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

GB/T 33392-2016

**Leather and fur -- Chemical tests -- Determination of
4-aminoazobenzene derived from azo colorants**

皮革和毛皮 化学试验 禁用偶氮染料中4-氨基偶氮苯的测定

(English Translation)

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Foreword

This Standard was drafted as per the rules specified in GB/T 1.1-2009.

This Standard adopts the redrafting method to modify and use ISO 17234-2:2011

Leather - Chemical Tests for the Determination of Certain Azo Colorants in Dyed

Leathers - Part 2.Determination of 4-Aminoazobenzene.

Compared with ISO 17234-2:2011, this Standard made the structural adjustment, and had the technical changes; Appendix D gives the list for the structural adjustment, technical changes and its causes for reference.

This Standard was proposed by China National Light Industry Council.

This Standard shall be under the jurisdiction of National Technical Committee for Standardization of Leather Industry (SAC/TC 252).

Drafting organizations of this Standard. State Center of Quality Supervision and Test for Leather (Zhejiang); Haining Mengnu Group Co., Ltd.; Zhejiang Tongtianxing Group

Joint-Stock Co., Ltd.; Haining Sende Leather Co., Ltd.; Taizhou Lilin Leather Co., Ltd.; Zhejiang Grammy Garments Co., Ltd.; and Zhejiang Institute of Standardization.

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Leather and Fur -

Chemical Tests - Determination of 4-

Aminoazobenzene Derived from Azo Colorants

1 Scope

This Standard specifies the test method of gas chromatography-mass spectrometry and high-performance liquid chromatography of 4-aminoazobenzene (CAS number. 60-09-3) that is decomposed from certain azo dyes in leather and fur and exists in free form.

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)

3 Principle

After degreasing, the specimen is placed in an enclosed container; and is reduced by sodium dithionite in an alkaline medium at a temperature of 40°C; and then the 4-aminoazobenzene cleaved by reduction is extracted by tert-butyl methyl ether. After purification, the extract liquor is determined by gas chromatography - mass spectrometry (GC-MS) or high-performance liquid chromatography with diode array detector (HPLC-DAD).

4 Reagents

Unless otherwise specified, only use reagents confirmed to be analytically-pure and Grade-3 water specified in GB/T 6682-2008.

5 Apparatus

Gas chromatography-mass spectrometry (GC-MS), with an electron impact ion source (EI).

Liquid chromatograph (HPLC), diode array detector (DAD).

6 Preparation of the Specimen

The leather is prepared in accordance with the provisions of QB/T 2716; while the fur is prepared in accordance with the provisions of QB/T 1272. The sampling process shall avoid the loss of hair.

The glue and attachments on the sample shall be removed as cleanly as possible.

7 Analysis Procedures

Add 9.0mL of sodium hydroxide solution (4.2) to the glass reactor; shake vigorously after sealing; so that the sample is immersed in the liquid.

Capillary chromatographic column. DB-5ms, 30m $\times$ 0.25mm $\times$ 0.25 μ m; or equivalent.

Inlet temperature. 250°C.

8 Calculation of the Results

According to the GC-MS analysis results, the content of 4-aminoazobenzene in the sample shall be calculated according to Formula (2).

9 Detection Limit and Recovery Rate

The detection limit in this method for 4-aminoazobenzene in the sample is 5mg/kg. The recovery rate of the method shall be no less than 60%.

10 Presentation of the Results

The relative deviation of the two independent determination results shall be less than 10%. The content of 4-aminoazobenzene is expressed by the arithmetic mean of the results of two parallel tests, in mg/kg, and the results are retained one digit after the decimal point.

11 Test Report

The test report should include the following.

Appendix A

(Informative)

HPLC-DAD Chromatogram and Spectrogram

The HPLC-DAD chromatogram and spectrogram can refer to Figures A.1 and A.2, respectively.

Appendix B

(Informative)

GC-MS Chromatogram and Mass-Spectrogram

The GC-MS chromatogram and mass-spectrogram can refer to Figures B.1~B.3.

Appendix C

(Informative)

Other Chromatographic Analysis Methods

For the detection of 4-aminoazobenzene in leather and fur samples, the following chromatographic methods and conditions are for reference and use.

Appendix D

(Informative)

Technical Differences and Causes between this Standard and ISO

17234-2.2011

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