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Textil – Bestämning av halten föreningar baserade på klorbensener och klortoluener

Textiles – Determination of the content of compounds based on chlorobenzenes and chlorotoluenes

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Europastandarden EN 17137:2018 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN 17137:2018.

The European Standard EN 17137:2018 has the status of a Swedish Standard. This document contains the official version of EN 17137:2018.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kemi, SIS/TK 160/AG 06.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 17137

November 2018

ICS 59.080.01

English Version

Textiles - Determination of the content of compounds based on chlorobenzenes and chlorotoluenes

Textiles - Détermination de la teneur de composés à base de chlorobenzènes et chlorotoluènes

Textilien - Bestimmung des Gehaltes von Verbindungen auf der Basis von Chlorbenzol und Chlortoluol

This European Standard was approved by CEN on 3 September 2018.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 17137:2018) 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of an identical text or by endorsement, at the latest by May 2019 and conflicting national standards shall be withdrawn at the latest by May 2019.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document is based on DIN 54 232, which was created by the Working Committee NA 062-05-12 AA “Textiles chemical test methods and fibre separation” of the Standard Committee Testing (NMP) in the DIN Deutsches Institut for Standardization.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document specifies a method of analysis for determining the content of chlorobenzenes and chlorotoluenes in textile products made of components such as outer fabric, interlining, lining, plastic slide fasteners, plastic buttons, labels, threads and appliques.

The method applies to a mass fraction of 0,1 mg/kg to 10 mg/kg per single isomer. Both higher and lower concentrations can be determined if the mass of the sample is selected accordingly or if appropriate dilutions are made during the analysis.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
component
individual part (one material) of the textile product sample

3.2
composite test specimen
test specimen composed from various sub-test specimens of components

4 Principle

The cut test specimen is extracted via ultrasonication in a closed vessel using dichloromethane. Interfering particles and fibres are removed by filtering through membrane filters. Without additional purification, the extract is analysed to determine the content of chlorobenzenes and chlorotoluenes by GC-MS with selected ion mode, using an internal standard for quantification.

5 Apparatus

5.1 Standard laboratory equipment.

5.2 Glass vials with tight closure.

NOTE Vial volumes of 40 ml to 100 ml have been found suitable.

5.3 Ultrasonic bath for extraction.

5.4 Analytical balance, resolution of at least 0,000 1 g.

5.5 Syringes with Luer Lock, 2 ml, with disposable syringe filters, made of polytetrafluoroethylene (PTFE) membrane.

NOTE Pore size of 0,45 µm has been found suitable.

5.6 Gas chromatograph with mass selective detector (MSD).

6 Reagents

6.1 Dichloromethane for residue analysis (analytical grade).

6.2 Reference substances

Reference substances are listed in Table 1.

Table 1 — Reference substances

Substance	CAS-Number [a]
2-Chlorotoluene	95–49–8
3-Chlorotoluene	108–41–8
4-Chlorotoluene	106–43–4
2,3-Dichlorotoluene	32768–54–0
2,4-Dichlorotoluene	95–73–8
2,5-Dichlorotoluene	19398–61–9
2,6-Dichlorotoluene	118–69–4
3,4-Dichlorotoluene	95–75–0
2,3,6-Trichlorotoluene	2077–46–5
2,4,5-Trichlorotoluene	6639–30–1
Pentachlorotoluene	877–11–2
1,2-Dichlorobenzene	95–50–1
1,3-Dichlorobenzene	541–73–1
1,4-Dichlorobenzene	106–46–7
1,2,3-Trichlorobenzene	87–61–6
1,2,4-Trichlorobenzene	120–82–1
1,3,5-Trichlorobenzene	108–70–3
1,2,3,4-Tetrachlorobenzene	634–66–2
1,2,3,5-Tetrachlorobenzene	634–90–2
1,2,4,5-Tetrachlorobenzene	95–94–3
Pentachlorobenzene	608–93–5
Hexachlorobenzene	118–74–1
2,4,5,6-Tetrachloro-m-Xylene (internal standard)	877–09–8

[a] Chemical Abstracts Service Number

7 Procedure

7.1 Sampling and test specimen preparation

The test specimen is usually a composite test specimen of sub-test specimens of different components. Attention has to be paid to take equivalent masses of the different selected components. For multicoloured and differently coloured products all available colours shall be selected and be tested.

Up to four colours may be tested together.

In order to gather four colours, the following rules shall be applied. The rules have been listed in order of preference:

— select the four colours from the same part of the textile article;

— if the four colours do not come from the same part of the textile article, select these four colours from textile parts made of the same type of textile fibre.

Each colour shall have approximately the same mass in order to obtain the total mass of 2 g.

If the rules cannot be applied (e.g. due to a complex printed pattern, plastic buttons), the sampling description of the selected test specimens shall be reported.

The test specimens are cut into pieces (approx. 5 mm wide) and stored in sealed glass vials until further processing.

7.2 Extraction

(2,0 ± 0,1) g of the cut test specimen are weighed in a vial, and overlaid with 20 ml dichloromethane. After adding 200 µl internal standard solution (e.g. at concentration of 10 µg/ml), the test specimen is extracted for (30 ± 1) min in an ultrasonic bath (starting at laboratory ambient temperature).

7.3 Filtration

1 ml of the extract is taken with a disposable syringe and cleaned of interfering particles and fibres with the help of membrane filters. After filtration, the extract is transferred into a vial.

7.4 Gas chromatographic determination

The substances of the extract are separated on a capillary column, and analysed using a mass-selective detector in selected ion mode (SIM).

Examples of instrumental parameters are shown in Annex A.

7.5 Calibration

Individual stock solutions are prepared for calibration of the reference substances (e.g. 10 mg to 10 ml of dichloromethane, mass concentration β = 1 mg/ml). Then a mixed standard is prepared (e.g. 1 ml of each stock solution to 100 ml of dichloromethane, mass concentration β = 10 µg/ml). This mixed calibration standard is used to prepare calibration solutions of various concentrations (e.g. 10, 50, 100, 200, 400, 600, 800, 1 000 ng/ml). Finally 1 ml of each individual calibration solution is mixed with 10 µl of the internal standard solution (e.g. at concentration of 10µg/ml) and analysed.

7.6 Calculation and expression of the results

The concentrations of chlorobenzenes or chlorotoluenes arise as a mass fraction in µg/ml of the following formula:

$$\beta_{sample} = \frac{F_{sample}}{F_{ISTD}} \times \frac{\beta_{ISTD}}{m} \quad (1)$$

where

F_{sample} is the measured value of the analyte (area value);

F_{ISTD} is the measured value of the internal standard (area value);

β_{sample} is the mass concentration of the analyte in the extract (µg/ml);

β_{ISTD} is the mass concentration of the internal standard in the extract (µg/ml);

m is the slope of the calibration.

The concentrations of the analytes are calculated as mass fraction w in mg/kg using the following formula:

$$w = \frac{\left(\beta_{sample} \times V\right)}{E} \quad (2)$$

where

w is the mass fraction (mg/kg);

V is the extraction volume (ml);

E is the initial mass (g).

8 Reliability of the method

For the reliability of the method, see Annex B.

9 Test report

The test report shall include at least the following particulars:

a) reference to this document;

b) identification of the submitted sample;

c) description of the sampling of individual components;

d) date of analysis;

e) content of the particular compound, the nature and content as a mass fraction in mg/kg of product;

f) any deviation from the given procedure.