Analysis of Potable Water for Trace Elements by ICP-OES

Application Note

Inductively Coupled Plasma-Optical Emission Spectrometers

Author

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Introduction

Water analysis is a typical application of Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES). ICP-OES is a well established analytical technique with large linear dynamic range, low detection limits, high precision and accuracy, which offers automation, rapid multi-element analysis for the determination of major, minor and trace elements in water samples. Most trace elements in fresh water are present in low concentrations which approach the detection limit of the instrument. It is not good practice to carry out routine analysis close to the detection limit where accurate results are not possible. To avoid this, sample pre-treatment — a ten-fold pre-concentration by evaporation, was applied [1]. To demonstrate the accuracy and the precision of the method, USEPA Pollution Quality Control water samples were analyzed in this study.

Agilent Technologies

Experimental

Instrumental

All measurements were performed on either the Agilent Liberty 100 or the Liberty 200 Inductively Coupled Plasma Atomic Emission Spectrometer, controlled by an IBM Personal Computer PS/2 Model 30/286. Both instruments have a 40.68 MHz RF Generator and a 0.75 m Czerny-Turner Monochromator with a 1800 grooves/mm Holographic grating. An argon saturator is standard. These instruments were chosen for their excellent resolution and stray light performance.

The resolution of the optical system is shown in Table 1.

Table 1.   The Resolution of the Liberty ICP-OE Spectrometer

Order	Resolution (nm)	Wavelength range (nm)
1	0.018	160–900
2	0.009	160–450
3	0.007	160–300
4	0.006	160–225

The system operating parameters are listed in Table 2.

Table 2.   Instrument Operating Parameters

Power	1.2 kW
Plasma gas flow	12.0 L/min
Auxiliary gas flow	0.75 L/min
Torch type	Standard one piece quartz torch with 1.4 mm id injector tube
Nebulizer type	Glass concentric
Nebulizer pressure	160 kPa
Pump tube	Grey-grey (inlet) Blue-blue (outlet)
Pump rate	15 rpm
Sample uptake rate	1.4 mL/min
Integration time	3 sec
Viewing height	Optimised for SBR
Background correction	Automatic
PMT voltage	650 V
Grating order	Default
Filter position	Default

Sample Preparation

Water samples were filtered and acid stabilized soon after collection. A 200 mL aliquot of a well-mixed, acid preserved sample was transferred to a beaker. 20 mL of concentrated HCl was added to the sample. The sample was heated (not boiled) on a steam bath until it was evaporated to near dryness. The beaker was rinsed thoroughly with 10% v/v HCl solution and the contents were transferred to a 20 mL volumetric flask, and made up to the mark with 10% v/v HCl solution.

Standard Preparation

All standard solutions were prepared from high purity ICP 10 000 ppm stock standards. Note that Al is often a contaminant of Mg compounds. According to the USEPA standard preparation specification [2], some elements are required to be grouped together due to their chemical compatibility. Therefore, standards were prepared as follows:

Standard 1   Mn, Be, Cd, Pb, Zn.
Standard 2   Cu, Fe, V, Co.
Standard 3   Al, Cr, Ni.

It is noted that the presence of Ca does not cause any spectral interferences on the Al 396 line. This is due to the superior resolution and low stray light of the Liberty system which has eliminated the wing broadening effect of Ca on Al. However, the elevated background is caused by the Ca continuum and can be treated by selecting automatic or dynamic background correction.

Multi-element standards were prepared in 10% v/v HCl and contained 5 mg/L of each element. The blank solution was 10% v/v HCl solution.

Analytical Line Selection

Due to the possible presence of high levels of Ca and Mg in a 10-fold pre-concentrated water sample, the choice of analytical line for Al was studied. The scan traces of 0.5 ppm Al and 0.5 ppm Al in 200 ppm Ca at Al 396.152 nm line are shown in Figure 1.
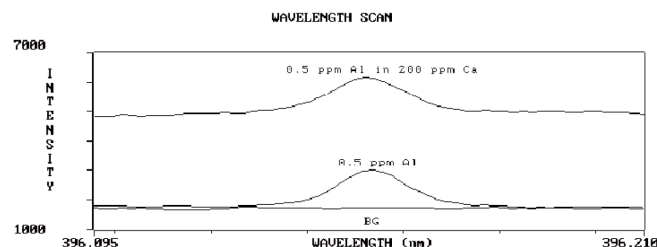

Figure 1.   Scan traces of 0.5 ppm Al and 0.5 ppm Al in 200 ppm Ca at the Al 396.152 nm line.

