concentration ranges can vary enormously from sample to sample. The upper-range limit is termed the Linear Dynamic Range (LDR). Method 200.8 defines the upper LDR to be the concentration at which an observed signal deviates by less than 10% from that extrapolated by the lower standards. Sample dilution can facilitate the measurement of high concentrations, but at additional effort, cost and error. The XSERIES 2 has a specialized interface design, the Xt interface, and a sensitivity attenuation technology in the form of a switchable resolution mode to allow calibrations for matrix elements, e.g. Na, K, Mg and Ca, well in excess of 100 mg/L.

### **Pre-run Validation**

The instrument performance must be validated prior to the sample run according to the 200.8 Method. This includes a 30 minute instrument warm-up, mass calibration, peak resolution and stability check using the tuning solution (a multi-element mix spanning the mass range, e.g. Be, Mg, Co, Y, In, Ba, Tb, Pb, U). Peak scanning of  $^{24,25,26}\text{Mg}$  and  $^{206,207,208}\text{Pb}$  is used to verify the mass calibration of these low and high mass elements and the peak width at 5% peak height is used as a resolution check. If the position of the measured peak maximum is  $>0.1$  amu from the datum position this indicates that a mass calibration must be performed to correct the mass position. The resolution must be set to approximately 0.75 amu at 5% peak height. The X Series' PlasmaLab software allows for these parameters to be conveniently checked using an automated sequence called a *Performance Report*.

### **Internal Standards**

ICP-MS methods use internal standardization to correct for matrix effects, particularly with environmental samples where high mg/L levels of matrix elements, relative to the calibration standards in dilute nitric acid, are frequently found. Internal standards also correct for long-term sensitivity drift associated with deposition of refractory matrix-based material around the cone orifices. The Xt interface on the XSERIES 2 was specifically designed to resist such matrix deposition, improving long-term stability. Method 200.8 lists nine candidate isotopes for internal standard use:  $^6\text{Li}$ ,  $^{45}\text{Sc}$ ,  $^{89}\text{Y}$ ,  $^{103}\text{Rh}$ ,  $^{115}\text{In}$ ,  $^{159}\text{Tb}$ ,  $^{165}\text{Ho}$ ,  $^{175}\text{Lu}$ , and  $^{209}\text{Bi}$ . It requires that at least three are used, spanning the mass range of interest and generally associated with analytes of similar mass. For acceptance as a valid 200.8 run, the responses for all the internal standards, relative to the original response of the calibration blank ("x") must stay within  $(x + 25\%)$  or  $(x - 40\%)$ . Anything outside of this range mandates greater dilution, rinsing or re-analysis. Internal standard is added either manually as a multi-element mix to each blank, standard and sample or may be combined automatically using a mixing "tee".

## Quality Control

Method 200.8 specifies a variety of quality control standards. These are summarised in Table 3.

CHECK CODE	CHECK NAME	PURPOSE	FREQUENCY	LIMITS
-	Tune: (Mass calibration and resolution verification)	Ensures correct mass is measured at its maximum and that peaks are properly resolved	Prior to analysis	masses measured must not deviate by more than 0.1 amu from their nominal position and peak width should be approximately 0.75 amu at 5 % peak height
	Tune: (Stability and precision verification)	Ensures the instrument is properly optimized and thermally stable	Prior to analysis	RSD < 5 % over 5 measurements
QCS	Quality Control Sample	Checks the accuracy of the calibration with a second source standard	Post calibration	90-110% recovery
LRB	Laboratory Reagent Blank	Checks the laboratory reagents and sample preparation process for contamination	1 per batch of 20 or fewer samples	No specific requirements
LFB	Laboratory Fortified Blank	Checks the recovery of analytes by spiking a known quantity into a blank	1 per batch of samples	85-115 % recovery or within $\pm 3$ standard deviations of the mean recovery
LFM	Laboratory Fortified Matrix	Checks recovery of analytes in a matrix by spiking a known quantity into a sample	1 in 10 samples	70-130 % recovery
CCV	Continuing Calibration Verification	A continuing check of accuracy and drift normally done by re-measuring a calibration standard as a sample	Every 10 analyses and at the end of the run	90-110 % recovery
CCB	Continuing Calibration Blank	A continuing check of the blank level by re-measuring the calibration blank as a sample	Every 10 analyses and at the end of the run	No specific requirements

Table 3: Summary of Method QC Requirements

## Experimental

### Equipment

An XSERIES 2 ICP-MS (Thermo Fisher Scientific, Bremen, Germany) was used in conjunction with an ASX-510 autosampler (Cetac, Omaha, Nebraska, USA). Internal standard was added on-line using a Y-piece (Thermo Scientific on-line internal standard addition kit P/N 4600431). The instrument was optimized using the autotune function when required. Typical instrument parameters are given in Table 4.

