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Textiles — Dyestuffs —

Part 1:
General principles of testing coloured textiles for dyestuff identification

Textiles — Colorants —

Partie 1: Principes généraux d'essais des textiles colorés pour l'identification des colorants

Reference number
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Foreword

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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).

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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 16373-1 was prepared by the European Committee for 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).

ISO 16373 consists of the following parts, under the general title *Textiles — Dyestuffs*:

- *Part 1: General principles of testing coloured textiles for dyestuff identification*
- *Part 2: General method for the determination of extractable dyestuffs including allergenic and carcinogenic dyestuffs (method using pyridine-water)*
- *Part 3: Method for determination of certain carcinogenic dyestuffs (method using triethylamine/methanol)*

Introduction

The ISO 16373- series deals with dyes used in textiles for qualification and quantification.

This part of ISO 16373 includes the definition of the classes of dyes, the description of some procedures to identify qualitatively the class of dyes used in textile material.

The other parts of ISO 16373 are related to the quantification of some dyes.

— The principle of the test method in ISO 16373-2 is based on the extraction using pyridine-water solution, which has been found to be the most efficient solution to extract a large range of dyes, including allergenic and carcinogenic dyes.

— The principle of the test method in ISO 16373-3 is based on the extraction using triethylamine-methanol solution. This solution has been found efficient to extract some dyes in some cases.

Additional information related to the recovery rates (to characterize the extraction efficiency) obtained from the application of ISO 16373-2 and ISO 16373-3 are summarized in Annex B.

It is important to note that there are other test methods related to azo dyes, for which a reduction of the extracted azo dyes leads to the release of some aromatic amines to be detected and determined using chromatography (See Bibliography/Aromatic amines determination).

Textiles — Dyestuffs —

Part 1:
General principles of testing coloured textiles for dyestuff identification

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

1 Scope

This part of ISO 16373 gives the definition of the colourant classes and the relationship to textile fibres.

It describes some procedures to identify qualitatively the colourant class used in textile material.

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.

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

ISO 5089, *Textiles — Preparation of laboratory test samples and test specimens for chemical testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
colourant
dye or pigment

3.2
dye
dyestuff
water-insoluble or water-soluble molecule which has dyeing affinity with fibre

Note 1 to entry: The defining difference between a dye and a pigment is its solubility in an aqueous medium. In this part of ISO 16373, the term “affinity” is used as a qualitative expression, although affinity is the quantitative expression of substantivity and usually expressed in Joules per mole, where substantivity is the attraction between a substrate and a dye or other substance under precise conditions where the latter is selectively extracted from the application medium by the substrate.

3.3
pigment
water-insoluble molecule which has no dyeing affinity with fibre

Note 1 to entry: The defining difference between a dye and a pigment is its solubility in an aqueous medium. In this part of ISO 16373, the term “affinity” is used as a qualitative expression, although affinity is the quantitative expression of substantivity and usually expressed in Joules per mole, where substantivity is the attraction between a substrate and a dye or other substance under precise conditions where the latter is selectively extracted from the application medium by the substrate.

4 Fibre identification

Prior to any colourant identification (including dye class), the fibre nature of the textile product shall be known. The fibre nature may be based on information given by manufacturer, etc. or can be identified, using one or more techniques as described in ISO/TR 11827, for example.

An explanatory table of colourants used in various textile materials is given in Annex A.

5 Dye classes, based on the method of applying the dye

5.1 Acid dye

Acid dye is water-soluble anionic dye using neutral to acid dye baths. Attachment to the fibre (e.g. protein fibres and polyamide fibres) is attributed, at least partly, to salt formation between anionic groups in the dye and cationic groups in the fibre.

5.2 Metal complex acid dye

A metal complex dye is an acid dye that has a coordinate metal atom in its molecule.

5.2.1 1:1 Metal complex dye

1 molecule of dye is combined with 1 metal ion.

5.2.2 1:2 Metal complex dye

2 molecules of dye are combined with 1 metal ion. 1:2 metal complex dyes are the most frequent.

5.3 Azoic dye

Azoic dye is water-insoluble dye which is formed on the fibre by coupling a water-soluble diazo compound with a water soluble coupling compound having affinity for cellulose.

NOTE 1 The definition of the class “azoic dye” (‘dyers’ classification) is distinct from the definition of the class “azo dye” (see Clause 6, chemist classification).

