ICS 59.080.01

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GB

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 01, 2019**

Issued by: State Administration for Market Regulation;

Standardization Administration of PRC.

Table of Contents

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:

- MODIFY the standard name into "Textiles - Determination of surfactant - Alkylphenols and alkylphenol ethoxylates";
- ADD the CAS number information of the standard product (see 4.9, 4.10, 4.11, 4.12);
- 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;
- MOVE the reversed-phase high-performance liquid chromatography and normal-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.

This standard replaces the standard previously issued as follows:

- GB/T 23322-2009.

Textiles - Determination of surfactant - Alkylphenols and alkylphenol ethoxylates

Caution - The personnel using this standard shall have practical experience in formal laboratory work. This standard does not address all possible safety issues. It is the user's responsibility, to take appropriate safety and health measures AND to ensure compliance with the conditions, which are stipulated by relevant national regulations.

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-C_6H_4-OH. AP in this standard refers to the common octylphenol [OP, C_8H_{17}-C_6H_4-OH] and nonylphenol [NP, C_9H_{19}-C_6H_4-OH]. The general molecular structure of APnEO is: R-C_6H_4-$(OC_2H_4)_n$OH. APnEO in this standard refers to the commonly used octylphenol ethoxylates [OPnEO, C_8H_{17}-C_6H_4-$(OC_2H_4)_n$OH] and nonylphenol ethoxylates [NPnEO, C_9H_{19}-C_6H_4-$(OC_2H_4)_n$OH].

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.

4.9 Standard octylphenol (OP, CAS No.140-66-9, purity ≥ 97%).

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 single-component 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.