### Calibration Solutions

High purity reagents were used throughout. Ultra pure water of resistivity >18 M  $\Omega$  cm (Milli-Q) was used, along with ultra pure grade hydrochloric acid and super pure grade nitric acid (Romil, Cambridge, UK). All analytical solutions were prepared from ICP-MS grade stock standards from the EPA Method 200.8 Productivity Pack solutions and reference samples (NIST, Gaithersburg, MD, USA and LGC Promochem, Teddington, UK) were analyzed along with unknown water samples. Table 5 gives the calibration concentrations.

ANALYTES	WCS-1	WCS-2	WCS-3
Be, Al, V, Cr, Mn, Co, Ni, As, Se, Mo, Ag, Cd, Sb, Tl, Pb, Th, U	10	20	100
Na, Ca	10000	20000	100000
Mg, K	5000	10000	50000
Fe, Cu, Zn, Ba	100	200	1000
Hg	0.5	1	5

Table 5: Calibration Concentrations (all in ug/L)

PARAMETER	VALUE
RF Power (W)	1400
Cool Gas Flow (L/min)	13
Auxiliary Gas Flow (L/min)	0.8
Nebuliser Gas Flow (L/min)	0.85-0.90
Sample Uptake Rate (mL/min)	0.4 approx.
Sample Introduction System	Concentric nebulizer with low-volume impact bead spraychamber (not cooled) and one-piece torch (1.5mm ID injector)
Cones	Xt Design
Detector	Simultaneous pulse/analogue
Uptake Time	Monitored uptake, minimum 10 seconds, maximum 25 seconds at 50 rpm
Stabilization Delay	10 seconds at 15 rpm
Wash Time	Monitored, minimum 30 seconds, maximum 300 seconds at 50 rpm
Survey Runs	1 - scanning
Main Runs	3 - peak jumping
- Number of Points per Peak	1
- Dwell Time / Point	5 - 50 ms
- Number of Sweeps / Replicate	25
Internal Standardization Technique	$^6\text{Li}$ , Sc, In, Tb, Bi with interpolation.
Total Time per Sample	2:45 minutes (average)

Table 4: XSERIES 2 Parameters

## Instrument Checks

To ensure that the mass-calibration, resolution and stability requirements are met, the XSERIES 2 has critical hardware and PlasmaLab software features built-in. The hardware has two variable resolution modes allowing high and standard resolution settings to be defined. Standard resolution is typically set to give peaks of approximately 0.75 amu width at 5 % peak height, whilst high resolution mode is typically set to give around 0.4 amu. Normally the excellent abundance sensitivity specification of the XSERIES 2 quadrupole will allow low concentration analytes to be measured next to very large interferences at peak width settings of 0.75 amu. However, these settings may be adjusted within the software to ensure no peak-tailing from high concentration species affects measurement of adjacent mass low concentration analytes. PlasmaLab allows the instrument to be automatically mass-calibrated whenever required. The mass calibration, the peak widths, and the precision over five measurements may be checked using a Performance Report. The Performance Report is a user definable test that may be run as part of an analytical method or separately as a setup function. The 200.8 Productivity Pack supplies a custom Performance Report designed to meet the requirements of method 200.8. Figures 1 and 2 show data from a Performance Report in the format in which it is generated.

