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Textiles – Determination of Short Chain 4-(1,1,3,3-Tetramethylbutyl) Phenol Ethoxylate

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Table of Contents

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Foreword

This Standard was drafted as per the rules specified in GB/T 1.1-2009.

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

This Standard shall be under the jurisdiction of National Technical Committee for Standardization of Textiles (SAC/TC 209).

Drafting organizations of this Standard: Ningbo Inspection and Quarantine Institute of Science and Technology, CTTC Inspection and Certification Co., Ltd., Ningbo Yinzhou China Inspection Co., Ltd., and Haosha Industry (Fujian) Co., Ltd.

Chief drafting staffs of this Standard: Bao Qibei, Fu Kejie, Zhang Hui, Ren Qingqing, Qian Dan, Lian Cefeng, Xu Jianlin, Yang Lisheng, Feng Yun, Fu Chunlin, and Chang Xiangzhen.

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Textiles – Determination of Short Chain 4-(1,1,3,3-Tetramethylbutyl) Phenol Ethoxylate

Warning – Personnel using this Standard shall have practical working experience in the formal laboratory. This Standard doesn't indicate all possible safety issues. The users shall be responsible for taking appropriate safety and health measures, and ensure to conform to the specified conditions of relevant national regulations.

1 Scope

This Standard specifies the method of adopting the normal phase High Performance Liquid Chromatography (HPLC) to determine the 3 kinds of short chain 4-(1, 1, 3, 3-tetrametylbutyl) phenol ethoxylate, namely, 4-*tert*-octylphenol monoethoxylate, 4-*tert*-octylphenol diethoxylate, 4-*tert*-octylphenol triethoxylate in the textiles.

This Standard is applicable to all types of textile products.

2 Normative References

The following documents are essential to the application of 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) are applicable to this document.

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

3 Principle

Take methanol as the extraction solvent; use ultrasonic wave to extract short chain 4-(1, 1, 3, 3-tetrametylbutyl) phenol ethoxylate in the specimen; concentrate the extracting solution, then use normal phase high performance liquid chromatography with fluorescence detector to determine; use external standard method to determine the quantity.

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6 Sample Pre-Treatment

Select the representative sample; cut into 5mm × 5mm pieces, mix evenly. Weigh 2g of specimen, accurate to 0.01g; place into the stoppered conical flask (5.4); add 50mL of methanol (4.1), extract for 40min under the room temperature in the ultrasonic-wave generator (5.2); transfer the extracting solution into the round bottom flask (5.5); then use 10mL of methanol (4.1) to wash the slag, add into the extracting solution. Concentrate to 1mL at 40°C rotary evaporator (5.6); then use isopropanol (4.3) to make the constant volume of 10mL. Filter the solution through the organic phase filter membrane (5.7), then serve as the sample solution to be tested by the normal phase high performance liquid chromatography.

7 Determination

7.1 Normal phase high performance liquid chromatography conditions

Since the test results depend on the instrument used; it is not possible to give general parameters for chromatographic analysis. The following parameters have been proved to be appropriate for the test:

a) Chromatographic column: Dikma platisil silica gel column; 250mm × 4.6mm, 5.0μm or equivalent.

b) Chromatographic column temperature: 35°C.

c) Mobile phase:

--- Mobile Phase A: n-hexane + isopropanol (85:15);

--- Mobile Phase B: isopropanol + water (85:15).

d) Fluorescence detector: excitation wavelength of 230nm; emission wavelength of 296nm.

e) Flow rate: 0.5mL/min.

f) Sample injection volume: 10μL.

g) Gradient elution program, refer to Table 1.

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