

Environmental Series - US EPA Method 200.7 using the iCAP 6500 Duo ICP

Bill Spence¹ and Matthew Cassap²

¹Thermo Fisher Scientific, Ion Path, Road 3, Winsford, Cheshire, CW7 3GA, UK

²Thermo Fisher Scientific, Solaar House, 19 Mercers Row, Cambridge, CB5 8BZ, UK

Introduction

This Application Note describes the use of the Thermo Scientific iCAP 6500 Duo for the analysis of waters using the US EPA method 200.7.

2. 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. 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 Development and Application of Method 200.7

The Safe Drinking Water Act (SDWA) (last amended 1996) gave the US EPA power to set and regulate national standards for the quality of supplied drinking water and drinking water resources, 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 measurement and their MCLs.

Contaminant	MCL	Contaminant	Level
Antimony	0.006	Aluminum	0.05 to 0.2
Arsenic	0.01	Copper	1
Barium	2	Iron	0.3
Beryllium	0.004	Manganese	0.05
Cadmium	0.005	Silver	0.1
Chromium	0.1	Sulfate	250
Copper	1.3	Zinc	5
Lead	0.015		
Mercury	0.002		
Selenium	0.05		
Thallium	0.002		
Uranium	0.03		

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

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

The approved ICP method for the determination of metallic contaminants for compliance measurements is the EPA Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry". Optical ICP is 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. Under the Arsenic Rule (part of 66 FR 6976, 2001) the EPA stated that as of January 2006 optical ICP methods were to be withdrawn from approval for the determination of arsenic since the typical detection limit of the technique was not generally thought to be routinely low enough to measure confidently at the MCL level of 10 µg/L. 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 vapor generation atomic absorption spectrophotometry (CV-AAS). This technique yields a lower detection limit than optical ICP, but has the disadvantage of allowing the determination of only one analyte at a time and of having a slower analysis time per sample.

Thermo Scientific: Your partner for enhanced environmental analysis

For information on other Thermo Scientific offerings for environmental trace element analysis or to keep up to date with legislative and environmental analysis developments, please visit our Environmental Resource Centre (ERC) at www.thermo.com/erc

Key Words

- ICP
- Water
- Environmental
- EPA Method 200.7

ICP method 200.7 is also extensively used for regulatory analysis of wastewater samples for compliance with the permits issued within the National Pollutant Discharge Elimination System (NPDES) under the Clean Water Act (CWA) (40 CFR part 136).

Large numbers of water samples are analyzed using this method. These include supplied waters, natural waters and waste waters. Some states also require well waters within properties to be analyzed prior to purchase of real estate and the method is commonly used for this purpose. Method 200.7 has also been used as the basis of water analysis methods by ICP across the world, especially in regions whose environmental monitoring industries developed later than that of the US.

Method 200.7 Summary

Method 200.7 describes a method for the determination of 32 elements in water samples. It suggests preferred wavelengths, calibration and quality control procedures as well as specifying procedures for determining method performance characteristics, such as detection limits and linear ranges. This section provides a brief overview of the procedures described.

Method Detection Limit

Method 200.7 describes a protocol for determining the Method Detection Limit (MDL). The instrument hardware and method are set up as intended for the analysis. A reagent blank solution spiked at 2-3 times the estimated instrument detection limit is subjected to seven replicate analyses. The Standard Deviation (SD) of the measured concentrations is determined and multiplied by 3.14 (the Student's t value for a 99 % confidence interval for 6 degrees of freedom) to arrive at the MDL. It is important that contamination is kept under control, especially for environmentally abundant elements such as Al and Zn, since any contamination will degrade the MDL.

Interference corrections also affect the MDL, since they employ the monitoring of additional lines and propagate the measurement errors accordingly.

Linear Dynamic Range

The upper linear range limit of a calibration is termed the Linear Dynamic Range (LDR). Method 200.7 defines the upper LDR to be the concentration at which an observed signal deviates by less than 10 % from that extrapolated from lower standards. Sample dilution can facilitate the measurement of high concentrations, but with additional effort, cost and error and therefore a wide LDR is desirable.

Quality Control

Method 200.7 specifies a variety of quality control standards. These are summarised in Table 3.

Experimental

3.1 Equipment

A Thermo Scientific iCAP 6500 Duo ICP was used in conjunction with an ASX-520 autosampler. Internal standard (5ppm Y) was added on-line using a Y-connector. Typical instrument parameters are given in Table 4.

3.2 Instrumental Method

Wavelengths were selected based on those suggested in method 200.7. Additional wavelengths were measured in some cases. Plasma views were selected using the iTEVA software's automated view selection function. Axial and radial views were selected to provide optimum data quality by avoiding easily ionized element interferences in the axial view where necessary. Table 5 shows the elements, wavelengths and views used.