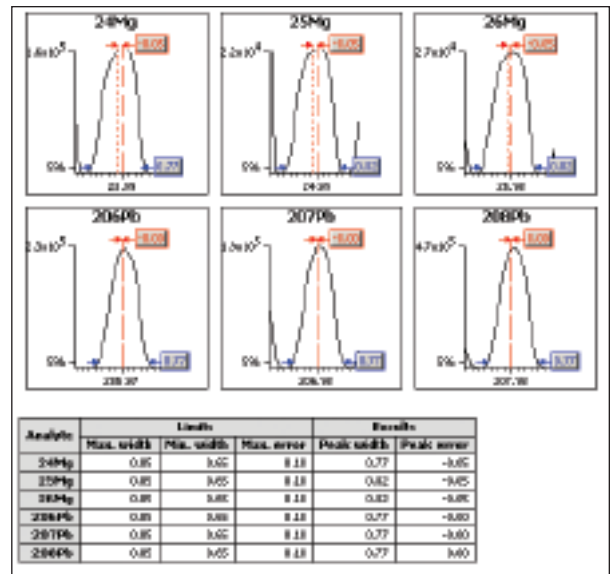


Figure 1: Plasmalab Performance Report for Mmethod 200.8, showing mass calibration and resolution check

Run	Time	58kg	7Li	9Be	24Mg	25Mg	26Mg	59Co	137Ba++	101Bkg
Dwell (mSecs)		100.0	3.0	3.0	3.0	3.0	3.0	3.0	2.0	100.0
Limits	9σRSD	-	5.0%	-	5.0%	-	-	-	-	-
	Concntrate	<1	>5010	-	>5000	>5000	>500	>500	-	<1
1	18:21:32	0.100	124819.73	23870.415	170884.91	22508.522	27188.663	25888.54	1760.093	0.810
2	18:21:43	0.400	122112.38	24291.168	169834.26	23216.158	27161.953	259917.70	1520.063	0.810
3	18:21:55	0.100	122206.40	25285.838	168177.58	22842.309	27302.344	259890.62	1560.073	0.480
4	18:22:06	0.400	121790.03	24818.465	169160.79	22950.530	26134.598	26055.419	1420.060	0.480
5	18:22:17	0.100	123777.93	24003.940	173067.83	22628.685	27729.716	25600.450	1460.064	0.810
x		0.160	122961.29	24473.964	170365.08	23029.241	27163.455	25905.111	1544.072	0.320
σ		0.22	1339.35	585.21	2142.79	581.00	398.60	1804.39	132.22	0.33
%RSD		136.131	1.088	2.391	1.258	2.523	1.467	0.697	8.563	104.513

Run	Time	115In	137Ba	140Ce	156Ce O	206Pb	207Pb	208Pb	220Bkg	238U
Dwell (mSecs)		3.0	3.0	3.0	3.0	3.0	3.0	3.0	101.0	2.0
Limits	9σRSD	5.0%	-	-	-	-	-	5.0%	-	-
	Concntrate	>5000	-	-	-	>10000	>10100	>10000	-	<1
1	18:21:32	664078.24	75075.376	631778.25	9415.992	233477.31	193139.30	477970.11	0.000	910910.59
2	18:21:43	655993.96	72825.426	629162.02	8188.678	234477.87	194865.89	476018.92	0.000	920882.18
3	18:21:55	657602.24	73736.084	619282.12	7881.863	226610.16	192330.03	474609.42	0.000	904895.15
4	18:22:06	641111.36	74740.548	627030.58	8108.639	230475.97	195985.57	472949.90	0.000	918109.34
5	18:22:17	651447.47	76056.522	623298.99	8762.303	225005.22	195864.16	476077.07	0.000	903840.16
x		653644.65	74488.790	626098.38	8471.485	232009.31	194436.99	474423.09	0.000	911747.16
σ		8463.87	1248.00	4928.33	619.57	3480.21	1638.76	2865.43	0.00	7579.12
%RSD		1.295	1.675	0.787	7.315	1.504	0.845	0.604	0.000	0.842

Ratio results			
Run	Time	137Ba++ / 137Ba	156Ce O / 140Ce
Ratio limits		<0.0100	<0.0200
1	18:21:32	0.023	0.015
2	18:21:43	0.021	0.013
3	18:21:55	0.021	0.013
4	18:22:06	0.019	0.013
5	18:22:17	0.019	0.014
x		0.0207	0.0135
σ		0.00	0.00
%RSD		8.6686	6.8902

Result : The performance report passed.

Figure 2: Performance Report showing precision check

## Pre-run Validation

In order to validate the performance of the instrument, a linear dynamic range (LDR), instrument detection limit (IDL) and a method detection limit (MDL) study was performed. The IDL and MDL study was performed in the chloride-containing digestion matrix of 0.8 % HNO<sub>3</sub>, 0.4 % HCL, as in method 200.2, in order to give a worst case result due to the effect of chloride-based interferences. Table 6 shows the results.

