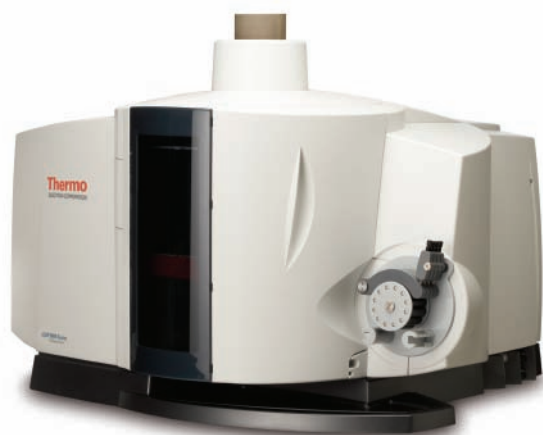


US EPA SW-846 Method 6010B using an iCAP 6500 Duo

Key Words

- ICP
- iCAP 6500
- Environmental Analysis
- SW-846 Method 6010b
- US-EPA



Introduction.

The Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) OF 1981 gave the US Environmental Protection Agency responsibility for cleanup of hazardous waste disposal sites. In order to assess the potential contamination and monitor cleanup of these sites, the EPA created the Contract Laboratory Program. This program directed the collection of routine environmental laboratory analytical data, to assure that quality standards were met.

Routine samples are analyzed using a fixed set of protocols with specified quality assurance and control measures.

This application note describes the performance of the iCAP 6500 Duo for EPA method 6010b. The method is suitable for the determination of 31 elements in ground waters, TCLP & EP extracts, industrial and organic wastes, soils, sludges, and sediments. All samples except filtered groundwater need to be digested prior to analysis using EPA method 3050b or similar.

Experimental

The iCAP 6000 series uses a Charged Injection Device (CID) detector, offering greater photosensitive area and lower noise for improved detection limits and better stability.

The CID detector is a non-blooming device and has the ability to measure high concentrations of matrix elements and low levels of contaminants at the same time without saturation.

Instrument conditions used for analysis are shown in Table 1 below.

Parameter	Setting	
Nebulizer	Glass Concentric	
Spray Chamber	Glass Cyclonic	
Center tube	2 mm	
Pump tubing (Tygon)	Sample Orange- White	Drain White-White
Nebulizer Gas Flow	0.7 l/min	
Plasma Gas Flow	12 l/min	
Auxiliary Gas Flow	0.5 l/min	
RF Power	1150 W	
Sample Flush Time	45 s	
Pump Speed	45 rpm	
Integration Time	Low (166 -230 nm) 15 s	High (230-847 nm) 5 s

Table 1: Instrument Parameters used for the analysis

Calibration standards were prepared in 5 % v/v HCl and 1 % v/v HNO₃ at concentrations selected to cover the desired range for each element. Elements were separated as specified in 6010b to minimize interference.

Quality control solutions were run at intervals specified by the method and any necessary corrective actions were taken automatically.

The iCAP 6500 Duo allows for elements expected at low concentration to be read axially where the best sensitivity is required; the radial view was used for elements at higher concentration or for those elements that suffer from easily-ionized element interference.

An internal standard of 5 ppm scandium was automatically added on-line with the internal standard mixing kit. Details of wavelengths and plasma view for each of the analyte and internal standard elements are shown in Table 2. The analyte elements were referenced to an internal standard wavelength on the same slit and plasma view.

Element	Wavelength	Plasma View	Internal Standard
Ag	328.068 nm	Axial	Sc 361.384 nm
Al	308.215 nm	Radial	Sc 361.384 nm
As	189.042 nm	Axial	Sc 227.318 nm
Ba	455.403 nm	Radial	Sc 361.384 nm
Be	313.107 nm	Radial	Sc 361.384 nm
Ca	315.887 nm	Radial	Sc 361.384 nm
Cd	214.438 nm	Axial	Sc 227.318 nm
Co	228.616 nm	Axial	Sc 227.318 nm
Cr	267.716 nm	Axial	Sc 361.384 nm
Cu	324.754 nm	Axial	Sc 361.384 nm
Fe	271.441 nm	Radial	Sc 361.384 nm
K	766.490 nm	Radial	Sc 631.384 nm
Mg	279.079 nm	Radial	Sc 361.384 nm
Mn	260.569 nm	Axial	Sc 361.384 nm
Na	589.592 nm	Radial	Sc 361.384 nm
Ni	231.604 nm	Axial	Sc 227.318 nm
Pb	220.353 nm	Axial	Sc 227.318 nm
Sb	206.833 nm	Axial	Sc 227.318 nm
Se	196.090 nm	Axial	Sc 227.318 nm
Tl	190.856 nm	Axial	Sc 227.318 nm
V	292.402 nm	Axial	Sc 361.384 nm
Zn	206.200 nm	Axial	Sc 227.318 nm

Table 2: Element Wavelengths

Significant interference from spectral overlaps can be observed due to the complex nature of the matrix for these types of samples. Major elements, such as Al, Ca, Fe, Mg, Si and P, were checked for spectral contributions on other analyte elements during method development. Once the interfering elements were identified, high-purity single element solutions were measured and the instrument software automatically calculated interference correction factors using the Interfering Element Correction (IEC) function of iTEVA software. Interferences observed are shown in Table 3.

