SHIMADZU

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.

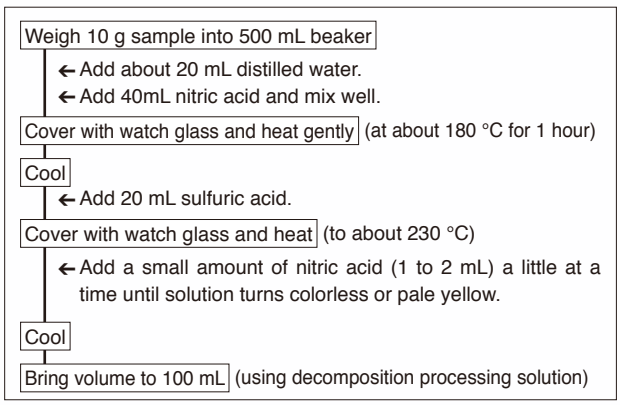

Fig. 1 Sample Decomposition Procedure Flow Chart

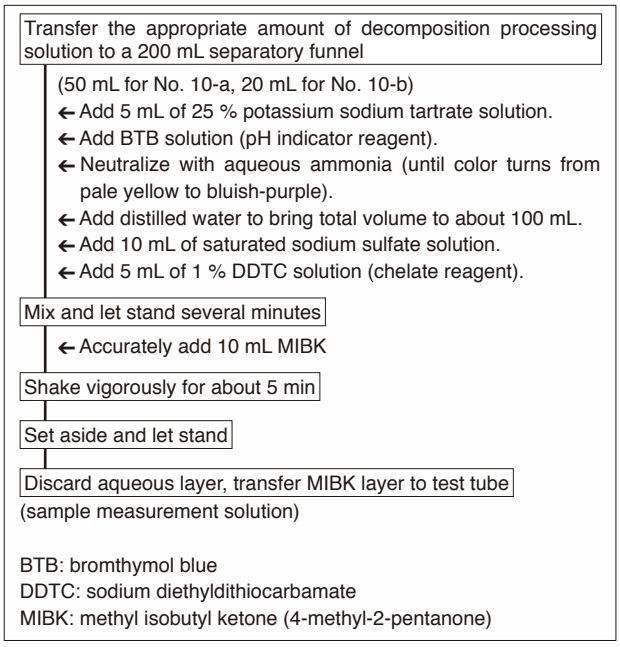

Fig. 2 Flow Chart of Organic Solvent Extraction Process

■ 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_2H_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.

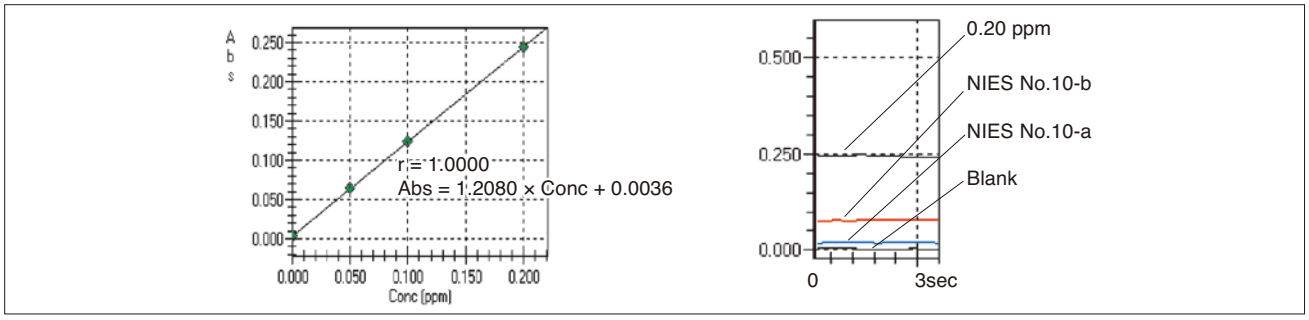

Fig. 3 Calibration Curve and Peak Profiles without Atom Booster

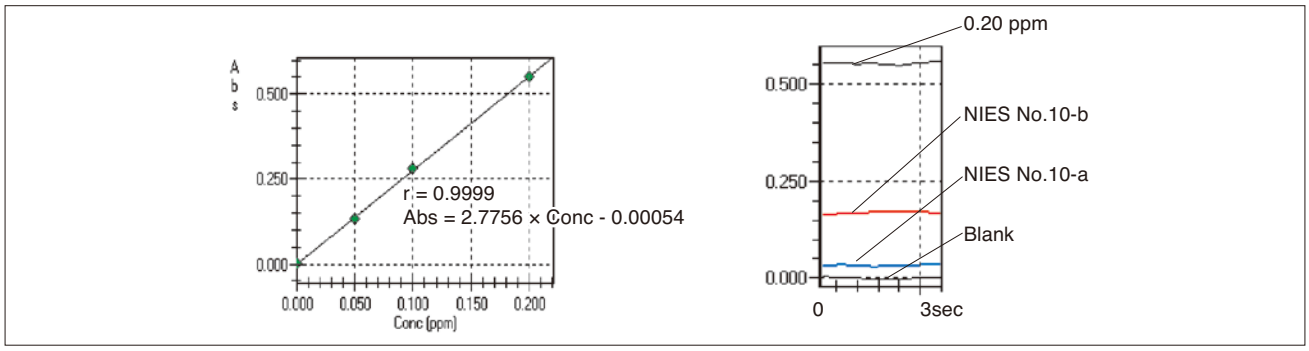

Fig. 4 Calibration Curve and Peak Profiles with Atom Booster

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 (0.023 ± 0.003 mg/kg)	NIES No.10-b (0.32 ± 0.02 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"

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