SLOVENSKI STANDARD
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Tekstilije - Metode za doloļevanje nekaterih aromatskih aminov, izvirajoļih iz azo barvil - 2. del: Zaznavanje prisotnosti nekaterih azo barvil, dostopnih z ekstrahiranjem vlaken

Textiles - Methods for determination of certain aromatic amines derived from azo colorants - Part 2: Detection of the use of certain azo colorants accessible by extracting the fibres

Textilien - Verfahren für die Bestimmung bestimmter aromatischer Amine aus Azofarbstoffen - Teil 2: Verwendungsnachweis bestimmter Azofarbstoffe durch Extraktion der Fasern

Textiles - Méthodes de détermination de certaines amines aromatiques dérivées de colorants azoiques - Partie 2: Détection de l'utilisation de certains colorants azoiques accessibles par l'extraction des fibres

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English version

Textiles - Methods for determination of certain aromatic amines derived from azo colorants - Part 2: Detection of the use of certain azo colorants accessible by extracting the fibres

Textiles - Méthodes de détermination de certaines amines aromatiques dérivées de colorants azoïques - Partie 2: Détection de l'utilisation de certains colorants azoïques accessibles par l'extraction des fibres

Textilien - Verfahren für die Bestimmung bestimmter aromatischer Amine aus Azofarbstoffen - Teil 2: Verwendungsnachweis bestimmter Azofarbstoffe durch Extraktion der Fasern

This European Standard was approved by CEN on 1 September 2003.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document (EN 14362-2:2003) 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 April 2004, and conflicting national standards shall be withdrawn at the latest by April 2004.

Annexes A, C, D and E are informative. Annex B is normative.

This European Standard calls for the use of substances and/or procedures that may be injurious to health if appropriate precautions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This part of EN 14362 describes a procedure to detect the use of certain azo colorants that may not be used in the manufacture or treatment of certain commodities made of synthetic fibres dyed with extractable dyes.

For the direct test method, see EN 14362-1 *Detection of the use of certain azo colorants accessible without extraction*.

For certain fibre blends both parts of this standard may need to be applied.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references subsequent amendments to, or revisions of, any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

ISO 4787, *Laboratory glassware — Volumetric glassware – Methods for use and testing of capacity*

3 General

Certain azo colorants may release, by reductive cleavage of azo group(s), one or more of the following aromatic amines, which are proscribed under Directive 2002/61/EC.

Table 1 – Aromatic amines proscribed under Directive 2002/61/EC.

No.	CAS number	Index number	EC number	Substances
1	92-67-1	612-072-00-6	202-177-1	biphenyl-4-ylamin 4-aminobiphenyl xenylamine
2	92-87-5	612-042-00-2	202-199-1	benzidine
3	95-69-2		202-441-6	4-chloro-o-toluidine
4	91-59-8	612-022-00-3	202-080-4	2-naphthylamine
5*	97-56-3	611-006-00-3	202-591-2	o-aminoazotoluene 4-amino-2',3-dimethylazobenzene 4-o-tolylazo-o-toluidine
6*	99-55-8		202-765-8	5-nitro-o-toluidine
7	106-47-8	612-137-00-9	203-401-0	4-chloroaniline
8	615-05-4		210-406-1	4-methoxy-m-phenylenediamine

9	101-77-9	612-051-00-1	202-974-4	4,4'-methylenedianiline 4,4'-diaminodiphenylmethane
10	91-94-1	612-068-00-4	202-109-0	3,3'-dichlorobenzidine 3,3'-dichlorobiphenyl-4,4'-ylenediamine
11	119-90-4	612-036-00-X	204-355-4	3,3'-dimethoxybenzidine o-dianisidine
12	119-93-7	612-041-00-7	204-358-0	3,3'-dimethylbenzidine 4,4'-bi-o-toluidine
13	838-88-0	612-085-00-7	212-658-8	4,4'-methylenedi-o-toluidine
14	120-71-8		204-419-1	6-methoxy-m-toluidine p-cresidine
15	101-14-4	612-078-00-9	202-918-9	4,4'-methylene-bis-(2-chloro-aniline) 2,2'-dichloro-4.4'-methylene-dianiline
16	101-80-4		202-977-0	4,4'-oxydianiline
17	139-65-1		205-370-9	4,4'-thiodianiline
18	95-53-4	612-091-00-X	202-429-0	o-toluidine 2-aminotoluene
19	95-80-7	612-099-00-3	202-453-1	4-methyl-m-phenylenediamine
20	137-17-7		205-282-0	2,4,5-trimethylaniline
21	90-04-0	612-035-00-4	201-963-1	o-anisidine 2-methoxyaniline
22**	60-09-3	611-008-00-4	200-453-6	4-aminoazobenzene