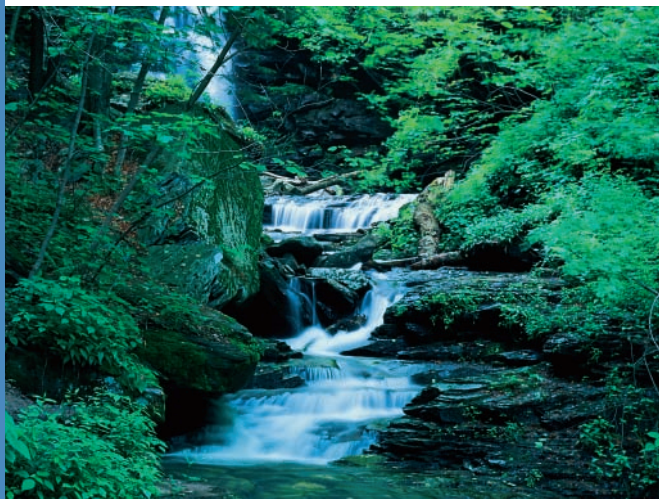
Element	Wavelength	Interfering Elements
Ag	328.068 nm	-
Al	308.215 nm	-
As	189.042 nm	Al, Fe
Ba	455.403 nm	-
Be	313.107 nm	-
Ca	315.887 nm	-
Cd	214.438 nm	Al, Fe
Co	228.616 nm	-
Cr	267.716 nm	-
Cu	324.754 nm	Fe
Fe	271.441 nm	-
K	766.490 nm	-
Mg	279.079 nm	-
Mn	260.569 nm	-
Na	589.592 nm	-
Ni	231.604 nm	-
Pb	220.353 nm	Al, Fe
Sb	206.833 nm	Al, Fe
Se	196.090 nm	Al, Fe
Tl	190.856 nm	Al, Fe
V	292.402 nm	-
Zn	206.200 nm	-

Table 3: Interelement Interferences

Analysis Results

Initial Performance Checks

Method detection limits (MDLs) were established by measuring a blank solution (5 % HCl, 1 % HNO₃). The solution was analyzed seven times with each analysis having three replicates, the mean of 3x the standard deviation value from all of the runs was calculated. Results obtained are given in Table 4.



Element	Wavelength	Plasma View	MDL (ppb)
Ag	328.068 nm	Axial	0.88
Al	308.215 nm	Radial	22
As	189.042 nm	Axial	1.6
Ba	455.403 nm	Radial	0.39
Be	313.107 nm	Radial	0.29
Ca	315.887 nm	Radial	6.6
Cd	214.438 nm	Axial	0.051
Co	228.616 nm	Axial	0.26
Cr	267.716 nm	Axial	0.39
Cu	324.754 nm	Axial	0.45
Fe	271.441 nm	Radial	73
K	766.490 nm	Radial	40
Mg	279.079 nm	Radial	37
Mn	260.569 nm	Axial	0.17
Na	589.592 nm	Radial	9.3
Ni	231.604 nm	Axial	0.31
Pb	220.353 nm	Axial	0.76
Sb	206.833 nm	Axial	1.2
Se	196.090 nm	Axial	1.9
Tl	190.856 nm	Axial	0.66
V	292.402 nm	Axial	0.39
Zn	206.200 nm	Axial	0.20

Table 4: Method Detection Limits

Quality Control Procedure

Method 6010b requires that a very strict quality control procedure should be followed to ensure validity of sample data. Quality control checks are carried out following instrument calibration, during sample analysis and at the end of the analytical run. All checks must meet the required criteria for the sample data to be acceptable.

The instrument was set up using the parameters shown in Table 1 and allowed to stabilize for 30 minutes prior to calibration.

Immediately after calibration an Initial Calibration Verification (ICV) solution, calibration blank and Continuing Calibration Verification (CCV) solution were run. The calibration blank readback must be within 3 times the method detection limit for each element, while the calibration verification solutions must be within 10 % of the actual values. The standard deviation of a minimum of 2 re-samples of each verification solution must be less than 5 % for the data to be acceptable.

Analysis of the CCV solution and calibration blank was then repeated every 10 samples to ensure the instrument remained in calibration, the results for the first CCV are shown in table 5.

