

ICS 59.080.01
CCS W 04

SN

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 5244-2020

**Import and export textile processing detergents. Determination
of alkyl phenol and alkyl phenol polyoxyethylene ether. High
performance liquid chromatography**

进出口纺织加工洗涤剂 烷基苯酚及烷基苯酚聚氧乙烯醚的测定 高效液相色谱法
(English Translation)

Issued on: August 27, 2020

Implemented on: March 1, 2021

Issued by: General Administration of Customs

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Foreword

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

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organizations of this standard. Shijiazhuang Customs of the People's Republic of China, Nanjing Customs of the People's Republic of China.

1 Scope

This standard specifies the high performance liquid chromatography method for the determination of alkylphenols and alkylphenol polyoxyethylene ethers in textile processing detergents.

This standard applies to the determination of alkylphenol and alkylphenol polyoxyethylene ether content in textile processing detergents and products with similar functions.

set.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analytical laboratory water specifications and test methods GB/T 8170 Numerical rounding rules and the expression and determination of limit values

3 Terms and definitions

The following terms and definitions apply to this document.

Alkylphenols

The molecular structure of alkylphenol is $R-C_6H_4-OH$. Octylphenol ($C_8H_{17}-C_6H_4-OH$, OP for short) is common among such substances and nonylphenol ($C_9H_{19}-C_6H_4-OH$, referred to as NP).

Alkylphenol polyethoxylates, APnEO the molecular structure of alkylphenol polyoxyethylene ether is $R-C_6H_4-(OC_2H_4)_nOH$. Octylphenol polyoxyethylene Alkenyl ether [$C_8H_{17}-C_6H_4-(OC_2H_4)_nOH$, referred to as OPnEO] and nonylphenol polyoxyethylene ether [$C_9H_{19}-C_6H_4-(OC_2H_4)_nOH$, referred to as NPnEO].

Alkylphenol polyoxyethylene ethers can be used as surfactants in textile processing detergents. Alkylphenols are alkylphenol polyoxyethylene ethers.

4 Principle

The detergent samples were extracted by ultrasonic extraction with methanol as the extraction solvent. After centrifugation, the extraction liquid was the spectrometer is used for analysis and the external standard method is used for quantification.

5 Reagents and materials

Unless otherwise specified, only use analytically pure reagents and tertiary water or equivalent purity water that meets the requirements of GB/T 6682.

5.1

Methanol. chromatographically pure.

5.2

Acetonitrile. chromatographically pure.

5.3

Octylphenol standard product. OP, purity $\geq 97\%$.

5.4

Nonylphenol standard product. NP, purity $\geq 99\%$.

5.5

Standard product of octylphenol polyoxyethylene ether. OPnEO, average degree of polymerization $n=9\sim 10$, excellent grade pure.

5.6

Nonylphenol polyoxyethylene ether standard product. NPnEO, average degree of polymerization $n=9\sim 10$, purity $\geq 95\%$.

5.7

Alkylphenol standard stock solution. accurately weigh appropriate amounts of octylphenol (5.3) and nonylphenol (5.4) standards, and use Alcohol (5.1) was prepared into a standard stock solution with a concentration of 1 mg/mL.

5.8

Alkylphenol polyoxyethylene ether standard stock solution. accurately weigh appropriate amounts of octylphenol polyoxyethylene ether (5.5) and nonylbenzene respectively Phenol polyoxyethylene ether (5.6) standard products were prepared with methanol (5.1) to prepare standard stock solutions with a concentration of 1 mg/mL.

5.9

Standard working solution. Pipette the alkylphenol standard stock solution (5.7) and alkylphenol polyoxyethylene with a concentration of 1 mg/mL respectively Enether standard stock solution (5.8) has an appropriate volume and diluted with methanol (5.1) to prepare a standard working solution of the required concentration.

Note: The standard solution should be stored below 4 °C and protected from light. The standard stock solution is valid for 12 months, and the standard working solution is valid for 3 months.

6 Apparatus and equipment

6.1

High performance liquid chromatograph. equipped with fluorescence detector.

6.2

Analytical balance. Sensitivity is 0.1 mg and 0.01 g respectively.

6.3 50 mL plastic centrifuge tube (with lid can be sealed).

6.4

Measuring cylinder. 10 mL.

6.5

Volumetric flask. 25 mL.

6.6

Ultrasonic extraction device. controllable temperature $(60 \pm 1) ^\circ\text{C}$.

6.7

Centrifuge. ≥ 8000 r/min.

7.1 Sample preparation

Mix the sample thoroughly before sampling, and take out 5.0 g~10.0 g sample.

Note: viscous samples should be shaken sufficiently to ensure uniform mixing.

7.2 Extraction

Weigh 1.0 g of the above sample, accurate to 0.01 g, add it to the centrifuge tube (6.3), add 10 mL directly to the weighed sample Methanol (5.1), seal and shake and mix, ultrasonically extract at $(60 \pm 1) ^\circ\text{C}$ for 30 min, centrifuge and transfer the supernatant extract to 25 mL Volumetric flask (6.5), add 10 mL of methanol (5.1) to the centrifuge tube (6.3) and shake well to wash, after centrifugation, transfer the supernatant for extraction Fill it to the volumetric flask (6.5), and use methanol (5.1) to make the volume constant. Dilute the sample solution 4 times and transfer it to a sample bottle for liquid chromatography measurement.

7.3.1 Liquid chromatography conditions

Since the test results depend on the instrument used, it is impossible to give general parameters for liquid chromatography analysis. The set parameters shall be guaranteed SN/T 5244-2020 to prove that the measured component can be effectively separated from other components during chromatographic determination, the following parameters prove to be feasible.

a) Chromatographic column. C18 reverse phase column, Diamonsil C18 (2), 3.0 mm x 150