GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 59.080.01

CCS W 04

GB/T 28189-2023

Replacing GB/T 28189-2011

Textiles - Determination of polycyclic aromatic hydrocarbons

纺织品 多环芳烃的测定

Issued on: May 23, 2023 **Implemented on: March 01, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the People's Republic of China.

Table of Contents

Textiles - Determination of polycyclic aromatic hydrocarbons

WARNING -- Personnel using this document shall have practical experience in formal laboratory work. This document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This document describes a method for the determination of 24 polycyclic aromatic hydrocarbons in textiles by gas chromatography-mass spectrometry (GC-MS).

This document applies to all types of textile products.

2 Normative references

This document has no normative references.

3 Terms and definitions

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

3.1 polycyclic aromatic hydrocarbons; PAHs

Aromatic hydrocarbons containing two or more benzene rings.

NOTE: There are two main combinations of PAHs. One is the non-fused ring type, including biphenyl, biphenyl and polyphenyl aliphatic hydrocarbons. The other is the fused ring type, that is, two carbon atoms are shared by two benzene rings.

4 Principle

The specimen is extracted by ultrasonic waves. If necessary, the extract is purified by a silica gel solid-phase extraction column, concentrated, and set to a constant volume. Use GC-MS to determine. Use the selected ion monitoring mode. Carry out quantification by external standard method.

5 Reagents and materials

Unless otherwise stated, all reagents used are chromatographically pure.

5.1 24 polycyclic aromatic hydrocarbon standard substances: in compliance with the provisions of Annex A. Purity is≥96%. Commercially available mixed standard solutions can also be used.

5.2 N-hexane: CAS No. 110-54-3.

5.3 Acetone: CAS No. 67-64-1.

5.4 Extraction solvent: n-hexane + acetone (volume ratio is 1:1).

5.5 Standard stock solution: use the standard substances listed in 5.1 to prepare a standard stock solution with a mass concentration of about 1000 mg/L with n-hexane (5.2).

NOTE: Store the standard stock solutions in the dark at 0°C~4°C. Be valid for 6 months.

5.6 Standard working solution: pipette an appropriate amount of the 24 polycyclic aromatic hydrocarbon standard stock solutions (5.5) into the same volumetric flask. Use n-hexane (5.2) to prepare a mixed standard working solution with a mass concentration of 10 mg/L. Then use n-hexane (5.2) to prepare a standard working solution with an appropriate concentration according to the needs of the work.

NOTE: Store the standard working solution in the dark at 0°C~4°C. Be valid for 3 months.

6 Instruments and equipment

6.1 Gas chromatography-mass spectrometry (GC-MS): equipped with an electron impact ionization source (EI).

6.2 Temperature-controllable ultrasonic generator: the temperature control accuracy is ±2°C at 60°C. The working frequency is 35 kHz ~ 45 kHz.

6.3 Rotary evaporator: the temperature control accuracy is ±2°C at 35°C.

6.4 Analytical balance: the division values are 0.1 mg and 0.01 g, respectively.

6.5 Extractor: with screw cap, about 50 mL, made of hard glass.

6.6 Flat bottom flask: 150 mL.

6.7 Organic phase needle filter membrane (PTFE): the pore size is 0.45 μm.

7 Analysis steps

7.1 Specimen preparation

Select at least 2 g of representative specimen. Cut into about 5 mm × 5 mm pieces. Mix well.

7.2 Extraction

Accurately weigh (1.0±0.01) g of the shredded specimen (7.1). Placed in a 50 mL extractor (6.5) with a screw cap. Add 30 mL of extraction solvent (5.4). After sealing, ultrasonically extract for 60 min in an ultrasonic generator (6.2) in a water bath at (60±2)°C. After cooling to room temperature, the extract is completely transferred to a 150 mL flat-bottomed flask (6.6). Conduct rotary evaporation under reduced pressure in a water bath at (35±2)°C until nearly dry. Dilute to 2 mL with n-hexane (5.2).

If necessary, the extract can be purified. Refer to Annex B for the purification method.

