

Application News

Spectrophotometric Analysis

No.A427

Determination of Cadmium in Brown Rice by Flame Atomic Absorption Spectrometry

■ Introduction

The standard for cadmium in rice based on Japan's Food Sanitation Act has been strengthened, with the allowable concentration revised downward from 1.0 mg/kg or less to a maximum of 0.4 mg/kg according to the Ministry of Health, Labour and Welfare Notification No. 183 (April 8, 2010). The revised standard will take effect on February 28, 2011. Up to now, this type of restriction was applied only to brown rice, but polished rice is now included in this revision.

Here we introduce examples of analysis of cadmium in standard reference materials of powdered brown rice (NIES No.10-a,-b) using the typical flame AA method, in addition to a high-sensitivity method using an atom booster.

■ Sample Preparation

Sample preparation was conducted based on the Ministry of Health, Labour and Welfare Notification No. 370 (1959) using thermal decomposition with acid followed by solvent extraction.

The decomposition process is shown in the flow chart of Fig. 1, and the solvent extraction process is shown in the Fig. 2 flow chart. The standard solution was prepared by diluting a 1000 mg/L standard solution for atomic absorption analysis to obtain a 1 mg/L standard solution, and add this little by little into a separatory funnel, and conduct solvent extraction in the same way as for the decomposition process.

Weigh 10 g sample into 500 mL beaker

← Add about 20 mL distilled water.
← Add 40mL nitric acid and mix well.

Cover with watch glass and heat gently (at about 180 °C for 1 hour)

← Add 20 mL sulfuric acid.

Cover with watch glass and heat (to about 230 °C)

← Add a small amount of nitric acid (1 to 2 mL) a little at a time until solution turns colorless or pale yellow.

Cool

Bring volume to 100 mL (using decomposition processing solution)

Fig. 1 Sample Decomposition Procedure Flow Chart

Transfer the appropriate amount of decomposition processing solution to a 200 mL separatory funnel

(50 mL for No. 10-a, 20 mL for No. 10-b)

- ← Add 5 mL of 25 % potassium sodium tartrate solution.
- ← Add BTB solution (pH indicator reagent).
- ← Neutralize with aqueous ammonia (until color turns from pale yellow to bluish-purple).
- ← Add distilled water to bring total volume to about 100 mL.
- ← Add 10 mL of saturated sodium sulfate solution.
- ← Add 5 mL of 1 % DDTC solution (chelate reagent).

Mix and let stand several minutes

← Accurately add 10 mL MIBK

Shake vigorously for about 5 min

Set aside and let stand

Discard aqueous layer, transfer MIBK layer to test tube (sample measurement solution)

BTB: bromthymol blue

DDTC: sodium diethyldithiocarbamate

MIBK: methyl isobutyl ketone (4-methyl-2-pentanone)

Fig. 2 Flow Chart of Organic Solvent Extraction Process

Analytical Method and Conditions

Measurement was conducted using both the typical flame atomic absorption spectrometry, and for high sensitivity, using an atom booster attached on top of the burner head. The atom booster is a 15 cm-long quartz tube with slits cut into and along the top and bottom of the tube. This tube is mounted on top of the burner, extending the time that atoms are retained in the flame. This augments the density of atoms, thereby improving absorption. It is effect for measurement not only of Cd, but for Pb, Cu, Mn and Ni, as well. For details regarding the atom booster, please refer to the reference document 1, cited below. The main measurement parameters are shown in Table 1 and 2. Since organic solvent nebulization is conducted, the acetylene flowrate should be set to a lower value than that for an aqueous sample.

Table 1 Analytical Conditions

Analysis Wavelength	228.8 nm
Slit Width	0.7 nm
Current	8 mA
Lamp Mode	BGC-D2

Table 2 Atomization Parameters

Flame Type	Air-C ₂ H ₂	
Acetylene Flowrate	0.8 L/min	
Burner Height	9 mm (without booster), 13 mm (with booster)	

■ Results

Fig. 3 and 4 show the respective calibration curves and measurement peak profiles. It is clear that the absorbance using the booster is more than twice that without the booster in the typical flame atomic absorption analysis. The standard solution concentration is the value for the solvent after extraction processing.

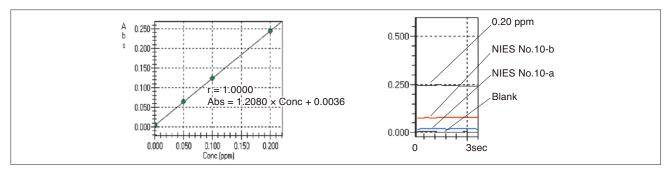


Fig. 3 Calibration Curve and Peak Profiles without Atom Booster

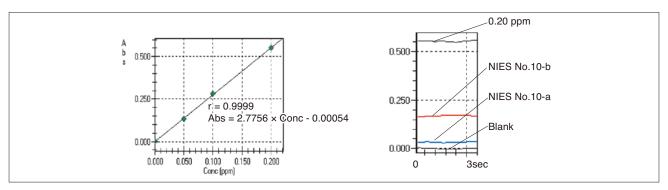


Fig. 4 Calibration Curve and Peak Profiles with Atom Booster

Table 3 shows the measurement results. The results obtained using both methods matched the certified value. Aside from the flame atomic absorption spectrometry, this analysis can also be conducted

using the furnace atomic absorption spectrometry as well as ICP emission spectrometry. Please refer to the reference documents 2 and 3 regarding these other methods.

Table 3 Measurement Results for Cd in Brown Rice

	NIES No.10-a	NIES No.10-b
	(0.023 ± 0.003 mg/kg)	$(0.32 \pm 0.02 \text{ mg/kg})$
Without booster	0.024 mg/kg	0.31 mg/kg
With booster	0.024 mg/kg	0.31 mg/kg

■ References

- 1: Shimadzu Application News No. A277 "Flame Atomic Absorption Spectrometry Using an Atomic Booster"
- 2: Shimadzu Application News No. A348 "Measurement of Cadmium in Rice"
- 3: Shimadzu Application News No. J87 "Multi-Element Simultaneous Determination of Nutrient as well as Hazardous Elements in Brown Rice by ICPE-9000"

