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Textiles - Determination of the Content of Ortho-Phenylphenol

纺织品 邻苯基苯酚的测定

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WARNING: Persons using this Standard shall have practical experience working in formal laboratory. This Standard does not point out all possible safety issues. Users shall assume the responsibility to take appropriate safety and health measures, ensuring to meet the conditions stipulated by relevant national regulations.

1 Scope

This Standard specifies the gas chromatography-mass selective detector (GC-MSD) method for determining the content of ortho-phenylphenol (OPP) in textiles.

Method 1 in this Standard is applicable to the determination and confirmation of the ortho-phenylphenol content in various textile materials and their products; Method 2 in this Standard is applicable to determination and confirmation of the ortho-phenylphenol and its salts and esters contents in various textile materials and their products.

2 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for Laboratory Use – Specifications (GB/T 6682-1992, neq ISO 3696:1987)

3 Principles

3.1 Method 1

The specimen is extracted by ultrasonic wave of methanol. After the extracting solution is concentrated to a constant volume, it is measured by a gas chromatograph equipped with a mass selective detector (GC-MSD). The selective ion detection is used for

6 months; while the mixed standard working solution is stored in the refrigerator at 0°C~4°C for a valid period of 3 months.

5 Instruments and Apparatuses

5.1 Gas chromatograph: equipped with a mass selective detector (MSD)

5.2 Ultrasonic generator: working frequency of 40 kHz.

5.3 Centrifuge: 4000 r/min.

5.4 Rotary evaporator.

5.5 Anhydrous sodium sulfate column: 7.5 cm × 1.5 cm (inner diameter); containing 4cm of high anhydrous sodium sulfate.

5.6 Conical flask: with ground stopper, 100mL.

5.7 Centrifuge tube: with ground stopper, 15mL.

5.8 Concentration bottle: 100mL.

6 Analytical Procedures

6.1 Method 1

6.1.1 Extraction

Take a representative sample; shred it 5mm × 5mm below and mix well. Take 1.0g (accurate to 0.01g) of the specimen; place it in a 100mL conical flask with a stopper; add 50mL of methanol; and extract for 20min in an ultrasonic generator. Filter the extracting solution. The residue is ultrasonically extracted by 30mL of methanol for 5min; combine the filtrate; and dehydrate by anhydrous sodium sulfate column; collect in a 100mL concentration bottle; concentrate to near dryness in a 40°C water bath rotary evaporator; dissolve by acetone and make constant volume of 5.0mL for confirmation and determination of gas chromatography-mass spectrometry.

6.1.2 Determination

6.1.2.1 Gas chromatography-mass spectrometry conditions

Since the test results depend on the used instrument, it is impossible to give general parameters for chromatographic analysis. The set parameters shall ensure that the measured component may be effectively separated from other components during chromatographic determination. The parameters given below prove to be feasible.

6.2.1 Extraction

Take a representative sample; cut it to a size of 5mm × 5 mm or less, and mix evenly. Take 1.0g (accurate to 0.01g) of the sample; place it in a 100mL conical flask with a stopper; add 50mL of methanol; and extract for 20min in the ultrasonic generator. Filter the extracting solution in a 100mL concentrator bottle. The residue was ultrasonically extracted by 30mL of methanol for 5min; combine the filtrate; then concentrate to near dryness on a 40°C water bath rotary evaporator. Use 8mL of potassium carbonate solution to dissolve the concentrated solution and totally transfer into a 15mL centrifuge tube.

6.2.2 Acetylation

Add 1mL of acetic anhydride; shake for 2min. Accurately add 5.0mL of n-hexane; shake for 2min; and centrifuge at 4000 r/min for 3min. Use a pointed pipette to extract the lower aqueous phase. Add 10mL of sodium sulfate solution; shake for another 1min; and centrifuge at 4000 r/min for 3min. The n-hexane phase is used for determination and confirmation of gas chromatography-mass spectrometry.

6.2.3 Preparation of standard working solution

Accurately transfer a certain volume of standard solution of appropriate concentration in a 15mL centrifuge tube; dilute to 8 mL by potassium carbonate solution. Add 1mL of acetic anhydride; and follow the procedures in 6.2.2 then.

6.2.4 Determination

6.2.4.1 Gas chromatography-mass spectrometry conditions

Since the test results depend on the used instrument, it is impossible to give general parameters for chromatographic analysis. The set parameters shall ensure that the measured component may be effectively separated from other components during chromatographic determination. The parameters given below prove to be feasible.

a) Chromatographic column: DB-17 MS 30m × 0.25mm × 0.1μm or equivalent;

b) Temperature of chromatographic column:

$$50\,℃\,(2\ \text{min}) \xrightarrow{20\,℃/\text{min}} 200\,℃\,(1\ \text{min}) \xrightarrow{10\,℃/\text{min}} 270\,℃\,(5\ \text{min})$$

c) Inlet temperature: 270°C;

d) Chromatography-mass spectrometer interface temperature: 260°C;

e) Carrier gas: helium, purity ≥99.999%, 1.4 mL/min;

X - the content of ortho-phenylphenol in the sample, in mg/kg;

A - Peak area (or peak height) of ortho-phenylphenol or ortho-phenylphenol acetic ester in the sample solution;

A_s - the peak area (or peak height) of ortho-phenylphenol or ortho-phenylphenol acetic ester in the standard working solution;

c - the concentration of ortho-phenylphenol or equivalent ortho-phenylphenol in the standard working solution, in mg/L;

V - volume of sample solution, in mL;

m - the amount of sample represented by the final sample solution, in g.

8 Lower Limit of Detection, Recovery Rate and Precision

8.1 Lower limit of detection

The lower limit of detection for Method 1 is 0.10 mg/kg; while the lower limit of detection for Method 2 is 0.05 mg/kg.

8.2 Recovery rate

The recovery rate of Method 1 and Method 2 is 85% ~ 110%.

8.3 Precision

In the same laboratory, the same operator uses the same equipment, the same test method, and the same test object is tested independently in a short time. The absolute difference between the two independent test results obtained by Method 1 and Method 2 shall be no greater than 10% of the arithmetic mean of the two measured values. Provided that the value greater than 10% of the arithmetic mean of the two measured values, it shall be on the premise of not exceeding 5%.

9 Test Report

The test report shall at least give the following contents:

a) Sample description;

b) The used standard;

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