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Textiles — Dyestuffs —

Part 2:
General method for the determination of extractable dyestuffs including allergenic and carcinogenic substances

Textiles — Colorants —

Partie 2: Méthode générale pour la détermination de colorants extractibles dont les substances allergènes et carcinogènes

ICS 59.080.01

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the European Committee for Standardization (CEN), and processed under the **CEN-lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five-month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 16373-2 was prepared by Technical Committee ISO/TC 38.

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*

— *Part 3: Method for determination of carcinogenic extractable dyestuffs*

Textiles — Dyestuffs —

Part 2:
General method for the determination of extractable dyestuffs including allergenic and carcinogenic substances

1 Scope

The analyses described in this standard are used to detect extractable dyestuffs in textile products. For all kind of fibres and types of dyestuffs the extraction is done with pyridine/water (1:1).

Annex A and B list the allergenic and carcinogenic dyestuffs which can be analysed with this method. These lists of dyestuffs are expandable.

NOTE The percentage of recovery using this method is shown in Annex F for the dyestuff classes (as defined in Part 1) acid, basic, direct, disperse, solvent dyestuffs and "mordant dyestuffs" on different textile fibres.

2 Normative references

The following referenced documents are indispensable for the application 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.*

3 Terms and definitions

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

3.1
allergenic dyestuff
dyestuff which may cause an allergic skin reaction

3.2
carcinogenic dyestuff
dyestuff which is classified as carcinogenic substance

NOTE Harmonized classification according to *Globally harmonized system of classification and labelling of chemicals (GHS)* [2] (incorporated in EU Regulation 1272/2008 (CLP) [3]).

4 Principle

A coloured test specimen is selected from the textile article and extracted with pyridine/water at 100 °C. The extract is analysed by liquid chromatography/diode array detector (LC/DAD) and/or by liquid chromatography/mass spectrometry (LC/MS).

5 Safety precautions

WARNING —The substances listed in Tables A.1, B.1 and B.2 are classified as substances known to be or suspected to be human allergens or carcinogens.

Ensure that any handling and disposal of these substances is 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.

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

NOTE Attention is drawn to any national and local safety regulations.

6 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

6.1 Pyridine.

6.2 Acetonitrile, chromatographic grade.

6.3 Ammonium acetate.

6.4 Tetrabutylammonium dihydrogen phosphate.

6.5 Deionized water, grade 3 according to ISO 3696.

6.6 Pyridine/water (1:1), mix 500 ml pyridine (6.2) and 500 ml water (6.6). Keep the solution in a brown glass bottle.

NOTE It is recommended to use reference substances (including those listed in Annex A and Annex B) of the highest purity grade available on the market. The given purity has to be considered for the calculation (9).

7 Apparatus

7.1 Apparatus and auxiliaries for sample preparation

7.1.1 Standard laboratory equipment.

7.1.2 Analytical balance, resolution at 0,01 g.

7.1.3 Glass vials (20 ml to 40 ml), with tight closure.

7.1.4 Heating source that generates 100 °C ± 2 °C (thermal block or laboratory sand-bath, controllable).

7.1.5 Autosampler glass vials, with tight closure.

7.2 Chromatographic equipment selected from the following:

7.2.1 Equipment for LC/DAD

— High performance liquid chromatograph (HPLC),

— DAD detector,

— separating column,

— guard column.

7.2.2 Equipment for LC/MS

— High performance liquid chromatograph (HPLC),

— electrospray ion source,

— MS detector,

— separating column,

— guard column,

8 Procedure

8.1 Preparation of test specimen

Cut the specimen into small pieces and weigh 0,5 g into a glass vial.

8.2 Extraction

Add 7,5 ml of pyridine/water (1:1) (6.7) to the test specimen and close the vial tightly. Heat the vial in the heating source until the solvent reaches a temperature of 100 °C ± 2 °C, and keep at this temperature for 35 min ± 5 min.

Check the time taken for the solvent to reach the required temperature using a blank.

Cool down to at 40°C or below before opening the vial. Transfer about 1 ml to a small vial for further analysis.

NOTE This step could be done with a syringe through the closed septum to minimize contact with pyridine.

8.3 Detection and quantification of the dyestuffs

Dyestuffs detection can be performed using the chromatographic techniques listed in 7.2. If other analytical techniques are used it shall be reported.

Dyestuffs quantification is performed by means of HPLC/DAD or HPLC/DAD/MS.

9 Calculation and expression of the results

Amounts of dyestuffs are usually calculated by means of a software program. The calculation can be carried out manually as described in Annex C.

Amounts of dyestuffs are expressed in mg dyestuff per kg textile (mg/kg).

If the detected amount of dyestuff is over 100 mg/kg it shall be assumed that a certain dyestuff was used, see Tables A.1, B.1 and B.2.

