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Determination of formamide in toy materials - High performance liquid chromatography-mass spectrometry

玩具材料中甲酰胺的测定　高效液相色谱-质谱法

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IMPORTANT NOTE: The use of this document may involve certain hazardous materials, procedures and equipment; but not all safety issues related thereto are recommended. It is the user's responsibility to, before applying this document, establish appropriate safety and protection measures AND determine the applicability of relevant regulatory limitations.

1 Scope

This document describes a method for the determination of formamide in toy materials using high performance liquid chromatograph-mass spectrometer.

This document is applicable to the determination of formamide in foam materials such as ethylene-vinyl acetate copolymer (EVA), polyethylene (PE), chemically cross-linked polyethylene (XPE), and polyvinyl chloride (PVC) in toy products. It is used as a reference for the determination of formamide in other foam materials.

2 Normative references

The contents of the following documents, through normative references in this text, constitute indispensable provisions of this document. Among them, for dated references, only the edition corresponding to that date applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

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

Use water to ultrasonically extract the prepared samples. After filtering the extract, use high performance liquid chromatograph-mass spectrometer to determine; use external standard method to quantify.

8 Sample extraction

Weigh two specimens of about 0.1 g (accurate to 0.1 mg) into the glass sample vial (6.4); accurately add 10 mL of water (5.1) and seal. After fully infiltrating the specimen, place it in an ultrasonic generator (6.2) for ultrasonic extraction for 3.5 h; cool to room temperature; use an organic microporous membrane (6.6) to filter it; wait for the determination.

9 Determination

9.1 Reference working conditions for high performance liquid chromatograph-mass spectrometer

Since the setting of working conditions depends on the instrument used, the set parameters shall ensure the effective separation of formamide chromatographic peaks. Appendix A gives the reference working conditions of high performance liquid chromatograph-mass spectrometer for the determination of formamide.

9.2 Qualitative analysis

According to the above analysis conditions (9.1), analyze the standard working solution (5.6) and the sample extract; use the retention time of characteristic ions and chromatographic peaks (see Figure A.1 in Appendix A) for qualitative analysis. If the selected ion proton number/charge number (m/z) is 46.0, the retention time of the detected chromatographic peak is consistent with that of the standard solution; it can be considered that the sample contains the target component.

9.3 Quantitative analysis

Use the external standard method for quantitative analysis. Use the mass concentration of the standard working solution as the abscissa; use the quantitative ion peak area of the compound as the ordinate; draw the standard working curve; obtain the regression equation of the standard curve, to calculate the formamide content in the sample.

If the specimen response value exceeds the linear range, it is necessary to dilute the specimen by an appropriate multiple AND re-inject the specimen for analysis.

9.4 Blank test

Except that no specimen is added, follow the above operation steps.

10 Result calculation

According to the formula (1), calculate the content of formamide in the specimen.

$$w = \frac{(c - c_0) \times V \times D}{m} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

Where:

w - The content of formamide in the specimen, in milligrams per kilogram (mg/kg);

c - The mass concentration of formamide in the sample solution, in milligrams per liter (mg/L);

c_0 - The mass concentration of formamide in the blank test, in milligrams per liter (mg/L);

V - The volume of the extract, in milliliters (mL);

D - The dilution factor of the extract;

m - The mass of specimen weighed, in grams (g).

The calculation result retains 3 significant figures.

11 Method limit of quantitation

When the sampling size is 0.1 g and the extraction solution volume is 10 mL, the quantitation limit of this method is 10.0 mg/kg.

12 Precision

In the same laboratory, using the same equipment by the same operator, according to the same test method, within a short period of time, by independently testing the same measured object; the absolute difference between the two independent test results obtained is not greater than 10% of the arithmetic mean of the two measured values; provided that no more than 5% of the cases are greater than 10% of the arithmetic mean of these two measured values.

See Appendix B for the precision test data of the method in this document.

13 Test report

The test report shall at least provide the following content:

a) Specimen description;

b) Number of this document;

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