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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 37040-2018

Limits and determination of carcinogenic dyes in dye products

染料产品中致癌染料的限量和测定

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1 Scope

China's national standard for the limits and determination of carcinogenic dyes in dye products. It specifies the requirements, the principle, the test methods, the minimum detection limit, precision and recovery, and the test report, with a normative annex listing the dyes. It is the companion to the standard on sensitizing dyes and the more serious of the two. The dyes listed are those classified as carcinogenic in their own right - distinct from the azo dyes that are restricted because they can cleave to release carcinogenic amines, which is a separate control. Several have been used in enormous quantity for a century, and some remain in use in countries where the restrictions do not apply. Testing the dye product rather than the finished textile is the point of both standards: a mill that buys a compliant dyestuff cannot produce a non-compliant fabric from it, whereas testing the fabric finds the problem after the cloth has been made and the dye bath discharged.

This Standard specifies limits and determination methods of 12 carcinogenic dyes (see Annex A) in dye product. This Standard is applicable to various formulations of dye products.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682-2008, Water for analytical laboratory use - Specification and test methods (ISO 3696:1987, MOD)

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

3 Requirements

The content of each carcinogenic dye (see Annex A) in the dye product shall not exceed 150mg/kg.

4 Principle

After the sample is dissolved (or extracted), use high performance liquid chromatography-diode array detector (HPLC-DAD) to determine the nature; use peak area external standard method to quantify carcinogenic dyes. For Prepare the single-component carcinogenic dye standard stock methanol solution listed in Annex A of which the effective concentration is 200mg/L. Store it in the freezer of a refrigerator from light. It shall be valid for 1 year.

5.4.2 Standard working solution Group A: Take 0.5mL from the basic violet 14 standard stock solution and place it in a 50mL volumetric flask. Use methanol to dilute and set it to a constant volume. Group B: Respectively take 0.5mL from the remaining 11 standard stock solutions and place them in 50mL volumetric flasks. Standard working solution can be prepared into other suitable concentrations as required. Prepare when required.

5.5 Preparation of sample solution Methanol soluble dye: Weigh 0.5g of sample (to the nearest of 0.0001g). Place it in a 50mL volumetric flask. Use methanol to dissolve. Dilute and set it to a constant volume. Vibrate in an ultrasonic generator. Fully dissolve and make it ready for use. Methanol insoluble dye: Weigh 0.5g of sample (to the nearest of 0.0001g). Place it in a 100mL conical flask with grinding mouth stopper. Accurately add 50mL of methanol. In an ultrasonic generator, conduct 30min extraction. Cool to room temperature. Use 0.45μm PTFE membrane filter head to filter the extract for use.

5.6.1 HPLC-DAD analysis method

5.6.1.1 HPLC-DAD chromatographic conditions 5.6.1.1.1 Since the test results depend on the instrument used, it is impossible to give general parameters for chromatographic analysis. The following parameters have been proved to be suitable for testing. It may, according to different instruments and equipment, select the best analysis conditions. 5.6.1.1.2 Chromatographic column: Stainless steel column of which the length is 150mm, the inner diameter is 3.9mm, the stationary phase is RP18, the particle size is 5μm. 5.6.1.1.3 Detection wavelength: 200nm~700nm. 5.6.1.1.4 Quantitative wavelength: 400nm, 500nm, 540nm, 590nm, 620nm. Turn on the liquid chromatography-mass spectrometer. After the various operating conditions of the instrument are stable, use a micro syringe to pipette 20μL of sample solution and standard working solutions A, B to respectively inject

into injection valves. After the last component flows out (see Annex B), use chromatography workstation to process the result. Through characteristic ion and comparation on the retention time of the chromatographic peak of the sample and the standard sample, determine the nature.

5.7 Chromatogram Refer to Annex B for the liquid chromatogram of carcinogenic dyes determined by this method.

6 Minimum detection limit, precision and recovery rate

6.1 Minimum detection limit The minimum detection limit of each carcinogenic dye in this method is 10mg/kg.

6.2 Precision In the same laboratory, the same operator uses the same equipment according to the same test method. Within a short time, independently test the same testing object. The absolute difference between the two independent test results obtained is not more than 10% of its arithmetic mean.

6.3 Recovery rate Use standard addition method. Add 1mL of the standard stock solution to the sample that has been determined by this method not contain carcinogenic dyes. Operate according to Clause 5. The measured recovery rate of carcinogenic dyes shall be 70%~130%.

7 Test report

The test report shall at least give the following information:

a) Sample description;

b) Standard reference number used;