ICS 59.080.01
CCS W 04

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 23322-2018

Replacing GB/T 23322-2009

Textiles - Determination of surfactant - Alkylphenols and alkylphenol ethoxylates

纺织品 表面活性剂的测定 烷基酚和烷基酚聚氧乙烯醚

Issued on: December 28, 2018 **Implemented on: July 1, 2019**

Issued by: State Administration for Market Regulation;
Standardization Administration of PRC..

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces GB/T 23322-2009 "Textiles - Determination of surfactant - Alkylphenol ethoxylates". Compared with GB/T 23322-2009, the main technical changes are as follows:

- ADD the CAS number information of the standard product (see 4.9, 4.10, 4.11,

- ADD the analytical method for alkylphenols;

- ADD the liquid chromatography-mass spectrometry (LC-MS) determination method;

- MODIFY the method of calculation and presentation of results;

- ADD the determination method of hydrophilic interaction chromatography in the Appendix;

phase high-performance liquid chromatography, from the main text to Appendix A and Appendix B.

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

This standard shall be under the jurisdiction of the National Textile Standardization Technical Committee (SAC/TC 209).

Drafting organizations of this standard: Zhejiang Provincial Institute of Inspection and Quarantine Science and Technology, Zhejiang Entry-Exit Inspection and Quarantine Bureau, China Textile Standard Inspection and Certification Co., Ltd., Zhejiang Sci- Tech University, Shenzhen Entry-Exit Inspection and Quarantine Bureau.

The main drafters of this standard: Chen Xiaomei, Lv Chunhua, Liu Haishan, Zhang Hui, Chen Haixiang, Zhu Ying, Zhang Weiya, Zhao Shanhong.

1 Scope

This standard specifies the liquid chromatography-mass spectrometry detection method of alkylphenol (AP) and alkylphenol ethoxylates (APnEO, n = 2 ~ 16), in textiles.

This standard applies to all types of textile products.

Note 1: The general molecular structure of AP is: R-C6H4-OH. AP in this standard refers to the common octylphenol [OP, C8H17-C6H4-OH] and nonylphenol [NP, C9H19-C6H4-OH]. The general molecular structure of APnEO is: R-C6H4-(OC2H4)nOH. APnEO in this standard refers to the commonly used octylphenol ethoxylates [OPnEO, C8H17-C6H4-(OC2H4)nOH] and nonylphenol ethoxylates [NPnEO, C9H19-C6H4-(OC2H4)nOH].

Note 2: For the determination method of reversed-phase high-performance liquid chromatography, normal-phase high-performance liquid chromatography, hydrophilic

interaction chromatography, of the alkylphenol and alkylphenol ethoxylate, refer to Appendix A, Appendix B, Appendix C, respectively.

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) is applicable to this standard.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

3 Principles

The AP and APnEO in the specimen are extracted by methanol ultrasonically. After the extract is concentrated and purified, it was determined by liquid chromatography-mass spectrometer, quantified by external standard method.

4 Reagents or materials

4.1 Unless otherwise specified, the reagents used in this method are all analytically pure; the water is the grade-1 water, which is specified in GB/T 6682.

4.2 Methanol (HPLC grade).

4.3 Acetonitrile (HPLC grade).

4.4 n-hexane (HPLC grade).

4.5 Isopropanol (HPLC grade).

4.6 Dichloromethane (HPLC grade).

4.7 Methanol-aqueous solution (3 + 2): Accurately take 300 mL of methanol (4.2) and 200 mL of water. Mix well. Prepare for later use.

4.8 Methanol-dichloromethane solution (1 + 4): Accurately take 100 mL of methanol (4.2) and 400 mL of dichloromethane (4.6). Mix well. Prepare for later use.

4.10 Nonylphenol standard (NP, CAS No.25154-52-3, excellent grade pure).

4.11 Standard product of octylphenol ethoxylates (OPnEO, CAS No.9002-93-1, average degree of polymerization n = 9, excellent grade pure).

4.12 Standard product of nonylphenol ethoxylates (NPnEO, CAS No.9016-45-9, average degree of polymerization n = 9, purity ≥ 99%).

4.13 Standard stock solution: Accurately weigh an appropriate amount of OP (4.9), NP

(4.10), OPnEO (4.11), NPnEO (4.12), respectively. Use methanol, to prepare a singlecomponent standard stock solution, which has a concentration of 10 mg/mL.

4.14 Standard working solution: Pipette an appropriate volume of OP, NP, OPnEO, NPnEO standard stock solution (4.13), respectively. Place in the same volumetric flask. Use methanol to dilute it. Prepare a mixed standard working solution of the required concentration.

4.15 Solid phase extraction column: The filler is lipophilic divinylbenzene and hydrophilic N-vinylpyrrolidone copolymer, 60 mg, 3 mL. Use 2 mL of methanol and 4 mL of water, to activate it in sequence, before use.

4.16 Organic filter membrane: 0.22 μm.

5 Instruments and equipment

5.1 Liquid chromatography-mass spectrometer: Equipped with an electrospray

5.2 Analytical balance: Sensitivity is 0.0001 g.

5.3 Analytical balance: Sensitivity is 0.01 g.

5.4 Ultrasonic cleaner: The temperature can be controlled at (70 ± 2) °C.

5.5 Rotary evaporator.

5.6 Solid phase extraction device.

5.7 Nitrogen blower.

6 Measurement steps

6.1 Extraction and purification of specimens

Take a representative specimen. Cut it into pieces of about 5 mm × 5 mm. Mix well. Weigh 1 g of the cut specimen (accurate to 0.01 g). Place it in a 50 mL centrifuge tube. Add 30 mL of methanol. Extract ultrasonically at (70 ± 2) °C, for (60 ± 5) min. Concentrate the extract to near dryness, at below 40 °C, by a rotary evaporator. Add 2 mL of methanol accurately, to dissolve the residue. Pass it through a 0.22 μm filter membrane (4.16). Use it for liquid chromatography-mass spectrometry determination.

When impurities in the specimen (such as silk) interfere with the detection, the following purification methods are used. Use 10 mL of methanol-water solution (4.7), to dissolve the residue in the above concentration bottle. Transfer all to the solid phase extraction column