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Method of Test for Selenium in Cosmetics

1. Scope

This method is applicable for the determination of selenium in cosmetics.

2. Method

After microwave assisted acid digestion, selenium is determined by inductively coupled plasma mass spectrometry (ICP-MS).

2.1. Equipment

2.1.1. Inductively coupled plasma mass spectrometer.

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

2.1.3. Acid steam cleaning system.

2.2. Chemicals

Nitric acid, ultrapure grade and reagent grade;
Isopropanol, analytical reagent;
Hydrogen peroxide (30%), ultrapure grade;
Deionized water, resistivity ≥ 18 MΩ • cm (at 25°C);
Selenium, 1000 μg/mL, reference standard, ICP grade;
Rhodium, 1000 μg/mL, internal 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. 10% (w/w) nitric acid

Add 100 mL of nitric acid (ultrapure grade) slowly into 500 mL of deionized water, and dilute to 1000 mL with deionized water.

2.5. Internal standard solution preparation

Accurately transfer 0.5 mL of rhodium internal standard to a 50-mL volumetric flask, make up to volume with 10% (w/w) nitric acid, and transfer to a storage

tube as the internal standard stock solution. When to use, dilute appropriate amount of the internal standard stock solution with 10% (w/w) nitric acid to 1000 ng/mL as the internal standard solution.

2.6. Standard solution preparation

Accurately transfer 0.5 mL of selenium reference standard to a 50-mL volumetric flask, make up to volume with 10% (w/w) nitric acid, and transfer to a storage tube as the standard stock solution. When to use, mix appropriate amount of the standard stock solution, internal standard solution and isopropanol, dilute with 10% (w/w) nitric acid to 0~200 ng/mL (containing 20 ng/mL internal standard and 0.5%(v/v) isopropanol), and transfer to storage tubes as the standard solutions.

2.7. Standard curve preparation

Inject the standard solutions into the ICP-MS at the appropriate rate, and operate according to the following conditions. Establish the standard curve of selenium by the ratio of the signal intensity of selenium to that of rhodium vs. the added concentration.

ICP-MS operating conditions (Note):

Radiofrequency power: 1550 W.
Plasma argon flow rate: 15 L/min.
Auxiliary argon flow rate: 0.9 L/min.
Nebulizer argon flow rate: 1.0 L/min.
Collision gas and flow rate: helium; 4.5 mL/min
Atomic mass (m/z): selenium: 78, 82;
rhodium: 103

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

2.8. Sample solution preparation

Transfer about 0.25 g of the homogenized sample accurately weighed into a microwave digestion flask, add 1 mL of the internal standard solution, 12 mL of nitric acid (ultrapure grade) and 3 mL of hydrogen peroxide (ultrapure grade), 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 to the same volumetric flask, then add 0.25 mL of isopropanol 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, and 1 mL of the internal standard solution, 12 mL of nitric acid (ultrapure grade) and 3 mL of hydrogen peroxide (ultrapure grade), 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 (℃)
1	1800	25	20	210

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

2.9. Quantification

Inject the sample solution, the blank solution and the standard solutions into the ICP-MS respectively at the appropriate rate, and operate according to the conditions described in section 2.7. Calculate the amount of selenium in the sample based on the ratio of the signal intensity of selenium to that of rhodium by the following formula:

$$\text{The amount of selenium in the sample (ppm)} = \frac{(C - C_0) \times V}{M \times 1000}$$

Where,

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

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

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

M: the weight of the sample (g)

Remark

1. The limit of quantification (LOQ) for selenium is 2.0 ppm.
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 standard reference material (SRM), or validation of the method should be performed.

Reference

1. U.S. Food and Drug Administration. 2020. 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. Larsen, E. H. and Stürup, S. 1994. Carbon-enhanced inductively coupled plasma mass spectrometric detection of arsenic and selenium and its application to arsenic speciation. J. Anal. At. Spectrom. 9: 1099-1105.