Check Code	Check Name	Purpose	Frequency	Limits
QCS	Quality Control Standard	Checks the accuracy of the calibration with a second source standard	Post calibration	95-105 % recovery
SIC	Spectral Interference Check Solution(s)	Checks for the presence of spectral interference and the effectiveness of inter-element corrections	Periodically	No specific requirements
IPC	Instrument Performance Check	A continuing check of accuracy and drift normally done by re-measuring a standard as a sample	Every 10 analyses and at the end of the run	95-105 % recovery immediately following calibration; 90-110 % recovery thereafter
BLANK	Check 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	<IDL
LRB	Laboratory Reagent Blank	Checks the laboratory reagents and sample preparation process for contamination	1 per batch of 20 or fewer samples	< 2.2 x MDL
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 ± 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 real sample	1 in 10 samples	85-115 % recovery

Table 3: Summary of method 200.7 QC requirements

Parameter	Setting
Pump Tubing	Tygon Orange/White sample White/White drain
Pump rate	50 rpm
Nebulizer	Concentric
Nebulizer Argon Pressure	0.65 L/min / 26 MPa
Spray Chamber	Cyclonic
Centre tube	2.0 mm
Torch Orientation	Duo
RF Forward Power	1150 W
Coolant flow	12 L/min
Auxiliary flow	0.5 L/min
Integration time	15 seconds (UV and VIS)

Table 4: iCAP Parameters

3.3 Solution Preparation

Ultra pure water of resistivity >15MΩ cm (Milli-Q) was used, along with Fisher Scientific Primar grade hydrochloric acid and nitric acid (Thermo Fisher Scientific, Loughborough, UK). All analytical standard solutions were prepared from Fisher Scientific stock standards (Thermo Fisher Scientific) and reference samples (NIST, Gaithersburg, MD, USA and LGC Promochem, Teddington, UK) were analyzed along with various unknown water samples. All samples were preserved in a mixture of 2 % nitric acid and 2 % hydrochloric acid.

3.4 Calibration Solutions

Table 6 gives the calibration solution concentrations.

Calibration Conc. (mg/L)	Analytes
0.25	Ag
1	As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V, Zn
1.2	Hg
2	Al, Ba, Be, Li, Mg, Na
4	B
10	Ca, K, P, SO ₄ , SiO ₂

Table 6: Calibration Concentrations

Element	Wavelength (nm)	View	Element	Wavelength (nm)	View
Aluminum (Al)	308.215	Radial	Molybdenum (Mo)	202.030, 203.844	Axial
Antimony (Sb)	206.833	Axial	Nickel (Ni)	221.647, 231.604	Axial
Arsenic (As)	189.042, 193.759	Axial	Phosphorus (P)	177.495, 214.914	Axial
Barium (Ba)	493.409	Radial	Potassium (K)	766.490	Radial
Beryllium (Be)	313.042, 313.107	Radial	Selenium (Se)	196.090	Axial
Boron (B)	249.678	Radial	Silica (SiO ₂)	251.611	Radial
Cadmium (Cd)	226.502, 228.802	Axial	Silver (Ag)	328.068	Radial
Calcium (Ca)	315.887	Radial	Sodium (Na)	588.995	Radial
Chromium (Cr)	205.552	Axial	Strontium (Sr)	407.771, 421.552	Radial
Cobalt (Co)	228.616	Axial	Sulfate (SO ₄)*	182.034	Axial
Copper (Cu)	324.754	Radial	Thallium (Tl)	190.856	Axial
Iron (Fe)	259.940	Radial	Tin (Sn)	189.989	Axial
Lead (Pb)	220.353	Axial	Titanium (Ti)	334.904, 334.941	Radial
Lithium (Li)	670.784	Radial	Vanadium (V)	292.402	Radial
Magnesium (Mg)	279.553	Radial	Zinc (Zn)	213.856	Axial
Manganese (Mn)	257.610	Radial	Yttrium (Y) [#]	224.306	Axial
Mercury (Hg)	184.950, 194.227	Axial	Yttrium (Y) [#]	371.030	Radial

* Not specified in method 200.7, but frequently of interest

Internal standard

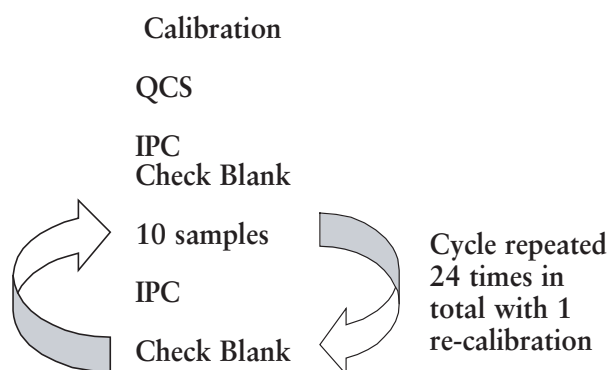
Table 5: Elements, wavelengths and views selected

3.5 Analytical Procedure

A linear dynamic range (LDR) and method detection limit (MDL) study was performed as described in method 200.7. The MDL study was performed with a reagent blank spiked with low concentrations of each element.

An interference study was performed using single element SIC solutions as described in method 200.7.