NOTE 2 For the French version, the term “azoic dye” should be translated by “colorant azoïque insoluble” (known as well as “colorant naphtol”) in order to make a clear distinction with the translation of “azo dye” by “colorant azoïque”.

5.4 Basic dye (also called cationic dye)

Basic dye is water-soluble cationic dye using neutral to acid dye baths. Attachment to the fibre (e.g. acrylic) is attributed, at least partly, to salt formation between cationic groups in the dye and anionic groups in the fibre.

5.5 Mordant dye

Dye capable of forming a chelate complex with a metal ion (e.g. chromium) thus forming a metal complex dye *in situ* within the fibre.

NOTE Due to the toxicity of residual chromium ions in waste water, this kind of treatment is usually strictly controlled.

5.6 Direct dye

An anionic dye that interacts with the fibre (e.g. cellulosic fibre, polyamide fibres) by mechanical adsorption, normally applied from an aqueous bath containing electrolyte

5.7 Disperse dye

A water insoluble dye having affinity for hydrophobic fibres (e.g. polyester and acetate fibres).

NOTE The dyes are finely ground in the presence of a dispersing agent and sold as a paste, or spray-dried and sold as a powder. The very fine particle size gives a large surface area that aids dissolution to allow uptake by the fibre.

5.8 Reactive dye

5.8.1 General

Reactive dye contains specific reactive groups that are capable of chemically reacting with the fibre substrate to form a covalent chemical bond between the dye and the fibre.

5.8.2 Anthraquinone reactive dye

Anthraquinone reactive dye is a sub-class of reactive dyes where the chromophore part is based on anthraquinones.

5.9 Sulfur dye

Sulfur dye is water insoluble. It has to be treated with a reducing agent and an alkali at temperature of around 80 °C where the dye breaks into small particles which then becomes water soluble and hence can be absorbed by the fibre. After this, the textile product is removed from the dye solution and then taken for oxidation. During the oxidation step the small particles of dye once more form the parent dye which is insoluble in water.

5.10 VAT dye

VAT dye is essentially insoluble in water and incapable of dyeing fibres directly. However, reduction in alkaline liquor produces the water soluble alkali metal salt of the dye, which, in this leuco form, has an affinity for the textile fibre. Subsequent oxidation reforms the original insoluble dye.

6 Complementary dye class, based on chemist classification: Azo dye

Azo dye is dye bearing the functional group $R\text{-}N = N\text{-}R'$, in which R and R' can be either aryl or alkyl. The $N = N$ group is called an azo group.

NOTE 1 For example, some acid dyes (5.1, 5.2), some direct dyes (5.6), some disperse dyes (5.7), some reactive dyes (5.8), etc. are also azo dyes.

NOTE 2 The definition of the class “azo dye” (chemist classification) is distinct from the definition of the class “azoic dye” (5.3, dyers’ classification).

7 Reagents

Use only reagents of recognized analytical grade.

7.1 Water, grade 3 in accordance with ISO 3696.

7.2 Ammonia solution, concentrated.

7.3 Acetic acid, 5 % or 30 % in weight.

7.4 Tannic acid.

7.5 Sodium hydroxide solution, 5 % or 20 % in weight.

7.6 Sodium formaldehyde sulfoxylate.

7.7 Sulfuric acid, concentrated or 5 % in weight.

7.8 Carbazole.

7.9 Hydrogen peroxide, 30 % in weight.

7.10 Sodium dithionite.

7.11 Hydrochloric acid, 16 % in weight.

7.12 Magnesium ribbon.

7.13 Zinc dust.

7.14 Lead acetate paper.

7.15 Hypochlorite solution, at various commercial concentrations.

7.16 Pyridine.

7.17 Pyridine-water solution, 50:50.

7.18 Non-ionic detergent.

7.19 Scoured wool fabric.

7.20 Scoured cotton fabric.

7.21 Scoured secondary acetate fabric.

7.22 Ethylene diamine tetra-acetic acid disodium salt.

7.23 Glycerol.

7.24 Diethyl ether.

7.25 Liquid paraffin.

7.26 Toluene.

8 Apparatus

8.1 Test tubes, watch glass, and other laboratory glassware.

8.2 Ceramic crucible.

8.3 Balance, accurate to 0,1 g.

9 Conditioning and testing atmosphere

Because dye classes are determined, it is unnecessary to condition the specimen. The analysis is carried out under ordinary room conditions.

10 Preparation of the test specimens

The test specimens are prepared in accordance with ISO 5089.

