

Application News

Spectrophotometric Analysis

No.A426

Determination of Cadmium in the Tap Water by Flameless Atomic Absorption Spectrometry

■ Introduction

The standard for the allowable concentration of cadmium in tap water has been revised downward from 0.01 mg/L or less to 0.003 mg/L or less according to Japan's "Ministerial Ordinance for Partial Revision of the Ordinance Concerning the Water Quality Standard" (The Ministry of Health, Labour and Welfare Ordinance No.18) (February 17, 2010). The revised standard has been in effect since April 1, 2010.

Of the original analysis methods specified, the flame atomic absorption spectrometry has been removed, with the following 3 methods remaining and specified for use.

1. Flameless atomic absorption spectrometry
2. ICP emission spectrometry
3. ICP mass spectrometry

Here we introduce an example of flameless atomic absorption analysis of tap water spiked with cadmium at 1/10 the concentration specified in the revised ordinance, using the certified river water reference material JAC 0302 (spiked) provided by The Japan Society for Analytical Chemistry. In addition, the convenient automatic dilution and re-analysis function is introduced.

■ Instruments and Analytical Conditions

Table 1 shows the instruments and analytical conditions used.

Table 1 Analytical Conditions Used for Flameless AA

Instrument	Main unit: AA-7700 Atomizer: GFA-7000 Autosampler: ASC-7000, ASK-7000
Analysis Wavelength	228.8 nm
Slit Width	0.7 nm
Current	8 mA
Lamp Mode	BGC-D2
Tube Type	Pyro-coated graphite tube
Injection Volume	2 to 20 μ L (total injection volume is 25 μ L)
Temperature Program	Drying: 120 $^{\circ}$ C Ashing: 500 $^{\circ}$ C Atomizing: 1800 $^{\circ}$ C Cleaning: 2400 $^{\circ}$ C
Standard Solution Concentration	Upper limit concentration: 0.0012 mg/L (1.2 μ g/L)
Matrix Modifier	Palladium nitrate aqueous solution: 5 μ L (palladium content equivalent to 100 ppm)

■ Results

Fig. 1 shows the calibration curve. Making use of the autosampler's automatic dilution and addition function for generating the calibration curve, all that is required to generate the calibration curve is to set the diluent, source solution (2 ppb) of the standard solution, and interference inhibitor (palladium nitrate solution) in the autosampler. Table 2 shows the standard solution mixing conditions for generating the calibration curve using the autosampler. The concentrations are based on an injection volume of 20 μ L. Fig. 2 shows the peak profiles of the standard solutions.

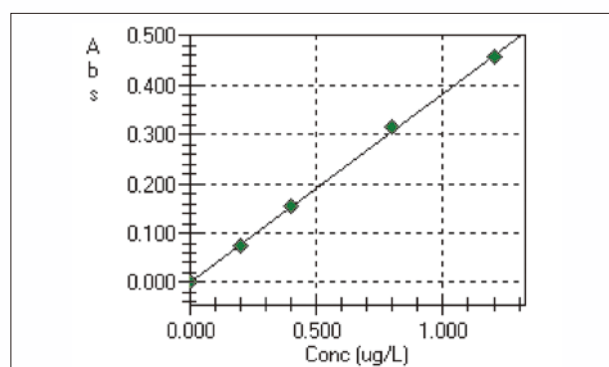


Fig. 1 Calibration Curve of Cadmium

Table 2 Mixing Conditions of Standard Solutions

Concentration	Diluent	2 ppb	Palladium Nitrate	Total Injection Volume
Blank	20 μ L	0 μ L	5 μ L	25 μ L
0.2 ppb	18 μ L	2 μ L	5 μ L	25 μ L
0.4 ppb	16 μ L	4 μ L	5 μ L	25 μ L
0.8 ppb	12 μ L	8 μ L	5 μ L	25 μ L
1.2 ppb	8 μ L	12 μ L	5 μ L	25 μ L

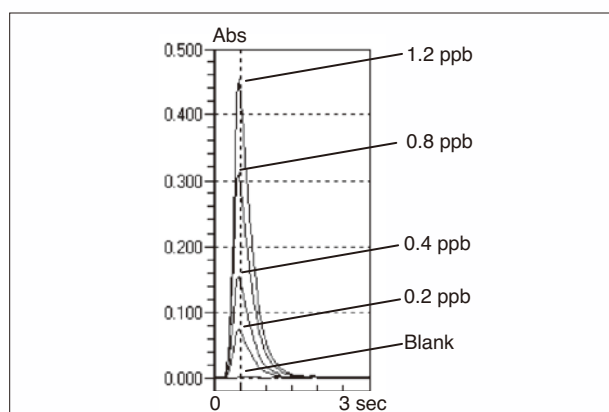


Fig. 2 Peak Profiles of Standard Solutions (Extracted)

