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Method of Test for Heavy Metals in Bottled (Packaged) Drinking Water and Ice Cubes

1. Scope

This method is applicable for the determination of arsenic, lead, cadmium and mercury in bottled (packaged) drinking water, antimony in drinking water packed in PET containers, and arsenic, lead and mercury in ice cubes.

2. Method

After dilution, 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. Acid steam cleaning system.

2.2. Chemicals

Nitric acid, ultrapure grade and reagent grade;

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

Arsenic, lead, cadmium, mercury, antimony and gold, 1000 μg/mL, reference standards, ICP grade;

Rhodium, 1000 μg/mL, internal standard, ICP grade.

2.3. Apparatus

2.3.1. Volumetric flask(Note): 20 mL and 50 mL.

2.3.2. Storage tube: 50 mL, PP.

2.3.3. 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

Add 50 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 and 5 mL of gold reference standard to a 50-mL volumetric flask, make up to volume with 5% (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 5% (w/w) nitric acid to 1000 ng/mL, and transfer to a storage tube as the internal standard solution.

2.6. Standard solution preparation

Accurately transfer 50 μL of arsenic, lead, cadmium, mercury and antimony reference standards to each 50-mL volumetric flask, make up to volume with 5% (w/w) nitric acid, and transfer to storage tubes as the standard stock solutions. When to use, mix appropriate amount of the standard stock solutions and the internal standard solution, dilute with 5% (w/w) nitric acid to 0-20 ng/mL (containing 10 ng/mL internal standard), 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 curves of arsenic, lead, cadmium, mercury and antimony by the ratios of the signal intensity of arsenic, lead, cadmium, mercury or antimony to that of rhodium vs. the added concentrations.

ICP-MS operating conditions(Note):

Radiofrequency power: 1500 W.

Plasma argon flow rate: 15 L/min.

Auxiliary argon flow rate: 0.9 L/min.

Nebulizer argon flow rate: 1.0 L/min.

Atomic mass (*m/z*): arsenic, 75;
lead, 208, 206, 207;
cadmium, 114, 112, 111;
mercury, 202, 200;
antimony, 121, 123;
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 10 g of the mixed sample (ice cubes must completely melt into liquid) accurately weighed into a 20-mL volumetric flask,

add 0.2 mL of the internal standard solution and 1 mL of nitric acid (ultrapure grade), and dilute 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 volumetric flask, add 10 mL of deionized water, 0.2 mL of the internal standard solution and 1 mL of nitric acid (ultrapure grade), and perform the same procedure described above as the blank solution.

2.9. Quantification

Inject the sample solution, the blank solution and the standard solutions into the ICP-MS separately at appropriate rate, and operate according to the conditions in section 2.7. Calculate the amount of arsenic, lead, cadmium, mercury or antimony in the sample based on the ratio of the signal intensity of arsenic, lead, cadmium, mercury or antimony to that of rhodium by the following formula:

The amount of arsenic, lead, cadmium, mercury or antimony in the sample (mg/kg) = $\frac{(C - C_0) \times V}{M \times 1000}$

Where,

C: the concentration of arsenic, lead, cadmium, mercury or antimony in the sample solution calculated by the standard curve (ng/mL)

C_0:.the concentration of arsenic, lead, cadmium, mercury or antimony 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) for arsenic, lead, cadmium, mercury and antimony are all 0.0005 mg/kg.
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

Felipe-Sotelo, M., Henshall-Bell, E. R., Evans, N. D. M. and Read, D. 2015. Comparison of the chemical composition of British and Continental European bottled waters by multivariate analysis. J. Food Compos. Anal. 39: 33-42.