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Method of Test for Heavy Metals in Metal Canned Foods - Test of Tin

1. Scope

This method is applicable for the determination of tin in metal canned foods.

2. Method

After microwave assisted acid digestion, tin is determined by inductively coupled plasma optical emission spectrometry (ICP-OES).

2.1. Equipment

2.1.1. Inductively coupled plasma optical emission spectrometer.

2.1.2. Blender.

2.1.3. Microwave digester: > 1000 W with temperature control and pressure feedback system.

2.1.4. Acid steam cleaning system.

2.2. Chemicals

Nitric acid, ultrapure grade (67-70%) and reagent grade;

Hydrochloric acid (34-37%) and hydrogen peroxide (30%), ultrapure grade;

Deionized water, resistivity ≥ 18 MΩ • cm (at 25°C);

Tin, 1000 μg/mL, reference standard, ICP grade.

2.3. Apparatus

2.3.1. Microwave digestion flask(Note): quartz or Teflon.

2.3.2. Volumetric flask(Note): 50 mL.

2.3.3. Storage tube: 50 mL, PP.

2.3.4. Membrane filter: 0.45 μm, PTFE.

Note: After cleaning, use an acid steam cleaning system to clean the apparatus with nitric acid (reagent grade) vapor for 2 hours, or soak the apparatus in nitric acid (reagent grade) : water (1:1, v/v) overnight. Take the apparatus out, wash away the residual nitric acid with deionized water and dry.

2.4. 5% (w/w) nitric acid containing 0.2% (w/w) hydrochloric acid

Add 50 mL of nitric acid (ultrapure grade) and 5 mL of hydrochloric acid (ultrapure grade) slowly into 500 mL of deionized water, and

dilute to 1000 mL with deionized water.

2.5. Standard solution preparation

Accurately transfer 0.5 mL of tin reference standard to a 50-mL volumetric flask, make up to volume with 5% (w/w) nitric acid containing 0.2% (w/w) hydrochloric acid, and transfer to a storage tube as the standard stock solution. When to use, take appropriate amount of the standard stock solution, dilute with 5% (w/w) nitric acid containing 0.2% (w/w) hydrochloric acid to 0-2 μg/mL, and transfer to storage tubes as the standard solutions.

2.6. Sample solution preparation

All contents were taken out and homogenized after opening the metal can. Transfer about 0.5 g of the homogenized food sample or 0.4 g of the edible fat and oil sample accurately weighed into a microwave digestion flask, add 6 mL of nitric acid (ultrapure grade) and 1 mL of hydrogen peroxide, and then digest according to the following conditions. After cooling to room temperature, transfer to a 50-mL volumetric flask. Wash and rinse the residue in the digestion flask with 5 mL of deionized water several times. Add the washings and 0.25 mL of hydrochloric acid to the same volumetric flask, and make up to volume with deionized water. Transfer to a storage tube, filter with a membrane filter, and take the filtrate as the sample solution. Take an empty microwave digestion flask, add 6 mL of nitric acid (ultrapure grade) and 1 mL of hydrogen peroxide, and perform the same procedure described above as the blank solution.

Microwave digester operating conditions(Note):

Condition / Step	Power (W)	Heating time (min)	Duration time (min)	Temperature (°C)
1	1000	5	0	100
2	1000	15	5	220
3	1800	10	10	240

Note: All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

2.7. Quantification

Inject the sample solution, the blank solution and the standard solutions into the ICP-OES separately at the appropriate rate, and

operate according to the following conditions. Calculate the amount of tin in the sample based on the signal intensity of tin by the following formula:

$$\text{The amount of tin in the sample (mg/kg)} = \frac{(C - C_0) \times V}{M}$$

Where,

C: the concentration of tin in the sample solution calculated by the standard curve (μg/mL)

C_0: the concentration of tin in the blank solution calculated by the standard curve (μg/mL)

V: the final make-up volume of the sample (mL)

M: the weight of the sample (g)

ICP-OES operating conditions(Note):

Radiofrequency power: 1200 W.

Plasma argon flow rate: 12 L/min.

Auxiliary argon flow rate: 1.0 L/min.

Nebulizer argon flow rate: 0.7 L/min.

Wavelength: tin, 189.925 nm.

Note: All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

Remark: 1. The limits of quantification (LOQs) for tin are 5 mg/kg in metal canned foods, and 10 mg/kg in metal canned edible fats and oils.

2. Further validation should be performed when interference compounds appear in samples.

3..When the test is conducted by other instruments, verification by the certified reference material (CRM) or the standard reference material (SRM), or validation of the method should be performed.

Reference

1. U.S. Food and Drug Administration. 2015. Elemental analysis manual for food and related products. 4.7. Inductively coupled plasma - mass spectrometric determination of arsenic, cadmium, chromium, lead, mercury, and other elements in food using microwave assisted digestion.

[https://www.fda.gov/media/87509/download]

2. Morte, E. S. B., Barbosa, I. S., Santos, E. C., Nóbrega, J. A. and Korn, M. G. A. 2012. Axial view inductively coupled plasma optical emission spectrometry for monitoring tin concentration in canned tomato sauce samples. Food Chem. 131: 348-352.