



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN 14362-3:2012.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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

Textilien - Verfahren für die Bestimmung bestimmter
aromatischer Amine aus Azofarbstoffen - Teil 3: Nachweis
der Verwendung gewisser Azofarbstoffe, die 4-
Aminoazobenzol freisetzen können

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Foreword

This document (EN 14362-3:2012) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2012, and conflicting national standards shall be withdrawn at the latest by December 2012.

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1 Scope

Azo colorants that are able to form 4-aminoazobenzene, generate under the conditions of EN 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 part of EN 14362 is supplementary to Part 1 and describes a special procedure to detect the use of certain azo colorants in commodities, which may release 4-aminoazobenzene,

- accessible to reducing agent without extraction, particularly concerning textiles made of cellulose and protein fibres (e.g. cotton, viscose, wool, silk);
- accessible by extracting the fibres (e.g. polyester or imitation leather).

For certain fibre blends both parts of this standard (without or with extraction) may need to be applied.

The procedure detects as well 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, Authorisation 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 14362-1:2012, *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*

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

3 General

Certain azo colorants may release, by reductive cleavage of azo group(s), 4-aminoazobenzene, which is proscribed under *Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards Annex XVII*.

Table 1 — 4-aminoazobenzene proscribed under Regulation REACH 1907/2006/Annex XVII

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

4 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 the dyes (see EN 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.

5 Safety precautions

5.1 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.

5.2 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.

5.3 Good laboratory practice should be followed. Wear safety glasses in all laboratory areas and a single-use dust respirator while handling powder colorants.

5.4 Users should comply with any national and local safety regulations.

6 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

6.1 aqueous sodium dithionite solution, $\rho = 200 \text{ mg/ml}^{1)}$, freshly prepared, to use immediately after resting for one hour in a closed vessel

6.2 sodium hydroxide aqueous solution, $\omega = 2 \text{ \%}^{2)}$

6.3 *n*-pentane

6.4 methanol

6.5 chlorobenzene

6.6 *t*-butyl methyl ether

6.7 sodium chloride

1) ρ = mass concentration

2) ω = mass portion (% by weight)

6.8 4-aminoazobenzene, highest available defined purity standard

6.9 internal standards for gas chromatography (IS), e.g.:

IS1: benzidine-d8, CAS No.: 92890-63-6

IS2: naphthalene-d8, CAS No.: 1146-65-2

IS3: 2,4,5-trichloroaniline, CAS No.: 636-30-6

IS4: anthracene-d10, CAS No.: 1719-06-8.

6.10 standard solutions

6.10.1 internal standard solution, IS in *t*-butyl methyl ether, $\rho = 10,0 \mu\text{g/ml}$

6.10.2 4-aminoazobenzene calibration solution for checking the experimental procedure and preparation of calibration solutions

4-aminoazobenzene in methanol, $\rho = 500 \mu\text{g/ml}$

6.11 grade 3 water, complying with EN ISO 3696.

7 Apparatus

7.1 reaction vessel (20 ml to 50 ml) of heat-resistant glass, with tight closure

7.2 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;
- heating source.

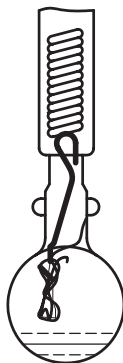


Figure 1 — Apparatus

NOTE Similar apparatus can be used, if the same results are obtained.

7.3 heating source that generates a temperature of $(40 \pm 2) ^\circ\text{C}$