APPLICATION NOTE 74146

Fast, accurate, and robust analysis of environmental samples according to US EPA Method 6010D

Authors: Jianfeng Cui[1] and Timothy Traynor[2]

[1]Thermo Fisher Scientific, Germany

[2]Pace Analytical Services, IDEA Laboratory, United States

Keywords: EPA Method 6010D (SW-846), environmental analysis, inter-element correction (IEC), EPA 3015A, EPA 3051A, microwave assisted acid digestion

Goal

This application note describes the performance of the Thermo Scientific™ iCAP™ PRO XP ICP-OES Duo system for the analysis of environmental samples by following the US Environmental Protection Agency (EPA) Method 6010D (SW-846)[1]. Samples were digested in two different ways—one used hot plate digestion according to EPA Method 3010A[2] for water samples and 3050B[3] for soil samples, and the other used microwave assisted acid digestion according to EPA Method 3015A[4] for water samples and 3051A[5] for soil samples. The iCAP PRO XP ICP-OES Duo system achieves short analysis times per sample while maintaining compliance with the EPA Method protocol.

Introduction

In response to growing environmental issues, the EPA has developed various methods to test the contaminants in environmental samples. On 21 October 1976, the United States Congress enacted the Resource Conservation and Recovery Act (RCRA), which governs the disposal of solid and hazardous waste. Guideline methods for the analysis of these types of samples are collated under "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," more commonly known as SW-846. One of the specified methods is EPA Method 6010D (SW-846) "Inductively Coupled Plasma - Atomic Emission Spectrometry." This method prescribes the use of inductively coupled plasma-optical emission spectrometry (ICP-OES) instrumentation for the determination of target elements in groundwaters, industrial and organic wastes, soils, sludges, and sediments. Although EPA Method 6010D (SW-846) is used mainly within the US for the analysis of environmental samples, variants of this method are widely used in other regions and for other sample types. Therefore, the ability to undertake the analysis of samples using this method represents an important benchmark for any ICP-OES instrument.

Experimental

Instrumentation

The iCAP PRO XP ICP-OES Duo system, in combination with a Teledyne™ CETAC™ ASX 560 autosampler and the *ASXPRESS*™ sprint valve system, was used to analyze environmental samples within the quality control framework of EPA Method 6010D (SW-846). The total analysis time achieved with the setup above was only 1 minute and 28 seconds per sample. The duo configuration was selected as it enables elements expected at low concentrations to be measured axially for maximum sensitivity, while elements expected at high concentrations can be measured radially. The radial view also allows the dynamic range of the instrument to be extended. The instrument parameters used for this analysis are shown in Table 1.

Table 1. Instrument parameters

Parameter	Setting
Pump tubing	Sample: Tygon™ orange/white Drain: Tygon™ white/white Internal standard: Tygon™ orange/blue
Pump speed	45 rpm
Spray chamber	Glass cyclonic
Nebulizer	Glass concentric
Nebulizer gas flow	0.55 L·min^{-1}
Coolant gas flow	12.5 L·min^{-1}
Auxiliary gas flow	0.5 L·min^{-1}
Center tube	2 mm
RF power	1,250 W
Replicates	3
Autosampler	Teledyne CETAC ASX-560 with AS*XPRESS PLUS* rapid sample introduction system
Sample loop	2 mL
Time per sample	1 min 28 s
Exposure time	Axial: 7 s Radial: 7 s

Sample and standard preparation

Water and soil samples

Twenty water samples and 12 soil samples, provided by Pace Analytical Services, LLC (US), were digested using the hot plate acid digestion procedures according to EPA Methods 3010A and 3050B, respectively. Soil samples were digested using HNO_3, H_2O_2, and HCl, whereas the water samples were digested using HNO_3 and HCl only.

Two water samples and two soil samples provided by Pace Analytical Services. In addition, two Standard Reference Materials (SRM® 2781 – Domestic Sludge, NIST; SRM® 2709a – San Joaquin Soil, NIST), were digested according to EPA Method 3015A and 3051A using the ETHOS™ EZ SK10 (Milestone, Italy) microwave system. Two most common digestion methods have been used in this note to demonstrate the suitability of these methods.

Calibration standards

Calibration standards were prepared in 2% HNO_3, with analyte concentrations covering the range expected in the samples. A 10 mg·L^{-1} yttrium internal standard was introduced online via the *ASXPRESS*® *PLUS* rapid sample introduction system. The internal standard wavelengths were appropriately matched to analyte wavelengths for each of the viewing modes, Axial and Radial.

Spectral-Interference Check (SIC) solutions

Individual element SIC solutions

These solutions are used to evaluate possible spectral interferences and to set interelement corrections if necessary. Individual element SIC solutions for each of the major and trace elements were prepared using 1,000 mg·L^{-1} and 10,000 mg·L^{-1} single element standards (SPEX CertiPrep™, Metuchen, NJ, US) to meet the requirements of different concentration ranges.

Mixed element SIC solution

This solution is used as an ongoing daily check of freedom from spectral interferences. The mixed element SIC solution contains aluminum, 500 mg·L^{-1}; calcium, 500 mg·L^{-1}; iron, 200 mg·L^{-1}; and magnesium, 500 mg·L^{-1} and is made up in an acid solution equivalent to the calibration standards.

