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Textiles — Dyestuffs —

Part 3:

Method for determination of certain carcinogenic extractable dyestuffs (method using triethylamine/methanol)

Textiles — Colorants —

Partie 3: Méthode de détermination de certains colorants cancérigènes extractibles (méthode en utilisant la triéthylamine/méthanol)

ICS 59.080.01

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO-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.

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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 16373-3 was prepared by Technical Committee ISO/TC 38, *Textiles*.

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

- *Part 3: Method for determination of certain carcinogenic extractable dyestuffs (method using triethylamine/methanol)*
- *Part 1: General principles of testing coloured textiles for dyestuff identification" under VA CEN lead (CEN TC248 WG 26) – Preliminary work item*
- *Part 2: General method for the determination of certain extractable dyestuffs" under VA CEN lead (CEN TC248 WG 26)*

Introduction

Under the circumstances of the consumers concern for the safety and hygiene, many countries have been introducing the regulations for the carcinogenic dyestuffs in the textile articles, especially important for the children and infant use. The testing methods for such carcinogenic dyestuffs used in the textile fabrics have not been prepared in the international standard organization (ISO). To support the regulations all over the world the test method to secure the regulation is very important in the present time. This standard gives the one method for one of such carcinogenic dyestuffs in the first time in ISO.

The series of ISO 16373 deal with dyestuffs used in textile for qualification and quantification.

- ISO 16373 - 1 includes the definition of the dyestuff classes the description of some procedures to identify qualitatively the dyestuff class used in textile material. The other parts of ISO 16373 are related to the quantification of some dyestuffs.
- ISO 16373-2, Textiles – Dyestuffs - Part 2: General method for determination of certain extractable dyestuffs including allergenic and carcinogenic. The principle of the test method 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 dyestuffs, including allergenic and carcinogenic dyestuffs.
- ISO 16373-3, Textiles – Dyestuffs - Part 3: Method for determination of certain carcinogenic extractable dyestuffs. The principle of the test method is based on the extraction using triethylamine-methanol solution. This solution has been found efficient to extract some dyestuffs in some cases.

Additional information related to the recovery rate (to characterize the extraction efficiency) obtained from the application of Part 2 and Part 3 are summarized in the informative Annex B of Part 1.

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 release some aromatic amines to be detected and determine using chromatography (cf. Bibliography / Aromatic amines determination)".

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Textiles — Dyestuffs —

Part 3:

Method for determination of certain carcinogenic extractable dyestuffs (method using triethylamine/methanol)

1 Scope

This International Standard specifies a method for the detection and quantitative determination of the presence of carcinogenic dyestuffs as listed below in dyed, printed or coated textile products by chromatographic analysis of their extracts.

- C.I. Basic Red 9, CAS No. 569-61-9
- C.I. Disperse Orange 11, CAS No. 82-28-0
- C.I. Disperse Yellow 3, CAS No. 2832-40-8
- C.I. Acid Red 114, CAS No. 6459-9-5
- C.I. Acid Red 26, CAS No. 3761-53-3
- C.I. Direct Black 38, CAS No. 1937-37-7
- C.I. Direct Red 28, CAS No. 573-58-0

2 Terms and definitions

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

2.1

textile

woven fabrics, knitted fabrics, etc., formed by the interlocking of fibres and yarns having a certain cohesion and which are generally intended for clothing or furniture applications

NOTE Often includes certain types of nonwovens textile of the definition

2.2

carcinogenic dyestuff

dyestuff classified as a substance known to be or suspected of being human carcinogens or allergens.

3 Principle

The dyestuff of a coloured test specimen is extracted by means of a solvent in an ultrasonic bath under specified conditions. The extract is analyzed using either a high performance liquid chromatography photodiode array detector (HPLC-DAD) or a mass spectrometer (HPLC-MSD).

4 Safety precautions

4.1 General

Warning-The dyestuffs targeted in this standard are classified as substances known to be or suspected of being human carcinogens or allergens.

4.2 Handling

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, etc.

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

Users should comply with any national and local safety regulations.

