



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

GB/T 34436-2023

**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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Foreword

This document was issued on 17 March 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 October 2023.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 34436-2023 covers the measurement of formamide in toy materials by high performance liquid chromatography-mass spectrometry, aimed at the foam materials the method names. The document walks the analytical chain in order: the principle, in which water is used to extract the prepared sample ultrasonically and the filtered extract is then measured and quantified against external standards; the reagents and the apparatus; sample preparation, and the extraction itself, where two specimens of about 0.1 g are weighed into a glass vial, 10 mL of water is added, and the sealed vial is held under ultrasound for 3.5 h; the determination, whose instrument working conditions are supplied only as a reference set in an appendix because they depend on the instrument in use; and

the calculation of the result. Three further clauses bound what a number produced this way may be claimed to mean: the method limit of quantitation, stated as 10.0 mg/kg for a 0.1 g sample size and a 10 mL extract volume; precision, expressed as the tolerated difference between independent results obtained in one laboratory; and the test report. Laboratories testing toys for chemical compliance, the toy makers commissioning that testing, and the certification bodies reading the reports are the readers.

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.

10 Result calculation

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

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);

c₀ - 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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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.