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

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**Determination of chloroform and other halogenated
hydrocarbons in detergents - Gas chromatography-mass
spectrometry**

洗涤剂中氯仿等多种卤代烃的测定 气相色谱质谱法

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part

1. Structure and Drafting Rules of Standardization Documents". Drafting. Please note that some content in this document may involve patents. The issuing organization of this document assumes no responsibility for identifying patents. This document was proposed by the China National Light Industry Council. This document is under the jurisdiction of the National Technical Committee on Standardization of Surfactants and Detergents (SAC/TC272). This document was drafted by: Shanghai Municipal Institute of Quality Supervision and Inspection Technology Co., Ltd., Chongqing Institute of Metrology and Quality Inspection, and China Daily. The companies involved are Chemical Research Institute Co., Ltd., China Light Industry Inspection and Certification (Taiyuan) Co., Ltd., and Henan Sanmenxia Ecological Environment Monitoring Center. The main drafters of this document are: Zhou Yu, Hu Shoujiang, Li Genrong, Li Xiaorui, Gu Yuxiang, Qiao Huanhuan, He Xiaoling, Lu Jiali, Ran Lu, and Yang Qian. Determination of chloroform and other halogenated hydrocarbons in detergents Gas chromatography-mass spectrometry

1. Scope This document describes a headspace-gas chromatography-mass spectrometry method for determining the content of various halogenated hydrocarbons, such as chloroform, in detergents. This document applies to detergents containing 1,1-dichloroethylene, dichloromethane, 1,1-dichloroethane, chloroform, 1,1,1-trichloroethane, carbon tetrachloride, etc. Determination of the contents of 1,2-dichloroethane, 2-chloropentane, 1-chloropentane, 1,1,2-trichloroethane,

1,1,1,2-tetrachloroethane, and 1,1,2,2-tetrachloroethane. This document does not apply to dry cleaning products.

3. Terms and Definitions This document does not contain any terms or definitions that need to be defined.

4. Principles Under certain temperature conditions, volatile haloalkanes in the sample within the headspace vial volatilize into the liquid space, generating a certain vapor pressure, and reaching... After reaching gas-liquid equilibrium, a gaseous sample is taken and separated by gas chromatography, then analyzed by mass spectrometry. The analysis is based on retention time, fragment ion mass-to-charge ratio, and... Qualitative analysis was performed using different ion abundance ratios, and quantitative analysis was performed using the internal standard method.

5. Reagents and Materials Unless otherwise specified, only reagents confirmed to be of analytical grade shall be used in the analysis.

5.1 All test water was Grade I water as specified in GB/T 6682.

5.2 Methanol, chromatographic grade.

5.3 Sodium chloride, baked in a muffle furnace at $(550 \pm 5)^{\circ}\text{C}$ for $\geq 3\text{h}$.

5.4 Halogenated hydrocarbon standard samples/standard substances (1000 mg/L), see Appendix A for relevant information.

5.5 Internal standard (1000 mg/L), see Appendix A for details.

5.3

g) sequentially. Add 40.0 μL of internal standard intermediate solution (5.7), quickly seal with aluminum cap, vortex evenly, sonicate for 10 min, and then test.

7.1.2 Laundry Detergent Pods Randomly select one laundry detergent pod, weigh it (accurate to 0.1g), and dissolve it in 40 times its volume of water, stirring gently with a glass rod. Weigh the dissolved product.

8.2 g (m_2 , weighed to

0.01

g) of the liquid was added to 1 g of sodium chloride (5.3) and 40.0 μL of internal standard intermediate solution (5.7) in 20 mL. The sample is placed in a headspace vial, quickly sealed with an aluminum cap, vortexed evenly, and sonicated for 10 minutes before testing.

5.6 Halogenated hydrocarbon standard intermediate solution (

10.0 mg/L). Take appropriate amounts of each haloalkanes standard sample/standard substance (5.4) and dilute with methanol (5.2) to

10.0 mg/L. Store in a sealed bottle at -18°C. Store away from light. Shelf life is 1 month.

5.7 Intermediate solution of internal standard (

50.0 mg/L). Take appropriate amounts of each internal standard (5.5) and dilute with methanol (5.2) to

50.0 mg/L. Store in sealed bottles at -18°C or below, protected from light. Stored, valid for one month.

5.8 Standard and Internal Standard Mixed Series Working Solutions. Take 5 headspace vials, first add 8 mL of water and 1 g of sodium chloride (5.3) to each, then prepare... Add 10.0 µL, 20.0 µL, 40.0 µL, 100 µL, and 200 µL of the intermediate standard solution of haloalkanes (5.6), and then add the intermediate solution of the internal standard for each. 40.0 µL of solution (5.7) was quickly sealed with an aluminum cap and vortexed to mix. The absolute masses of the standard series working solutions were 0.500 µg, 1.00 µg, and... 2.00 µg, 5.00 µg, and 10.0 µg, where the absolute mass of the internal standard is 2.00 µg. Prepare fresh before use.

6 Instruments and Equipment

6.1 Headspace-gas chromatography-mass spectrometry (electron impact ionization source, EI source).

6.2 Analytical balance, with an accuracy of

0.1 g and

0.1 mg.

6.3 Vortex Oscillator.

6.4 Muffle furnace [temperature controllable (550±5)°C].

6.5 Headspace autosampler.

7.1.1 Products other than laundry detergent pods Weigh

0.2 g (m1, accurate to

0.001

g) of sample into a 20 mL headspace vial, and add 8 mL of water and 1 g of sodium chloride (

7.2 Instrument Conditions

7.2.1 Headspace Reference Conditions The following are the reference conditions.

- a) Equilibrium temperature. 80°C;
- b) Injection temperature. 180°C;
- c) Transmission line temperature. 180°C;
- d) Equilibrium time. 30 min;
- e) Quantitative loop. 1 mL.

7.2.2 Chromatographic Reference Analysis Conditions The following are the reference conditions.

- a) Chromatographic column. DB-624 capillary column (60m x 0.32mm x 1.8μm), or other equivalent performance;
- c) Inlet temperature. 180°C;
- d) Injection method. split injection, split ratio 10:1;
- e) Programmed temperature rise. Initial temperature 40°C, hold for 0 min, increase to 180°C at 5°C/min, increase to 200°C at 20°C/min, hold 5 minutes.

7.2.3 Mass spectrometry reference analysis conditions The following are the reference conditions.

- a) Ion source temperature 230°C;
- b) Transmission line temperature 220°C;

7.3 Measurement

7.3.1 Qualitative determination Perform sample determination according to the instrument conditions in 7.2. If the ion chromatographic peaks of the sample solution and the standard appear at the same retention time, and Furthermore, the mass-to-charge ratio of the corresponding mass spectrometry fragment ions is consistent with that of the standard, and their abundance ratio should conform to the requirements of Table 1 compared with the standard sample (5.4). Therefore, it is judged that... The sample contains the corresponding analyte.

7.3.2 Quantitative determination Sample measurements were performed according to the instrument conditions described in 7.2, with the standard working curve solution (5.8) measured from low to high concentrations. The ratio of the mass concentration of the target analyte to its corresponding internal standard is plotted on the x-axis, and the ratio of the peak area of the target analyte to its corresponding internal standard is plotted on the y-axis. Working curve. Based on the peak area ratio, find the mass ratio of the corresponding components from the standard curve and calculate the content of each halogenated hydrocarbon in the sample. Ensure the sample... If the content of halogenated hydrocarbons in the sample bottle is within the range of the standard curve, and the test