ANALYTE	m/z	LDR (ppm)	IDL (ppb)	MDL (ppb)
Ag	107	300	0.01	0.01
Al	27	800	0.01	0.02
As	75	5000	0.02	0.06
Ba	137	2000	0.02	0.07
Be	9	5000	0.009	0.04
Cd	111	2000	0.007	0.02
Co	59	700	0.005	0.01
Cr	52	800	0.03	0.02
Cu	65	3000	0.01	0.02
Hg	202	0.02	0.025	0.019
Mn	55	400	0.007	0.01
Mo	98	1500	0.01	0.03
Ni	60	3000	0.01	0.03
Pb	206, 207, 208	150	0.002	0.006
Sb	123	1000	0.02	0.03
Se	82	5000	0.2	0.2
Th	232	150	0.004	0.01
Tl	205	150	0.002	0.005
U	238	150	0.002	0.007
V	51	800	0.01	0.02
Zn	66	3000	0.03	0.08
Ca*	44	5000	1	4
Fe*	56	5000	3	3
K*	39	5000	3	2
Mg*	25	5000	0.5	1
Na*	23	1500	1	3

\* Not included in the method 200.8 analyte list, but measured for information

Table 6: LDR, IDL and MDL Results

## Performance Evaluation

The performance of the XSERIES 2 ICP-MS for method 200.8 was evaluated by analyzing various certified reference water samples (see Table 7) under the method's QC protocol. Results for the reference samples, QCs and spikes are given in the next section.

REFERENCE MATERIAL	DESCRIPTION
ERML-CA010	Hard drinking water
ERML-CA021	Soft drinking water
NWTMRAIN-95	Simulated rain water
BCR-610	Ground water
BCR-616	Artificial ground water
NRCORMS-2	Hg in river water
SPS-WW1	Waste water
LGC6175	Landfill leachate
LGC6177	Landfill leachate
SLRS-4	River water
NIST 1640	River water
NIST 1641d	Hg in water

Table 7: Reference Samples Analysed

## Results and Discussion

ANALYTE	QCS (N=6)			CCV (N=15)		
	X	SD	REC %	X	SD	REC %
Ag	47.9	1.05	96	19.8	0.204	99
Al	49.1	1.57	98	19.9	0.389	100
As	49.9	1.33	100	20.7	0.401	103
Ba	488	10.6	98	194	3.67	97
Be	50.0	2.09	100	20.1	0.884	101
Cd	47.3	1.28	95	19.9	0.513	100
Co	49.4	1.60	99	20.2	0.263	101
Cr	48.3	1.54	97	19.6	0.319	98
Cu	478	13.0	96	196	3.03	98
Hg	2.43	0.08	97	1.00	0.03	100
Mn	49.5	1.42	99	19.8	0.265	99
Mo	49.4	1.15	99	19.8	0.259	99
Ni	47.3	0.917	95	19.7	0.443	99
Pb	48.3	1.30	97	19.0	0.263	95
Sb	49.6	1.19	99	20.0	0.394	100
Se	48.0	0.676	96	20.4	0.670	102
Th	52.1	2.56	104	19.6	0.819	98
Tl	49.4	1.37	99	19.5	0.397	98
U	51.8	2.40	104	20.0	0.345	100
V	48.0	1.32	96	19.5	0.148	97
Zn	477	9.88	95	199	2.74	100
Ca*	49.0	1.81	98	19.8	0.274	99
Fe*	494	16.8	99	198	2.50	99
K*	24.7	1.04	99	9.9	0.222	99
Mg*	25.0	0.886	100	10.0	0.196	100
Na*	49.8	1.78	100	19.9	0.275	100

Table 8: Results for QCS, and CCV Solutions

The XSERIES 2 produced consistently accurate results with little response drift, as shown by the results for the QCS and CCV solutions. The results were consistently within the allowed range of 90-110 % of the known value.