To demonstrate the performance of the iCAP 6500 Duo for typical routine analysis of a variety of water samples with method 200.7, a sequence was set up as follows:



The 10 samples analysed between each IPC and blank pair consisted of a variety of aqueous matrices. Table 7 gives details of these samples. The samples were analyzed multiple times throughout the analysis, replicating a run consisting of a total number of 240 samples (>300 samples, including QC and calibration solutions). Samples denoted 'unknown' in Table 7 were also spiked for analysis as a laboratory fortified matrix (LFM). Spike recoveries for these are given in the following section.

Reference Material/Sample	Description
NIST 1640	River water CRM
BCR-610	Ground water CRM
ERML-CA010A	Hard drinking water CRM
ERML-CA021A	Soft drinking water CRM
SPS-WW1	Waste water CRM
SPS-WW2	Waste water CRM
LGC6177	Landfill leachate CRM
NIST 1641d	Mercury in water CRM
Unknown drinking water sample	Hard tap water
Unknown leachate sample	Landfill leachate diluted 1:10
Unknown saline sample	Estuarine water diluted 1:5
Unknown soil digest sample	Aqua regia soil extract, 0.5 g/L

Table 7: Reference materials and samples analyzed

Results and Discussion

Analyte and line (nm)	SIC Solution	Contribution (mg/L)
Al 308.2	50 ppm V	0.8
Al 308.2	50 ppm Mo	0.4
Sb 206.8	50 ppm Cr	0.9
Zn 213.8	50 ppm Ni	0.2

Table 8: Major interferences and their contributions

Analyte	View*	LDR (mg/L)	Achievable 3-s IDL with blank **	Robust 3.14-s MDL with MDL solution	Drinking Water Level of Interest
Ag 3280 nm	R	>2	4 (0.7)	10	100
Al 3082 nm	R	>200	40 (5)	100	50-200
As 1890 nm	A	>50	2	4	10
As 1937 nm	A	>50	2	4	10
B_ 2496 nm	R	>50	6 (1)	10	-
Ba 4934 nm	R	>50	1.5	3	2000
Be 3130 nm	R	>50	0.2 (0.03)	1	4
Be 3131 nm	R	>50	0.3 (0.05)	4	4
Ca 3158 nm	R	>500	8 (1)	20	-
Cd 2265 nm	A	>50	0.1	0.2	5
Cd 2288 nm	A	>50	0.1	0.2	5
Co 2286 nm	A	>50	0.3	0.5	-
Cr 2055 nm	A	>50	0.1	0.2	100
Cu 3247 nm	R	>50	3 (0.5)	4	1000
Fe 2599 nm	R	>300	2 (0.3)	3	300
Hg 1849 nm	A	>2	0.5	2	2
Hg 1942 nm	A	>2	0.5	1	2
K_ 7664 nm	R	>200	40 (5)	100	-
Li 6707 nm	R	>50	1 (0.1)	2	-
Mg 2795 nm	R	>50	0.05 (0.008)	0.1	-
Mn 2576 nm	R	>50	0.5 (0.08)	0.8	50
Mo 2020 nm	A	>50	0.3	0.5	-
Mo 2038 nm	A	>50	0.5	1	-
Na 5889 nm	R	>200	20 (3)	70	-
Ni 2216 nm	A	>50	0.1	0.2	-
Ni 2316 nm	A	>50	0.3	0.5	-
P_ 1774 nm	A	>50	1	2	-
P_ 2149 nm	A	>50	2	6	-
Pb 2203 nm	A	>50	1	2	15
S_ 1820 nm	A	>50	2	10	250,000
Sb 2068 nm	A	>50	1	4	6
Se 1960 nm	A	>50	2	4	50
Si 2516 nm	R	>50	8 (1)	20	-
Sn 1899 nm	A	>50	0.4	2	-
Sr 4077 nm	R	>50	0.07 (0.01)	0.2	-
Sr 4215 nm	R	>50	0.2 (0.02)	0.4	-
Ti 3349 nm	R	>50	2 (0.3)	4	-
Ti 3349 nm	R	>50	2 (0.3)	3	-
Tl 1908 nm	A	>50	1	2	2
V_ 2924 nm	R	>50	3 (0.5)	6	-
Zn 2138 nm	A	>50	0.2	0.6	5000

*R = Radial, A = Axial

** Bracketed figures show axial view values for radially viewed elements

Table 9: LDR, IDL and MDL results (µg/L, except where indicated)

