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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 36908-2018

Limits and determination of sensitizing dyes in dye products

染料产品中致敏染料的限量和测定

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1 Scope

China's national standard for the limits and determination of sensitizing dyes in dye products. It specifies the requirements, the principle, the test method, the minimum detection limit, precision and recovery, and the test report, with a normative annex identifying the dyes concerned. Sensitizing dyes are those that provoke an allergic reaction on skin contact - not through toxicity but by sensitising the immune system, so that a person who has reacted once reacts to every subsequent exposure, often for life. Most are disperse dyes used on synthetic fibres, and the reason they matter more than other dyes is that disperse dyes are held in the fibre rather than bonded to it, so they migrate out with sweat and warmth onto skin. The European restrictions and the Oeko-Tex requirements have made this a trade barrier as much as a health matter, which is why the standard controls the dye product rather than the finished textile: the honest place to stop a restricted dye is before it is applied.

This Standard specifies the limit requirements and determination methods for 20 kinds of sensitizing dyes (see Appendix A) in dye products. This Standard is applicable to various formulations of dye products.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this document.

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 sensitizing dye (see Appendix A) in the dye products shall be no greater than 150mg/kg.

4 Principle

The sensitizing dyes in the dyes are dissolved out with methanol as solvent; and each sensitizing dye is separated and qualitatively determined by high performance liquid chromatography-diode array detector (HPLC-DAD), and quantified by the peak area external standard method. The qualitative determination of the detected and uncertain samples was carried out by liquid chromatography-tandem mass spectrometry (HPLC-MS/MS).

5 Test Method

5.1 General provisions Unless otherwise specified, only use reagents confirmed to be analytically pure and the Class-III water specified in GB/T 6682-2008. The test result is judged according to the rounding value comparison method in

4.3.3 of GB/T 8170-2008.

5.2 Reagents and materials

5.2.1 Standard product of sensitizing dye (see Appendix A).

5.2.2 Acetonitrile: chromatographically pure.

5.2.3 Methanol: chromatographically pure.

5.2.4 Sodium acetate aqueous solution: 0.01mol/L.

5.2.5 PTFE membrane filter head: 0.45 μ m.

5.3 Apparatus

5.3.1 Liquid chromatograph: infusion pump - flow range of 0.1mL/min~5.0mL/min, within this range its flow stability is $\pm 1\%$; with diode array detector (DAD).

5.3.2 Chromatographic column: a stainless-steel column with a length of 150mm and an inner diameter of 3.9mm; the stationary phase is RP18, and the particle size is 5 μ m.

5.3.3 Chromatography workstation or integrator.

5.3.4 Flat-head micro syringe or autosampler.

5.3.5 Ultrasonic generator.

5.3.6 Conical flask with abrasive stopper: 150mL.

5.4 Analysis conditions of liquid chromatography

5.4.1 Flow rate: 0.8mL/min.

5.4.2 Column temperature: 40°C. many types of liquid chromatography-mass spectrometers, the qualitative methods of instruments with different principles shall be different. The conditions provided in

5.5 are the parameters of the high-performance liquid chromatography-tandem mass spectrometer; other types of instruments may be set according to the characteristics of the instrument itself, as long as it can meet the accuracy of the qualitative. See Appendix A for the qualitative characteristic ions of 20 kinds of sensitizing dyes.

6 Minimum Detection Limit, Precision and Recovery

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

6.2 Precision In the same laboratory, the absolute difference between the two independent test results obtained by the same operator using the same equipment and the same test method against the same object under test in a short period of time shall be no greater than 10% of its arithmetic average.

6.3 Recovery rate Use the standard addition method, add 1mL of the standard stock solution into the sample, determined by this method, not containing sensitizing dyes. Operate according to Clause 5, the measured recovery rate of sensitizing dye shall be 80%~120%.

7 Test Report

The test report shall give at least the following contents:

- a) Sample description;
- b) The used standard number;
- c) test results;
- d) The name and model of the used instrument;
- e) Deviations from the standards;