ANALYTE	UNSPIKED		SPIKED		SPIKE LEVEL	SPIKE RECOVERY %
	X	SD	X	SD		
Ag	2.03	0.016	50.5	0.136	50	97
Al	2.54	0.001	50.3	0.729	50	95
As	2.12	0.033	53.6	0.830	50	103
Ba	27.4	0.332	552	8.26	500	105
Be	1.95	0.066	51.4	1.43	50	99
Cd	2.00	0.024	51.6	0.416	50	99
Co	1.97	0.038	51.1	0.904	50	98
Cr	1.91	0.017	50.0	0.927	50	96
Cu	23.6	0.508	502	10.7	500	96
Hg	0.08	0.03	2.54	0.04	2.50	98
Mn	1.99	0.011	51.8	0.961	50	100
Mo	2.10	0.003	52.3	0.774	50	100
Ni	2.02	0.052	49.4	1.30	50	95
Pb	1.90	0.005	51.4	0.161	50	99
Sb	2.21	0.050	52.8	1.15	50	101
Se	2.25	0.130	53.1	1.47	50	102
Th	1.95	0.172	53.3	2.53	50	103
Tl	1.98	0.045	53.8	1.26	50	104
U	2.10	0.041	52.5	0.514	50	101
V	2.21	0.224	50.5	0.603	50	97
Zn	20.7	0.236	511	7.02	500	98
Ca*	49.5	0.462	100	1.61	50	102
Fe*	15.3	0.775	516	8.68	500	100
K*	2.07	0.031	26.6	0.355	25	98
Mg*	3.25	0.032	27.8	0.254	25	98
Na*	6.46	0.044	55.5	0.125	50	98

Table 9: Results of LFM

The accurate results for the LFM sample show that quantitative recovery can be achieved in both blank and matrix-containing solutions. The LFM was performed on a hard drinking water sample (see Table 9).

ANALYTE	ERML-CA010				ERML-CA021				TMRAIN-95			
	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %
Ag	5.90	0.05	6.2	95	7.55	0.02	7.6	99	0.31	0.02		
Al	188	2	208	90	185	4	193	96	1.70	0.04	1.7	100
As	57.3	0.7	55	104	10.5	0.4	9.9	106	1.10	0.03	1.07	103
Ba	117	1	116	100	99	1	101	98	0.67	0.06	0.73	92
Be	<MDL	0.01			<MDL	0.008			0.26	0.03	0.27	97
Cd	<MDL	0.01			4.57	0.09	4.4	104	0.48	0.01	0.48	99
Co	0.08	0.01			0.02	0.01			0.21	0.01	0.22	97
Cr	41.6	0.6	48	87	43.5	0.7	45.3	96	0.73	0.02	0.79	92
Cu	72	2			1930	51	1975	98	6.0	0.2	6.2	96
Mn	48.0	0.9	48	100	47.9	0.7	48.2	99	6.1	0.1	6.1	100
Mo	0.76	0.02			0.04	0.01			0.13	0.02	0.17	74
Ni	43	2	48	90	17.1	0.6	18.3	93	0.75	0.03	0.8	93
Pb	91.92	0.07	95	97	21.9	0.2	23.7	92	0.24	0.01	0.29	83
Sb	12.0	0.2	11.9	101	5.2	0.2	4.9	106	0.35	0.03	0.35	100
Se	10.4	0.2	9.5	109	10.1	0.2	9.5	106	0.55	0.04	0.74	74
Th	<MDL	0.01			<MDL	0.01			<MDL	0.01		
Tl	<MDL	0.007			<MDL	0.007			0.30	0.01		
U	0.41	0.02			<MDL	0.006			0.230	0.007	0.25	92
V	1.0	0.3			0.3	0.2			0.66	0.04	0.64	103
Zn	572	14	542	106	562	10	514	109	11.0	0.2	11.1	99
Ca*	79.6	0.9	83.2	96	14.4	0.2	14.7	98	0.617	0.004		
Fe*	224	3	236	95	183	3	189	97	22	1	24.2	91
K*	4.93	0.04	5.1	97	1.17	0.02	1.16	101	0.040	0.002		
Mg*	4.15	0.03	4.2	99	1.23	0.02	1.27	97	0.178	0.001		
Na*	21.3	0.2	21.9	97	8.19	0.09	8.1	101	0.132	0.004		

