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Surface active agents - Determination of volatile organic compounds residues - Headspace gas chromatography-mass spectrometry (GC-MS)

表面活性剂 挥发性有机化合物

残留量的测定 顶空气相色谱质谱

(GC-MS)联用法

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China.**

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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 by China Petroleum and Chemical Industry Federation.

This Standard shall be under the jurisdiction of Subcommittee on (Special) Surfactant of National Technical Committee on Chemicals of Standardization Administration of China (SAC/TC 63/SC 8).

The drafting organizations of this Standard: Shanghai Tianxiang Quality Technology Service Co., Ltd., Zhejiang Real Madrid Technology Co., Ltd.

Main drafters of this Standard: Ning Qiaoyu, Ling Lijun, Xie Xueqin, Zhang Jingjie, Huang Sheng, Tang Fuwei, Xia Yichu.

Surface active agents - Determination of volatile organic compounds residues - Headspace gas chromatography-mass spectrometry (GC-MS)

1 Scope

This Standard specifies the method for determining the volatile residues of organic compounds in surfactants by headspace gas chromatography-mass spectrometry.

This Standard is applicable to various surfactants.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 13173, *Surface active agents - Detergents - Testing methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 volatile organic compounds; VOCs

any organic compound having an initial boiling point lower than or equal to 250°C at a standard pressure of 101.3 kPa

4 Principle

The testing sample is placed in a headspace bottle and sealed. After heating in the headspace sampler for a certain period of time, the volatile components in the sample are directly introduced into the gas chromatograph for separation and detection. Calculate the concentration of organic compounds with a boiling point below 250°C by using the response coefficient of toluene.

8.3 Preparation of standard curve

Prepare 6 headspace bottles. Add 1.00 mL of standard solution A, B, C, D, E to each of the 5 headspace bottles. Add 1.00 mL of marker working solution to the other headspace bottle. Add 1 mL of water separately. Immediately cover and seal. The toluene contents shall be 0.1 µg, 0.5 µg, 2.0 µg, 10 µg, and 20 µg, respectively.

9 Procedures

9.1 Testing conditions

The testing conditions shall meet the following requirements:

- Inlet temperature: 230°C;
- Carrier gas: helium, flow rate at 1 mL/min;
- Column temperature: temperature programmed, held at 50°C for 2 min, raised to 100°C at 12°C/min, then raised to 230°C at 35°C/min;
- Detector: MSD;
- Ionization mode: electron bombardment ionization (EI), ionization energy 70 eV;
- Scan mode: full scan;
- Headspace parameters: 80°C, balance for 40min, injection time is 1min, injection volume is 1mL.

9.2 Testing

Prepare samples and standard curves as described in 8.2, 8.3. Put the headspace vial into the headspace sampler. Analyze according to the chromatographic conditions given in 9.1. Scan to obtain a chromatogram and record the peak area of the compound peaking before the marker.

10 Result expression

10.1 Calculate the content of volatile organic compounds (i) peaking in the sample before the marker according to formula (1):

$$m_i = \frac{x_i}{M} \dots\dots\dots (1)$$

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