The extract is filtered through an organic needle filter membrane (6.7) with a pore size of 0.45 μm, and then analyzed by GC-MS (6.1).

7.3 GC-MS analysis conditions

Annex C gives examples of test parameters for GC-MS detection.

7.4 Qualitative and quantitative analysis

Under the same test conditions, take the specimen solution (7.2) and the standard working solution (5.6) to measure. Refer to conditional test analysis in Annex C. The analyte in the specimen has the same retention time as the standard substance detected at the same time (the allowable deviation is within ±0.25 min). The relative abundance of the qualifier ion is compared with the relative abundance of the corresponding qualifier ion in the spectrum of the mixed standard working solution with similar concentration. If the maximum allowable deviation does not exceed the range specified in Table 1, it can be determined that there is a corresponding analyte in the sample.

Table 1 -- Maximum allowable deviation of relative ion abundance for qualitative confirmation

Relative ion abundance/%	＞50	＞20～50	＞10～20	≤10
Maximum allowable deviation/%	±10	±15	±20	±50

According to the content of the analyte in the specimen, select the standard working solution with similar concentration for determination. If the detection response value of the sample solution exceeds the linear range of the instrument detection, it can be measured after appropriate dilution. Carry out quantification by external standard

Annex B

(informative)

Example of sample purification steps

B.1 Reagents and materials

Unless otherwise stated, all reagents used are chromatographically pure.

B.1.1 N-hexane: CAS No. is 110-54-3.

B.1.2 Dichloromethane: CAS No. is 75-09-02.

B.1.3 Purification and elution solvent: n-hexane + dichloromethane (volume ratio is 3:2).

B.1.4 Silica gel solid phase extraction column: 500 mg/3 mL or equivalent.

B.1.5 Organic phase needle filter membrane (PTFE): pore size is 0.45 μm.

B.2 Instruments and equipment

B.2.1 Solid phase extraction device.

B.2.2 Nitrogen blowing device.

B.3 Sample purification steps

Use a silica gel column to carry out sample purification. Select 500 mg/3 mL or equivalent silica gel solid phase extraction column (B.1.4). Use 5 mL of n-hexane (B.1.1) to pre-rinse before use to keep it moist. Transfer the sample solution treated in 7.2 to the silica gel solid-phase extraction column (B.1.4). Control the flow rate to 0.5 drops/s. Then add 5 mL of n-hexane (B.1.1) to rinse. Discard the above eluent. Then add 5 mL of purification and elution solvent (B.1.3) for elution. Collect the eluate. In a water bath at 35°C, blow slowly to nearly dry with a nitrogen blower (B.2.2). Dilute to 2 mL with n-hexane (B.1.1). After filtering through an organic needle filter membrane (B.1.5) with a pore size of 0.45 μm, it is analyzed by GC-MS (6.1).

NOTE: Selectively purify the samples with interfering substances according to the situation. It is not necessary to purify each sample.

Annex C

(informative)

Example of GC-MS detection test parameters

Since the test results are related to the instruments and conditions used, it is impossible to give general parameters for instrumental analysis. The following parameters have been found to be suitable for the test.

a) Gas chromatographic column: DM-PAH quartz capillary column, 30 m × 0.25 mm (inner diameter) × 0.25 μm (film thickness), or equivalent.

b) Heating program: the initial temperature is 70°C. Keep for 1 min. Rise to 230°C at 10°C/min. Then it is raised to 260°C at 3°C/min. Then ramp up to 280°C at 1°C/min. Finally, it is raised to 330°C at 10°C/min. Keep for 12 min.

c) Injection port temperature: 270°C.

d) Injection volume: 1 μL.

e) Sampling method: splitless injection.

f) Carrier gas: helium (purity is ≥ 99.999%); flow rate is 1.2 mL/min.

g) Transfer line temperature: 280°C.

h) Ion source temperature: 250°C.

i) Ionization method: EI.

j) Ionization energy: 70 eV.

k) Solvent delay: 4 min.

l) Selection of mass number: selected ion monitoring mode (SIM). See Table C.1 for the selection of mass spectrometry parameters.

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

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