Initial Calibration Verification (ICV) Standard

According to the EPA Method 6010D requirement, this QC control solution should differ from those used for the preparation of the calibration standards, and consist of a second-source reference material from a different commercial lot or vendor. In this application work, an already-prepared, second-source reference material from VHG (P/N 1600590) was used as the ICV standard.

Method development

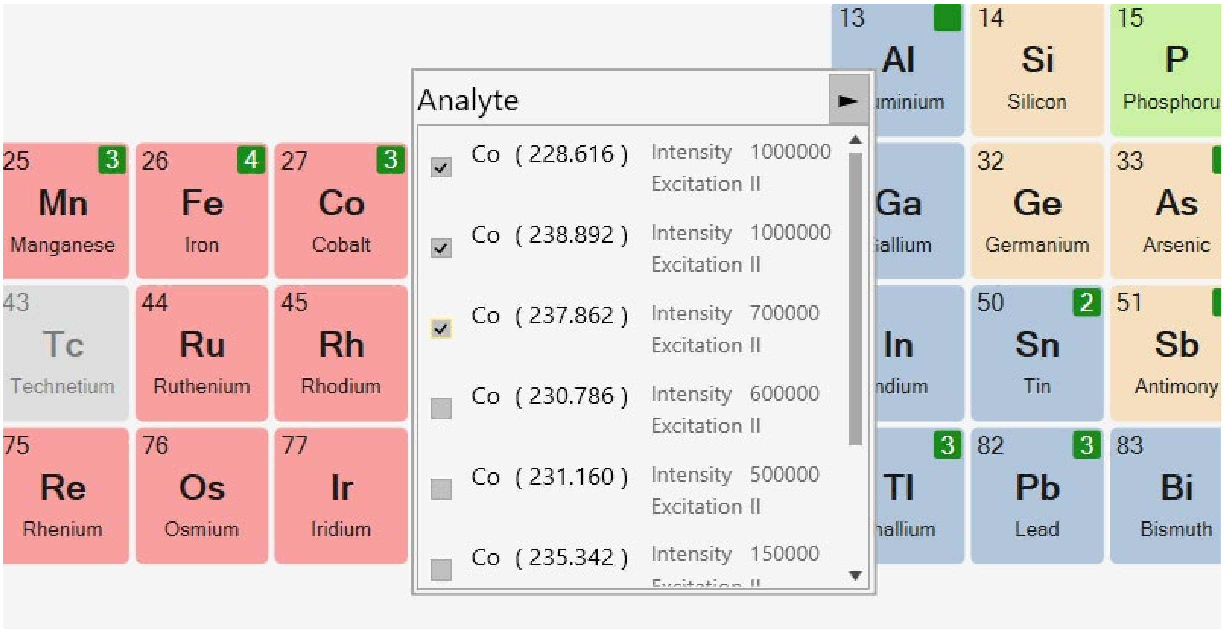
A LabBook was set up using the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software for the analysis.

Wavelength selection within the Qtegra ISDS Software is simple. The wavelength with the least interferences and the strongest signal is automatically recognized and ranked by the software for the analyst to select as shown in Figure 1 for cobalt.

Due to the very complex matrix of some environmental samples, significant spectral overlaps are possible and must be corrected for accurate results. The Qtegra ISDS Software includes an automatic feature for the correction of interferences based on concentration. Single element solutions for each of the major interferents were analyzed as SIC solutions to check for interferences on each analyte. For concentrations of individual element SIC solutions, see Table 2. Once identified, the Inter-Element Correction (IEC) function was used to calculate the interference correction factors based on concentration.

Table 2. Single element SIC solutions

Element	Concentration (mg·L^{-1})
Al	500
B	50
Ba	50
Ca	500
Cu	50
Fe	200
Mg	500
Mn	50
Mo	20
Na	1,000
Ni	20
Se	20
Si	200
Sn	20
V	20
Zn	20



