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NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 17592-2011

Replacing GB/T 17592-2006

Textiles - Determination of the Banned Azo Colourants

纺织品禁用偶氮染料的测定

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Jointly issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China (AQSIQ);
Standardization Administration of China (SAC).

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Foreword

This Standard is drafted in accordance with regulations given in GB/T 1.1-2009.

This Standard is the revision of GB/T 17592-2006 "Textiles - Determination of the banned azo colourants". Compared with GB/T 17592-2006, the main technical changes are as follows:

- The normative reference GB/T 23344 "Textiles-Determination of 4-aminoazobenzene" has been added (See Chapter 2);
- In Chapter 5, temperature-control ultrasonic generator has been deleted (See 5.1 of the 2005 edition);
- The pre-treatment provisions for polyester products have been deleted, which act as the notes in 6.1 (See 6.1.1 of 6.1 in the 2006 edition);
- Preparation methods for mixing standard working solution have been added (See 4.5.4);
- The expressions of gradient i) in the analysis methods for HPLC/ DAD have been amended (See 6.4.1; 6.4.1 of 2006 edition);
- Footnote a in Appendix A has been added; Note 2 has been amended (See Appendix A);
- Appendix B has been changed into informative appendix (See Appendix B; Appendix B of 2006 edition);
- The liquid chromatogram of carcinogenic aromatic amines standard substance has been added (See Appendix C).

This Standard was proposed by China National Textile and Apparel Council.

This Standard shall be under the jurisdiction of basic standard subcommittee of Technical Sub-Committee on Textiles of Standardization Administration of China (SAC/TC 209/SC 1).

Drafting organizations of this Standard: Shanghai Textile Science Research Institute, Standardization Institute of Textile Industry, and Standardization Institute of Textile Industry.

Chief drafting staffs of this Standard: Chen Yun, Zheng Yuying, Yang Haiying, Zhu Ying, and Zhang Xiaoyan.

Textiles - Determination of the Banned Azo Colourants

WARNING: The personnel using this Standard shall have practical experiences in regular laboratory work. This Standard does not point out all the possible safety problems. The users shall be responsible for taking appropriate safety and health measures and ensure to comply with the conditions specified in the relevant laws and regulations of the nation.

1 Scope

This Standard specifies the detection methods for the banned azo colourants (in textile products) which can decompose carcinogenic aromatic amines (see Appendix A).

This Standard is applicable to the textile products after dyeing and printing.

2 Normative References

The following documents are essential to the application of this document. For dated references, only the dated edition of the document is applicable to this Standard. For undated references, the latest edition (including all the amendments) of the document is applicable to this Standard.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 23344 Textiles-Determination of 4-aminoazobenzene

3 Principle

Textile sample uses sodium dithionite for reduction and decomposition to generate potential carcinogenic aromatic amine in the medium of citrate buffer solution; extract aromatic amine in the solution with proper solution - solution partition column. And scale the volume with appropriate organic solvent after concentrating; carry out determination with gas chromatograph (GC/MSD) which is equipped with mass selective detector. If necessary, select another method or more methods to determine the isomer. Carry out quantification with high pressure liquid chromatography - diode array detector (HPLC /DAD) or gas chromatography - mass spectrograph.

4 Reagents and Materials

4.1 General rules

disodium hydrogen phosphate in 1000 mL of Grade 2 water, pH =6.9;

i) Gradient: at starting, use 15% mobile phase A and 85% mobile phase B; then linearly change the gradient into 80% mobile phase A and 20% mobile phase B. Maintain for 5 min.

Accurately transfer 1 mL of methanol or other appropriate solvent to add it into the round-bottom flask (6.2.2) that has been concentrated to nearly dry. Mix the solution uniformly. Stand at still. Then take 1 μL of standard working solution (4.6.2) and sample solution respectively. Inject them in the chromatograph. Operate according to the above-mentioned conditions. Carry out qualitative determination by external standard method.

Note: When the above-mentioned analytical conditions are adopted, chromatogram of carcinogenic aromatic amine standard substance HPLC is detailed in Figure C.2 in Appendix C.

6.4.2 GC/MSD analysis method

Accurately transfer 1 mL of internal standard solution to add it into the round-bottom flask (6.2.2) that has bee concentrated to nearly dry. Mix the solution uniformly. Stand at still. And then take 1 μL of mixing standard working solution and sample solution respectively. Inject them into the chromatograph. Operate according to the conditions of 6.3.1. Ion mode may be selected to carry out thee qualitative determination. Grouping of internal standard quantitative is detailed in Appendix D.

7 Result Calculation and Expression

7.1 External standard method

The aromatic amine *i* content separated from the sample shall be calculated according to Formula (1):

$$X_i = \frac{A_i \times c_i \times V}{A_{iS} \times m} \quad (1)$$

Where:

X_i - Content of aromatic amine *i* separated from the sample, (mg/kg);

A_i - Peak area (or peak height) of aromatic amine *i* in sample solution;

c_i - Concentration of aromatic amine *i* in standard working solution, (mg/L);

V - Final volume of sample solution, (mL);

A_{iS} - Peak area (or peak height) of aromatic amine *i* in standard working solution;

m - Sample mass, (g).

7.2 Internal standard method

The aromatic amine *i* content separated from the sample shall be calculated according to Formula (2):

$$X_i = \frac{A_i \times c_i \times V \times A_{iSC}}{A_{iS} \times m \times A_{iSS}} \quad (2)$$

Where,

X_i - Content of aromatic amine i separated from the sample, (mg/kg);

A_i - Peak area (or peak height) of aromatic amine *i* in sample solution;

c_i - Concentration of aromatic amine *i* in standard working solution, (mg/L);

V - Final volume of sample solution, (mL);

A_{iSC} - Peak area of internal standard in standard working solution;

A_{iS} - Peak area (or peak height) of aromatic amine *i* in standard working solution;

m - Sample mass, (g).

A_{iSS} - Peak area of internal standard in sample solution.

7.3 Result expression

Test results shall be expressed with the inspection results of various aromatic amines. The calculated results shall be expressed to integer. If the test results are lower than the lower limit of the determination, the test results are deemed as not detectable.

8 Lower Limit of Determination

The lower limit of the determination in this method is 5 mg/kg.

9 Test Report

The following contents at least shall be given in the test report:

a) Sample source and description;

b) Adopted pre-treatment method of sample;

c) Adopted quantitative method;

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