INTERNATIONAL STANDARD

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Textiles — Methods for determination of certain aromatic amines derived from azo colorants —

Part 3: Detection of the use of certain azo colorants, which may release 4-aminoazobenzene

Textiles — Méthodes de détermination de certaines amines aromatiques dérivées de colorants azoïques —

Partie 3: Détection de l'utilisation de certains colorants azoïques susceptibles de libérer du 4-aminoazobenzène

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html

This document was prepared by the European Committee Standardization (CEN) Technical Committee CEN/TC 248, *Textiles and textile products*, in collaboration with ISO Technical Committee TC 38, *Textiles*, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 14362-3 cancels and replaces ISO 24362-3:2014, which has been technically revised.

The following is a list of the major technical changes between this edition and ISO 24362-3:2014:

— addition of a new Clause 3 and renumbered;

— changes to 7.1 to clarify the preparation and use of sodium dithionite solution;

— changes to Clause 9 "Procedure" to improve the method, including using xylene as substitute for chlorobenzene (reasons: lower toxicity and lower adverse environmental effect of xylene).

A list of all parts in the ISO 14362 series can be found on the ISO website.

Textiles — Methods for determination of certain aromatic amines derived from azo colorants —

Part 3: Detection of the use of certain azo colorants, which may release 4-aminoazobenzene

1 Scope

Azo colorants that are able to form 4-aminoazobenzene, generate under the conditions of ISO 14362-1, the amines aniline and 1,4-phenylenediamine. The presence of these 4-aminoazobenzene colorants cannot be reliably ascertained without additional information (e.g. the chemical structure of the colorant used) or without a special procedure.

This document is supplementary to ISO 14362-1 and describes a special procedure to detect the use, in commodities, of certain azo colorants, which may release 4-aminoazobenzene, and that are

— accessible to reducing agent without extraction, particularly concerning textiles made of cellulose and protein fibres (e.g. cotton, viscose, wool, silk), and

— accessible by extracting the fibres (e.g. polyester or imitation leather).

For certain fibre blends, in 9.3 and 9.4 (with and without extraction) may need to be applied.

The procedure also detects 4-aminoazobenzene (Solvent Yellow 1), which is already available as free amine in commodities without reducing pre-treatment.

The use of certain azo colorants, which may release, by reductive cleavage of their azo group(s), one or more of the other aromatic amines listed in the *Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH) as regards Annex XVII*, except 4-aminoazobenzene, cannot be determined quantitatively with this method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14362-1:2017, *Textiles — Methods for determination of certain aromatic amines derived from azo colorants — Part 1: Detection of the use of certain azo colorants accessible with and without extracting the fibres*

3 Terms and definition

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 General

Certain azo colorants may release, by reductive cleavage of azo group(s), 4-aminoazobenzene.

Table 1 — 4-aminoazobenzene[a]

No.	CAS number	Index number	EC number	Substance
22	60–09–3	611–008–00–4	200–453–6	4-aminoazobenzene

[a] Proscribed aromatic amine under Regulation (EC) No 1907/2006 of the European Parliament and of the Council of 18 December 2006 concerning the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH), establishing a European Chemicals Agency.

5 Principle

After selection of a coloured test specimen from the textile article, the test specimen is tested according to the method of the colorant extraction for disperse dyes and/or the method of the direct reduction for the other classes of colorants (pigments and/or dyes) (see ISO 14362-1).

The textile sample or the residue of the sample extraction is treated with sodium dithionite in an alkaline solution at 40 °C in a closed vessel. 4-aminoazobenzene, which is released in the process, is transferred to a t-butyl methyl ether phase by means of liquid-liquid extraction. An aliquot of the *t*-butyl methyl ether phase is used for analysis. The detection and determination of 4-aminoazobenzene can be performed using chromatography (see Annex A).

If 4-aminoazobenzene is detected by one chromatographic method, then confirmation shall be made using one or more alternative methods.

6 Safety precautions

WARNING — 4-aminoazobenzene is classified as a substance known to be or suspected to be human carcinogen.

Any handling and disposal of these substances shall be in strict accordance with the appropriate national health and safety regulations.

It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Consult manufacturers for specific details, such as material safety data sheets and other recommendations.

7 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

7.1 Aqueous sodium dithionite solution, ρ = 200 mg/ml freshly prepared: rest the solution in a closed vessel for (55 ± 1) min, transfer it into an open glass beaker, rest for 5 min (−0 min, + 5 min) and then use within 10 min.

7.2 Sodium hydroxide aqueous solution, ω = 2 %.[1)]

1) ω = a mass fraction of 2%.

7.3 Methanol.

7.4 Xylene (mixture of isomers) CAS No 1330-20-7.

7.5 *t*-butyl methyl ether.

7.6 Sodium chloride.

7.7 4-aminoazobenzene, highest available defined purity standard.

7.8 Internal standards for gas chromatography (IS), e.g. in the case of GC-MS analysis, one of the following internal standards can be used:

— IS1: naphthalene-d8, CAS No.: 1146-65-2;

— IS2: 2,4,5-trichloroaniline, CAS No.: 636-30-6;

— IS3: anthracene-d10, CAS No.: 1719-06-8.

7.9 Standard solutions.

7.9.1 Internal standard solution, IS in *t*-butyl methyl ether, ρ = 10,0 µg/ml.

7.9.2 4-aminoazobenzene calibration solution for checking the experimental procedure and preparation of calibration solutions

4-aminoazobenzene in methanol, ρ = 500 µg/ml.

7.10 Grade 3 water, complying with ISO 3696.

8 Apparatus

8.1 Extraction apparatus, according to Figure 1, consisting of

— coil condenser NS 29/32,

— a hook made from an inert material to hold the specimen in place so that the condensed solvent drips onto the specimen,

— 100 ml round bottom flask NS 29/32, and

— heating source.

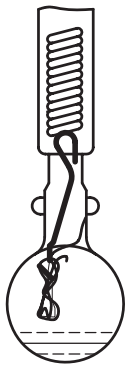

Figure 1 — Apparatus