☑ Co (228.616) Intensity 1000000 Excitation II

☑ Co (238.892) Intensity 1000000 Excitation II

☑ Co (237.862) Intensity 700000 Excitation II

☐ Co (230.786) Intensity 600000 Excitation II

☐ Co (231.160) Intensity 500000 Excitation II

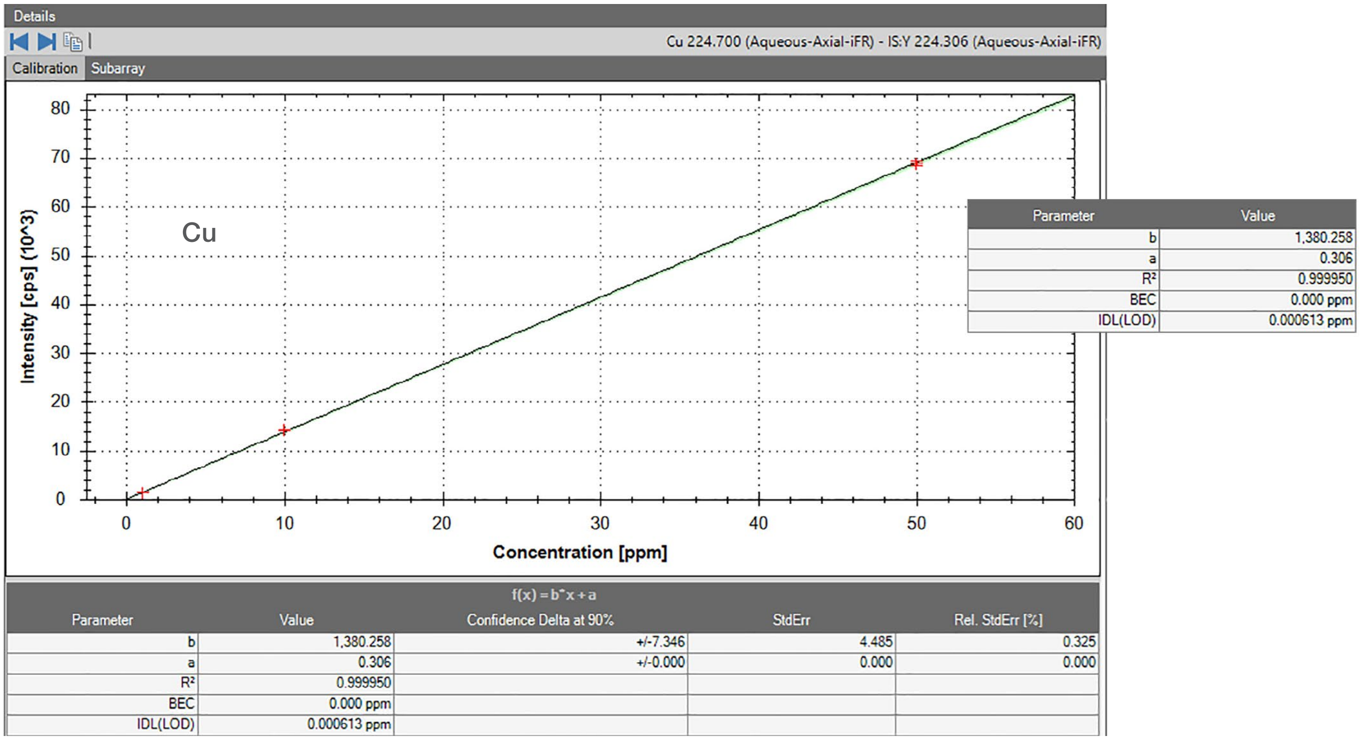
☐ Co (235.342) Intensity 150000

Figure 1. The wavelength selection tool within Qtegra ISDS Software showing the wavelength for Co, in order of sensitivity

Results and discussion

Linearity

The linearity of the target analytes was demonstrated using a four-point calibration curve. Calibration curves for all analytes were established in the same run. A linear range study was completed for all target elements to establish the highest concentration that can be reported without dilution of a sample. Excellent calibration curves were obtained for all target elements, with correlation coefficients of >0.9997 as shown in Figures 2 and 3 for copper and sulfur, respectively.