5 Apparatus

5.1 Ultrasonic bath, controllable heating

5.2 Coil condenser, proper size

5.3 Vacuum rotary evaporator with vacuum control and water bath

5.4 Round bottom flask, proper size

5.5 Pipettes, proper size

5.6 Measuring flask, necessary size

5.7 High Performance Liquid Chromatography

HPLC system and diode array detector (DAD) or mass spectroscope (MSD)

NOTE Refer to Annex A, details of the High Performance Liquid Chromatography equipment

5.8 Analytical balance, with resolution of 0,001 g.

6 Reagents

Unless otherwise specified, analytical grade have to be used.

6.1 Acetonitrile

6.2 Methanol

6.3 Hexane

6.4 0,25 % tri-ethylamine methanol solution

tri-ethylamine of 2,5 ml is added in the methanol of 1 l.

6.5 10 mmol/l ammonium acetate aqueous solution

ammonium acetate of 0,77 g is added in the water of 1 l.

6.6 Carcinogenic dyestuffs

use only the highest purity grade available in the market.

6.7 Standard solution of carcinogenic dyestuffs

The standard substance of 1 mg to 10 mg of carcinogenic dyestuffs are measured and placed in the 10 ml measuring flask, and then add in methanol (the standard solution: 100 µg/ml to 1 000 µg/ml). The standard solution may be dilute properly and made 4 solutions with known concentrations to draw the calibration curve. As an example, the range of the concentration of standard solutions for calibration curve may be recommended 1 µg/ml -100 µg/ml.

7 Test specimen sampling and preparation

7.1 General

The test specimen shall be selected based on the following criteria:

- Parts of the textile article;
- Nature of the fibre component (fibre composition);
- Colours.

Prepare a test specimen of 1,0 g max by cutting the laboratory sample up into small pieces no larger than 1 cm². Determine the mass of the test specimen to the nearest 0,01 g and record it as m_E (see 8.2)

8 Procedure

8.1 Extraction

8.1.1 Cleansing

If required remove oil, grease or other fatty matter from the surface of the test specimen by soaking it in 100 ml hexane (6.3) for 5 min in an ultrasonic bath (5.1) at ambient temperature.

Remove and drain the test specimen.

8.1.2 Extraction of dyestuff

Place 1,0 g of the test specimen in a 100 ml tube that can be hermetically sealed. Add 100 ml of the 0,25 % tri-ethylamine methanol solution (6.4) and seal the tube. Heat the tube in an ultrasonic bath until a temperature of $50\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ reached and maintain this temperature for 3 h.

8.1.3 Concentration of extract and preparation of analysis solution

Transfer the extract obtained in 8.1.2 to a 200 ml round bottom flask (5.4) and place in a vacuum rotary evaporator (5.3) in the water bath at $40\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until all liquid has evaporated.

Dissolve the residue in sufficient methanol to obtain a 1 ml solution. Filter the solution through a $0,45\text{ }\mu\text{m}$ PTFE filter. If the resultant measurement is higher than the calibrated range of the chromatograph, further dilute the solution with methanol.

8.2 Carcinogenic dyestuffs detection and quantification

The detection of carcinogenic dyestuffs is executed by the chromatographic analysis using the apparatus described in 5.7. When the carcinogenic dyestuffs were identified, the quantification is executed using calibration curve which is drew by using more than 4 points of standard solution and the correlation of the 4 points are more than 0,99 in the range concentration of $1\text{ }\mu\text{g/ml}$ to $100\text{ }\mu\text{g/ml}$. Quantification is executed by the method of HPLC/DAD. When the foreign substances are found a lot, HPLC/MSD is recommended to identify and quantify.

The carcinogenic dyestuff concentration in the specimen is calculated as mass of the specimen $w\text{ }\mu\text{g/g}$ according to the following formula

$$w = \frac{\rho_s \times V}{m_E}$$

Here

ρ_s interpolated concentration of carcinogenic dyestuff in $\mu\text{g/ml}$

V final solution volume made up to according to 8.1.2 in ml;

m_E mass of the test specimen in g.

9 Test report

The test report shall include the following: