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

GB 19601-2013

Replacing GB 19601-2004

**Limit and determination of 23 harmful aromatic amines in dye
products**

染料产品中23种有害芳香胺的限量及测定

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Foreword

All the technical content of this Standard is mandatory. This Standard is drafted in accordance with the rules of GB/T 1.1-2009. This Standard replaces GB 19601-2004 "Limit and determination of 23 harmful aromatic amines in dye products". Compared with GB 19601-2004, except editorial changes, the major technical changes are as follows: - DELETE the analysis methods of gas chromatographic (

4.4.3.2 in 2004 edition); - Quantitative methods have been transformed from the external-standard method into the internal-standard method (see 5.5.3,

4.4.4.2 in 2004 edition); - DELETE the column extraction from the sample-preparation methods (

4.4.1 in 2004 edition); - MODIFY the sample-preparation methods (see 5.5.1,

4.4.1 in 2004 edition); - COMBINE qualitative parts and quantitative parts to analysis steps (see 5.5.2,

4.4.3 in 2004 edition); - REMOVE the reserved time from Appendix A (Appendix A in 2004 edition); - DELETE the recovery-rate table (Table 2 in 2004 edition). This Standard is

proposed by China Petroleum and Chemical Industry Federation. This Standard is administered by the National Technical Committee of Dye Standardization (SAC/TC 134)

This Standard is drafted by organizations. Shenyang Chemical Institute Co., Ltd., Xuzhou Kaida Fine Chemical Co., Ltd., State-Key Laboratory of Fine Chemicals of Dalian University

of Technology, Jiangsu Yabang Dye Co., Ltd., Beijing Institute of Clothing, Institute of Product Quality Supervision and Inspection of Taizhou City, and National Dye Quality Supervision and Inspection Center. Chief drafters of this Standard. Ji Hao, Piao Kezhuang, Fu Ping, Peng Xiaojun, Zheng Junliang, Shen Rijong, Ji Lanqin, Zhou Yongkai, and Li Xinggen. The standard of previous version replaced by this Standard is. -GB 19601-2004. Limit and determination of 23 harmful aromatic amines in dye products Warning. Personnel using this Standard shall have practical working experience in qualified laboratory. This Standard does not point out all possible safety problems. Users are responsible for taking appropriate safety and healthy measures, and to ensure compliance with the relevant conditions of the national regulations.

1 Scope

GB 19601-2013 is the Chinese national standard on limit and determination of 23 harmful aromatic amines in dye products, in the field of paint and colour industries. It carries no /T suffix, which in the Chinese system means compliance is mandatory: a product or a practice within its scope has to meet it to be lawfully made, sold or carried out in China. It was issued on 6 September 2013 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 October 2014. Classification: ICS 87.060.10, CCS G55. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Standard specifies the allowable limit and determination methods of harmful aromatic amines in dye products (see Appendix A). This Standard is applicable to all kinds of commercial dyes, dye products, dye intermediates and textile-dye auxiliaries.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated edition is applicable for this document. For undated documents, the latest version (including all amendments) applies 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 judgment of limiting values

3 Requirements

The content of harmful aromatic amines (see Appendix A) in dye products shall be less

than 150mg (aromatic amines)/kg (product),

4 Principles

In dielectric of citrate buffer solution (pH=6.0), The dye sample uses sodium hyposulfite to achieve the reductive decomposition, and then uses the organic solvent to directly extract the aromatic amines from the lysis solution.

5 Experimental methods

5.1 General provisions Unless otherwise specified, it shall only use the reagents confirmed as analytical reagents and grade 3 water specified by GB/T 6682-2008. Judgment of the inspection results shall be conducted according to

4.3.3 rounding-value-comparison method in GB/T 8170-2008.

5.2 Reagents and materials

5.2.4 Glacial acetic acid.

5.2.5 Citrate buffer solution.

0.06 mol/L aqueous solution, pH = 6 (TAKE 12.526g citric acid and 6.320g sodium hydroxide, DISSOLVE them in water, and then DILUTE to 1000 mL). 5

2.6 Sodium hyposulfite. 200 g/L aqueous solution, when using, TAKE solid sodium hyposulfite (content of $\text{Na}_2\text{S}_2\text{O}_4 \geq 85\%$) and DISSOLVE it in water for preparation.

5.3 Instruments and equipment.

5.3.1 Balance. precision is 0.00002g.

5.3.2 Gas chromatography - mass spectrometry (GC/MS).

5.3.3 Constant-temperature water bath. temperature ranges from room temperature to 100°C.

5.3.4 Rotating evaporator.

5.4 Preparation of standard solution

5.4.1 Preparation of single standard solution WEIGH each 0.01g (accurate to 0.00002g) of standard material and internal standard substance listed in Appendix A and Appendix B respectively, USE chloroform to dissolve them and DILUTE into 10 mL volumetric flask;

5.5 Analysis steps

5.5.1 Pre-treatment of samples WEIGH 0.2g of sample (accurate to 0.0002g), PLACE in a 100mL grinding-mouth conical flask, ADD 24mL of citrate buffer solution, ADD 1.0mL of

internal standard solution, COVER the stopper. PUT the conical bottle into the water-bath pan in which the temperature is $70^{\circ}\text{C}\pm 2^{\circ}\text{C}$ to preheat for about 15 min, KEEP shaking from time to time, LET the sample to be completely dissolved as possible.

5.5.2 Chromatography

5.5.2.1 Operating conditions of gas chromatography-mass spectrometry Because test result depends on the instrument to be used, so it is impossible to provide general parameters of chromatography. Using following parameters (refer to Table 2) is proved to be suitable for test.

5.5.3 Result calculation The content of harmful aromatic amines in sample shall be represented by mass fraction ω , the value represented by mg/kg, calculated according to formula (1).

5.5.4 Spectrum The total ion chromatogram of gas chromatography-mass spectrometry of harmful aromatic amines determined by this Method refers to Appendix C.

6 Minimum qualitative limit, recovery and precision

6.1 Minimum qualitative limit The minimum qualitative limit of this Method is 20mg/kg, the determination result is "undetected" when it is below this limit.

6.3 Precision In same laboratory, USE the same instrument by same operator, according to same test method, the absolute difference values of two independent test results obtained from the independent tests of same object in a short period of time, shall not be greater than 30% of the arithmetic mean of these two determined values.

7 Test report

The test report shall provide following contents at least.

Appendix A

(Normative) The table of contents of harmful aromatic amines Table A.1 Name and related information of harmful aromatic amines