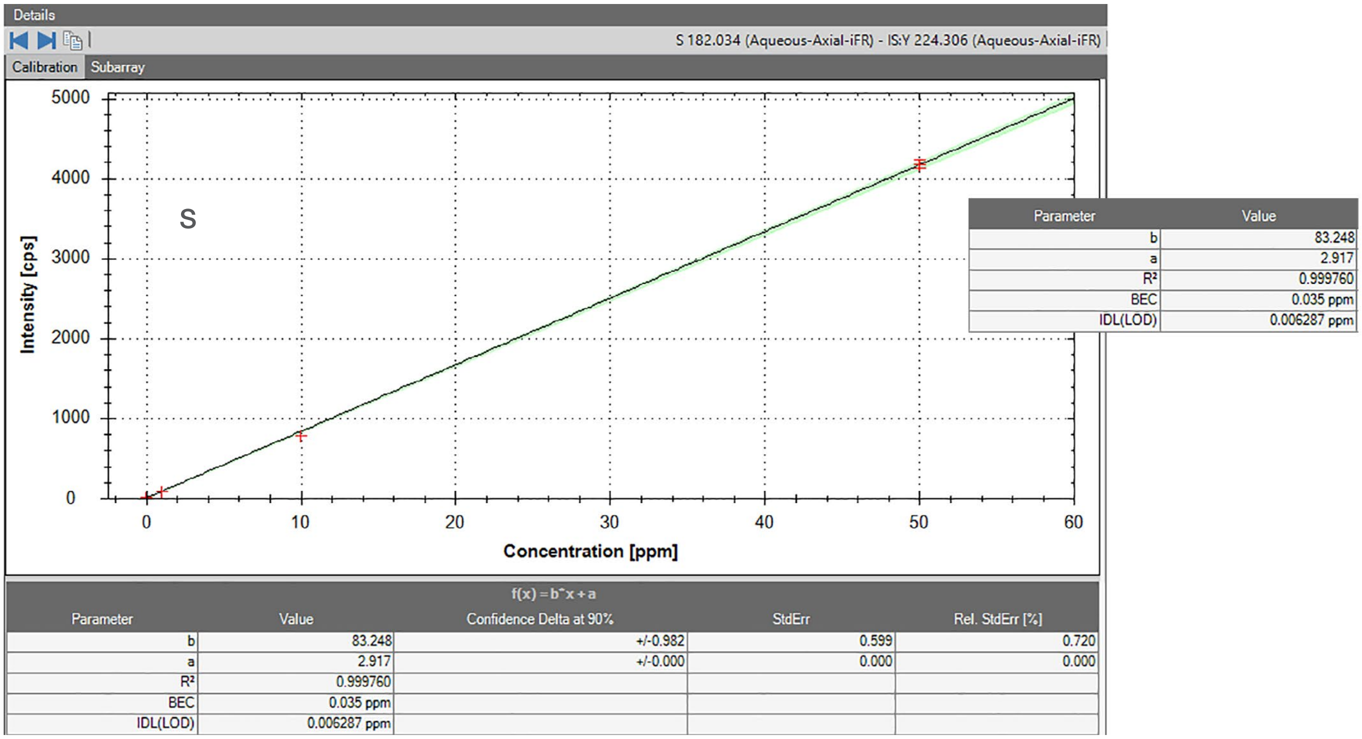


Parameter	Value
b	1,380.258
a	0.306
R²	0.999950
BEC	0.000 ppm
IDL(LOD)	0.000613 ppm

f(x) = b*x + a

Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	1,380.258	+/-7.346	4.485	0.325
a	0.306	+/-0.000	0.000	0.000
R²	0.999950			
BEC	0.000 ppm			
IDL(LOD)	0.000613 ppm			

Figure 2. Linearity of copper assessed over a concentration range of 1–50 mg·L^{-1}



Parameter	Value
b	83.248
a	2.917
R²	0.999760
BEC	0.035 ppm
IDL(LOD)	0.006287 ppm

f(x) = b*x + a

Parameter	Value	Confidence Delta at 90%	StdErr	Rel. StdErr [%]
b	83.248	+/-0.982	0.599	0.720
a	2.917	+/-0.000	0.000	0.000
R²	0.999760			
BEC	0.035 ppm			
IDL(LOD)	0.006287 ppm			

Figure 3. Linearity of sulfur assessed over a concentration range of 1–50 mg·L^{-1}

Instrument detection limits

Instrument detection limits (IDL) are useful means to evaluate the instrument noise level and response changes over time. For each analyte, a series of reagent blanks were analyzed to obtain a calculated concentration. IDLs were determined using the iCAP PRO XP ICP-OES Duo operating conditions given in Table 1. The detection limits were calculated using the raw intensity data from the standard and the blank as follows:

$$IDL = 3 \times SD_{blk} \frac{STD_{conc}}{STD_x - BLK_x}$$

Where:

IDL is the instrument detection limit

SD_{blk} is the standard deviation of the intensities of the multiple blank measurements

STD_{conc} is the concentration of the standard

STD_x is the mean signal for the standard

BLK_x is the mean (calculated from n=7 replicate injections) signal for the blank

The IDLs can be seen in Table 3.

Quality control procedure and results

EPA Method 6010D (SW-846) requires that certain quality control protocols be followed to ensure the validity of sample data. Quality control checks should be carried out following instrument calibration, during sample analysis, and at the end of the analytical run. All checks must meet the method required criteria for the sample data to be valid.

The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) are used to test the accuracy of the calibration and to check for potential contaminants in the reagents used to prepare the calibration standards. Immediately after calibration, an ICB solution and an Initial Calibration Verification (ICV) solution were analyzed. The ongoing validity of the calibration was verified through the analysis of CCB solution and Continuing Calibration Verification (CCV) solution after every 10 samples, and at the end of each analysis batch run as well. In this application work, the CCV solution was measured after every 5 samples to monitor the stability of the instrument and the ability to eliminate interferences.

Table 3. IDLs achieved by iCAP PRO XP ICP-OPS Duo system under typical laboratory conditions

Element	Wavelength (nm)	View	IDL ($mg \cdot L^{-1}$)
Ag	328.068	Axial	0.00037
Al	396.152	Radial	0.01365
As	189.042	Axial	0.00170
B	249.773	Axial	0.00015
Ba	455.403	Radial	0.00025
Be	234.861	Axial	0.00012
Bi	223.061	Axial	0.00248
Ca	315.887	Radial	0.00249
Cd	226.502	Axial	0.00004
Co	228.616	Axial	0.00025
Cr	284.325	Axial	0.00092
Cu	224.700	Axial	0.00061
Fe	259.940	Radial	0.00034
K	766.490	Radial	0.01515
Li	670.791	Radial	0.00155
Mg	279.079	Radial	0.00312
Mn	257.610	Radial	0.00025
Mo	203.844	Axial	0.00166
Na	589.592	Radial	0.00633
Ni	231.604	Axial	0.00066
P	178.284	Axial	0.00072
Pb	220.353	Axial	0.00228
S	182.034	Axial	0.00629
Sb	206.833	Axial	0.00148
Se	196.090	Axial	0.00316
Si	251.611	Radial	0.00438
Sn	189.989	Axial	0.00237
Sr	421.552	Axial	0.00004
Th	283.730	Axial	0.00180
Ti	334.941	Radial	0.00096
Tl	190.856	Axial	0.00331
V	292.402	Axial	0.00037
Zn	213.856	Radial	0.00050
Zr	343.823	Axial	0.00009

The ICB must return a result less than 0.5 times the Lower Limit of Quantification (LLOQ), which in most cases is the lowest concentration in the calibration curve, while the ICV and CCV solutions must be within ±10% of the true values. The CCB must not contain target analytes above the LLOQ. Analysis of the CCB and CCV was then repeated every 10 samples and at the end of the analysis run to ensure the instrument remained in calibration. In this work, the CCB and CCV solutions were repeated after every five samples and at the end of the analysis, all results were within the expected method limits. The results of the ICB and CCB solutions are shown in Table 4.

