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**Textile -- Determination of alpha-bromocinnamaldehyde and
1,3-propanesultone**

纺织品 α -溴代肉桂醛和 1,3-丙烷磺酸内酯的测定

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Table of Contents

Foreword	3
1 Scope	4
2 Normative references	4
3 Terms and Definitions	4
4 Principles.....	4
5 Reagents and materials.....	4
6 Instruments and equipment	5
7 Analysis steps.....	5
8 Result calculation	7
9 Limit of quantitation and precision	7
10 Test report.....	8
Appendix A (Informative) GC-MS analysis conditions for 1,3-propanesultone and alpha-bromocinnamaldehyde	9
Appendix B (Informative) Total ion chromatogram of GC-MS analysis of 1,3-propanesultone and alpha-bromocinnamaldehyde	10

Textile -- Determination of alpha-bromocinnamaldehyde and 1,3-propanesultone

Warning -- The personnel using this document shall have practical experience in formal laboratory work. This document does not indicate all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This document describes a method for the determination of the content of alpha-bromocinnamaldehyde and 1,3-propanesultone in textiles by using the gas chromatography-mass spectrometry (GC-MS).

This document applies to all types of textile products.

2 Normative references

There are no normative references in this document.

3 Terms and Definitions

There are no terms and definitions that need to be defined in this document.

4 Principles

After ultrasonic extraction and filtration of the samples with ethyl acetate, determine it by the gas chromatography-mass spectrometer, qualitatively analyze it by the selected ion mode, and then quantitatively analyze it by the external standard method.

5 Reagents and materials

Unless otherwise specified, the reagents used shall be of analytical grade.

5.1 Ethyl acetate.

5.2 Alpha-bromocinnamaldehyde (CAS No.: 5443-49-2): purity $\geq 99\%$.

5.3 1,3-propanesultone (CAS No.: 1120-71-4): purity $\geq 95\%$.

5.4 Standard stock solution: Accurately weigh an appropriate amount of α -bromocinnamaldehyde (5.2) and 1,3-propanesultone (5.3) reference materials respectively, and dilute them with ethyl acetate (5.1) respectively to prepare the standard stock solutions with a concentration of about 500 mg/L.

Note: The standard stock solution shall be stored in the dark at 0 °C~4 °C, and the validity period is 6 months.

5.5 Mixed standard working solution: Accurately pipette a certain volume of standard stock solution (5.4) as needed, and dilute it stepwise with ethyl acetate (5.1) into a series of mixed standard working solutions with appropriate concentrations.

Note: The mixed standard working solution shall be stored in the dark at 0 °C~4 °C, and it is valid for 1 month.

6 Instruments and equipment

6.1 Gas chromatography-mass spectrometer: It shall be equipped with an electron impact ion source (EI).

6.2 Balance: The division value shall be 0.1 mg and 0.01 g.

6.3 Temperature-controlled ultrasonic instrument: The temperature control range shall be 20 °C~60 °C, and the working frequency shall be (40 $\pm$ 5) kHz.

6.4 Glass extraction tube with stopper: 100 mL.

6.5 Concentration bottle: It shall be 100 mL with a ground-glass stopper.

6.6 Rotary evaporator.

6.7 Nitrogen blower.

6.8 Organic filter membrane: 0.45 μ m.

7 Analysis steps

7.1 Preparation and treatment of specimens

Take a representative sample, cut it into pieces with a size of less than 5 mm $\times$ 5 mm, and mix well. Accurately weigh 1.0 g of the above sample, and the weight shall be accurate to 0.01 g; place it in a 100 mL stoppered glass extraction tube (6.4), add 20 mL of ethyl acetate (5.1), and perform ultrasonic extraction for 20 min at (40 $\pm$ 5) °C.

8 Result calculation

8.1 Calculate the content of alpha-bromocinnamaldehyde or 1,3-propanesultone in the sample according to formula (1):

$$X = \frac{A \times c \times V}{A_s \times m} \dots\dots\dots (1)$$

Where:

X --- The content of alpha-bromocinnamaldehyde or 1,3-propanesultone in the sample, in milligrams per kilogram (mg/kg);

A --- The peak area of alpha-bromocinnamaldehyde or 1,3-propanesultone in the sample solution;

c --- The mass concentration of alpha-bromocinnamaldehyde or 1,3-propanesultone in the standard working solution, in milligrams per liter (mg/L);

A_s --- The peak area of alpha-bromocinnamaldehyde or 1,3-propanesultone in the standard working solution;

V --- The volume of the sample solution that is accurate to the scale, in milliliters (mL);

m --- The mass of the sample, in grams (g).

8.2 The measurement results of this method are expressed as the calculation results of alpha-bromocinnamaldehyde and 1,3-propanesultone respectively, and the calculation results are rounded to one decimal place.

9 Limit of quantitation and precision

9.1 Limit of quantitation

In this method, the limit of quantification of alpha-bromocinnamaldehyde is 0.2 mg/kg, and the limit of quantification of 1,3-propanesultone is 0.5 mg/kg.

9.2 Precision

In the same laboratory, the same operator uses the same equipment, according to the same test method, and tests the same test object independently within a short period. The absolute difference between the two test results obtained shall be not greater than 10% of the arithmetic mean of the two measured values; take the confidence coefficient

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