ICS 59.080.01
CCS W04

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 28189-2023

Textiles - Determination of polycyclic aromatic hydrocarbons

纺织品 多环芳烃的测定

Issued on: May 23, 2023 **Implemented on: March 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

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Foreword

This document was issued on 23 May 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 1 March 2024.

It is a GB/T standard: recommended rather than compulsory, but it is the text a Chinese reviewer applies when assessing a submission.

1 Scope

GB/T 28189-2023 covers the determination of 24 polycyclic aromatic hydrocarbons, the aromatic compounds of two or more benzene rings, in textile products of every type by gas chromatography-mass spectrometry - the principle, in which the specimen is extracted under ultrasound, purified where needed on a silica gel solid-phase extraction cartridge, concentrated and made up to volume, then measured in selected ion monitoring mode; the reagents and materials, chromatographic grade and with the reference substances of not less than 96 percent purity; the instruments, the GC-MS with an electron impact source and a temperature-controlled ultrasonic bath; the analysis steps, from cutting at least 2 g of representative material into small pieces through to qualitative and quantitative measurement against the standard working solution; the calculation and presentation of results; the limit of quantitation and the precision; and the test report. Annex A carries the

chemical information for the 24 compounds, while Annexes B and C give worked purification steps and detection parameters. These compounds arrive with carbon black pigments, mineral oils and rubberised prints, several are carcinogenic, and skin contact with clothing is prolonged. Written for textile and garment testing laboratories, dyehouses, and buyers auditing chemical compliance.

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 um.

7 Analysis steps

7.1 Specimen preparation

Select at least 2 g of representative specimen. Cut into about 5 mm x 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 um, 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

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

Relative ion abundance/%

Maximum allowable deviation/%