Matrix interference correction accuracy

High salt concentrations can cause analyte signal suppression and cause interferences. The accuracy of IEC factors should be verified through the analysis of SIC solutions. Interference check solutions were analyzed prior to the start of the sample analysis to verify the accuracy of the IEC factors and the background correction points. A mixed SIC solution (SIC-A) was prepared containing 500 $mg \cdot L^{-1}$ each of aluminum, calcium, and magnesium and 200 $mg \cdot L^{-1}$ of iron. Then, a spiked SIC solution (SIC-AB) was prepared by spiking the SIC-A solution with concentrations of 0.05–1 $mg \cdot L^{-1}$ (recommended by EPA Method 6010D) for the analyte elements. The concentrations measured for SIC-AB must be within 20% of the true spiked concentration for the data to be acceptable. The obtained data is shown in Table 4; all results are within the acceptance criteria.

To test the effect of the matrix on method performance, matrix spike (MS) and matrix spike duplicate (MSD) samples should be prepared by spiking samples with target analytes prior to digestion. In this study, a water and soil sample were spiked at different levels: 1000 $\mu g \cdot L^{-1}$, 2000 $\mu g \cdot L^{-1}$, and 5000 $\mu g \cdot L^{-1}$. The MS recoveries must not exceed ±25% of the target element spike concentration. All results shown in Tables 5 and 6 are within the acceptance criteria.

Method validation check - NIST SRM recoveries

Method performance was also verified by analyzing two NIST Standard Reference Materials: SRM 2781 – Domestic Sludge and SRM 2709a – San Joaquin Soil, which were digested using microwave assisted acid digestion according to EPA Method 3051A. Table 7 shows the results for both materials. All results were within ±10% of the certified values.

Method validation check – digestion methods

To determine the performance of different digestion methods with complex matrices, the same samples were digested by hot plate acid digestion according to the EPA Methods 3010A and 3050B, and microwave digestion method according to EPA Methods 3015A and 3051A. The results showed that samples prepared by microwave digestion had improved analyte recoveries as compared to the hot plate approach, indicating a possible loss of analytes to the atmosphere or through adsorption to digestion vessel walls. Analyte recoveries are typically better using microwave digestion since higher pressure and temperatures are used, allowing more complete decomposition of the matrix and retention of the analytes in solution.

In a hot plate digestion the temperature is limited to the boiling point of the acid. It is worth mentioning that whether it is water or soil samples, the experimental results showed the same trend. Table 8 shows the results of one water sample and one soil sample that were digested by two methods mentioned above. It should be noted that under the EPA Methods Update Rule (MUR) 2021, the EPA is allowing the use of microwave digestion as equivalent to hotplate digestion without the alternate test procedure (ATP) validation.

Table 4. Results of the Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Spectral-Interference Check (SIC) solutions and recoveries. Wavelengths and measurement modes for each element as per Table 3.

Element	ICB	CCB	SIC-A			SIC-AB		
	Measured (mg·L^{-1})	Measured (mg·L^{-1})	Expected (mg·L^{-1})	Measured (mg·L^{-1})	Recovery (%)	Expected (mg·L^{-1})	Measured (mg·L^{-1})	Recovery (%)
Ag	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.2	0.227	113
Al	<0.5*LLOQ	<LLOQ	500	516	103	500	527	105
As	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.1	0.109	109
B	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Ba	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.567	113
Be	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.510	102
Bi	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Ca	<0.5*LLOQ	<LLOQ	500	493	99	500	503	101
Cd	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	1	1.049	105
Co	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.459	92
Cr	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.512	106
Cu	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.451	90
Fe	<0.5*LLOQ	<LLOQ	200	181	91	200	185	93
K	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Li	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Mg	<0.5*LLOQ	<LLOQ	500	492	98	500	502	100
Mn	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.514	103
Mo	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Na	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Ni	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	1	0.966	97
P	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Pb	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.05	0.045	90
S	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Sb	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.6	0.622	104
Se	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.05	0.051	102
Si	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Sn	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Sr	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Th	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Ti	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	-
Tl	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.1	0.092	92
V	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0.5	0.460	92
Zn	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	1	0.982	98
Zr	<0.5*LLOQ	<LLOQ	0	<LLOQ	-	0	<LLOQ	

Table 5. Results and recoverires of the water sample analysis with different levels of spikes

