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Determination of isothiazolinone preservatives in toys - High performance liquid chromatography-tandem mass spectrometry

玩具中异噻唑啉酮类防腐剂的测定

高效液相色谱-串联质谱法

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WARNING -- Persons using this document should have practical experience with formal laboratory work. This document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures, and to ensure compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Standard specifies high performance liquid chromatography-tandem mass spectrometry for determination of isothiazolinone preservatives in toys.

This Standard applies to determination of 2-methyl-4-isothiazolin-3-one (MIT), 5-chloro-2-methyl-4-isothiazolin-3-one (CMIT) and 1,2-benzisothiazoline- 3-ketone (BIT) in toys. Other preservatives can be determined with reference to this document after verification.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682, *Water for analytical laboratory use - Specification and test methods*

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle

After the sample is prepared, use methanol to conduct ultrasonic extraction at room temperature. The extract is filtered by a filter membrane and then determined by high performance liquid chromatography-tandem mass spectrometer. Quantify by internal standard method.

5 Reagents and materials

5.1 Water: Grade 1 water specified in GB/T 6682.

5.2 Methanol: Chromatographically pure.

5.3 Preservative standard material: Purity is ≥95%; Annex A gives the specific information of MIT, CMIT and BIT.

5.4 Internal standard substance: 2-methyl-4-isothiazolin-3-one-d_3 hydrochloride salt, referred to as MIT-d_3, CAS No. is 1329509-49- 0; Purity is ≥95%.

5.5 Internal standard substance: 5-chloro-2-methyl-4-isothiazolin-3-one-d_3, referred to as CMIT-d_3, CAS No. is 1329611-34-8); Purity is ≥95%.

5.6 Internal standard substance: 1,2-benzisothiazolin-3-one-$^{13}C_6$, referred to as BIT-$^{13}C_6$, CAS No. is 1329616-16-1; Purity is ≥95%.

5.7 Standard stock solution: Respectively and accurately weigh an appropriate amount of preservative reference material (5.3). Use methanol (5.2) to prepare a standard stock solution with a mass concentration of 100.0mg/L.

5.8 Internal standard mixed solution: Accurately weigh an appropriate amount of internal standard substances MIT-d_3 (5.4), CMIT-d_3 (5.5) and BIT-$^{13}C_6$ (5.6). Use methanol (5.2) to dilute and obtain internal standard mixed solutions with mass concentrations of 1.0mg/L, 3.0mg/L and 1.0mg/L, respectively.

5.9 Standard mixed working solution: Pipette an appropriate volume of the preservative standard stock solution (5.7) into a volumetric flask. Use methanol (5.2) to set volume to the scale mark. Obtain a standard mixed solution with a mass concentration of 10.0mg/L. Use methanol (5.2) and stepwise dilution method to dilute the standard mixed solution and obtain a series of standard mixed working solutions with mass concentrations of 0.0005mg/L, 0.001mg/L, 0.005mg/L, 0.01mg/L, 0.02mg/L and 0.05mg/L. Take 1.0mL of a series of standard mixed working solutions into the injection bottle. Add 0.050mL of internal standard mixed solution (5.8). Mix well.

5.10 Microporous membrane: Organic phase, with a pore size of 0.22μm.

6 Instruments and equipment

6.1 High performance liquid chromatography-tandem mass spectrometer: Equipped with electrospray ionization (ESI).

6.2 Ultrasonic extractor.

6.3 Analytical balance: Accuracy is 0.1 mg.

Keep 3 significant digits.

10 Method limits of quantification

The method limits of quantification for MIT, CMIT and BIT in this document are all 0.040mg/kg.

11 Precision

See Annex C for the precision test results of the method in this document.

12 Test report

The test report shall at least give the following information:

a) Specimen description;

b) Reference (including the year number) to this document;

c) Test results;

d) Variations from the prescribed analysis steps;

e) Anomalies observed in tests;

f) Test date.

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