10 Test report

The test report shall refer to this official method and state at least the following particulars:

a) reference to this International Standard;

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

c) date of receipt and date of analysis;

d) sampling procedure;

e) detection method and quantification method;

f) results reported as level and detection limit per dyestuff in mg/kg;

g) any deviation of the procedure.

Annex A
(normative)

List of carcinogenic dyestuffs

Table A.1 — Reference carcinogenic dyestuffs

Number[a]	Carcinogenic dyestuff[b]	C. I. Number[c]	CAS Number	Molecular formula
1	Disperse Blue 1	64500	2475-45-8	$C_{14}H_{12}N_4O_2$
2	Solvent Yellow 1 4-aminoazobenzene	11000	60-09-4	$C_{12}H_{11}N_3$
3	Solvent Yellow 2	11020	60-11-7	$C_{14}H_{15}N_3$
4	Solvent Yellow 3 o-aminoazotoluene	11160	97-56-3	$C_{14}H_{15}N_3$
5	Basic Red 9	42500	569-61-9	$C_{19}H_{17}N_3HCl$
6	Basic Violet 14	42500	632-99-5	$C_{20}H_{19}N_3HCl$
7	Disperse Yellow 3	11855	2832-40-8	$C_{15}H_{15}O_2N_3$
8	Acid Red 26	16150	3761-53-3	$C_{18}H_{14}N_2Na_2O_7S_2$
9	Direct Black 38	30235	1937-37-1	$C_{34}H_{25}N_9Na_2O_7S_2$
10	Direct Blue 6	22610	2602-46-2	$C_{32}H_{24}N_6O_{14}S_4Na_4$
11	Direct Red 28	22120	573-58-0	$C_{32}H_{22}N_6Na_2O_6S_2$
12	Disperse Orange 11	60700	82-28-0	$C_{15}H_{11}NO_2$
13	Acid Red 114	23635	6459-9-5	$C_{37}H_{28}N_4Na_2O_{10}S_3$

[a] Numbering used in Tables D.1, D.4, D.5

[b] Classified according to (GHS), see Reference [2] (and to CLP, see Reference [3])

[c] Colur Index number (Reference [4])

Annex B
(normative)

List of allergenic and other dyestuffs

Table B.1 — Reference disperse dyestuffs

Number[a]	Allergenic dyestuff	C.I.-Number	CAS-Number	Molecular formula
A1	Disperse Blue 1	64500	2475-45-8	$C_{14}H_{12}N_4O_2$
A2	Disperse Blue 3	61505	2475-46-9	$C_{17}H_{16}N_2O_3$
A3	Disperse Blue 7	62500	3179-90-6	$C_{18}H_{18}N_2O_6$
A4	Disperse Blue 26	63305	3860-63-7	$C_{16}H_{14}N_2O_4$
A5	Disperse Blue 35	-	56524-77-7	$C_{15}H_{12}N_2O_4$
A6		-	56524-76-6	$C_{16}H_{14}N_2O_4$
A7	Disperse Blue 102	11945	12222-97-8	$C_{15}H_{19}N_5O_4S$
A8	Disperse Blue 106	111935	12223-01-7	$C_{14}H_{17}N_5O_3S$
A9	Disperse Blue 124	111938	61951-51-7	$C_{16}H_{19}N_5O_4S$
A10	Disperse Brown 1	11152	23355-64-8	$C_{16}H_{15}N_4O_4Cl_3$
A11	Disperse Orange 1	11080	2581-69-3	$C_{18}H_{14}N_4O_2$
A12	Disperse Orange 3	11005	730-40-5	$C_{12}H_{10}N_4O_2$
A13	Disperse Orange 37/76/59	11132	13301-61-6	$C_{17}H_{15}N_5O_2Cl_2$
A14	Disperse Red 1	11110	2872-52-8	$C_{16}H_{18}N_4O_3$
A15	Disperse Red 11	62015	2872-48-2	$C_{15}H_{12}N_2O_3$
A16	Disperse Red 17	11210	3179-89-3	$C_{17}H_{20}N_4O_4$
A17	Disperse Yellow 1	10345	119-15-3	$C_{12}H_9N_3O_5$
A18	Disperse Yellow 3	11855	2832-40-8	$C_{15}H_{15}N_3O_2$
A19	Disperse Yellow 9	10375	6373-73-5	$C_{12}H_{10}N_4O_4$
A20	Disperse Yellow 39	480095	12236-29-2	$C_{17}H_{16}N_2O$
A21	Disperse Yellow 49	-	54824-37-2	$C_{22}H_{22}N_4O_2$

[a] Numbering used in Tables D.2 and D.3

NOTE All dyestuffs in Table B.1 are not clinically verified as allergenic.