SHIMADZU
Excellence in Science

Application News

No. A583

Atomic Absorption Spectrophotometry

Measurement of Lead (Pb) Content in L-Ascorbic Acid (Vitamin C) According to the Ninth Edition of Japan's Specifications and Standards for Food Additives

Japan's Specifications and Standards for Food Additives was updated from the eight edition to the ninth edition in 2018. For many additives, the heavy metals limit test as part of the purity test which was based on turbidity is now replaced by the measurement of lead by atomic absorption spectrophotometry.

This article introduces a measurement of the lead content in L-ascorbic acid (vitamin C), a widely known food additive, by performing preparation and flame atomic absorption spectrophotometry as described in the ninth edition.

T. Kawakami

Preparation

The ninth edition specifies preparation methods 1 to 5 as indicated in Table 1. For many additives, one of these five methods is specified. The basic method is to add sulfuric acid to the sample, carbonize it on a hot plate, then incinerate it in an electric furnace, and finally dissolve the ashes in acid (Methods 1 and 2). For additives that have a high salt content, the sample is further subjected to chelate extraction using ammonium pyrrolidine dithiocarbamate (APDC) and butyl acetate after dissolution or digestion (Methods 3 to 5). Depending on the additive, the preparation method may be defined separately.

L-ascorbic acid is prepared using Method 1 and the obtained test solution undergoes measurement by flame atomic absorption spectrophotometry. The sample weight is 2 g and the lead content is to be no more than 2 µg/g when in solid form (no more than 0.4 mg/L in the test solution).

Fig. 1 shows the preparation procedure used for L-ascorbic acid this time. Fig. 2 shows the sample as it is being carbonized and the sample after incineration.

Table 1 Description of Preparation Methods

Name	Description
Method 1 and Method 2	The sample is added sulfuric acid, carbonized by heating, and then incinerated in an electric furnace. The resulting ashes are dissolved in acid.
Method 3	The sample is added sulfuric acid, carbonized by heating, and then incinerated in an electric furnace. After dissolving the resulting ashes in acid, the sample is subjected to chelate extraction using APDC and butyl acetate.
Method 4	After wet digestion, chelate extraction is performed using APDC and butyl acetate.
Method 5	After dissolving, chelate extraction is performed using APDC and butyl acetate.

1. Weigh 2.0 g of the sample and place it in a quartz (or porcelain) crucible.
2. Moisten the entire sample by adding a small amount of sulfuric acid (1 in 4 solution).
3. Heat the sample on a hot plate until it is completely carbonized.
4. Place a cover on the sample and place it in an electric furnace.
5. Gradually raise the temperature to 550 to 600 °C and maintain until completely incinerated.
6. After cooling, add 10 mL of hydrochloric acid (1 in 4 solution) to the residues.
7. Dry the solution by evaporation using a heated bath.
8. Add a small amount of nitric acid (1 in 100 solution) to the residues and dissolve by heating.
9. After cooling, add nitric acid (1 in 100 solution) to make exactly 10 mL. (Test solution)
10. Measure using flame atomic absorption spectrophotometry.

Fig. 1 Preparation Procedure

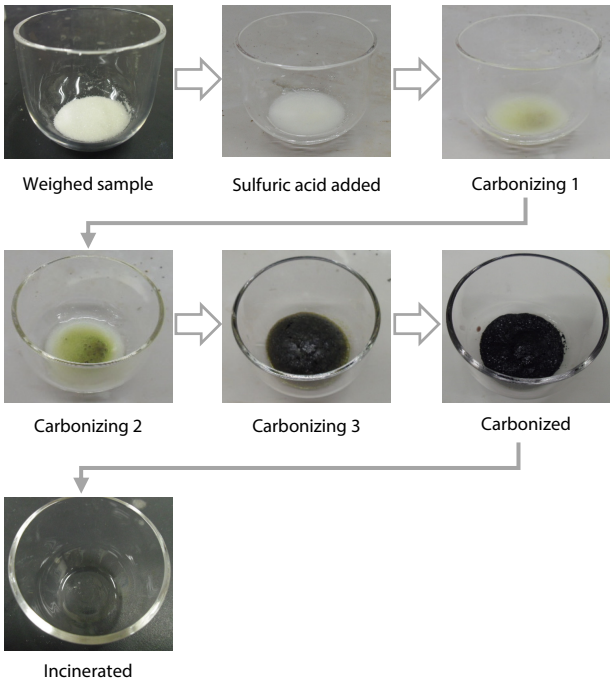

Fig. 2 Sample Appearances During Carbonization and After Incineration

Instrument Configuration and Measurement Conditions

We used the flame model AA-7000F atomic absorption spectrophotometer (Fig. 3) for measurement.

By adding optional units, the AA-7000F can be upgraded to a dual atomizer system (Fig. 4) that is also capable of high-sensitivity furnace analysis.