Analyte	Mean	QCS (n=2)		Mean	Known	IPC (n=25)		SD	RSD %
		Known	Rec %			Rec %			
Ag 3280 nm	0.249	0.25	100	0.359	0.35	103	0.02	4.4	
Al 3082 nm	1.07	1	107	1.95	2	98	0.08	3.9	
As 1890 nm	0.982	1	98	1.98	2	99	0.07	3.6	
As 1937 nm	0.987	1	99	1.99	2	99	0.07	3.5	
B_ 2496 nm	1.06	1	106	3.88	4	97	0.07	1.8	
Ba 4934 nm	0.991	1	99	1.95	2	98	0.05	2.4	
Be 3130 nm	0.983	1	98	1.91	2	95	0.06	3.2	
Be 3131 nm	0.991	1	99	1.93	2	96	0.07	3.9	
Ca 3158 nm	1.04	1	104	2.03	2	101	0.06	3.0	
Cd 2265 nm	0.983	1	98	1.92	2	96	0.06	3.2	
Cd 2288 nm	0.986	1	99	1.90	2	95	0.05	2.7	
Co 2286 nm	0.985	1	99	1.95	2	97	0.06	3.3	
Cr 2055 nm	0.994	1	99	2.00	2	100	0.03	1.6	
Cu 3247 nm	1.00	1	100	1.88	2	94	0.06	3.3	
Fe 2599 nm	0.984	1	98	1.84	2	92	0.02	1.1	
Hg 1849 nm	0.603	0.6	101	1.19	1.2	99	0.06	4.8	
Hg 1942 nm	0.604	0.6	101	1.20	1.2	100	0.05	4.2	
K_ 7664 nm	5.32	5	106	9.93	10	99	0.15	1.5	
Li 6707 nm	1.03	1	103	2.08	2	104	0.07	3.3	
Mg 2795 nm	1.04	1	104	1.97	2	99	0.08	4.0	
Mn 2576 nm	1.02	1	102	1.91	2	96	0.05	2.6	
Mo 2020 nm	0.995	1	99	1.99	2	100	0.03	1.6	
Mo 2038 nm	0.985	1	99	2.00	2	100	0.07	3.7	
Na 5889 nm	1.11	1	111	2.03	2	102	0.10	4.8	
Ni 2216 nm	0.991	1	99	1.98	2	99	0.05	2.5	
Ni 2316 nm	0.984	1	98	1.98	2	99	0.07	3.6	
P_ 1774 nm	4.98	5	100	10.1	10	101	0.31	3.1	
P_ 2149 nm	4.91	5	98	10.0	10	100	0.40	4.0	
Pb 2203 nm	0.982	1	98	1.94	2	97	0.08	4.1	
S_ 1820 nm	5.00	5	100	10.2	10	102	0.17	1.7	
Sb 2068 nm	0.985	1	98	1.94	2	97	0.06	3.0	
Se 1960 nm	0.981	1	98	1.98	2	99	0.07	3.4	
Si 2516 nm	4.95	5	99	9.43	10	94	0.29	3.0	
Sn 1899 nm	0.993	1	99	1.97	2	99	0.05	2.7	
Sr 4077 nm	1.01	1	101	1.98	2	99	0.04	2.0	
Sr 4215 nm	1.00	1	100	1.99	2	99	0.05	2.3	
Ti 3349 nm	1.01	1	101	1.88	2	94	0.05	2.4	
Ti 3349 nm	1.02	1	102	1.93	2	96	0.08	3.9	
Tl 1908 nm	1.01	1	101	2.05	2	102	0.08	3.9	
V_ 2924 nm	1.01	1	101	1.88	2	94	0.05	2.8	
Zn 2138 nm	0.985	1	98	1.92	2	96	0.07	3.5	

Table 10: Results for QCS (post-calibration), and IPC (ongoing QC) measurements. All concentrations are expressed as mg/L.

Analyte	NIST 1640 River Water			CRM610 Ground Water		
	Mean	Known	Rec %	Mean	Known	Rec %
Ag 3280 nm	0.008	0.00762	103	0.007	0.0076	98
Al 3082 nm	<MDL	0.052	N/A	0.124	0.193	64
As 1890 nm	0.024	0.02667	89	0.006	0.0099	63
As 1937 nm	0.025	0.02667	92	0.008	0.0099	85
B_ 2496 nm	0.298	0.301	99	0.975	1.013	96
Ba 4934 nm	0.146	0.148	99	0.103	0.101	102
Be 3130 nm	0.034	0.03494	98	N/A	N/C	N/A
Be 3131 nm	0.034	0.03494	97	N/A	N/C	N/A
Ca 3158 nm	7.37	7.045	105	15.4	14.7	105
Cd 2265 nm	0.020	0.02279	89	0.004	0.0044	81
Cd 2288 nm	0.020	0.02279	88	0.004	0.0044	82
Co 2286 nm	0.019	0.02068	94	N/A	N/C	N/A
Cr 2055 nm	0.036	0.0386	92	0.045	0.0453	99
Cu 3247 nm	N/A	N/C	N/A	1.89	1.975	96
Fe 2599 nm	0.021	0.0343	62	0.180	0.196	92
K_ 7664 nm	0.921	0.994	93	1.10	1.16	95
Li 6707 nm	0.049	0.0507	96	N/A	N/C	N/A
Mg 2795 nm	5.71	5.819	98	1.24	1.27	98
Mn 2576 nm	0.117	0.1215	96	0.047	0.0482	97
Mo 2020 nm	0.045	0.04675	97	N/A	N/C	N/A
Mo 2038 nm	0.045	0.04675	96	N/A	N/C	N/A
Na 5889 nm	27.0	29.35	92	8.09	8.1	100
Ni 2216 nm	0.027	0.0274	98	0.017	0.0183	95
Ni 2316 nm	0.026	0.0274	93	0.017	0.0183	93
Pb 2203 nm	0.026	0.02789	93	0.024	0.0237	101
Sb 2068 nm	0.012	0.01379	88	0.005	0.0049	93
Se 1960 nm	0.023	0.02196	104	0.010	0.0095	103
Si 2516 nm	9.79	10.13	97	N/A	N/C	N/A
Sr 4077 nm	0.127	0.1242	102	N/A	N/C	N/A
Sr 4215 nm	0.124	0.1242	100	N/A	N/C	N/A
V_ 2924 nm	0.013	0.01299	100	N/A	N/C	N/A
Zn 2138 nm	0.053	0.0532	100	0.520	0.514	101

