

Analysis of Cadmium in Brown Rice by Flame Atomic Absorption Spectrometry

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User Benefits

- ◆ Cadmium in brown rice can easily be analyzed by a diluted acid extraction method.
- ◆ Positioning the atom booster above the burner head increases how long atoms are delayed inside the flame, which increases absorbance by about 2 to 3 times by increasing the atomization density.

Introduction

Cadmium is found in many foods, including rice, vegetables, fruits, meat, and fish. In Japan, the highest intake of cadmium comes from rice, which is estimated to account for about 40% of total intake. In 2010, the Food Sanitation Act was amended to specify that "brown rice and polished rice should not contain more than 0.4 ppm (mg/kg) of cadmium," with the regulation was strengthened from the previous standard of less than 1.0 ppm (mg/kg).

The current official method requires the wet ashing of brown rice samples with strong acid (sulfuric acid and nitric acid) followed by extraction with methyl isobutyl ketone using a chelating agent (sodium diethyldithiocarbamate). That method has problems with taking a long time for decomposition, poor operating efficiency, analyst exposure to hazardous materials, and disposal of liquid waste.

This article describes using a simple screening method (not compliant with the official method) that can quickly provide high sensitivity cadmium measurement results by extracting and filtering brown rice components with diluted acid and measuring it directly in an AA-7800 atomic absorption spectrophotometer.

Sensitive Analysis with Atom Booster

There are two basic methods for atomic absorption spectrometry, either the flame method or the graphite furnace method. When microanalysis is required, the graphite furnace method is often used, but there are many cases where the rapid and reliable flame method is preferable.

The atom booster is a quartz tube with an overall length of about 15 cm that has vertical slits at the top and bottom. (The lower slit is 100 mm long and the upper slit is 80 mm long.) The upper slit is shorter than the lower slit, so atoms stay longer in the flame. That increases the density of atomized elements, leading to improved absorbance. It is especially effective for elements that atomize at relatively low temperatures (Cd, Pb, Cu, etc.).



Fig. 1 Atom Booster

Sample and Pretreatment

Sample: A certified reference material of brown rice powder for cadmium analysis (NMIJCRM 7531-a) was used as a sample.

Sample Pretreatment: 2 g of brown rice powder was placed in a container, 20 mL of 1 mol/L nitric or hydrochloric acid was added, the lid was closed, and the container was shaken at room temperature for 1 hour. Then the contents were filtered through a filter paper and the filtrate was used as the analytical sample.

System Configuration and Analytical Conditions

A Shimadzu AA-7800 atomic absorption spectrophotometer was used. The main analytical conditions for the spectrophotometer and flame analysis are shown in Table 1. The analysis was performed by the calibration curve method.

Table 1 Analytical Conditions for Flame Analysis

Element	Cd
Analytical Instrument	AA-7800F
Analysis Wavelength	228.8 nm
Slit Width	0.7 nm
Lighting Mode	BGC-D2
Height of Burner	7 mm (When using atom booster: 13 mm)
Type of Flame	Air-C ₂ H ₂
C ₂ H ₂ Flowrate	1.8 L/min
Integration Time	5 s
Repetitions	3 times

Analytical Results

Analytical results of the samples are shown in Table 2. Dilute nitric acid and dilute hydrochloric acid were compared as extractants and measurements with and without the atom booster were compared.

A comparison of the 1 mol/L solutions of nitric acid and hydrochloric acid as extractants determined that the extraction rate of nitric acid was higher, with almost all the cadmium in brown rice extracted.

When using the atom booster, the absorbance increased by about 2.5 times compared to the normal flame method. The calibration curves for Cd are shown in Fig. 2 and Fig. 3. All calibration curves showed good linearity.

Table 2 Cd Analytical Result

Acid Type	Atom Booster	Analytical Result (mg/L)	SD	Concentration (mg/kg)
1 mol/L HNO ₃	No	0.0297	0.0006	0.299
	Yes	0.0295	0.0005	0.298
1 mol/L HCl	No	0.0284	0.0003	0.288
	Yes	0.0271	0.0002	0.275
Certified Value (mg/kg)		0.308 ± 0.007		

Table 3 Limit of Quantification (Cd)

Acid Type	Atom Booster	LOQ in Solid (mg/kg)
1 mol/L HNO ₃	No	0.07
	Yes	0.03
1 mol/L HCl	No	0.1
	Yes	0.04
Reference Value	0.4 ppm (mg/kg)	

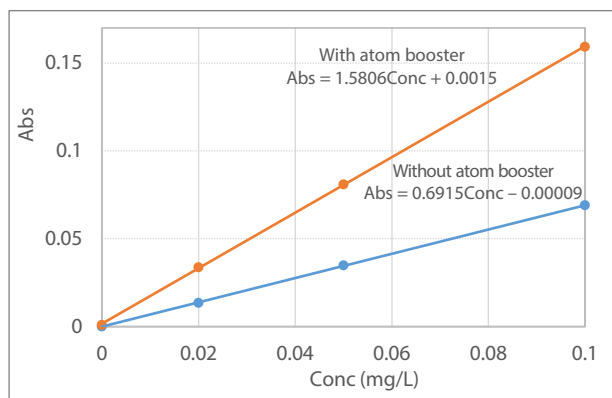


Fig. 2 Calibration Curve of Cd (HNO₃ Extract)

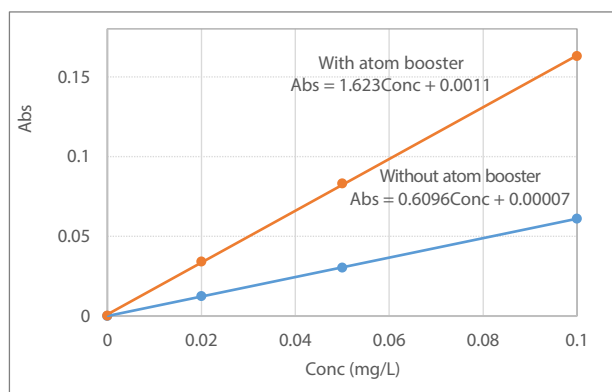


Fig. 3 Calibration Curve of Cd (HCl Extract)

Table 3 shows the limit of quantification (LOQ) for Cd in solids. The LOQ is expressed as the value of 10σ calculated from the standard deviation (SD) obtained by measuring the calibration curve blank 10 times and converting the results for the actual sample. Using the atom booster, the LOQ was less than 1/10 of the reference value, which was a good result.

Conclusion

Cadmium in brown rice can be easily analyzed by a simple screening method using an AA-7800 atomic absorption spectrophotometer (however, not in compliance with official methods). Formal analysis must be compliant with official methods, but simple screening can be used prior to official methods to quickly determine the probability of exceed the threshold.

Also, by setting the atom booster on the burner head, the absorbance can be improved by about 2 to 3 times compared to the normal flame method.

AA-7800 series spectrophotometers can be upgraded with additional units to evolve the system according to what is analyzed.



AA-7800F Flame Model

References

- 1) Food Safety Division, Pharmaceutical and Food Safety Bureau, the Ministry of Health, Labour and Welfare, Q & A on "Cadmium in Foods" Revised July 2010
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- 3) Standard Methods of Analysis in Food Safety Regulation: Japan Food Hygiene Association
- 4) Application News A427