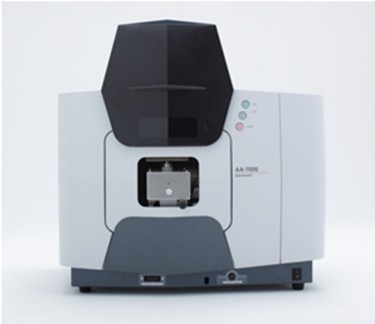

Fig. 3 Flame Model AA-7000F

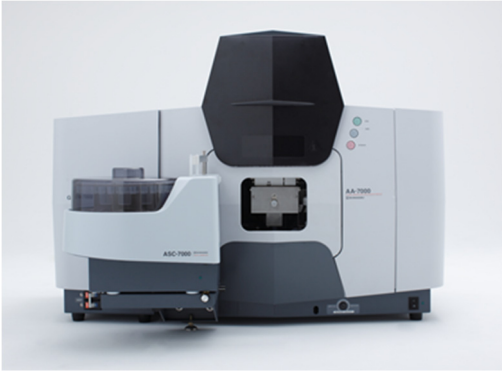

Fig. 4 AA-7000F/AAC Dual Atomizer System (With Options)

Table 2 lists the main measurement conditions.

The ninth edition judges lead content by comparison with a 0.4 mg/L standard solution (equivalent to 2 ppm for L-ascorbic acid in solid form). This time, we prepared five standard solutions including a blank solution. We also measured a solution prepared by adding 0.2 mg/L (1 µg/g in solid form) of the standard to the test solution before carbonization so as to determine the spike and recovery rate.

Table 2 Measurement Conditions

Measurement Element	: Lead (Pb)
Analytical Wavelength	: 283.3 nm
Slit Width	: 0.7 nm
Background Correction	: Deuterium lamp method
Flame Type	: Air-acetylene
Burner Height	: 7 mm
Burner Angle	: 0°
Integration Time and Repetition Times	: 3 s × three times
Standard Concentration	: 0 mg/L (0.0 µg/g)* 0.1 mg/L (0.5 µg/g) 0.2 mg/L (1.0 µg/g) 0.3 mg/L (1.5 µg/g) 0.4 mg/L (2.0 µg/g)

*: Values in parentheses are solid concentration conversions.

Measurement Results

The measurement results of the standard solutions are listed in Table 3 and the calibration curve is shown in Fig. 5. The quantitation limit of lead was 0.1 mg/L.

Table 4 shows the measurement results of L-ascorbic acid. The lead concentration was below the quantitation limit (less than 0.5 µg/g in solid form) and the spike and recovery rate was favorable at 95 %.

Table 3 Measurement Results of the Standard

Set Concentration	Absorbance (n=3)	%RSD (n=3)	Standard Deviation (n=3)
0 mg/L	0.0003	46	0.0002
0.1 mg/L	0.0029	7.3	0.0002
0.2 mg/L	0.0055	3.8	0.0002
0.3 mg/L	0.0083	3.0	0.0003
0.4 mg/L	0.0107	1.6	0.0002

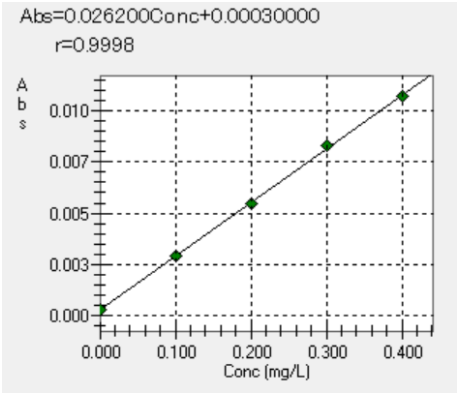

Fig. 5 Calibration Curve of Lead (Pb)

Table 4 Measurement Result of Lead (Pb) Content in L-Ascorbic Acid

Concentration in Test Solution	Concentration in Solid	Spiked Sample Concentration in Test Solution	Spike and Recovery Rate
<0.1 mg/L	<0.5 µg/g	0.19 mg/L	95 %

Conclusion

We measured the lead content in L-ascorbic acid (vitamin C) using the Method 1 preparation procedure (acid dissolution after incineration) and flame atomic absorption spectrophotometry as directed by the ninth edition of Japan's Specifications and Standards for Food Additives. The spike and recovery rate was favorable and we confirmed that quantitation of 2 µg/g, which is the criterion, is possible.

The AA-7000 incorporates a double-beam optical system. With low noise, it achieves a stable baseline immediately after lamp lighting.

The burner head is made of titanium and highly corrosion-resistant. In addition, the air-cooled burner head can be easily mounted/dismounted and cleaned, facilitating maintenance work.

First Edition: Nov. 2018

Shimadzu Corporation
www.shimadzu.com/an/