11 Procedures (examples)

11.1 Pigment identification

Look at the fibres under a microscope: if there is a layer of dyes visible on the surface of the fibre and if the fibre is not dyed deep inside, it is typical of pigment dyeing.

11.2 Finishing removal treatment

Remove final chemical finishes by treating the sample twice in a boiling solution of 1 % of hydrochloric acid during about 5 min, then rinse thoroughly.

11.3 Acid dyes, basic, direct dyes and reactive dyes

See Table 1.

11.4 VAT dyes, sulfur dyes, reactive dyes, aniline black, azoic dyes, direct dyes, developed disperse dyes and chrome dyes

See Table 2.

11.5 Metal-complex dyes and disperse dyes

See Table 3.

11.6 Extraction test

See Table 4.

11.7 Ash test

See Table 5.

11.8 Miscellaneous tests

See Table 6.

12 Testing report

The test report shall include the following information:

a) the number and year of publication of this International Standard, i.e. ISO 16373-1:2015;

b) kind, origin and designation of the specimen (partial specimen, if applicable);

c) principle of the applied testing;

d) identified colourant class: pigment or class of the identified dye(s);

e) any deviation, by agreement or otherwise, from the procedure specified.

Table 1 — Acid dyes, basic, direct dyes and reactive dyes

<table>
<tr><td colspan="4">Treat 0,5 g of test specimen for 1 min in boiling 1 % ammonia solution (7.2).</td></tr>
<tr><td rowspan="3">A considerable amount of dye bleeds into solution.
Discard the test specimen.
Acidify the solution with 30 % acetic acid (7.3).
Add 0,5 g of scoured wool (7.19) and warm for 5 min to 10 min at 80°°C to 90 °C.
Wool is dyed: Acid dye</td><td colspan="3">Little or no dye bleeds into solution.
Treat 0,5 g of fresh test specimen for 1 min with boiling 5 % acetic acid (7.3).</td></tr>
<tr><td rowspan="2">The solution is distinctly coloured.
Divide the solution into 2 parts:
(1) To one part, add tannic acid (7.4). Formation of precipitate: Basic dye
(2) to the other part, add scoured wool (7.19) and warm for 4 min to 5 min. Staining of wool: Basic dye</td><td colspan="2">The solution is not coloured.
Treat 0,5 g of fresh test specimen for 1 min to 2 min with boiling 5 % sodium hydroxide solution (7.5).
Considerable amount of dye is stripped.
Divide the solution into 2 parts:
(1) To one part, add scoured cotton (7.20) and warm for 5 to 10 min. Deep staining: Direct dye. Little or no staining: Reactive dye
(2) To the other part, add sodium formaldehyde sulfoxylate (7.6). the solution is decolourised and original colour is not restored by oxidation: Direct dye is confirmed (non-dischargeable direct dyes will not respond to this treatment)</td></tr>
<tr><td>Direct dyes treated with formaldehyde.
Warm 0,5 g of test specimen in 5 % sulfuric acid (7.7). Allow it to cool. Discard the test specimen. Then add 0,1 % of carbazole (7.8) dissolved in concentrated sulfuric acid to this extract, drop by drop; the formation of a blue precipitate indicates the presence of formaldehyde.</td><td>Direct dyes treated with copper, chromium or nickel.
Take 1 ml of hydrogen peroxide (7.9) in a watch glass (8.1) and add 2 to 3 drops of concentrated ammonia solution (7.2). After the evolution of bubbles has ceased, add about 0,1 g of test specimen. If any vigorous action is observed, it indicates the presence of copper, chromium or nickel.</td></tr>
<tr><td colspan="4">NOTE Direct dyes treated with resins are not identified by the treatment given in this table, since the resin present will not have been removed in the treatment given for the preparation of the test specimen for the identification.</td></tr>
</table>

Table 2 — VAT dyes, sulfur dyes, reactive dyes, aniline black, azoic dyes, direct dyes, developed disperse dyes and chrome dyes