Table 11: Results of analysis of river water and ground water CRMs (n=2). All concentrations are expressed as mg/L.

Analyte	CA010A Hard Tap Water			CA021A Soft Water		
	Mean	Known	Rec %	Mean	Known	Rec %
Ag 3280 nm	N/A	N/C	N/A	0.007	0.0076	98
Al 3082 nm	0.164	0.208	79	0.124	0.193	64
As 1890 nm	0.050	0.055	90	0.006	0.0099	63
As 1937 nm	0.050	0.055	92	0.008	0.0099	85
B_ 2496 nm	N/A	N/C	N/A	0.975	1.013	96
Ba 4934 nm	0.118	0.116	102	0.103	0.101	102
Ca 3158 nm	85.1	83.2	102	15.4	14.7	105
Cd 2265 nm	N/A	N/C	N/A	0.004	0.0044	81
Cd 2288 nm	N/A	N/C	N/A	0.004	0.0044	82
Cr 2055 nm	0.043	0.048	90	0.045	0.0453	99
Cu 3247 nm	N/A	N/C	N/A	1.89	1.975	96
Fe 2599 nm	0.218	0.236	92	0.180	0.196	92
K_ 7664 nm	4.94	5.1	97	1.10	1.16	95
Mg 2795 nm	4.18	4.2	100	1.24	1.27	98
Mn 2576 nm	0.047	0.048	98	0.047	0.0482	97
Na 5889 nm	20.7	21.9	94	8.09	8.1	100
Ni 2216 nm	0.047	0.048	98	0.017	0.0183	95
Ni 2316 nm	0.046	0.048	95	0.017	0.0183	93
Pb 2203 nm	0.092	0.095	96	0.024	0.0237	101
Sb 2068 nm	0.010	0.0119	83	0.005	0.0049	93
Se 1960 nm	0.009	0.0095	100	0.010	0.0095	103
Zn 2138 nm	0.559	0.542	103	0.520	0.514	101

Table 12: Results of analysis of hard and soft drinking water CRMs (n=2). All concentrations are expressed as mg/L.

Analyte	WW1 Waste Water			WW2 Waste Water		
	Mean	Known	Rec %	Mean	Known	Rec %
Al 3082 nm	1.99	1.95	102	9.87	10.0	99
As 1890 nm	0.091	0.1	91	0.471	0.5	94
As 1937 nm	0.092	0.1	92	0.471	0.5	94
Cd 2265 nm	0.018	0.0201	89	0.090	0.1	90
Cd 2288 nm	0.017	0.0201	85	0.087	0.1	87
Co 2286 nm	0.057	0.06	95	0.282	0.3	94
Cr 2055 nm	0.196	0.2	98	0.985	1.0	99
Cu 3247 nm	0.382	0.401	95	1.87	2.0	93
Fe 2599 nm	0.960	0.996	96	4.85	5.0	97
Mn 2576 nm	0.387	0.4	97	1.93	2.0	97
Ni 2216 nm	0.978	1	98	4.85	5.0	97
Ni 2316 nm	0.969	1	97	4.81	5.0	96
P_ 1774 nm	0.994	1	99	5.01	5.0	100
P_ 2149 nm	0.962	1	96	4.86	5.0	97
V_ 2924 nm	0.096	0.0999	96	0.484	0.5	97
Zn 2138 nm	0.601	0.6	100	2.98	3.0	99

Table 13: Results of analysis of wastewater CRMs (n=2). All concentrations are expressed as mg/L.

