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Textiles – Determination of Phenol and Bisphenol A

纺织品 苯酚和双酚 A 的测定

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Textiles – Determination of Phenol and Bisphenol A

Warning: Personnel using this Document shall have practical working experience in formal laboratory. This Document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This Document describes a method for the determination of phenol and bisphenol A in textiles using high performance liquid chromatography.

This Document applies to all types of textile products.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

3 Terms and Definitions

For the purposes of this Document, no terms and definitions apply.

4 Principles

The specimen is ultrasonically extracted with methanol; and the extracting solution is filtered through a filter membrane; and then the specimen is determined by a high-performance liquid chromatograph equipped with a fluorescence detector (HPLC/FLD) or a high-performance liquid chromatograph equipped with a diode array detector (HPLC/DAD); then conduct the quantification by external standard method.

6.4 Glass extraction bottle: with a capacity of 40mL and a screw cap.

6.5 Polytetrafluoroethylene filter membrane: The pore size is 0.45μm.

7 Analysis Procedures

7.1 Preparation and extraction of specimens

Take a representative specimen; cut it to less than 5mm×5mm; and mix well. Take 1g from the mixed sample, accurate to 0.01g; put the specimen in the glass extraction bottle (6.4); add 30mL of methanol (5.2) into the glass extraction bottle (6.4); screw the cap tightly. Put the glass extraction bottle (6.4) in an ultrasonic cleaner (6.2); extract at 35°C for (40±2) min; then cool to room temperature; and filter the extracting solution through a polytetrafluoroethylene filter membrane (6.5) as a sample solution for analysis by the instrument.

7.2 Standard working curve

Pipette the mixed standard working solution (5.6); and measure it with reference to the instrument analysis conditions given in A.1 of Appendix A. Take the mass concentration of phenol or bisphenol A as the abscissa, and the corresponding chromatographic peak area as the ordinate, draw the working curve.

7.3 Qualitative and quantitative analysis

Qualitative analysis is carried out by comparing the retention time and spectrum of the measured substance with the standard substance in the chromatogram; and the external standard method is used for quantification. See A.1 for instrument analysis conditions. When the analysis conditions of A.1 are used, the liquid chromatograms of phenol and bisphenol A are shown in Figures A.1 and A.2.

8 Calculation and Representation of Result

Calculate the content of phenol and bisphenol A according to the formula (1), respectively, and the calculation results are accurate to one digit after the decimal point. The test results are represented by the content of phenol and bisphenol A, respectively.

$$X = \frac{c_s \times V \times f}{m} \dots\dots\dots (1)$$

Where:

X - the content of phenol and bisphenol A in the specimen, in mg/kg;

c_s - mass concentration of phenol and bisphenol A in the sample solution obtained through the

standard working curve, in mg/L;

V – extracting solution volume of specimen, in mL;

f – dilution factor;

m – mass of specimen, in g.

9 Quantitative Limit of Method

9.1 The quantitative limit of HPLC/FLD phenol is 1.0mg/kg; and the quantitative limit of bisphenol A is 2.0mg/kg.

9.2 The quantitative limit of HPLC/DAD phenol is 10.0mg/kg; and the quantitative limit of bisphenol A is 10.0mg/kg.

10 Precision

At the level of confidence at 95%, at the same laboratory, the same operator uses the same device, according to the same measurement method, and independently testing the same measured objects in a short time. The obtained absolute difference of the two independent test results shall be no more than 10% of the arithmetic mean.

11 Test Report

The test report should be given at least the following content:

- a) Use this Document;
- b) Sample source and description;
- c) Types of used detectors;
- d) Test results;
- e) Any details that deviate from this Document;
- f) Test date.

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