<table>
<tr><td colspan="6">Treat 0,5 g of test specimen for 2 min with 5 ml to 10 ml of boiling 20 % sodium hydroxide solution (7.5). Add 4 ml to 5 ml of water (7.1) and 15 mg to 35 mg of sodium dithionite (7.10) and boil for 1 min.</td></tr>
<tr><td colspan="2">If the colour of the sample is changed to another tone or the test specimen becomes colourless and on oxidation the original colour is restored: **VAT dye**, **Anthraquinone reactive dye** or **Sulfur dye**.</td><td rowspan="2">The colour of the test specimen readily becomes brown and is restored to black when exposed to atmosphere: **Aniline black**.
Warm the test specimen with concentrated sulfuric acid (7.7) for few seconds and pour the liquid into little cold water; a dull green colour is produced: **Aniline black** is confirmed</td><td colspan="2">The sample is permanently decolourised or is changed into another tone and the original colour is not restored on oxidation: **Azoic dye** or **certain Diazolisable Azoic dye** or **non-Anthraquinone reactive dye**</td><td rowspan="2">No colour change: **Chrome dye**.
Warm the test specimen for 1 min to 2 min with 16 % hydrochloric acid. Cool and add pyridine-water (50:50) (7.17). Dye bleeds into solution. Add wool mordanted with potassium dichromate to this extract, and warm for 2 min to 3 min.
Wool is dyed: **Chrome dye**.</td></tr>
<tr><td>Treat a fresh test specimen for 30 s with boiling 16 % hydrochloric acid. Cool and add 3 mg of magnesium ribbon (7.12) or pure zinc dust (7.13) and warm for 2 min to 3 min. Place a lead acetate paper (7.14) on the mouth of the test tube. The paper turns black and vapour give smell of hydrogen sulfide gas: **Sulfur dye**</td><td>Treat a fresh test specimen with cold diluted hypochlorite solution for 5 min to 10 min. The test specimen is either decoloured or its colour lighter in tone: **Anthraquinone reactive dye**. No change in colour: **VAT dye**.
(With blacks and greys, only after the absence of **Aniline black** has been confirmed)</td><td>Azoic dye or certain diazolisable azoic dyes
Treat 0,5 g of fresh test specimen for 1 min to 2 min with 5 ml of boiling pyridine (7.16). Profuse bleeding of colour: **Azoic dye**. Slight bleeding of colour: **developed disperse dye**.
Treat 0,5 g of fresh test specimen for 2 min to 3 min with 2 g/l of a non-ionic detergent (7.18) at 85 °C to 90 °C. Discard the test specimen, then add 0,5 g of scoured cotton (7.21) to the solution for 2 min to 3 min. Cotton is stained: **Direct dye**. No staining of the cotton: **developed Disperse dye**</td><td>Reactive dyes
Treat a fresh test specimen for 15 min under reflux with 1 ml of concentrated sulfuric acid (7.7) diluted to a litre with water and 2 g/l of sodium sulfate. Dye bleeds into solution. Add scoured wool (7.19). Wool is stained: **Reactive dye** (heterocyclic halogenated). No bleeding of colour: **Reactive dye** (other than heterocyclic halogenated).</td></tr>
</table>

Table 3 — Metal-complex dyes and disperse dye

Dissolve 4 g of ethylene diamine tetra-acetic acid disodium salt (7.22) in 100 g of glycerol (7.23). Heat the test specimen in this mixture at 110°C. Observe change in colour after 1 min to 2 min and after 20 min; Colour of the test specimen changes in 1 min to 2 min: **1:1 Metal-complex dye** Colour of the test specimen changes in 20 min: **1:2 Metal-complex dye**	Treat 0,5 g of test specimen for 5 min with boiling 5 % acetic acid (7.3). If some dye is stripped, cool, add diethyl ether (7.24) and shake well. Ether layer is coloured: **Disperse dye**. Treat a fresh test specimen in hot liquid paraffin (7.25) at 160°C for 5 min. If the dye is stripped, add scoured secondary acetate fabric (7.21). Tinting of acetate fabric: **Disperse dye**