Element	Initial Cal Check (ICV) (mg/L)		Continuing Cal Check (CCV) (mg/L)	
	Actual	Measured	Actual	Measured
Ag	0.5	0.495	1	0.998
Al	2.5	2.553	25	25.520
As	1.0	1.019	5	5.190
Ba	0.5	0.490	5	4.862
Be	0.5	0.481	2.5	2.410
Ca	10.0	10.140	25	25.100
Cd	0.5	0.510	5	4.985
Co	0.5	5.02	5	4.909
Cr	0.5	0.46	5	4.849
Cu	0.5	0.488	5	4.951
Fe	5.0	5.060	25	24.870
K	10.0	9.935	25	25.220
Mg	6.0	6.014	25	25.120
Mn	0.5	0.494	5	4.855
Na	10.0	10.000	25	25.300
Ni	0.5	0.501	5	4.902
Pb	1.0	1.006	5	4.895
Sb	1.0	1.000	5	9.933
Se	1.0	1.008	10	10.020
Tl	1.0	1.028	10	9.566
V	0.5	0.500	5	4.909
Zn	1.0	1.020	5	4.954

Table 5: Calibration Checks

The iCAP 6000 series of spectrometers feature a highly regulated temperature control system. This temperature control ensures that the spectrum position remains constant and peak analytical performance is maintained over extended time periods even with fluctuations in the laboratory conditions.

Interference check solutions were run prior to the start of the sample analysis to verify the accuracy of the inter-element corrections factors and background correction points. Interference Check Sample A (ICSA) was prepared containing 250 mg/L each of Al, Ca, Mg and 100 mg/L Fe. Interference Check Solution AB (ICSAB) was then prepared by spiking the ICSA solution with concentrations of 0.05 to 1 mg/L for the analyte elements. The values measured for ICSAB must be within 20 % of the true value for the data to be acceptable.

Data is shown in Table 6 below.

Element mg/L	ICSA (mg/L)	ICSAB (mg/L)	Target Value (mg/L)	% Recovery
Ag	<LOD	0.212	0.210	101.0
As	0.003	0.112	0.097	115.5
Ba	0.002	0.499	0.475	105.1
Be	<LOD	0.482	0.482	100
Cd	0.001	1.052	0.916	114.8
Co	0.003	0.509	0.455	111.9
Cr	0.040	0.516	0.506	102.0
Cu	0.017	0.532	0.537	99.1
Mn	0.016	0.494	0.483	102.3
Ni	0.013	1.037	0.930	111.5
Pb	0.005	0.059	0.051	115.7
Sb	0.004	0.667	0.585	114.0
Se	0.004	0.060	0.051	117.6
Tl	0.001	0.096	0.096	100.0
V	0.005	0.500	0.481	104.0
Zn	0.045	1.060	0.975	108.7

Table 6: Interference Checks

Internal laboratory quality control checks

Method performance was verified with two different Laboratory Control Samples, a water (LCSW) and a soil digest (LCSS). The results of this are shown in table 7. The water digest was prepared by the addition of nitric acid to the sample so that the final concentration of nitric acid in the sample was 10 %. The soil digest was prepared by digesting 1 gram of sample in a mixture of nitric and hydrochloric acid and diluting the sample so the final concentration of acid was 5 % nitric acid and 1 % hydrochloric.

Element	LCSS		LCSW	
	Measured ppm	Target ppm	Measured ppm	Target ppm
Ag	0.0223	0.0209	0.479	0.495
Al	0.2722	0.3090	2.600	2.482
As	1.1750	0.9300	1.019	0.996
Ba	0.0053	[0.0053]	0.491	0.502
Be	0.0183	0.0188	0.477	0.493
Ca	175.2000	184.0005	10.410	10.180
Cd	0.0448	0.0416	0.521	0.494
Co	0.1565	0.1400	0.523	0.496
Cr	0.0982	0.0965	0.495	0.490
Cu	6.7470	6.6800	0.499	0.490
Fe	21.4300	21.0000	5.083	5.107
K	0.0579	[0.1024]	9.924	10.008
Mg	112.1000	113.0000	6.091	6.003
Mn	0.1996	0.2010	0.500	0.495
Na	0.0536	[0.0928]	10.160	10.039
Ni	0.0622	0.0568	0.518	0.492
Pb	0.2655	0.2240	1.039	0.996
Sb	0.2702	0.2130	1.006	0.992
Se	0.0473	0.0370	0.942	1.005
Tl	0.0361	0.0381	1.044	1.027
V	0.0679	0.0658	0.510	0.501
Zn	0.1751	0.1750	1.071	1.000

Table 7: Internal laboratory control checks

Conclusions

The iCAP 6500 Duo far exceeds the requirements needed to meet EPA 6010b protocols.

The instrument has a high resolution optical system which minimizes spectral interferences and reduces stray light. It uses a next generation design of the exclusive Charged Injection Device detector which offers higher sensitivity and lower noise which results in better signal to background ratios.

The integrated structural castings and precision regulated optics ensure excellent long term stability.

The results of these developments are an instrument that has extreme stability and detection limit performance which meet the requirements for this type of analysis.

References

- USEPA SW-846 Method 3050b, Revision 2, December 1996.
- USEPA SW-846 Method 6010b, Revision 2, December 1996.

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