Analyte	LGC6177 Landfill Leachate (1:10)			NIST 1641D Mercury in Water		
	Mean	Known	Rec %	Mean	Known	Rec %
B_ 2496 nm	0.907	0.98	100	N/A	N/C	N/A
Ca 3158 nm	7.506	7.48	100	N/A	N/C	N/A
Cr 2055 nm	0.016	0.018	88	N/A	N/C	N/A
Fe 2599 nm	0.347	0.38	98	N/A	N/C	N/A
Hg 1849 nm	N/A	N/C	N/A	1.63	1.59	102
Hg 1942 nm	N/A	N/C	N/A	1.64	1.59	103
K_ 7664 nm	77.802	78	100	N/A	N/C	N/A
Mg 2795 nm	6.737	7.35	100	N/A	N/C	N/A
Mn 2576 nm	0.013	0.014	103	N/A	N/C	N/A
Na 5889 nm	123.099	175	70	N/A	N/C	N/A
Ni 2216 nm	0.019	0.021	90	N/A	N/C	N/A
Ni 2316 nm	0.018	0.021	90	N/A	N/C	N/A
P_ 1774 nm	1.362	1.15	114	N/A	N/C	N/A
P_ 2149 nm	1.311	1.15	114	N/A	N/C	N/A
Zn 2138 nm	0.026	0.026	101	N/A	N/C	N/A

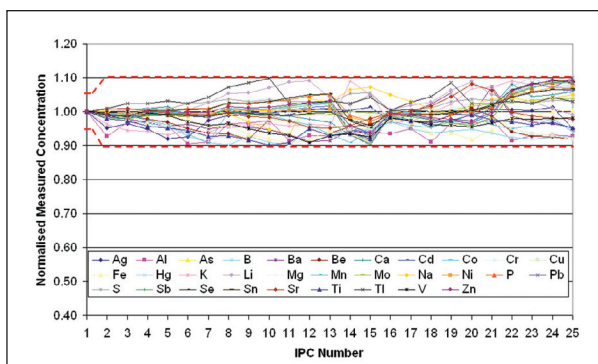
Table 14: Results of analysis of landfill leachate and mercury CRMs (n=2). All concentrations are expressed as mg/L.

Analyte	Spike Value	Hard Tap Water			Leachate (1:10)		
		Unspiked	Spiked	Rec %	Unspiked	Spiked	Rec %
Ag 3280 nm	0.08	<MDL	0.075	97	<MDL	0.077	100
Al 3082 nm	0.45	<MDL	0.449	103	<MDL	0.429	104
As 1890 nm	0.5	<MDL	0.486	97	0.008	0.497	98
As 1937 nm	0.5	<MDL	0.491	99	0.007	0.503	99
B_ 2496 nm	0.9	0.040	0.945	101	0.907	1.84	104
Ba 4934 nm	1	0.059	0.999	94	0.080	1.05	97
Be 3130 nm	0.45	<MDL	0.447	99	<MDL	0.453	101
Be 3131 nm	0.45	<MDL	0.448	100	<MDL	0.457	102
Ca 3158 nm	50	98.0	147	99	7.51	57.2	99
Cd 2265 nm	0.45	<MDL	0.455	101	<MDL	0.450	100
Cd 2288 nm	0.45	<MDL	0.466	104	<MDL	0.465	103
Co 2286 nm	0.45	<MDL	0.458	102	0.006	0.464	102
Cr 2055 nm	0.5	<MDL	0.475	95	0.016	0.492	95
Cu 3247 nm	0.9	0.484	1.37	98	0.008	0.925	102
Fe 2599 nm	0.45	0.010	0.436	95	0.347	0.774	95
Hg 1849 nm	0.1	<MDL	0.097	96	<MDL	0.094	95
Hg 1942 nm	0.1	<MDL	0.096	95	<MDL	0.095	94
K_ 7664 nm	20	1.68	21.6	100	77.8	98.7	104
Li 6707 nm	1.1	0.011	1.06	95	0.026	1.09	97
Mg 2795 nm	17	2.41	19.1	98	6.74	23.3	98
Mn 2576 nm	0.45	<MDL	0.424	94	0.013	0.446	96
Mo 2020 nm	0.5	<MDL	0.491	98	<MDL	0.491	98
Mo 2038 nm	0.5	<MDL	0.493	99	<MDL	0.490	98
Na 5889 nm	50	8.71	57.1	97	123	171	95
Ni 2216 nm	0.45	0.002	0.458	101	0.019	0.474	101
Ni 2316 nm	0.45	0.001	0.456	101	0.018	0.471	101
P_ 1774 nm	22	1.04	22.5	97	1.36	23.0	98
P_ 2149 nm	22	1.00	22.2	96	1.31	22.7	97
Pb 2203 nm	0.45	<MDL	0.457	101	<MDL	0.452	100
S_ 1820 nm	22	25.3	47.7	102	18.1	40.7	103
Sb 2068 nm	0.5	<MDL	0.476	95	<MDL	0.476	95
Se 1960 nm	0.5	<MDL	0.486	97	<MDL	0.485	97
Si 2516 nm	9	10.5	19.6	101	5.17	14.2	100
Sn 1899 nm	0.5	<MDL	0.481	96	0.004	0.481	95
Sr 4077 nm	1	0.381	1.41	102	0.094	1.14	104
Sr 4215 nm	1	0.379	1.40	102	0.093	1.11	102
Ti 3349 nm	0.5	<MDL	0.469	93	0.006	0.482	95
Ti 3349 nm	0.5	0.007	0.473	93	0.006	0.485	96
Tl 1908 nm	0.45	<MDL	0.467	104	<MDL	0.461	103
V_ 2924 nm	0.45	<MDL	0.450	100	0.008	0.463	101
Zn 2138 nm	1	0.019	1.01	99	0.026	1.02	99

Table 15: Results of analysis of unfortified and fortified tap water and leachate matrices with spike recoveries (n=16). All concentrations are expressed as mg/L.

