

Enhanced Detection of Trace Elements in Municipal Drinking Water per USEPA Method 200.7

Introduction

To safeguard human health and the environment, the United States Environmental Protection Agency (USEPA) adjusts regulations for maximum contaminant levels (MCLs) in water based on accepted scientific findings and guidelines. The current EPA Method 200.7 revision 4.4¹ describes criteria for the measurement of metals and trace elements in drinking water and wastewater using inductively coupled plasma optical emission spectrometry (ICP-OES).

This technical note describes the use of an ultrasonic nebulizer (USN) as a liquid sample introduction accessory for ICP-OES. Using a piezoelectric transducer for aerosol generation, ultrasonic nebulization improves liquid sample transport efficiency versus conventional pneumatic nebulization. Trace element sensitivity is enhanced, extending the lower detection range of the ICP-OES technique. The benchtop-size USN has an integrated desolvation system to ensure ICP stability and reduce solvent emission background; USN setup time is only 10 minutes with no computer control requirement.

Instrument detection limits (IDLs) for six spiked elements (As, Cd, Pb, Sb, Se, Tl) in a municipal drinking water matrix are measured using the USN with ICP-OES detection and per EPA Method 200.7 criteria. The IDLs measured are compared to USEPA maximum contaminant levels (MCLs).

Experimental

Instrument Conditions

Measurements were performed using a Teledyne CETAC U5000AT+ Ultrasonic Nebulizer (USN) coupled with a PerkinElmer Avio® 500 ICP-OES instrument. An interface kit consisting of a gas supply line and a sample out line with torch adapter was used to connect the USN to the ICP-OES. The host ICP-OES peristaltic pump was used to supply blank and standard solutions to the USN, and the ICP-OES nebulizer gas was used as the Ar carrier gas supply to the USN. A PerkinElmer S23 Autosampler was used to automate the introduction of blank and standard solutions.

A list of operating conditions is given in Table 1; a front view of the U5000AT+ USN is given in Figure 1.

Municipal Water Matrix

The municipal water sample was from the Platte West Water Treatment Plant (Elkhorn, NE) which serves the Omaha, Nebraska metropolitan area. The dissolved solid residue from an evaporated 400mL sample of the municipal water is shown in Figure 2.

The standard pneumatic nebulizer was used to measure major element concentrations in the municipal water sample; results are given in Table 2 for Ca, Na, Mg, K, Ba, and S.

Table 1. Operating Conditions of the Avio 500 ICP-OES with the U5000AT+ USN

Parameter	U5000AT+ USN
ICP Power	1500 W
Plasma Gas	10.0 L/min
Auxiliary Gas	0.2 L/min
Nebulizer Gas	0.50 L/min
Torch Injector	2 mm
Uptake Rate	1.25 mL/min
Cassette Position	-5.0
Resolution	Normal
Nebulizer Type	Piezoelectric
Spray Chamber	Conical
Heater Temp	140 °C
Cooler Temp	3 °C
Viewing	Axial
Integration Times	0.01 s min / 3 s max
Peak Area	3 pts / peak
Replicates	4



Figure 1. Teledyne CETAC U5000AT+ Ultrasonic Nebulizer

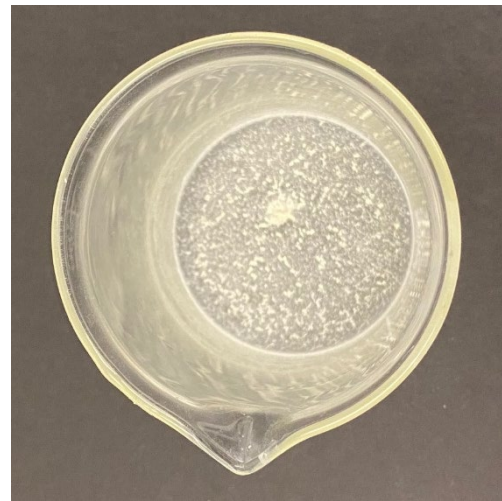


Figure 2. Evaporated municipal water sample residue

Table 2. Measured Major Elements in Municipal Water Matrix

Element	Wavelength (nm)	Measured Concentration
Ca	317.933	45.9 mg/L ± 0.9 mg/L
Na	330.237	34.4 mg/L ± 0.1 mg/L
Mg	285.213	98.3 mg/L ± 1.9 mg/L
K	766.490	8.6 mg/L ± 0.1 mg/L
Ba	493.408	85.6 µg/L ± 1.5 µg/L
Sr	421.552	45.9 µg/L ± 5.4 µg/L

Uncertainty is 2σ

Results

Instrument Calibration

Calibration standards were prepared in the described municipal drinking water matrix after acidification with double-distilled HNO₃. Five spiked water standards were set at concentrations of 5, 10, 20, 50 and 100 µg/L. An acidified municipal water blank was used as a reagent blank; no internal standard was used for calibration. Correlation coefficients must be 0.995 or better to be considered valid for analysis; coefficients measured using the U5000AT+ range from 0.9995 to 0.9999.

Analyte Emission Spectra

Selected U5000AT+ USN / ICP-OES calibration standard spectra for As and Pb spikes in the municipal water matrix are depicted in Figures 3a and 3b. Note the distinct emission peaks detected for the 5 µg/L As and Pb spikes with use of the U5000AT+ USN accessory.

