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**Textiles-Determination of adipate esters-Gas
chromatography-mass spectrometry method**

纺织品 己二酸酯的测定 气相色谱-质谱法

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Foreword

This document was drafted in accordance with the provisions of GB/T 1.1-2020 Directives for standardization - Part 1: Rules for the structure and drafting of standardizing documents.

Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents.

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Textiles - Determination of adipate esters - Gas chromatography-mass spectrometry method WARNING -- People who use this document shall have practical experience in regular laboratory work. This document does not point out all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring that the conditions stipulated by relevant national laws and regulations are met.

1 Scope

This document describes a method for the determination of five adipate esters in textiles by gas chromatography-mass spectrometry.

This document is applicable to the determination of the content of 5 types of adipate esters in various textile products.

2 Normative references

GB/T 8170

GB/T 10629

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

The test specimen is extracted with tetrahydrofuran by the ultrasonic method and then precipitated with a suitable solvent (acetonitrile, n-hexane). After centrifugation, the extract is detected by gas chromatography-mass spectrometer for 5 types of adipate esters, including diethyl adipate (DEA), diisobutyl adipate (DIBA), dibutyl adipate (DBA), bis(2-butoxyethyl) adipate (BBOEA) and bis(2-ethylhexyl) adipate (DEHA), which are quantified by using the external standard method.

5 Reagents

5.1 Tetrahydrofuran. chromatographic grade.

5.2 Precipitant. acetonitrile or n-hexane, chromatographic grade.

5.3 Adipate ester standards. The basic information of the five adipate ester compounds

is shown in Appendix A. The purity of all of them is $\geq 98\%$.

5.4 Standard stock solution. Weigh appropriate amounts of adipate ester standards (5.3)

(accurate to 0.1 mg) and prepare 1000 $\mu\text{g/mL}$ stock solutions with a precipitant (5.2).

The standard stock solutions should be stored in a refrigerator at $0\text{ }^{\circ}\text{C}$ – $4\text{ }^{\circ}\text{C}$ away from light and have a shelf life of 6 months.

5.5 Standard working solution. Take appropriate amounts of standard stock solutions

(5.4) of various adipate esters, and dilute into at least 5 mixed standard working solutions of appropriate concentrations using a mixture of tetrahydrofuran (5.1) and precipitant (5.2) (volume ratio of 1.2) as solvent as needed. The standard working solutions should be stored in a refrigerator at $0\text{ }^{\circ}\text{C}$ – $4\text{ }^{\circ}\text{C}$ away from light, and the validity period is 1 month.

6.1 Gas Chromatography-Mass Spectrometer (GC-MS). It is equipped with an electron

impact ionization source (EI).

6.2 Balance. Graduation values are 0.1 mg and 0.01 g.

6.3 Extraction bottle. with an airtight stopper, 50 mL, made of hard glass.

6.4 Ultrasonic generator. The operating frequency is $40\text{ kHz} \pm 5\text{ kHz}$. 6.5 Vortex mixer.

7.1 Test specimen preparation and extraction

Take a sufficient amount of representative sample according to the method described in GB/T 10629, cut it into pieces with a maximum size of no more than 5 mm, mix it, weigh 1.0 g (accurate to 0.01 g) from it using a balance (6.2) as the test specimen; place it in a 50 mL extraction bottle (6.3), add 10 mL of tetrahydrofuran (5.1), extract it in an ultrasonic generator (6.4) at $60\text{ }^{\circ}\text{C}$ for 30 min, let it stand, and cool it to room temperature. Use a pipette (6.8) to add 20 mL of precipitant (5.2) dropwise to the extraction bottle (6.3). Vortex it on a vortex oscillator (6.5) for at least 30 s, let it stand for 30 min, centrifuge it at 3000 r/min for 5 min, filter the supernatant through a $0.45\text{ }\mu\text{m}$ organic phase filter membrane (6.6), and then test it on the machine.

7.2 Qualitative analysis

Under the same experimental conditions, take equal volumes of the sample solution (7.1) and the standard working solution (5.5) for injection and measurement, and refer to the gas chromatography-mass spectrometry analysis conditions in Appendix B for test and analysis.

7.3 Quantitative analysis

Under the same determination conditions, the standard working curve is drawn with the concentration of the standard solution as the abscissa and the peak area of the quantitative ion as the ordinate. The content of the analyte in the test specimen is calculated by the external standard method. The target response value in the sample solution shall be within the linear range of the instrument detection. If the target Relative ion abundance/%

8 Result calculation and presentation

Calculate the content of each adipate ester in the test specimen according to formula (1). The calculation result shall be rounded to one decimal place in accordance with GB/T 8170.

9.2 Precision

In the same laboratory, the same operator uses the same equipment, according to the same test method, and conducts independent tests on the same object within a short period of time; the absolute difference between two independent test results shall not be greater than 15% of the arithmetic mean of the two measured values, and the premise is that the probability of exceeding the 15% of the arithmetic mean of the two measured values does not exceed 5%.

10 Test report

The test report shall at least include the following.

- a) The number of this document;
- b) Source and description of the sample;
- c) Analytical instruments used;
- d) Test results;
- e) Any details that deviate from this document;
- f) Date of the test.

Appendix A Appendix A

(Informative) Basic information on 5 adipate esters Table A.1 gives the basic information on