U5000AT+ Application Note

Determination of Trace Elements in a Natural Water Sample Using Ultrasonic Nebulization with Inductively Coupled Plasma Atomic Emission Spectrometry

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) is widely used for trace element determination in an extensive variety of samples. A number of important elements are more difficult to detect, including antimony, arsenic, lead, and selenium. The coupling of an efficient ultrasonic nebulizer accessory to a contemporary ICP-AES instrument can provide the necessary signal enhancement for accurate measurement of the above elements and others in a standard reference water sample.

Operating Conditions

Thermo IRIS Intrepid II XSP ICP-AES Instrument:
ICP Power: 1250 W
Auxiliary Gas Flow: 0.5 L/min
Nebulizer Gas Pressure: 25 psi
Viewing: Axial for all elements
Integration Time: 30 s
Replicates: 3

CETAC U5000AT+ Ultrasonic Nebulizer

CETAC U5000AT+ Ultrasonic Nebulizer:
Heater Temperature: 140^{0}C
Chiller Temperature: 3^{0}C
Sample Uptake Rate: 2.4 mL/min
Nebulizer Gas Flow: approx. 0.6 L/min at 25 psi pressure

Note: A flow restriction fitting is attached to the gas port of the ultrasonic nebulizer to regulate the flow of nebulizer gas from the ICP-AES instrument. An ICP-AES instrument equipped with a mass flow controller would not require this fitting.

Sample

The reference sample analyzed was NIST (National Institute of Standards and Technology) SRM 1640 Trace Elements in Natural Water. NIST SRM 1640 is composed of natural fresh water from Clear Creek, Colorado USA. The sample is filtered and stabilized with nitric acid to a concentration of 0.5M.

Sample Preparation and Calibration

Only an internal standard spike of 20 µg/L yttrium was added to the SRM 1640 natural water sample; no other sample treatment was performed. The ICP-AES instrument was calibrated with a reagent blank and two standards (also spiked with 20 µg/L yttrium) that covered the range of expected analyte concentrations. The three solutions were all made with high-purity nitric acid to a final concentration of 0.5M, matching the concentration in the natural water sample.

CETAC

A two-point background correction was performed for each analyte wavelength, and no interelement correction factors (IECs) were used for this sample matrix. Wavelengths were chosen for best performance for the analyte concentrations expected.

Note: Hydrogen peroxide can be added to samples and standards to correct for oxidation state variability for elements such as arsenic. The typical concentration of hydrogen peroxide is 0.4% (v/v) from 30% hydrogen peroxide. More details can be found in USEPA Method 200.15: "Determination of Metals and Trace Elements in Water by Ultrasonic Nebulization Inductively Coupled Plasma Atomic Emission Spectrometry".

Results

Table 1 lists measured values for certified elements and Table 2 lists measured values for reference elements. Values from the NIST certificate were converted from mass fractions (µg/kg) by multiplying by the density of SRM 1640 (1.0015 g/cm^3 +/- 0.0005 g/cm^3). Uncertainties are based on a 95% confidence interval. Overall agreement of the results obtained using the CETAC U5000AT$^+$ ultrasonic nebulizer with ICP-AES detection is excellent.

Table 1. Certified Values

Element	Wavelength (nm)	Certified (µg/L)	Measured (µg/L)
Ag	328.068	7.63 ± 0.25	7.79 ± 0.03
Al	396.152	52.1 ± 1.5	52.9 ± 0.3
As	189.042	26.71 ± 0.41	27.04 ± 0.32
Ba	233.527	148.2 ± 2.2	147.7 ± 0.8
Be	234.861	34.99 ± 0.41	34.56 ± 0.34
Cd	226.502	22.82 ± 0.96	22.78 ± 0.08
Co	228.616	20.31 ± 0.31	20.42 ± 0.18
Cr	267.716	38.6 ± 1.6	38.7 ± 0.18
Fe	238.204	34.3 ± 1.6	34.5 ± 1.3
Mn	257.610	121.7 ± 1.1	122.0 ± 1.0
Mo	202.030	46.82 ± 0.26	47.14 ± 0.16
Sb	206.833	13.81 ± 0.42	13.91 ± 0.54
Se	196.090	21.99 ± 0.51	21.69 ± 0.18
Sr	407.771	124.3 ± 0.7	124.6 ± 2.0
V	292.402	13.00 ± 0.37	12.90 ± 0.16

Table 2. Reference Values

Element	Wavelength (nm)	Certified (µg/L)	Measured (µg/L)
Cu	324.754	85.3 ± 1.2	84.3 ± 0.3
Li	670.784	50.8 ± 1.4	51.5 ± 0.1
Ni	231.604	27.4 ± 0.8	27.7 ± 0.4
K	766.491	995 ± 27	1005 ± 6
Zn	213.856	53.3 ± 1.1	53.6 ± 0.4

Element	Wavelength (nm)	Certified (mg/L)	Measured (mg/L)
Ca	184.006	7.055 ± 0.089	7.030 ± 00.010
Mg	285.213	5.828 ± 0.056	5.938 ± 0.022
Na	330.237	29.39 ± 0.31	30.13 ± 0.16