Table 3 shows the measurement results. The results of analysis of the river water reference material matched the certified value. As for the sample spiked with cadmium at 1/10 the concentration of the tap

Table 3 Measurement Results

	Certified Value	Measurement Value	%RSD
River Water Reference Material (spiked)	0.00101 mg/L	0.00100 mg/L	2.0 %
Tap Water (unspiked)	–	<0.00002 mg/L	–
Tap Water (spiked)	–	0.00031 mg/L	2.3 %

The measurement value shown is the average of 5 repeat measurements. %RSD is calculated from the values obtained in 5 repeat measurements.

water standard criteria value, excellent results were obtained for both accuracy and precision. Fig. 3 shows the respective peak profiles.

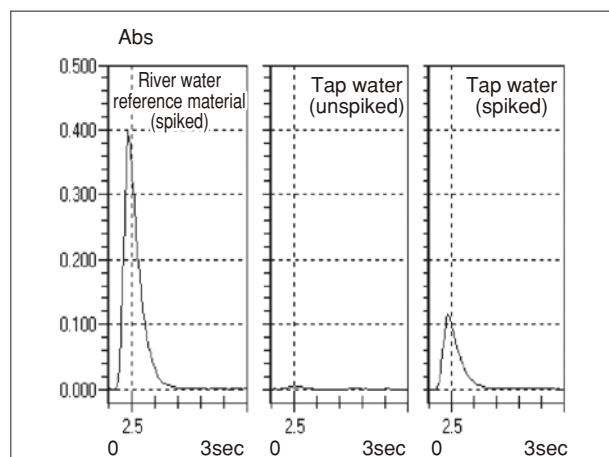


Fig. 3 Peak Profiles of Samples (Extracted)

The AA-7000 autosampler (ASC-7000+ASK-7000) is equipped with an automatic dilution and re-analysis function. Using this function, if the concentration of the unknown sample exceeds the upper limit setting, the amount of sample drawn is automatically reduced and re-analysis is conducted. Fig. 4 shows the window for setting the automatic dilution and re-analysis function. By entering the upper limit concentration of the unknown sample and selecting the automatic dilution and re-analysis checkbox, a smaller amount of sample is drawn and re-analyzed if the current sample measurement result exceeds the set upper limit concentration. Table 4 shows an example of automatic dilution and re-analysis. In this example, the upper

limit concentration of the calibration curve is set to 1.2 ppb, and the measurement of a 5 ppb standard solution is conducted as an unknown sample. In the first measurement, the upper limit concentration is exceeded, so the sample injection volume is automatically reduced from 20 μ L to 3 μ L, and the sample is re-analyzed. Using automatic dilution and re-analysis, the dilution factor is automatically entered in the automatic dilution column, and is reflected in the actual concentration column. This automatic dilution and re-analysis function reduces the time and effort normally expended by the analyst in conducting this re-analysis operation.

Measurement Sequence for Calibration Curve

No. of Lines

Auto Dilution & Remeasurement ☒ UNK. Sample Conc. Upper Limit:

	Action	Sample ID	True Value	Pos.	VOL (μ L)	Diluent R1	Cd2ppb R2	Pd100ppm R3	Reagent R4
	STD	Blank	0.0000	R1	20	0	0	5	0
	STD	Std-1	0.2000	R2	2	18	0	5	0
	STD	Std-2	0.4000	R2	4	16	0	5	0
	STD	Std-3	0.8000	R2	8	12	0	5	0
	STD	Std-4	1.2000	R2	12	8	0	5	0

Fig. 4 Setting for Automatic Dilution and Re-Analysis

Table 4 Example of Automatic Dilution and Re-Analysis

Action	Sample ID	Exclude	Conc.	Abs.	Pos.	Vol.	Diluent	Palladium Nitrate	Total	Dilution Factor	Auto Dilution	Actual Conc.	Conc. Unit
UNK1	5 ppb	X	3.475	1.332	6	20	0	5	25	1		3.47	ppb
UNK1-1	5 ppb		0.765	0.294	6	3	17	5	25	1	6.67	5.10	ppb
UNK1-2	5 ppb		0.757	0.291	6	3	17	5	25	1	6.67	5.05	ppb
UNK1-3	5 ppb		0.741	0.284	6	3	17	5	25	1	6.67	4.94	ppb
UNK1-4	5 ppb		0.727	0.279	6	3	17	5	25	1	6.67	4.85	ppb
UNK1-5	5 ppb		0.766	0.294	6	3	17	5	25	1	6.67	5.11	ppb
UNK1-AV	5 ppb		0.751	0.289	6	3	17	5	25	1	6.7	5.01	ppb



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