SHIMADZU

# 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<br>Atomizer: GFA-7000<br>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 µL (total injection volume is 25 µL) |
| Temperature Program | Drying: 120 °C<br>Ashing: 500 °C<br>Atomizing: 1800 °C<br>Cleaning: 2400 °C |
| Standard Solution Concentration | Upper limit concentration:<br>0.0012 mg/L (1.2 µg/L) |
| Matrix Modifier | Palladium nitrate aqueous solution: 5 µL<br>(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 µL. Fig. 2 shows the peak profiles of the standard solutions.

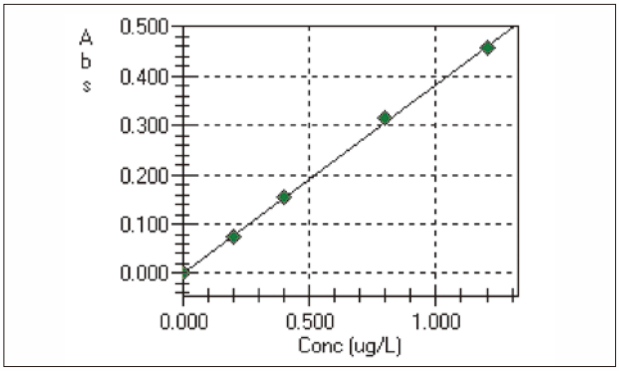


**Fig. 1 Calibration Curve of Cadmium**

**Table 2 Mixing Conditions of Standard Solutions**

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

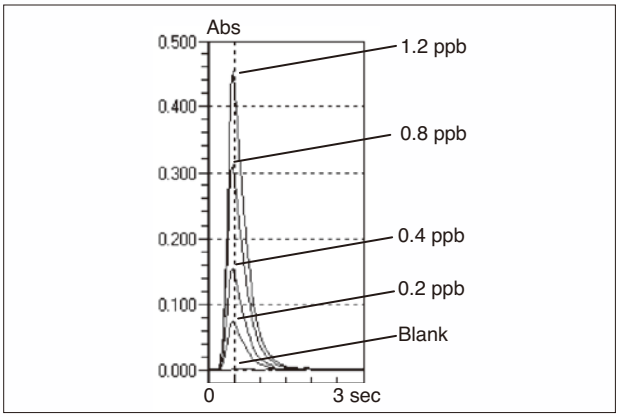


**Fig. 2 Peak Profiles of Standard Solutions (Extracted)**

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

**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.

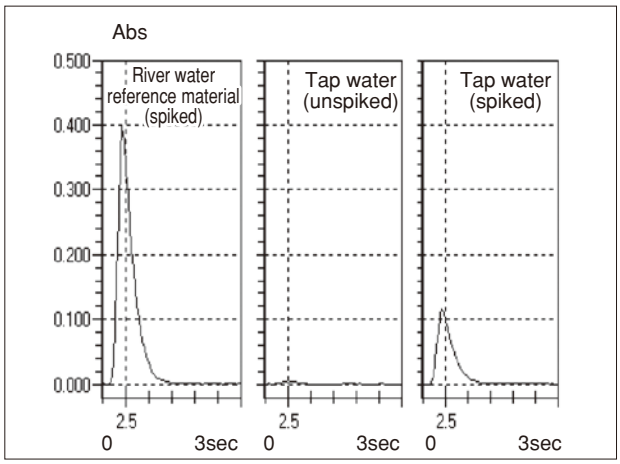


**Fig. 3 Peak Profiles of Samples (Extracted)**

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 µL to 3 µL, 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.

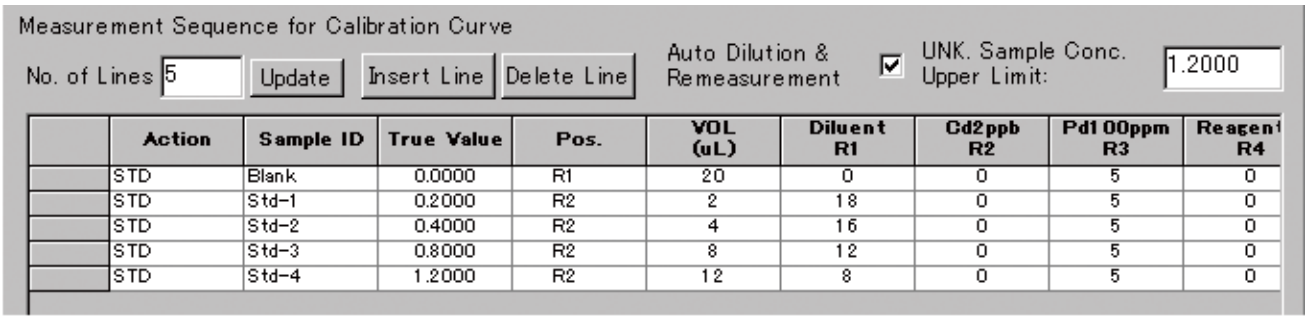


**Fig. 4 Setting for Automatic Dilution and Re-Analysis**

**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** |

SHIMADZU
SHIMADZU CORPORATION. International Marketing Division
3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO