GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 13.120
Y 41

GB/T 39181-2020

Consumer products - Polyester fiber and ABS material - Rapid determination method of fragrance allergens

消费品 聚酯纤维及 ABS 材质

致敏性芳香剂快速检测方法

Issued on: October 21, 2020 **Implemented on: October 21, 2020**

Issued by: State Administration for Market Regulation;

Standardization Administration of the PRC.

Table of Contents

Consumer products - Polyester fiber and ABS material - Rapid determination method of fragrance allergens

1 Scope

This Standard specifies the determination method of headspace gas chromatography-mass spectrometry for 54 kinds of fragrance allergens in consumer product polyester fiber and acrylonitrile-butadiene-styrene (ABS) material.

This Standard applies to the rapid determination of the content of 54 kinds of fragrance allergens in consumer product polyester fiber and ABS material.

2 Principle

CUT the polyester fiber into a headspace bottle; ADD a small amount of organic solvent. After crushing the ABS, use solvent dissolution-precipitation method to extract; PLACE the extracted trace solution into the headspace bottle. SET the headspace parameters. The fragrance in the sample volatilizes and quickly forms a dynamic equilibrium in the bottle (polyester fiber sample); or all is transferred to the headspace (ABS sample). At this time, the concentration of the fragrance in the headspace gas is proportional to its initial content in the sample. Introduce the headspace gas to the gas chromatograph-mass spectrometer for determination. By measuring the concentration of fragrances in the headspace gas, the content of fragrances in the sample can be obtained.

3 Reagents or materials

3.1 Acetone: Chromatographically pure.

3.2 Methanol: Chromatographically pure.

3.3 Fragrance reference materials: The purity is all greater than 90%. The material types are shown in Appendix A.

3.4 Single standard stock solution: Respectively weigh the appropriate amount of fragrance reference materials (3.3) accurately; USE acetone to prepare a single standard solution of appropriate concentration; STORE it in a refrigerator at 4 °C. The expiry date is 12 months.

FREEZE and crush the sample to a particle size of less than 1 mm; WEIGH 0.2 g (accurate to 0.1 mg) into a 20 mL glass bottle (4.6). ADD 5 mL of acetone to dissolve; then slowly add 5 mL of methanol while shaking. After the polymer precipitation is complete, take 40 μL of the supernatant and place it in a 20 mL headspace bottle (4.5). Quickly seal the headspace bottle; PLACE it in the headspace autosampler. Under the set headspace conditions, the fragrance is transferred to the gas phase. At this time, the concentration of the fragrance material in the headspace gas is proportional to its initial content in the sample. Introduce the gas in the quantitative loop into a gas chromatograph-mass spectrometer for analysis.

6 Analytical procedure

6.1 Headspace injection conditions

The test results depend on the apparatus used. General parameters for headspace injection cannot be given. The following operating conditions have been proven appropriate:

a) Equilibrium temperature: 180 °C~200 °C. High-temperature-resistant headspace bottle sealing caps and gaskets shall be used; otherwise the gasket may be damaged and leaking or even the bottle cap may burst.

b) Equilibrium time: 10 min (polyester fiber); 6 min (ABS).

c) Auxiliary gas pressurization pressure: 70 kPa (polyester fiber); 80 kPa (ABS).

d) Auxiliary gas pressurization time: 10 s.

e) Quantitative loop temperature: 5 °C~10 °C higher than the equilibrium temperature.

f) Transmission line temperature: 5 °C~10 °C higher than the quantitative loop temperature.

g) Volume of quantitative loop: 1 mL.

h) Equilibrium time of quantitative loop: 10 s.

i) Injection time: 20 s.

6.2 Determination conditions of gas chromatography-mass spectrometry

The test results depend on the apparatus used. General parameters for gas chromatography-mass spectrometry cannot be given. The set parameters shall

ensure that, during the determination, the measured component and other components can be effectively separated. The following parameters are available for reference:

a) Chromatographic column: Low-loss quartz capillary column (30 m×0.25 mm×0.25 μm) with a stationary phase of 35% phenyl and 65% dimethyl polysiloxane; or equivalent;

b) Temperature programming: KEEP at 40 °C for 1 min; RAISE to 210 °C at 5 °C/min; then raise to 300 °C at 10 °C/min;

c) Temperature of injection port: 280 °C;

d) Ion source temperature: 230 °C;

e) Transmission line temperature: 280 °C;

f) Carrier gas: Helium (purity≥99.999%). Flow rate is 1.0 mL/min;

g) Ionization method: EI ionization;

h) Ionization energy: 70 eV;

i) Injection mode: Split injection. Split ratio is 10:1;

j) Data acquisition mode: Selected ion monitoring (SIM). The analysis parameters of each material are shown in Appendix A;

k) Solvent delay: 2.5 min.

6.3 Drawing of standard curve

6.3.1 Polyester fiber

ADD 20 μL of standard working solution (3.6) to the headspace bottle (4.5) containing the blank sample of polyester fiber. Quickly press the bottle cap to prepare a series of standard samples with different fragrance content. Then, the standard samples are determined in order from low to high concentration. USE the peak area of the quantitative ion chromatographic peak as the ordinate; USE the corresponding fragrance mass (in micrograms) as the abscissa; DRAW the standard working curve. For the total ion chromatogram of 54 fragrance materials under the above analysis conditions, refer to Appendix B.

6.3.2 ABS

ADD 40 μL of standard working solution (3.6) to the headspace bottle (4.5). Quickly press the bottle cap to prepare a series of standard samples with

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------