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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 5229-2019

**Determination of isothiazolinone preservatives in toy materials
by liquid chromatography-mass spectrometry (LC-MS) method
(English Translation)**

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Foreword

This standard was drafted in accordance with the rules given in GB/T1.1-2009.

Please note that some content in this document may involve patents. Publication of this document the agency is not responsible for identifying these patents.

This part is proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organizations of this section. Tianjin Customs of the People's Republic of China, Nanjing Customs of the People's Republic of China.

1 Scope

This standard specifies the combined use of liquid chromatography-mass spectrometry (LC-MS method) for isothiazolinone preservatives in toy materials.

This standard applies to 2-methyl-4-isothiazolin-3-one (MI), 5-chloro-2-methyl-4-isothiazolin-3-one (MI) and 5-chloro-2-methyl-4-isothiazolin-3-one in toy materials.

Determination of ketone (CMI) and 1,2-benzisothiazolin-3-one (BIT).

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version is applicable to this file. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analyze laboratory water specifications and test methods.

3 Principle

Use deionized water as the extractant to extract isothiazolinone preservatives in the sample (as shown in Figure 1), using liquid chromatography-mass spectrometry (LC-MS) Analytical method for detecting isothiazolinone preservatives in toy materials.

Figure 1 Schematic diagram of the structure of isothiazolinone preservatives

4 Reagents and materials

Unless otherwise specified, the purity of the reagents used in the analysis is analytically pure, and the laboratory water meets the requirements of GB/T 6682.

4.1

Methanol (chromatographically pure).

4.2

Anhydrous acetic acid (chromatographically pure).

4.3

Acetic acid solution. Pipette anhydrous acetic acid to prepare an acetic acid solution with a volume fraction of 0.4%.

4.4 2-Methyl-4-isothiazolin-3-one (MI) standard material, CAS number, 2682-20-4, purity greater than or equal to 98%.

4.5 5-Chloro-2-methyl-4-isothiazolin-3-one (CMI) standard material, CAS number, 26172-55-4, purity greater than or equal 99%.

4.6 1,2-Benzisothiazolin-3-one (BIT) standard material, CAS number, 2634-33-51, purity greater than or equal to 99%.

4.7 0.22μm water phase filter membrane.

5 Apparatus and equipment

5.1

Liquid chromatography-mass spectrometer.

5.2

Ultrasonic cleaner, 40 kHz.

5.3

Analytical balance, with a sense of 0.0001g.

5.4

Vortex mixer.

6.1.1 Aqueous liquid

Accurately weigh 1.0 g sample, accurate to 1 mg, place it in a 50 mL polypropylene test tube, add water to 15 mL, and ultrasonically extract for 20 minutes. Filtered with 0.22 μ m filter membrane, to be tested.

6.1.2 Sticker samples with adhesive

Take representative samples of different colors from the sticker samples. Cut into small pieces less than 3mm on each side, and mix well. Accurately weigh 1.0 g the sample is accurate to 1 mg, placed in a 50 mL polypropylene test tube, add water to 15 mL, ultrasonically extract for 20 minutes, and filter with a 0.22 μ m filter membrane.

To be tested.

6.1.3 Modeling clay samples

Accurately weigh 1.0 g of the above sample, accurate to 1 mg, place it in a 50mL polypropylene test tube, add water to 15mL, and ultrasonically extract 20 min, 0.22 μ m filter membrane filter, to be tested.

6.1.4 Leather samples

Select representative samples from different colors or different parts of the leather samples, the sample area is not less than 10 cm². The sample is cut to a small length on each side in 3 mm small pieces, mix well. Accurately weigh 1.0 g of the above sample, accurate to 1 mg, place it in a 50mL polypropylene test tube, and add water to 15mL, ultrasonic extraction for 20 minutes, 0.22 μ m filter membrane filtration, to be tested.

6.1.5 Blank test

Except that no sample is added, other tests shall be carried out according to the test procedure 6.3.

6.2.1 Configuration of standard solution

Weigh 10 mg (accurate to 0.1 mg) of MI, CMI, and BIT standards respectively in a 50 mL volumetric flask, and use 0.4% acetic acid the solution (5.3) has a constant volume and is made into 3 standard stock solutions with a mass concentration of 200 mg/L.

6.2.2 Configuration of standard stock solution

Accurately pipette the above three standard stock solutions and dilute them with deionized water to obtain three preservatives with mass concentrations of 5.0 µg/L, 10 µg/L, 20 µg/L, 50 µg/L, 100 µg/L, 200 µg/L, 500 µg/L series of mixed standard solutions (If the sample concentration exceeds the concentration range, press Properly dilute the ratio; you can also choose the appropriate curve concentration range according to the actual situation).

6.3.1 LC-MS conditions

Depending on the instrument used, the optimal analysis conditions may be different, so it is impossible to give general parameters suitable for all LC-MS. set up the parameters should ensure that the tested combination is effectively separated and determined. The parameters listed below have been proven to be feasible, and the spectra can be found in Appendix A.

6.3.2 Determination

According to the data in Table 1, the mass spectrum peaks of each isothiazolinone preservative were collected.

7.1 Qualitative results

The sample was qualitatively judged by liquid chromatography-tandem mass spectrometry. Under the same test conditions, the mass color of the tested components in the sample the retention time of the spectral peak is consistent with the retention time of the mass chromatographic peak of the corresponding component in the standard solution; and the mass-to-charge ratio of the selected two pairs of product ions Consistent, the deviation between the relative abundance ratio of the selected monitoring ion pair in the sample chromatogram and the ion relative abundance ratio of the standard solution of equivalent concentration If it does not exceed the range specified in Table 2, it can be determined that the corresponding components are present in the sample.

7.2 Quantitative calculation

Measure the standard solution and sample solution under the same experimental conditions, make a standard curve, and use the content of isothiazolinone preservatives in the sample the external standard method is used for quantification and the content is calculated according to formula (1).