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Replacing GB/T 32612-2016

**Textiles -- Determination of ethylene glycol ethers and
related ester compounds**

纺织品 乙二醇醚类及相关酯类 化合物的测定

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Foreword

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

This document replaces GB/T 32612-2016 "Textiles -- Determination of ethylene glycol ethers and related ester compounds". Compared with GB/T 32612-2016, in addition to structural adjustments and editorial modifications, the main technical changes are as follows:

- a) The scope of application has been changed. The substances to be tested have been increased from 2-methoxyethanol and 2-ethoxyethanol to 19 glycol ethers and related ester compounds (see Chapter 1 and Annex A of this Edition; Chapter 1 of Edition 2016);
- b) The preparation of standard solution has been changed (see 5.3 and 5.4 of this Edition; 3.3.1~3.3.3 of Edition 2016);
- c) The specifications of extractors and filter heads have been changed (see 6.3 and 5.5 of this Edition; 4.3 and 4.4 of Edition 2016);
- d) The conditions for gas chromatography-mass spectrometry analysis have been changed (see 7.3.1 and 7.3.2 of this Edition; 5.3.1~5.3.2 of Edition 2016);
- e) The result calculation and quantification limit have been changed (see 8.1 and 9.1 of this Edition; 5.3.3 and 6.1 of Edition 2016);
- f) The recovery rate has been deleted (see 6.2 of Edition 2016);
- g) The test report has been changed (see Chapter 10 of this Edition; Chapter 7 of Edition 2016);
- h) The names and CAS numbers of 17 glycol ethers and related ester compounds have been added (see Annex A of this Edition).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. The issuing authority shall not be held responsible for identifying any or all such patent rights.

This document was proposed by China National Textile and Apparel Council.

This document shall be under the jurisdiction of National Technical Committee on Textiles of Standardization Administration of China (SAC/TC 209).

The drafting organizations of this document: Fujian Fiber Inspection Center, Zhejiang Boao New Materials Co., Ltd., Xiamen Laihe Lace Co., Ltd., China Textile Standard

Textiles -- Determination of ethylene glycol ethers and related ester compounds

WARNING -- The personnel who use this document should have practical experience in formal laboratory work. This document does not point out all possible safety issues. The user is 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 test method for the determination of glycol ethers and related esters in textile products using gas chromatography-mass spectrometry.

This document applies to all types of textile products.

2 Normative references

This document has no normative references.

3 Terms and definitions

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

4 Principle

19 glycol ethers and related ester compounds in the sample are extracted with methanol in an ultrasonic generator. The extracted solution of the sample is measured by gas chromatography-mass spectrometry. Full scan detection is used for qualitative analysis. Selected ions are used for quantitative analysis by external standard method.

5 Reagents and materials

Unless otherwise specified, all reagents are chromatographically pure.

5.1 Methanol: CAS No. 67-56-1.

5.2 Standard substance/standard sample: It shall comply with the provisions of Annex A, with a purity of $\geq 98.0\%$.

5.3 Standard stock solution: Use an analytical balance (6.2) with a graduation value of 0.0001 g to weigh each standard substance/standard sample (5.2). Dissolve and make up to the set volume with methanol (5.1). Prepare standard stock solutions with a mass concentration of 1000 mg/L. You can also directly purchase certified standard stock solutions. Either single component or mixed standard stock solutions can be used.

NOTE: The standard stock solution should be stored in a refrigerator at 0°C~4°C away from light. The validity period is 6 months.

5.4 Mixed standard working solution: Prepare according to work needs. Use methanol (5.1) to dilute the standard stock solution step by step into a series of mixed standard working solutions of at least 5 different mass concentrations (e.g., 0.1 mg/L, 0.2 mg/L, 0.5 mg/L, 0.8 mg/L, 1.0 mg/L, 2.5 mg/L).

NOTE: The mixed standard working solution should be stored in a refrigerator at 0°C~4°C away from light. The validity period is 1 month.

5.5 Organic phase needle filter: The pore size is 0.45 µm.

6 Instruments and equipment

6.1 Gas chromatograph-mass spectrometer: equipped with electron impact ionization source (EI source).

6.2 Analytical balance: graduation value is 0.01 g and 0.0001 g.

6.3 Extractor: 30 mL, tubular hard glass extractor with screw cap (with polytetrafluoroethylene gasket).

6.4 Ultrasonic generator: working frequency is (40±5) kHz, temperature is (45±2)°C.

7 Test steps

7.1 Specimen preparation and extraction

Take a representative sample. Cut it into pieces less than 5 mm×5 mm and mix well. Weigh 1.0 g of the specimen, accurate to 0.01 g. Place it in an extractor (6.3). Add 10 mL of methanol (5.1) to the extractor. Tighten the lid. Place the extractor in an ultrasonic generator (6.4). Extract in an ultrasonic water bath at (45±2)°C for (30±1) min. After cooling to room temperature, filter with an organic phase needle filter (5.5) to obtain the specimen extraction solution.

7.2 Blank test solution

Except for not adding the specimen, follow the procedures in 7.1.

Where,

X_i - Content of each component in the specimen, in milligrams per kilogram (mg/kg);

p_i - Mass concentration of the component solution obtained from the standard working curve, in milligrams per liter (mg/L);

p_0 - Mass concentration of the blank test solution obtained from the standard working curve, in milligrams per liter (mg/L);

V - Volume of the sample solution, in milliliters (mL);

m - Mass of the specimen, in grams (g);

f - Dilution factor.

8.2 Expression of results

The test results are expressed as the content of each target compound in the specimen. When it is below the limit of quantification (9.1), the test result is not detected.

9 Limit of quantification and precision of the method

9.1 Limit of quantification

The limit of quantification of this method for 19 glycol ethers and related ester compounds is 2.0 mg/kg.

9.2 Precision

The absolute difference between two independent test results obtained by the same operator using the same equipment and the same test method in the same laboratory and on the same object within a short period of time shall not exceed 10% of the arithmetic mean of the two measured values, provided that the absolute difference between the two independent test results shall not exceed 5% of the arithmetic mean of the two measured values.

10 Test report

The test report shall at least include the following:

- a) Reference to this document;
- b) A detailed description of the test sample, such as the storage method before the test and the packaging of the sample;

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