Element	Water ($\mu g\cdot L^{-1}$)	Water + 1000 $\mu g\cdot L^{-1}$ ($\mu g\cdot L^{-1}$)	Recovery (%)	Water + 2000 $\mu g\cdot L^{-1}$ ($\mu g\cdot L^{-1}$)	Recovery (%)	Water + 5000 $\mu g\cdot L^{-1}$ ($\mu g\cdot L^{-1}$)	Recovery (%)
Ag	<IDL	906	91	1951	98	4864	97
Al	45.6	1123	108	2260	111	5627	112
As	3.6	1092	109	2203	110	5456	109
B	<IDL	1051	107	2144	108	5393	108
Ba	1.0	1035	103	2083	104	5195	104
Be	<IDL	1073	107	2146	107	5243	105
Bi	<IDL	907	91	1812	91	4550	91
Ca	522407.8	-	-	-	-	-	-
Cd	0.2	1125	112	2247	112	5527	111
Co	<IDL	990	99	2004	100	4920	98
Cr	3.0	1051	105	2112	105	5361	107
Cu	0.8	974	97	1946	97	4819	96
Fe	0.9	1007	101	2069	103	5129	103
K	<IDL	1087	109	2166	108	5407	108
Li	5.5	1140	113	2312	115	5667	113
Mg	9249.4	10183	93	11377	106	14701	109
Mn	3.4	1043	104	2121	106	5263	105
Mo	0.5	1053	105	2121	106	5333	107
Na	88832.6	-	-	-	-	-	-
Ni	0.8	1040	104	2101	105	5207	104
P	<IDL	1077	108	2166	109	5405	108
Pb	<IDL	948	95	1905	95	4734	95
S	<IDL	1022	106	2097	107	5289	107
Sb	<IDL	1035	104	2090	105	5248	105
Se	<IDL	1050	105	2119	106	5303	106
Si	4759.6	5757	100	6752	100	9780	100
Sn	<IDL	993	99	2005	100	4981	100
Sr	0.2	1019	102	2022	101	4950	99
Th	<IDL	1016	102	2055	103	5068	101
Ti	<IDL	998	100	2023	101	5175	104
Tl	<IDL	914	91	1812	91	4470	89
V	0.3	993	99	1979	99	4947	99
Zn	12.6	1008	100	2032	101	5021	100
Zr	0.4	1067	107	2173	109	5469	109

Table 6. Results and recoverires of the soil sample analysis with different levels of spikes

Element	Soil (µg·L^{-1})	Soil + 1000 µg·L^{-1} (µg·L^{-1})	Recovery (%)	Soil + 2000 µg·L^{-1} (µg·L^{-1})	Recovery (%)	Soil + 5000 µg·L^{-1} (µg·L^{-1})	Recovery (%)
Ag	2	880	89	1900	95	4740	95
Al	<IDL	1100	110	2300	115	5520	111
As	200	1220	101	2320	106	5460	105
B	2	1020	101	2120	105	5200	104
Ba	<IDL	1040	104	2140	107	5220	104
Be	<IDL	980	98	2080	104	5060	101
Bi	2	920	92	1880	94	4540	91
Ca	6	1000	99	2100	104	5040	101
Cd	144	1260	112	2480	117	5820	114
Co	<IDL	1000	99	2120	106	5120	102
Cr	2	1040	104	2180	109	5220	104
Cu	710	1700	99	2780	104	5700	100
Fe	10	1100	110	2280	113	5480	109
K	20	1020	100	2140	106	5060	101
Li	<IDL	1120	113	2340	117	5560	111
Mg	<IDL	1000	103	2140	109	5100	103
Mn	<IDL	1140	114	2340	117	5640	113
Mo	<IDL	1020	101	2120	106	5240	105
Na	<IDL	1020	103	2280	115	5240	105
Ni	<IDL	1080	107	2240	112	5400	108
P	<IDL	980	99	2060	103	5100	102
Pb	12790	13700	91	14680	94	17540	95
S	<IDL	960	98	2020	101	5000	100
Sb	2	1000	99	2060	103	5100	102
Se	<IDL	1020	102	2120	106	5220	104
Si	8	960	95	1960	98	4860	97
Sn	<IDL	1000	101	2080	104	5120	102
Sr	<IDL	1060	106	2140	108	4920	98
Th	<IDL	980	98	2080	104	5060	101
Ti	<IDL	980	98	2060	103	4940	99
Tl	<IDL	980	99	2020	101	4900	98
V	<IDL	1060	106	2160	108	5240	105
Zn	9612	10700	108	11920	116	14900	106
Zr	<IDL	1000	100	2180	109	5360	107

Table 7. Method validation: SRM recoveries. Please note, the SRM samples did not contain all of the elements required by EPA Method 6010D (SW-846).

Element	SRM 2781 – Domestic Sludge, NIST			SRM 2709a – San Joaquin Soil, NIST		
	Measured (mg·kg^{-1})	Certified value (mg·kg^{-1})	Recovery (%)	Measured (mg·kg^{-1})	Certified value (mg·kg^{-1})	Recovery (%)
Al	16253	16000	102	70636	73700	96
As	8.1	7.81	104	11.2	10.5	107
Ba	-	-		992	979	101
Be	0.5820	0.6133	95	-	-	
Ca	39523	39000	101	18945	19100	99
Cd	11.83	12.78	93	0.348	0.371	94
Co	-	-		12.2	12.8	95
Cr	208	202	103	129	130	99
Cu	607.3	627.8	97	32.0	33.9	94
Fe	28357	28000	101	32508	33600	97
K	4962	4900	101	20649	21100	98
Mg	5953	5900	101	14925	14600	102
Mn	-	-		532	529	101
Mo	45.9	46.6	98	-	-	
Na	2215	2100	105	11979	12200	98
Ni	78.2	80.2	98	83	85	98
P	24722	24300	102	704	688	102
Pb	206	200.8	103	16.4	17.3	95
Sb	-	-		1.46	1.55	94
Se	17	16	106	-	-	
Tl	-	-	-	0.559	0.58	96
V	-	-	-	108	110	98
Zn	-	-	-	98	103	95