Analyte	Spike Value	Saline Water (1:10)			Soil Digest (1:10)		
		Unspiked	Spiked	Rec %	Unspiked	Spiked	Rec %
Ag 3280 nm	0.08	<MDL	0.072	95	0.026	0.101	93
Al 3082 nm	0.45	0.121	0.580	102	4.27	4.71	97
As 1890 nm	0.5	<MDL	0.502	100	0.558	1.04	97
As 1937 nm	0.5	<MDL	0.509	102	0.584	1.07	98
B_ 2496 nm	0.9	0.338	1.23	99	<MDL	0.616	100
Ba 4934 nm	1	0.011	0.977	97	0.032	0.996	96
Be 3130 nm	0.45	<MDL	0.443	98	0.000	0.451	100
Be 3131 nm	0.45	<MDL	0.446	99	<MDL	0.453	101
Ca 3158 nm	50	33.3	82.1	98	35.6	85.3	99
Cd 2265 nm	0.45	<MDL	0.446	99	0.041	0.500	102
Cd 2288 nm	0.45	0.000	0.474	105	0.020	0.492	105
Co 2286 nm	0.45	0.005	0.455	100	0.005	0.469	103
Cr 2055 nm	0.5	<MDL	0.472	95	0.028	0.505	95
Cu 3247 nm	0.9	0.004	0.911	101	1.03	1.95	102
Fe 2599 nm	0.45	0.258	0.682	94	258	258	N/A
Hg 1849 nm	0.1	<MDL	0.098	98	0.002	0.098	96
Hg 1942 nm	0.1	<MDL	0.096	96	0.004	0.099	96
K_ 7664 nm	20	32.7	53.2	102	1.5	22.3	104
Li 6707 nm	1.1	0.021	1.11	99	0.007	1.11	100
Mg 2795 nm	17	69.9	86.1	95	4.27	21.0	99
Mn 2576 nm	0.45	0.017	0.444	95	1.34	1.76	93
Mo 2020 nm	0.5	<MDL	0.492	98	0.002	0.487	97
Mo 2038 nm	0.5	<MDL	0.490	98	<MDL	0.485	97
Na 5889 nm	50	221	269	95	0.471	48.0	95
Ni 2216 nm	0.45	<MDL	0.449	100	0.032	0.496	103
Ni 2316 nm	0.45	<MDL	0.446	99	0.038	0.500	103
P_ 1774 nm	22	0.023	22.4	102	0.636	21.5	95
P_ 2149 nm	22	0.021	22.0	100	0.815	21.4	94
Pb 2203 nm	0.45	<MDL	0.440	98	7.87	8.25	82
S_ 1820 nm	22	247	271	108	998	1048	N/A
Sb 2068 nm	0.5	<MDL	0.484	97	0.039	0.505	93
Se 1960 nm	0.5	<MDL	0.493	99	<MDL	0.418	97
Si 2516 nm	9	0.637	10.1	105	2.68	11.6	99
Sn 1899 nm	0.5	<MDL	0.473	95	0.012	0.481	94
Sr 4077 nm	1	0.638	1.68	104	0.044	1.087	104
Sr 4215 nm	1	0.627	1.65	102	0.043	1.066	102
Ti 3349 nm	0.5	0.005	0.473	94	0.123	0.598	95
Ti 3349 nm	0.5	0.007	0.483	95	0.127	0.601	95
Tl 1908 nm	0.45	<MDL	0.423	94	0.015	0.479	103
V_ 2924 nm	0.45	<MDL	0.456	101	<MDL	0.427	100
Zn 2138 nm	1	0.001	1.00	100	4.60	5.62	102

Table 16: Results of analysis of unfortified and fortified saline water and soil digest matrices with spike recoveries (n=16). All concentrations in mg/L.



Graph 1: The stability of successive IPC measurements during the 240 sample analysis. Control limits are shown with dashed red lines.

Interference Study

Very few significant interferences were found during the analysis of the SIC solutions, showing that the instrument is relatively free of interferences. Those that were observed can easily be corrected by using inter-element corrections when necessary.

LDR

The high standards analyzed for the linear dynamic range check showed little deviation from their expected values, indicating linearity up to at least the levels indicated in Table 9. These levels are normally more than sufficient for the analysis of typical water samples.

MDL

The method detection limits calculated from analysis of the MDL solution are generally in the low ppb range for the majority of elements. All MDLs are sufficiently below the typical levels of interest for drinking water analysis, with the exception of antimony, mercury, thallium, and aluminium. The MDLs for these elements are of the same magnitude as the level of interest. For this reason ICP-MS may be a more appropriate alternative for regulatory drinking water measurements for these elements. The use of the axial view can, however, significantly improve the detection limit of some elements measured using the radial view in this study. MDLs for some elements, such as aluminium may be compromised by spot contamination in the sample tubes.