Table 4 — Extraction test

<table>
<tr><td colspan="4">Extract 0,5 g of test specimen with 15 ml of pyridine-water (50:50) (7.17) in a test tube by keeping it in a beaker of boiling water for 10 min to 15 min, or until sufficient amount of dye bleeds into the reagent. Discard the test specimen and note the colour of the extract. Pour the solution into a separating funnel and add 15 ml of toluene (7.26); shake well and allow the two layers to separate. The distribution of dyes between the two layers is as follows:</td></tr>
<tr><td colspan="2">Toluene layer
All disperse dyes
Some neutral-dyeing 1:2 metal-complex dyes
Some VAT dyes</td><td colspan="2">Water layer
All direct dyes
All basic dyes
All acid-dyeing 1:1: metal-complex dyes
All chrome dyes
All acid dyes
Logwood (natural dye from <i>Haematoxylum campechianum</i>)
Some neutral-dyeing 1:2 metal-complex dyes</td></tr>
<tr><td colspan="2">Toluene layer
If toluene layer is coloured, wash it with water three times.
Separate the toluene layer again and evaporate it.
Disperse the residue with a few drops of a 10 % solution of a dispersing agent in water. Add scoured wool and acetate to this extract and warm for 15 min.</td><td colspan="2">Water layer</td></tr>
<tr><td>Only wool is dyed: neutral-dyeing metal-complex dyes</td><td>If both wool and acetate are dyed: Disperse dye</td><td>(I) With Logwood, the pyridine-water extract is coloured dark cherry-red. On addition of 1–2 ml of concentrated hydrochloric acid it turns yellow brown; further, when shaken with toluene, dye remains in water layer</td><td>(II) With chrome dyes, water layer is coloured; but sometimes toluene layer is also stained to a different colour than original dyeing</td></tr>
</table>

Table 5 — Ash test

Ash approximately 0,2 g to 0,3 g of test specimen in a crucible. Add approximately 0,2 g to 0,3 g of flux composed of equal parts by mass of powdered sodium carbonate and sodium nitrate. Fuse the mixture and allow it to cool. Note the presence of any metals as indicated below:	
Chromium	Yellow
Cobalt	Royal Blue
Copper	Faint Turquoise
Manganese	Blue-Green
Nickel	Brown
Presence of cobalt or manganese: Neutral dyeing **1:2 Metal-complex dye**	
Presence of chromium: **Direct Dye** (aftertreated with chromium salt); **Chrome Dye** or **Metal-complex dye** (i.e. **1:1** and **1:2 Metal-complex dyes**)	
Presence of copper or nickel: **Direct Dye** (aftertreated with copper salt or nickel salt respectively)	

Table 6 — Miscellaneous tests

Pigments (printed pigments, excluding pigments of mass dyed fibre)	If pigments are found to be present by the microscope test and if azoic and VAT dyes, which look like pigments, are found to be absent by the relevant tests, then the pigment present may be either carbon black or of phthalocyanine type.	
	With carbon black: there will be no discolouration when the test specimen is treated with sodium hydroxide and sodium dithionite (7.10).	If phthalocyanine pigment is present, spotting the test specimen with concentrated nitric acid will produce a bright green tone.

Annex A
(informative)

Explanatory table of colourants (dyes and pigments) used in various textile materials

Table A.1 — Explanatory table of colourants (dyes and pigments) used in various textile materials

Classes of colourants		Dye classes (5)										Pigment
		Acid (5.1)	Metal complex (5.2)	Azoic (5.3)	Basic (5.4)	Chrome (5.5)	Direct (5.6)	Disperse (5.7)	Reactive (5.8)	Sulfur (5.9)	VAT (5.10)	
Natural fibres												
Animal fibres	Wool	XX	XX			XX	(X)		X			X
	Silk	XX	X	=	(X)	X	(X)		X	(X)	(X)	X
Cellulose based	Cotton, Hemp, Flax, Sisal, Ramie, Jute			(X)			XX		XX	XX	XX	X
Man-made fibres												
Polyester								XX				X
Polyamide		XX	XX			X	(X)	X	X	X	X	X
Triacetate								XX				X
Acetate, secondary acetate							XX	X	XX	(X)	(X)	X
Acrylic					XX			(X)				
Viscose				(X)			XX		XX	X	XX	X
Chlorofibre								X				X

X means that the dye class is used – (X) means that the dye class is used in exceptional cases – XX means that the dye class is commonly used.

Annex B
(informative)

Comparison between ISO 16373-2 and ISO 16373-3: Recovery rates

The data as reported in Table B.1 are based on a study which has been carried out by Japan in 2010/2011.

Table B.1 — ISO 16373-2 and ISO 16373-3: Recovery rates

Recovery rate %	Acid Red 114		Acid Red 26		Basic Red 9	Disperse Orange 11	Disperse Yellow 3	Direct Red 28	Direct Black 38
	Polyamide (1 %)	Wool (0,2 %)	Polyamide (1 %)	Wool (0,2 %)	Acrylic (0,75 %)	Polyester (1 %)	Polyamide (0,75 %)	Silk (0,75 %)	Cotton (7 %)
ISO 16373-2 (pyridine-water solution)	89	95	73	88	35	44	92	101	60
ISO 16373-3 (triethylamine methanol solution)	76	16	73	7,6	0,5	19	84	75	12

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