GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 59.080.01

W 04

GB/T 24281-2009

Textiles - Determination of Volatile Organic Compounds - Gas Chromatography / Mass Spectrography

纺织品 有机挥发物的测定

气相色谱-质谱法

Issued on: June 11, 2009 **Implemented on: January 1, 2010**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration of the People's Republic of China.

Table of Contents

Textiles - Determination of Volatile Organic Compounds - Gas Chromatography / Mass Spectrography

Warning: personnel using this Standard shall have practical experience of working in a regular laboratory. This Standard does not point out all the possible safety issues. Users of this Standard shall undertake the responsibility of taking appropriate safety and health measures; ensure the compliance with the conditions stipulated by relevant national regulations.

1 Scope

This Standard specifies the method of adopting solid-phase microextraction (SPME) - headspace sampler (HS) - gas chromatography / mass spectrometry (GC/MS) for the determination of total volatile organic compounds, total aromatic hydrocarbons and vinyl chloride, 1,3-butadiene, toluene, vinyl-cyclohexene, styrene and 4-phenylcyclohexene in textiles.

This Standard is applicable to all types of textiles.

2 Normative References

Through the reference in this Standard, the clauses of the following documents become clauses of this Standard. In terms of references with a specified date, all the subsequent modification sheets (excluding the corrected content) or revised versions are not applicable to this Standard. However, the various parties that reach an agreement in accordance with this Standard are encouraged to explore the possibility of adopting the latest version of these documents. In terms of references without a specified date, the latest version is applicable to this Standard.

GB/T 5009.67-2003 *Method for Analysis of Hygienic Standard of Product of Polyvinyl Chloride for Food Packaging*

GB/T 6682 *Water for Analytical Laboratory Use - Specification and Test Methods* (GB/T 6682-2008, ISO 3696:1987, MOD)

3 Terms and Definitions

The following terms and definitions are applicable to this Standard.

5.2 Internal Standard Substance

N-octane or *n*-heptane.

5.3 Organic Solvents

N-pentane, methanol and butanone.

5.4 Preparation of Aromatic Hydrocarbon Mixed Solution

Use weight reduction method to respectively weigh-take around 0.1 g of aromatic hydrocarbon standard substance. Place it in a 4 mL sample bottle with a stopper (6.7). Seal it up, then, shake it well.

NOTE: this mixed solution shall be sealed and stored in a refrigerator at 0 °C ~ 4 °C; the validity period is 2 weeks.

5.5 Preparation of Standard Stock Solutions of Toluene, Vinyl-cyclohexene, Styrene and 4-phenylcyclohexene

Transfer-take a small amount of *n*-pentane into a 100 mL brown volumetric flask (6.8). Use weight reduction method to respectively weigh-take around 0.4 g (accurate to 0.2 mg) of toluene, vinyl-cyclohexene, styrene and 4-phenylcyclohexene in volumetric flasks. Use *n*-pentane to reach a constant volume.

NOTE: the standard stock solutions shall be sealed and stored in a refrigerator at 0 °C ~ 4 °C; the validity period is 2 weeks.

5.6 Preparation of Standard Working Solutions of Toluene, Vinyl-cyclohexene, Styrene and 4-phenylcyclohexene

Transfer-take an appropriate amount of the above-mentioned standard stock solutions (5.5) into 10 mL brown volumetric flasks (6.8). Use *n*-pentane to dilute to the scale, so that the concentration of the calibration working solutions is respectively: 2,000 μg/mL, 1,000 μg/mL, 500 μg/mL, 200 μg/mL, 50 μg/mL and 10 μg/mL.

NOTE: the standard working solutions shall be sealed and stored in a refrigerator at 0 °C ~ 4 °C; the validity period is 2 weeks.

5.7 Preparation of Standard Working Solutions of Vinyl Chloride and 1,3-butadiene

Use butanone as the solvent. In accordance with the methods of 6.2.3 and 6.2.4 in GB/T 5009.67-2003, respectively prepare standard working solutions of vinyl chloride and 1,3-butadiene with the concentration of 100 mg/mL, 50 mg/mL, 20 mg/mL, 10 mg/mL and 5 mg/mL. Prepare the solutions before usage.

NOTE: the relative density of vinyl chloride and 1,3-butadiene is respectively: 0.912 g/mL

7.2 Purification of SPME Extraction Head

Insert SPME extraction head into the gas chromatography inlet or other purification devices. At 300 °C, purify it for 60 min. Then, immediately insert the SPME extraction head into the gas chromatography inlet for GC / MS analysis, till there is no target object or non-stability interference chromatographic peak in the analysis chromatogram.