Table 3. Correlation Coefficients in municipal water matrix

Element	Wavelength (nm)	Corr. Coefficient
As	188.797	0.9999
Cd	214.440	0.9999
Pb	220.353	0.9999
Sb	206.836	0.9999
Se	196.026	0.9995
Tl	190.801	0.9999

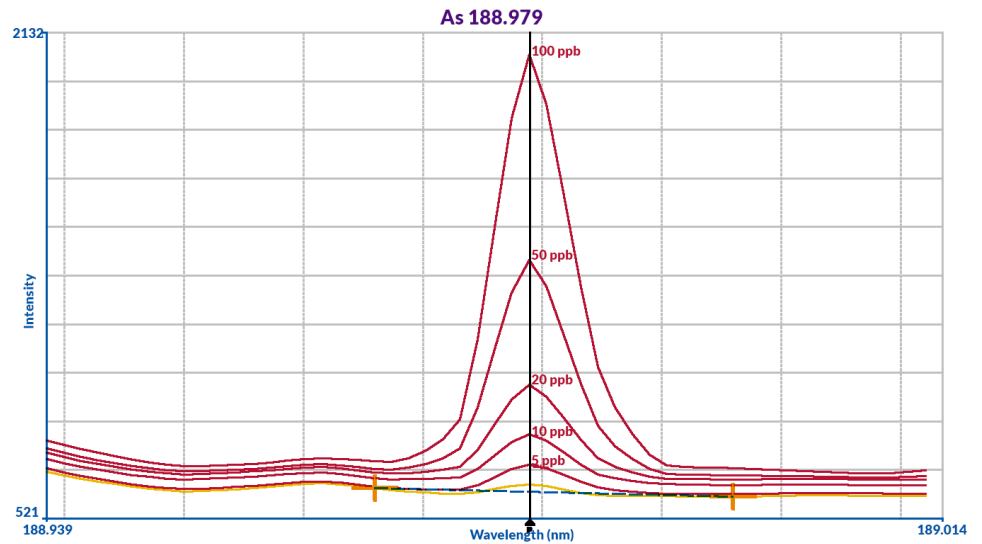


Figure 3a. Spectra overlay of As spikes in municipal water matrix using U5000AT+ USN

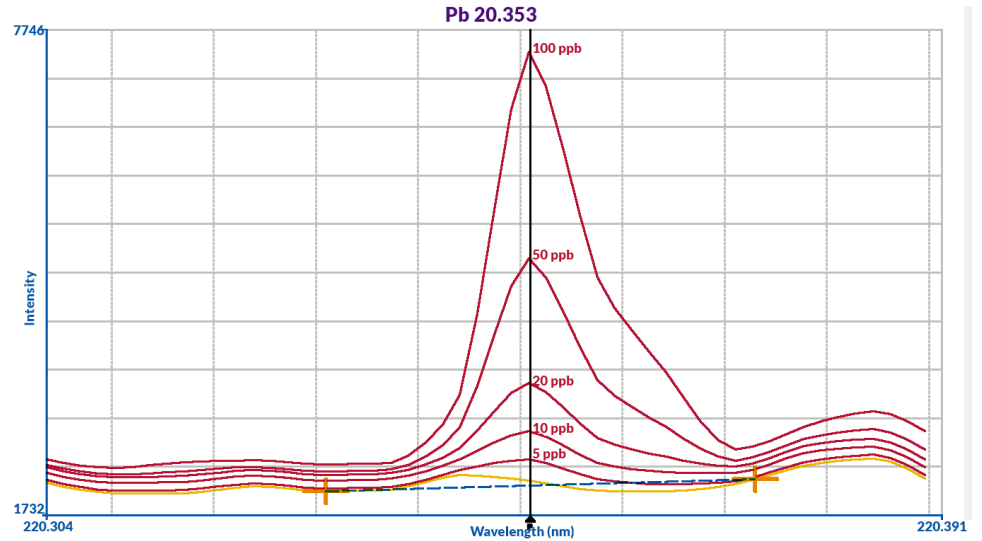


Figure 3b. Spectra overlay of Pb spikes in municipal water matrix using U5000AT+ USN

Detection Limits

Instrument detection limits (IDLs) were obtained for the six selected elements using the U5000AT+ USN with the host ICP-OES. Instrument detection limits were calculated per EPA Method 200.7.

After ICP-OES calibration, the reagent blank was measured 10 times as a sample; the individual IDLs defined as 3x the standard deviation of the reagent blank concentration. The mean of the 10 individual blank runs for each element was calculated as the final IDL per Method 200.7 criteria; a summary including the IDLs, limits of quantitation (LOQs) and the MCLs is given in Table 4. Note the limit of quantitation is 3.3x the IDL and that measured LOQs are all less than the MCL for the target six elements in a municipal water matrix.

Summary and Benefits

The use of the U5000AT+ USN accessory with ICP-OES detection enables LOQs at or below 2 µg/L in a municipal water matrix, meeting USEPA MCL criteria for six important regulated elements: As, Cd, Pb, Sb, Se, and Tl.

Setup of the compact U5000AT+ is fast (10 minutes) and easy with a supplied interface kit. No computer control of the U5000AT+ is required and introduction of samples can be done efficiently with an autosampler and the host ICP-OES peristaltic pump.

References

1. U.S. EPA. 1994. "Method 200.7: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 4.4. Cincinnati, OH

Element	Wavelength (nm)	U5000AT+ USN IDL (µg/L)	U5000AT+ USN LOQ (µg/L)	USEPA MCL in Drinking Water (µg/L)
As	188.797	0.44	1.45	10
Cd	214.440	0.07	2.31	8
Pb	220.353	0.28	0.92	15
Sb	206.836	0.26	0.86	6
Se	196.026	1.4	4.6	50
Tl	190.801	0.34	1.12	2

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