GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 71.100.01;87.060.01

CCS G 55

GB/T 41071-2021

Determination of polycyclic aromatic hydrocarbons in dye products

染料产品中多环芳烃的测定

Issued on: December 31, 2021 **Implemented on: July 1, 2022**

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

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Warning -- The personnel using this document shall have practical experience in formal laboratory work. This document does not indicate all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This document specifies a method for the determination of 18 polycyclic aromatic hydrocarbon residues in dye products by gas chromatography-mass spectrometry (GC/MS).

This document applies to the determination of polycyclic aromatic hydrocarbons in various types of dye products.

2 Normative references

The following documents contain the provisions which, through normative reference in this document, constitute the essential provisions of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is applicable to this document.

GB/T 8170-2008 Rules of rounding off for numerical values & expression and judgment of limiting values

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle of the method

In an ultrasonic bath, use n-hexane-acetone mixed solvent to extract the polycyclic aromatic hydrocarbons in the sample, and purify the extract by solid phase extraction (Florisil SPE column); after condensing the eluant and making up to the scale, use a gas chromatography-mass spectrometer (GC/MS) for separation and determination, and

adopt peak area external standard method for quantification.

5 Reagents or materials

5.1 The types of standard polycyclic aromatic hydrocarbons and the reference parameters for determination shall comply with the provisions of Appendix A.

5.2 Dichloromethane, chromatographically pure.

5.3 n-hexane, chromatographically pure.

5.4 Acetone, chromatographically pure.

5.5 n-hexane-acetone mixed solvent: The volume ratio of n-hexane and acetone is 1:1.

5.6 n-hexane-dichloromethane mixed solvent: The volume ratio of n-hexane and dichloromethane is 8:2.

5.7 Florisil column (SPE column): 1000 mg, 6 mL.

6 Instruments and equipment

6.1 Gas chromatography-mass spectrometer (GC/MS): with EI source.

6.2 Chromatographic column: 50% phenylmethyl polysiloxane solid phase, such as DB-17MS, 30 m×0.25 mm×0.25 μm, or equivalent.

6.3 Analytical balance: The accuracy shall be 0.01 mg.

6.4 Ultrasonic generator.

6.5 Extractor: a tube with a stopper, 10 mL.

6.6 Centrifuge: 4000 r/min.

6.7 Nitrogen-blowing concentrator.

6.8 Solid phase extraction device.

7 Preparation of standard solutions

7.1 Polycyclic aromatic hydrocarbon single standard stock solutions

Respectively weigh 10 mg of the standard substances listed in Appendix A, and the weights shall be accurate to 0.01 mg; dissolve with n-hexane and dilute to the mark in

collect the supernatant of the extract. If the sample is completely dissolved, the ultrasonic extraction step can be omitted. Analyze it later with instruments.

8.2 Concentrating and solvent replacement

Adopt the nitrogen blowing concentrating method, and the temperature of the nitrogen blowing concentrator shall be set to 30 °C; concentrate the extract (8.1) to 1 mL~2 mL with a small flow rate of nitrogen, wash the wall of the concentrating tube with about 5 mL~10 mL of n-hexane, and then concentrate the liquid to about 1 mL. Purify it later.

8.3 Purification

Fix the Florisil column on the solid-phase extraction device, rinse and purify the column with 4 mL of acetone, and then add 4 mL of n-hexane. After the column is filled, close the control valve and soak for 5 min; then, slowly open the control valve, and close the control valve before the filler is exposed to air. Transfer the concentrated solution (8.2) to the column, wash the concentrating tube several times with about 2 mL of n-hexane in total, and transfer all the washing liquor to the column. Slowly open the control valve and collect the effluent. Before the adsorbent is exposed to air, add about 8 mL of n-hexane-dichloromethane mixed solvent (5.6) for elution, and collect all the eluent.

8.4 Concentrating and making the volume up to mark

The purified eluent shall be concentrated by nitrogen blowing, made up to 1.0 mL with n-hexane, mixed well, and transferred to a sample bottle for analysis later.

8.5 Preparation of blank samples

Prepare blank samples following the same procedures as for sample solution preparation.

9 Analysis steps

9.1 Chromatographic analysis conditions

Since the test results depend on the instrument used, it is impossible to offer general parameters for chromatographic analysis. The use of the parameters in Table 1 has been proved suitable for testing. The optimal analysis conditions can be selected according to different instruments and equipment.

V --- The value of the metered volume of the sample solution, in milliliters (mL);

A_s --- The peak area value of the target quantitative ion in the standard working solution;

m --- The value of the mass of the sample, in grams (g).

The calculation result is retained to two decimal places.

The calculation results are rounded off according to the provisions of GB/T 8170-2008.

11 Detection limits, precision and accuracy

11.1 Detection limit

If Method One in Chapter 8 is used for the preparation of the sample solution, the method detection limit of 18 types of polycyclic aromatic hydrocarbons is 0.2 mg/kg; If Method Two is used for the preparation of the sample solution, the method detection limit is 1.0 mg/kg.

11.2 Precision

In the same laboratory, the same operator uses the same equipment to carry out independent tests on the same test object within a short period of time according to the same determination method; the absolute difference between two independent test results shall not be greater than 20% of the arithmetic mean of the two determination results.

11.3 Accuracy

Adopting the standard addition method, add an appropriate amount of polycyclic aromatic hydrocarbon standard solution to 1 g of dye sample, the concentration of the standard addition is 10 mg/kg, and operate according to the provisions of Chapter 8 and Chapter 9; the measured recovery of polycyclic aromatic hydrocarbons (except naphthalene) shall be 70%~120%, and the recovery of naphthalene shall be 60%~120%.

12 Test report

The test report shall at least include the following content:

a) Description of the sample;

b) The document number;

c) Test conditions;

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