7.3 Preparation of Headspace Sampler

Use methanol to clean the inner wall of the headspace sampler (6.2) and the sample holder; dry them. After raising the temperature to 120 °C, place 2 blank test pieces (7.1), cover with the top cover; equilibrate it for 60 min. Through the sampling port on the top cover, insert the already-purified SPME extraction head (7.2) into the headspace sampler; conduct extraction for 20 min. Then, immediately insert it into the gas chromatography inlet for GC / MS analysis, till there is no target object or non-stability interference chromatographic peak in the analysis chromatogram.

8 Test Procedures

8.1 Preparation of Test Pieces

From test sample, cut 2 test pieces with the area of 100 cm^2. Accurately weigh-take its mass (accurate to 1 mg). Use aluminum foil to seal it up.

NOTE: during the preparation, appropriate measures may be adopted to avoid test piece contamination that might be caused by grease or environmental organic matters.

8.2 Determination of Standard Working Curves of Toluene, Vinyl-cyclohexene, Styrene and 4-phenylcyclohexene

Wait till the headspace sampler (6.2) is raised to 120 °C, stack two blank test pieces (7.1) on the sample holder; cover with the top cover. Use 10 μL micro-injection needle to respectively transfer-take 4 μL of toluene, vinyl-cyclohexene, styrene and 4-phenylcyclohexene standard working solution (5.6). Through the sampling port on the top cover, promptly inject into the headspace sampler. Meanwhile, inject 4 μL of internal standard solution (5.8); equilibrate it for 60 min. Then, through the sampling port on the top cover, insert the already-purified SPME extraction head (7.2) into the headspace sampler; conduct extraction for 20 min. Then, immediately insert it into the gas chromatography inlet. In accordance with the chromatographic conditions (8.5), analyze, determine and draw a standard working curve.

8.3 Determination of Standard Working Curves of Vinyl Chloride and 1,3-butadiene

8.3.1 In accordance with the steps of 8.2, through the sampling inlet of the headspace aromatic hydrocarbons in the chromatographic peak.

8.7 Sample Determination

Wait till the headspace sampler (6.2) is raised to 120 °C, then, stack two test pieces (8.1) on the sample holder; cover with the top cover. Through the sampling port on the top cover, inject 4 μL of the internal standard solution (5.8); equilibrate it for 60 min. Then, through the sampling port on the top cover, insert the already-purified SPME extraction head (7.2) into the headspace sampler. Conduct extraction for 20 min, then, immediately insert it into the gas chromatographic inlet; in accordance with the chromatographic conditions (8.5), analyze it. Before the determination, the sample shall receive a blank test.

NOTE: when the sample simultaneously contains vinyl chloride and 1,3-butadiene, the selective ion scanning mode may be used for determination.

9 Calculation of Results

9.1 Establishment of Linear Calibration Formula

Respectively use the corresponding ratio of the peak area and mass of six monomers (vinyl chloride, 1,3-butadiene, toluene, vinyl-cyclohexene, styrene and 4-phenylcyclohexene) in the standard working solutions, and the peak area and mass of the internal standard substances. Use the least square fitting method to obtain linear calibration formula (1), where the linear correlation coefficient γ shall be greater than 0.995.

$$\frac{A_i}{A_s} = K_i \times \frac{m_i}{m_s} + b_i \qquad \cdots\cdots\cdots\cdots (1)$$

Where,

A_i---peak area of six monomers;

A_s---peak area of internal standard substances;

K_i---slope of linear calibration formula of six monomers;

m_i---mass of six monomers, expressed in (mg);

m_s---mass of internal standard substances, expressed in (mg);

b_i---intercept of linear calibration formula of six monomers on the *Y*-axis.

9.2 Calculation of Desorption

9.2.1 Calculation of desorption of vinyl chloride, 1,3-butadiene, toluene, vinyl-

W_{mc}---mass concentration of total volatile organic compounds, expressed in (mg/kg);

M_c---desorption of total volatile organic compounds, expressed in (mg);

m_0---mass of test piece, expressed in (kg).

11 Lower Determination Limits

The lower determination limits of this Method are: vinyl chloride 0.3 mg/m^2, 1,3-butadiene 0.3 mg/m^2, toluene 0.001 mg/m^2, vinyl-cyclohexene 0.005 mg/m^2, styrene 0.005 mg/m^2 and 4-phenylcyclohexene 0.0005 mg/m^2.

12 Test Report

Test report shall at least provide the following content:

a) Standard being used;

b) Sample source and description;

c) Instruments being used and composition of blank test pieces;

d) Test result;

e) Statement of any deviation from this Standard;

f) Date of test.

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