Accuracy, Precision and Stability

The iCAP produced consistently accurate results with minimal intensity drift, as shown by the results for the QCS and IPC solutions (see Table 10). The ongoing IPC results were consistently within the allowed range of 90-110 % of the known value, as shown in Graph 1. The precision of the 25 IPC measurements across the 240 sample run are also shown to be very good. Table 10 indicates that the relative standard deviation (RSD) of these measurements is well within 5 % across the duration of the run (16 hours).

The results in Tables 11-14 show that the iCAP consistently produced accurate and precise data in all of the reference material matrices. The vast majority of results were within 10 % of the certified concentration, the few exceptions tending to be when the measured value was close to the method detection limit.

The accurate results for the LFM samples (see Tables 15 and 16) show that quantitative recovery can be achieved in a variety of real environmental matrices. All spike recoveries were well within the allowable range of 85-115 %.

Conclusions

The iCAP 6500 Duo ICP demonstrated compliance with the requirements of EPA Method 200.7 for a wide range of water sample types and showed that it easily coped with the stringent AQC requirements of the method. A combination of specifically designed hardware and software tools enable and simplify compliant analysis as outlined below.

Wavelength verification is quick and easy with the autotape function and method and instrument optimization are automatically performed with the built-in optimization procedures. Along with the high transmission optical design and sensitive CID 86 detector, this helps to produce the optimum performance, as indicated by the excellent method detection limits obtained.

The lack of physical and spectral interference in environmental samples, demonstrated in the interference study, makes the iCAP ideal for analyzing waters and other environmental materials. This yields accurate results for all environmental sample types.

Careful attention was paid to the thermal conductivity of the instrument components during the design phase. This has produced an extremely stable system that continues to give good accuracy over long periods of time without frequent re-calibration and also produces good precision over extended runs. This is demonstrated by the consistent IPC results.

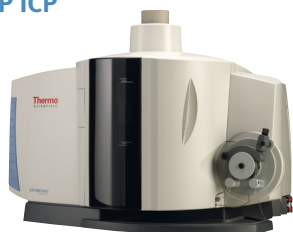
iTEVA software has a built-in QC checking capability that is designed to meet the requirements of EPA methods. The package also includes monitored uptake / washout features. This reduces the amount of non-productive time and maximizes useful analytical time. The productivity tools in iTEVA in combination with the speed of the iCAP and the low-volume sample introduction system result in rapid analysis times. Samples in this study were being processed at a speed of 1 sample every 3 minutes and 30 seconds, or 17 samples per hour. This makes the iCAP the ultimate ICP for cost-effective elemental analysis.

The use of an Inductively Coupled Plasma source (ICP) is the accepted and most powerful technique for the analysis and quantification of trace elements in both solid and liquid samples. Its applications range from routine environmental analyses to the materials industry, geological applications to clinical research and from the food industry to the semiconductor industry.

Thermo Fisher Scientific is the only instrument manufacturer to offer the full range of Inductively Coupled Plasma Spectrometers (ICP, Quadrupole and Sector ICP-MS) to satisfy every aspect of plasma spectrometry from routine to highly demanding research applications.

Develop your lab from the easy-to-use iCAP ICP to the high performance XSERIES 2 Quadrupole ICP-MS and up to the ultra-sophisticated ELEMENT2 and NEPTUNE Sector ICP-MS instruments. Each instrument combines leading-edge technology, fit for purpose and affordability with a tradition of quality, longevity, accuracy and ease of use.

**Thermo Scientific
iCAP ICP**



**Thermo Scientific
XSERIES 2 ICP-MS**



**Thermo Scientific
ELEMENT2 HR-ICP-MS**



**Thermo Scientific
NEPTUNE Multi-collector ICP-MS**



In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

- Africa**
+43 1 333 5034 127
- Australia**
+61 2 8844 9500
- Austria**
+43 1 333 50340
- Belgium**
+32 2 482 30 30
- Canada**
+1 800 530 8447
- China**
+86 10 8419 3588
- Denmark**
+45 70 23 62 60
- Europe-Other**
+43 1 333 5034 127
- France**
+33 1 60 92 48 00
- Germany**
+49 6103 408 1014
- India**
+91 22 6742 9434
- Italy**
+39 02 950 591
- Japan**
+81 45 453 9100
- Latin America**
+1 608 276 5659
- Middle East**
+43 1 333 5034 127
- Netherlands**
+31 76 579 55 55
- South Africa**
+27 11 570 1840
- Spain**
+34 914 845 965
- Sweden/Norway/
Finland**
+46 8 556 468 00
- Switzerland**
+41 61 48784 00
- UK**
+44 1442 233555
- USA**
+1 800 532 4752

www.thermo.com



FM 09032
Thermo Electron Manufacturing Ltd (Cambridge) is ISO Certified.

AN40853_E 06/08C

©2008 Thermo Fisher Scientific Inc. All rights reserved. Milli-Q is a registered trademark of Millipore Corporation. ASX-520 is a trademark of CETAC Corporation. All other trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.