Table 10: Results and Recoveries for Reference Samples

ANALYTE	BCR-610				BCR-616				SPS-WW1			
	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %
Ag	<MDL	0.02			<MDL	0.02			<MDL	0.1		
Al	147	3	159	92	212	3			1827	36	2000	91
As	11.8	0.3	10.8	109	<MDL	0.03			101	2	100	101
Ba	36.9	0.5			1.29	0.03			1.2	0.4		
Be	<MDL	0.01			<MDL	0.009			<MDL	0.05		
Cd	3.22	0.06	2.94	110	<MDL	0.009			19.9	0.5		
Co	<MDL	0.01			<MDL	0.008			58.5	0.9	60	98
Cr	<MDL	0.01			0.34	0.03			187	3	200	94
Cu	44	1	45.7	95	0.3	0.2			390	5	400	97
Mn	32.6	0.7			18.4	0.2	19.7	93	382	6	400	96
Mo	0.10	0.01			0.06	0.01			<MDL	0.04		
Ni	20.4	0.9			<MDL	0.03			942	23	1000	94
Pb	7.86	0.05	7.78	101	0.17	0.01			91	2	100	91
Sb	<MDL	0.01			<MDL	0.01			<MDL	0.06		
Se	0.4	0.1			<MDL	0.1			<MDL	0.7		
Th	<MDL	0.008			0.02	0.01			<MDL	0.05		
Tl	<MDL	0.007			<MDL	0.007			<MDL	0.03		
U	<MDL	0.007			<MDL	0.008			<MDL	0.03		
V	0.2	0.2			0.3	0.2			95	1	100	95
Zn	0.30	0.09			0.94	0.03			607	8	600	101
Ca*	74	2			35.9	0.5	38.5	93	<MDL	0.03		
Fe*	2526	56			48	2			937	13	1000	94
K*	6.0	0.2			0.59	0.01	0.58	101	<MDL	0.01		
Mg*	32.8	0.6			22.8	0.3	23.9	95	<MDL	0.01		
Na*	52	1			60.2	0.4	61.5	98	<MDL	0.04		

Table 11: Results and Recoveries for Reference Samples

ANALYTE	LGC 6175				LGC 6177				SLRS-4				NIST 1640			
	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %
Ag	0.1	0.1			<MDL	0.4			0.02	0.01			7.39	0.06	7.62	97
Al	29	1			122	2			52	1	54	97	53	1	52	101
As	9.3	0.9			100	3			0.79	0.09	0.68	116	27.7	0.6	26.67	104
Ba	497	11			761	10			12.9	0.3	12.2	106	145	3	148	98
Be	<MDL	0.1			<MDL	0.2			<MDL	0.002	0.007		34	1	34.94	98
Cd	<MDL	0.09			0.2	0.2			<MDL	0.001	0.012		23.6	0.4	22.79	104
Co	10.4	0.2			42	1			0.031	0.005	0.033	94	19.5	0.1	20.28	96
Cr	10.94	0.08			158	4	180	88	0.28	0.02	0.33	84	38.0	0.6	38.6	99
Cu	257	7			33	1			1.46	0.06	1.81	81	81	2	85.2	96
Mn	326	6	330	99	142	3	140	101	3.38	0.09	3.37	100	118	2	121.5	97
Mo	1.8	0.1			6.4	0.3			0.262	0.009	0.21	125	43.8	0.5	46.75	94
Ni	75	4	90	84	229	5	210	109	0.62	0.03	0.67	93	25	1	27.4	92
Pb	47.1	0.3			17.0	0.2			0.080	0.003	0.086	93	26.0	0.2	27.89	93
Sb	2.0	0.1			5.0	0.2			0.27	0.01	0.23	118	13.8	0.3	13.79	100
Se	19	1			39	6			<MDL	0.1			23.2	0.3	21.96	106
Th	0.01	0.06			<MDL	0.2			0.011	0.007			0.08	0.01		
Tl	<MDL	0.07			<MDL	0.1			<MDL	0.005			0.023	0.004		
U	<MDL	0.07			0.3	0.1			0.041	0.006	0.05	81	0.78	0.02		
V	4	2			65	2			0.35	0.04	0.32	109	12.5	0.1	12.99	96
Zn	268	6	280	96	252	7	260	97	1.04	0.05	0.93	112	55.6	0.7	53.2	104
Ca*	152	3	148	102	74	2	74.8	99	5.63	0.08	6.2	91	6.9	0.1	7.045	98
Fe*	1011	17	1050	96	3801	31	3800	100	93	2	103	90	32.5	0.5	34.3	95
K*	375	9	385	97	775	17	780	99	0.63	0.02	0.68	93	0.93	0.02	0.994	94
Mg*	215	4	221	97	73	1	73.5	99	1.57	0.02	1.6	98	5.64	0.09	5.189	109
Na*	835	11	860	97	1763	8	1750	101	2.20	0.01	2.4	92	28.7	0.3	29.35	98