*The CAS-numbers 97-56-3 (No. 5) and 99-55-8 (No. 6) are further reduced to CAS-numbers 95-53-4 (No. 18) and 95-80-7 (No. 19).

** Azo colorants that are able to form 4-aminoazobenzene, generate under the condition of this method aniline and 1,4-phenylenediamine. The presence of these colorants cannot be reliably ascertained without additional information, e.g. the chemical structure of the colorant used.

4 Principle

The colorant is extracted (see Figure 1) from the fibre in the headspace using appropriate solvents under reflux, for example for polyester fibres use chlorobenzene. The extract is concentrated, transferred with methanol, taken up in aqueous citrate-buffer solution and treated in an ultrasonic bath for dispersion of the colorant.

The amines formed by adding sodium dithionite are transferred to a t-butyl methyl ether phase by means of liquid-liquid extraction using diatomaceous earth columns. The extract is concentrated, and the residue is taken up in methanol or a solvent appropriate for detection and determination of the amines using chromatography.

If the amines are detected by one chromatographic method, then confirmation shall be made using one or more alternative methods.

5 Safety precautions

5.1 General

WARNING The substances [amines] listed in Table 1 are classified as substances known to be or suspected to be human carcinogens.

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

6.1 General

Unless otherwise specified, analytical grade chemicals have to be used.

6.2 chlorobenzene

6.3 methanol

6.4 ethyl acetate

6.5 t-butyl methyl ether

6.6 citrate/sodium hydroxide buffer solution, pH = 6, c = 0,06 mol/ml[1)]

6.7 aqueous sodium dithionite solution, ρ = 200 mg/m, l[2)] freshly (daily) prepared

6.8 diatomaceous earth

6.9 amine substances - amines 1 to 4, 7 to 21 (as specified in Table 1), and aniline and 1,4-phenylenediamine - all of highest available defined purity standard

6.10 standard solutions

6.10.1 calibration solution of amines (see 6.9) ρ = 15,0 µg of each amine per millilitre of an appropriate solvent

6.10.2 an appropriate mixture of internal standards in solution, ρ = 10,0 µg of each IS/ml of the appropriate IS solvent

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

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

IS3: 4-aminoquinaldine, CAS No.: 6628-04-2

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1) c is citrate concentration.

2) ρ is the mass concentration

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

6.10.3 solution of amine (see 6.9) for checking the experimental procedure, ρ = 30,0 µg of each amine per millilitre methanol

6.11 grade 3 water, complying with EN ISO 3696.

7 Apparatus

7.1 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 specimens

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. (See annex E.)

7.2 ultrasonic bath, controllable heating

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

7.4 heating source that generates (70 ± 2) °C

7.5 glass or polypropylene column, inside diameter 25 mm to 30 mm, length 140 mm to 150 mm, packed with 20 g of diatomaceous earth (6.8), fitted with glass fibre filter at the outlet. The diatomaceous earth columns are either bought pre-packed and used as is, or 20 g of diatomaceous earth can be packed into a glass or polypropylene column of the dimensions given.

7.6 vacuum rotary evaporator with water bath

7.7 pipettes 10 ml, 5 ml, 2 ml, 1 ml of class 1 complying with ISO 4787

7.8 chromatographic equipment selected from the following

7.8.1 thin layer chromatography (TLC) or high performance thin layer chromatography (HPTLC) equipment, including relevant detection

7.8.2 high performance liquid chromatography (HPLC) with gradient elution and diode array detector (DAD) or mass selective detector (MSD)

7.8.3 gas chromatography (GC) with flame ionisation detector (FID) or mass selective detector (MSD)