Scans of 1 ppm Al at the 167.081 nm, 308.215 nm and 396.152 nm lines have been examined for the effect of 1000 or 100 ppm Ca and 10 000 ppm Mg, respectively. Mg has no effect on any of these three Al lines. The effect of Ca is shown in Figures 2, 3 and 4.
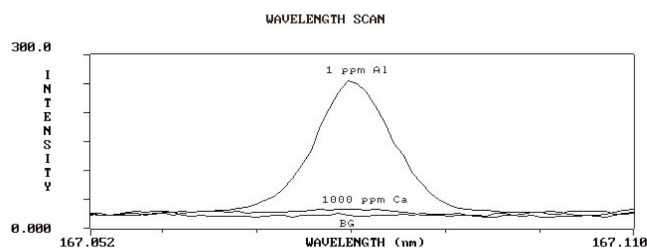

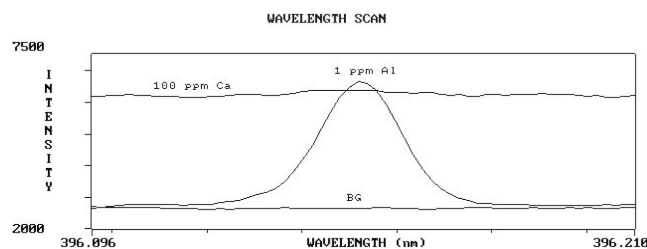Figure 2.   Scans of 1 ppm Al at 167.081 nm with the effect of 1000 ppm Ca.

Figure 3.   Scans of 1 ppm Al at 396.152 nm with the effect of 100 ppm Ca.
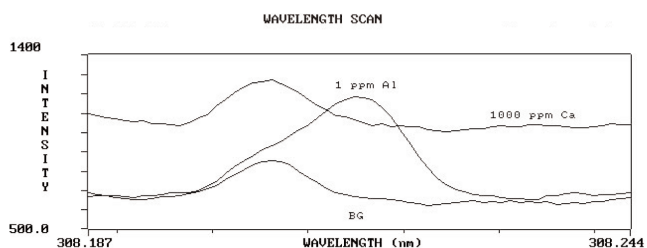

Figure 4.   Scans of 1 ppm Al at 308.215 nm with the effect of 1000 ppm Ca.

Calcium has no effect on the Al 167 line. It does cause elevated background on both the Al 396 nm and 308 nm lines, which can be treated by using background correction.

The disadvantage of using the Al 308.215 nm line is the structural background which is due to OH band emission. A narrower peak search window is required to exclude the OH peak for low level determination. This line is not recommended for any determinations too close to the detection limit.

Analytical Wavelength

The analytical wavelengths and detection limits of the elements of interest are listed in Table 3. The detection limit is defined as the concentration of an analyte which is equivalent to twice the standard deviation of the intensity of a blank.

Table 3.   Detection Limits

Element	Wavelength (nm)	Detection limit (ng/mL)
Al	167.081	1.5
Al	396.152	4
Al	308.215	12
Be	234.802	0.2
Cd	228.802	1.5
Co	238.893	5
Cr	267.716	4
Cu	324.754	2
Fe	259.940	1.5
Mn	257.610	0.3
Ni	231.604	6
Pb	220.353	14
V	292.402	4
Zn	213.856	0.9

Results

The results of the analysis of two USEPA Pollution Quality Control water samples No.4 and No. 5 are listed in Table 4. The precision of the measurements ranged from 0.1 to 0.8 %RSD.

Table 4.   Results of the Analysis

Element	Wavelength (nm)	No. 4 found values (ng/mL)	True values (ng/mL)	No.5 found values (ng/mL)	True values (ng/mL)
Al	396.152	65	60	477	450
Be	234.861	18.6	20	254	250
Cd	228.802	2.4	2.5	13.4	13
Co	238.893	18.3	20	79.4	80
Cr	267.716	11.7	10	80.8	80
Cu	324.754	13	11	53.6	50
Fe	259.940	27	20	86	80
Mn	257.610	15.5	15	77.8	75
Ni	231.604	34.2	30	85.6	80
Pb	220.353	26.9	24	130	122
V	292.402	71	70	254	250
Zn	213.856	18.9	16	85.1	80

Conclusion

The determination of trace elements in water by ICP-OES after a 10-fold pre-concentration has been described. The accuracy of the measured values are in good agreement with the EPA true values, while the precision of the measurement ranged from 0.1 to 0.8 %RSD.

References

1. M. Thompson, M. H. Ramsay, B. Pahlavanpour, Analyst, **1982**, 107, 1330-1334.

2. Inductively Coupled Plasma Atomic Emission Spectrometric Method for trace element analysis of water and wastes, Method 200.7 CLP-M, US Environmental Protection Agency, Cincinnati, Ohio 1985.

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