Table 8. Results of water and soil samples digested by two different methods

Element	**Water sample**		**Soil sample**	
	Hot plate ($\mu g \cdot L^{-1}$)	**Microwave ($\mu g \cdot L^{-1}$)**	**Hot plate ($mg \cdot kg^{-1}$ (solid sample))**	**Microwave ($mg \cdot kg^{-1}$ (solid sample))**
Ag	<IDL	<IDL	0.1	<IDL
Al	45.6	47.9	<IDL	<IDL
As	3.6	3.9	10.0	9.45
B	<IDL	<IDL	0.08	0.09
Ba	1.0	1.1	0.02	0.03
Be	<IDL	<IDL	<IDL	<IDL
Bi	<IDL	<IDL	0.06	0.12
Ca	522407	509801	0.20	0.22
Cd	<IDL	<IDL	7.16	6.15
Co	<IDL	<IDL	<IDL	<IDL
Cr	3.0	3.6	0.05	0.18
Cu	0.8	1.5	35.5	35.5
Fe	0.9	1.0	0.14	0.20
K	<IDL	<IDL	1.0	2.6
Li	5.5	3.8	<IDL	0.03
Mg	9249	8959	<IDL	0.05
Mn	3.4	4.5	<IDL	<IDL
Mo	<IDL	<IDL	<IDL	<IDL
Na	88832.6	86757	0.28	0.45
Ni	<IDL	<IDL	0.08	0.11
P	<IDL	3.7	0.03	0.16
Pb	<IDL	5.4	639	659
S	<IDL	<IDL	<IDL	<IDL
Sb	<IDL	<IDL	0.1	0.1
Se	<IDL	<IDL	<IDL	<IDL
Si	4759	5155	0.39	0.39
Sn	<IDL	<IDL	<IDL	0.06
Sr	0.2	<IDL	<IDL	<IDL
Th	<IDL	<IDL	<IDL	0.14
Ti	<IDL	<IDL	<IDL	0.02
Tl	<IDL	<IDL	<IDL	<IDL
V	<IDL	<IDL	<IDL	<IDL
Zn	12.6	12.9	480.6	442.4
Zr	0.4	0.5	<IDL	<IDL

Robustness

The iCAP PRO XP ICP-OES Duo instrument includes precise gas Mass Flow Controllers (MFCs) and a highly regulated temperature control system. This advanced temperature control technology ensures that the spectrum position remains constant with fluctuations in the laboratory conditions. This ensures that the long-term signal stability of the instrument is exceptional and that CCV samples are within acceptable levels for extended periods.
To demonstrate the long-term stability of the system, more than 500 environmental samples were analyzed continuously over a 15 hour period. During this analysis a CCV standard was analyzed after every five samples.

Figure 4 shows the recoveries of the CCV solution plotted against time (h). All results of the CCV were within the acceptance criteria of ±10%.

The recovery of the yttrium internal standard (a real time display generated by the Qtegra ISDS Software automatically) is shown in Figure 5. All wavelengths used as internal standards showed excellent recoveries within 80 and 120% over the entire duration of the experiment and in different types of sample matrices. This recovery demonstrates stability and accuracy through the run, with no detectable carryover or other effects from sample matrices (e.g., signal suppression or enhancement).

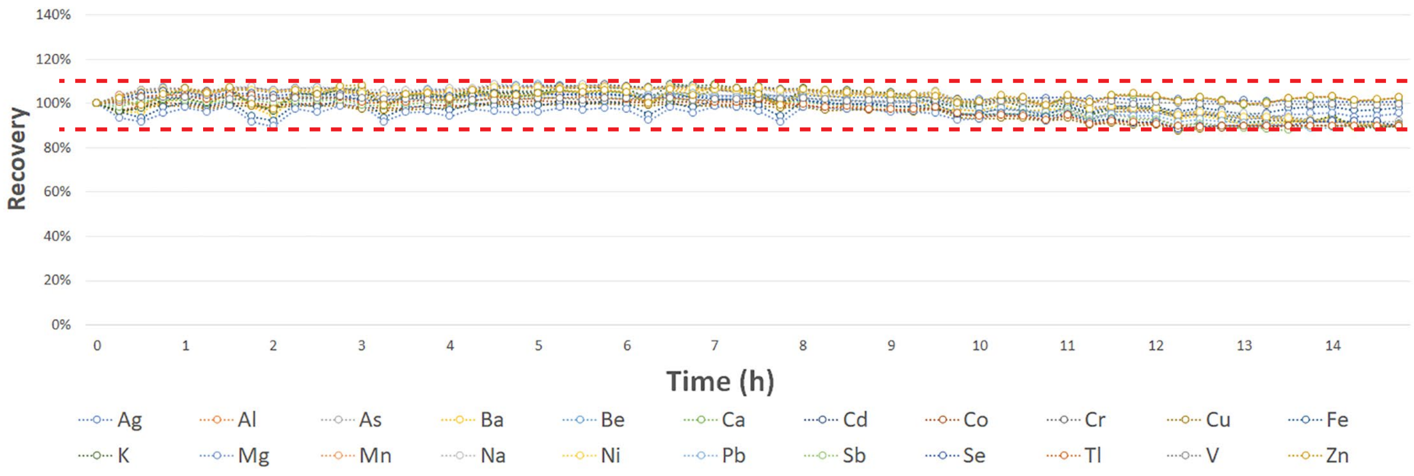


Figure 4. Analysis of CCV demonstrating long term recovery over 15 hours of continuous analysis

