Published, Aug 24, 2020
RA03H005.001

Method of Test for Heavy Metals in Cosmetics

1. Scope

This method is applicable to the determination of chromium, cobalt, nickel, arsenic, strontium, zirconium, cadmium, antimony, barium, mercury, and lead in cosmetics.

2. Method

After microwave assisted acid digestion, heavy metals are 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: power output > 1000 W, and with temperature control and pressure feedback system.

2.1.3. Acid steam cleaning system.

2.2. Chemicals

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

Hydrogen peroxide (30%), ultrapure grade;

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

Chromium, cobalt, nickel, arsenic, strontium, zirconium, cadmium, antimony, barium, mercury, lead, and gold, 1000 μg/mL, reference standards, ICP grade;

Geranium, rhodium, and bismuth, 1000 μg/mL, internal standards, 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 geranium, rhodium, and bismuth internal standard to each 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 250 ng/mL as the internal standard solution.

2.6. Gold standard solution preparation

Accurately transfer 5 mL of gold 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 gold standard stock solution. When to use, mix appropriate amount of the gold standard stock solution, dilute with 10% (w/w) nitric acid to 25 μg/mL as the gold standard solution.

2.7. Standard solution preparation

Accurately transfer 0.1 mL of chromium, cobalt, nickel, arsenic, strontium, zirconium, cadmium, antimony, barium, mercury and lead reference standards into each 50-mL volumetric flask, make up to volume with 10% (w/w) nitric acid, and transfer to a storage tube as the standard stock solutions. When to use, mix appropriate amount of the standard stock solution, internal standard solution and gold standard solution, dilute with 10% (w/w) nitric acid to 0-25 ng/mL for chromium, cobalt, nickel, arsenic, strontium, zirconium, cadmium, antimony, barium and lead to 0-10 ng/mL for mercury (containing 5 ng/mL internal standard and 500 ng/mL gold standard). Transfer to storage tubes as the standard solutions.

2.8. 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 each heavy metal by the ratio of the signal intensity of each heavy metal to that of internal standard vs. the concentration of each heavy metal.

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: 1.05 L/min.
Atomic mass (m/z):

Heavy metal		Internal standard	
Chromium	52	Geranium	74
Cobalt	59	Geranium	74
Nickel	60	Geranium	74
Arsenic	75	Geranium	74
Strontium	88	Geranium	74
Zirconium	90	Rhodium	103
Cadmium	114, 111, 112	Rhodium	103
Antimony	123, 121	Rhodium	103
Barium	138	Rhodium	103
Mercury	202, 200, 201	Bismuth	209
Lead	208, 206, 207	Bismuth	209

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

2.9. 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, 1 mL of the gold standard solution, 12 mL of nitric acid (ultrapure grade) and 2 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 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 1 mL of the internal standard solution, 1 mL of the gold standard solution, 12 mL of nitric acid (ultrapure grade) and 2 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 (℃)
1	1800	25	20	210

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

2.10. 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 2.8. Calculate the amount of heavy metals in the sample based on the ratio of the signal intensity of heavy metals to that of internal standards by the following formula:

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

Where,

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

C_0: the concentration of each heavy metal 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 limits of quantification (LOQs) are 0.1 ppm for chromium, cobalt, nickel, arsenic, strontium, cadmium, antimony, barium, mercury and lead, and 0.2 ppm for zirconium.
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. 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. U.S. Environmental Protection Agency. 2014. Inductively coupled plasma-mass spectrometry. Method 6020B. [https://www.epa.gov/esam/epa-method-6020b-sw-846-inductively-coupled-plasma-mass-spectrometry]