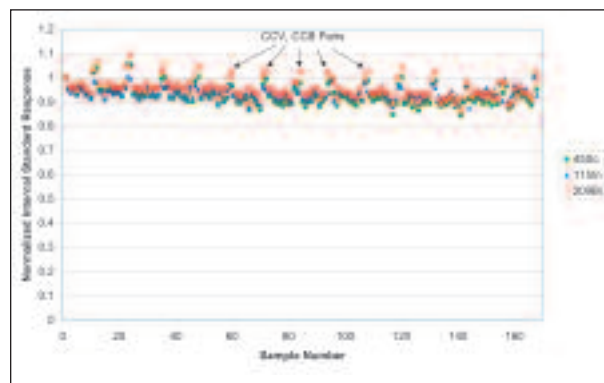
Table 12: Results and Recoveries for Reference Samples

ANALYTE	NRC ORMS-2				NIST 1641D (1:1000 DILUTION)			
	X	SD	CERTIFIED	RECOVERY %	X	SD	CERTIFIED	RECOVERY %
Hg	0.038	0.009	0.036	106	1.65	0.04	1.59	103

Table 13: Results and Recoveries for Reference Samples for Mercury

The results in Tables 10-13 show that the XSERIES 2 consistently produces accurate and precise data in all drinking water and environmental water matrices, from low ng/L levels up to high mg/L levels.

Graph 1 shows the recovery of the internal standard isotopes, relative to the calibration blank, during a typical analytical run. It is seen that all internal standards stay well within the allowed range of 60 – 125 %. This is due to the use of the Xt interface technology in the XSERIES 2. This promotes stability when running typical environmental samples, meaning that sensitivity is not lost with time, therefore MDLs are not compromised, and there is less drift, therefore more QCs pass and fewer samples require reanalysis. The samples analyzed were replicates of the hard water sample used for the LFM.



Graph 1: Internal Standard Behaviour for a Typical Run

## Conclusions

The XSERIES 2 demonstrates compliance with the requirements of EPA Method 200.8 for a wide range of water sample types and easily copes with its stringent AQC requirements. A combination of specifically designed hardware and software tools enables and simplifies compliant analysis as outlined below.

Mass calibration and resolution checking is made simple with the custom Performance Report and peaks are easily set to the required width using the variable resolution function. The Performance Report also monitors and records the precision over five measurements, allowing a "Tune" sample to be tagged to each analytical run. Any deviations from acceptable performance are clearly flagged in red and the report ends with a simple, unambiguous Pass or Fail statement.

The unique Xt interface design produces low background equivalent concentrations, resulting in very low instrument and method detection limits (as seen in Table 6). It reduces the effective contribution of polyatomic species allowing robust, reproducible interference correction, and enhances stability when analyzing solutions containing high levels of matrix components, e.g. Ca, Na, Fe, Mg, K. This is demonstrated by the consistent CCV results and the stability of the internal standards. The unique response properties of this interface technology coupled with the dual mode simultaneous detector and, when necessary, high resolution mode, allow unrivalled linear range. This further improves productivity by reducing the number of dilutions required.

The PlasmaLab software has a built-in QC checking capability that is specifically designed to meet the requirements of EPA methods. Each QC type (ICV, CCV, LFM, etc) is available as a default in the QC set-up page and the user can also define his/her own QC tests, as required. The results display page visually flags results that are outside the allowed range making validation a simple process. Percentage recoveries can be automatically calculated for any QC sample or spiked sample and percentage differences can be calculated for DUP and SER samples. A variety of user-selectable automated actions can be set-up to ensure fully compliant analysis is achieved during an unattended run. PlasmaLab enables automated initiation of measurement and completion of washout using the intelligent Monitored Uptake / Washout features. This reduces the amount of non-productive time and maximizes useful analytical time.

The productivity tools in PlasmaLab in combination with the rapidity of the XSERIES 2 quadrupole and the low-volume sample introduction system result in the fastest analysis with complete compliance. Samples in this study were being processed at a speed of 1 sample every 2 minutes and 45 seconds, or 22 samples per hour. This makes the XSERIES 2 the ultimate ICP-MS for cost-effective elemental analysis.

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