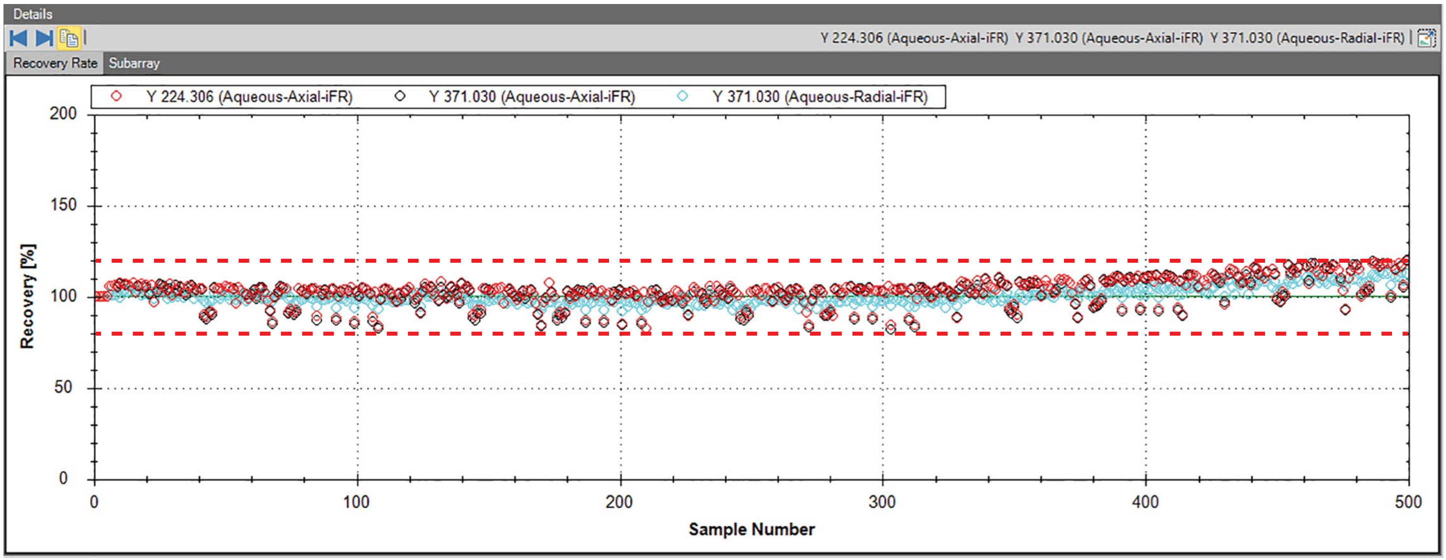


Figure 5. Recoveries of the internal standards wavelengths from the analysis of more than 500 samples over 15 hours showing recoveries within the acceptance criteria

Conclusions

This application note demonstrates that environmental samples can be analyzed accurately, precisely, and quickly using a Thermo Scientific iCAP PRO XP ICP-OES Duo system. The performance of the instrument exceeds the requirements needed to meet the EPA Method 6010D (SW-846) protocol.

- The CID 821 detector of the iCAP PRO XP ICP-OES Duo allows the measurement of the full wavelength range in a single measurement, giving the analyst the flexibility to choose from several wavelengths per analyte. Upon selection of the anlaytes, the wavelength library selects the optimal wavelength based on both signal intensity and freedom from interferences.
- High sensitivity achieved the detection limits of all target elements in the low ppb level, for several elements even in the sub-ppb level. The requirements of the EPA method were fully satisfied and even exceeded in many cases.
- Spectral interferences were easily corrected, as required by the EPA Method 6010D (SW-846), using the IEC function within the Qtegra ISDS Software, which automatically calculates IEC factors by concentration and applies them to the sample results.
- The simplified workflow of the Qtegra ISDS Software allows easy method development, implementation of Quality Control protocols, and corrective actions based on EPA methods.
- The recoveries of two SRM samples and spiked samples demonstrate the excellent accuracy of the method and the technique.
- Fast analysis (1 minute and 28 seconds per sample) for all target analytes utilizing both Axial and Radial modes was achieved by combining the iCAP PRO XP ICP-OES Duo system with the CETAC ASX 560 autosampler and *ASXPRESS PLUS* rapid sample introduction valve system. This enabled high sample throughput with minimal downtime, with no interaction from the analyst on the instrument. This is especially important for contract testing laboratories where hundreds of samples are analyzed daily.
- Robust and stable analytical performance was demonstrated through 15 hours of continuous sample analyses corresponding to >500 environmental samples with CCV and Internal Standard recoveries within the acceptance criteria.

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Acknowledgements

We thank Timothy Traynor from Pace Analytical Services, LLC IDEA Laboratory, Minneapolis, MN, USA for the excellent cooperation throughout the entire project.

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