



SLOVENSKI STANDARD
oSIST prEN 17137:2023
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Tekstilije - Določevanje spojin na osnovi klorobenzenov in klorotoluenov

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

Textilien - Bestimmung des Gehaltes von Verbindungen auf der Basis von Chlorbenzolen und Chlortoluolen

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

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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 draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 248.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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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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prEN 17137:2023 (E)**European foreword**

This document (prEN 17137:2023) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 17137:2018.

prEN 17137:2023 includes the following significant technical changes with respect to EN 17137:2018:

- The clauses have been reorganised and renamed according to CEN/CENELEC Internal Regulations Part 3:2019;
- Clause 1: The scope has been extended to α -chlorinated toluenes and rephrased;
- Clause 2: EN ISO 4787 has been added;
- Subclauses 3.1 and 3.2 have been deleted;
- Clause 4 has been rephrased;
- Clause 5 has been reorganised and rephrased;
- Subclause 5.2: Several α -chlorinated toluenes have been added in Table 1, and Table 1 has been reorganised.
- Clause 6 has been reorganised and rephrased;
- Subclause 6.6: Triple-quadrupole mass spectrometer has been added as a suitable detector;
- Clause 7 has been reorganised and rephrased;
- Clause 8 has been reorganised and rephrased;
- Subclause 9.2: A clause on how to calculate the sum of chlorobenzenes and chlorotoluenes has been added;
- Subclause 9.3: The limit of quantification of the test method is given;
- Clause 10 has been reorganised and rephrased;
- Annex A: A description on the occurrence of false positives has been added;
- Annex B: Test procedure if false positives are suspected has been added;
- Annex C: Former Annex A has become Annex C,
 - Annex C.2: Instrumental Conditions and characteristic masses for quantification with mass chromatograph/triple quad mass spectrometer (GC-MS/MS) have been added;
- Annex D: Former Annex B has become Annex D;
- Bibliography: Further references have been added.

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, Republic of North 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 test method using gas chromatography with mass selective detector (GC-MSD) for detection and quantification of chlorobenzenes, chlorotoluenes, and α -chlorinated toluenes in fibres, yarns, fabrics, coated fabrics and plastics.

NOTE 1 CEN/TR 16741 defines which materials are applicable to this test method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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

EN ISO 4787, *Laboratory glass and plastic ware - Volumetric instruments - Methods for testing of capacity and for use (ISO 4787:2021)*

3 Terms and definitions

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp/>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

The sample is cut into small pieces and extracted with dichloromethane in a sealed vial at a defined temperature in an ultrasonic bath. An aliquot of the extract is analysed for the substances listed in Table 1 using a GC-MSD.

5 Reagents

Unless otherwise specified, all reagents shall be of a recognised analytical grade.

5.1 Dichloromethane, CAS Registry Number^{®1} (CAS RN[®]): 75-09-2

¹ CAS Registry Number[®] (CAS RN[®]) is a trademark of CAS corporation. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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5.2 Reference substances

Reference substances are listed in Table 1.

Table 1 — Reference substances

Substance number	Substance	CAS RN®
	Chlorobenzenes	
1	Chlorobenzene	108-90-7
2	1,2-Dichlorobenzene	95-50-1
3	1,3-Dichlorobenzene	541-73-1
4	1,4-Dichlorobenzene	106-46-7
5	1,2,3-Trichlorobenzene	87-61-6
6	1,2,4-Trichlorobenzene	120-82-1
7	1,3,5-Trichlorobenzene	108-70-3
8	1,2,3,4-Tetrachlorobenzene	634-66-2
9	1,2,3,5-Tetrachlorobenzene	634-90-2
10	1,2,4,5-Tetrachlorobenzene	95-94-3
11	Pentachlorobenzene	608-93-5
12	Hexachlorobenzene	118-74-1
	Chlorotoluenes	
13	2-Chlorotoluene	95-49-8
14	3-Chlorotoluene	108-41-8
15	4-Chlorotoluene	106-43-4
16	2,3-Dichlorotoluene	32768-54-0
17	2,4-Dichlorotoluene	95-73-8
18	2,5-Dichlorotoluene	19398-61-9
19	2,6-Dichlorotoluene	118-69-4
20	3,4-Dichlorotoluene	95-75-0
21	3,5-Dichlorotoluene	25186-47-4
22	2,3,4-Trichlorotoluene	7359-72-0
23	2,3,5-Trichlorotoluene	56961-86-5
24	2,3,6-Trichlorotoluene	2077-46-5
25	2,4,5-Trichlorotoluene	6639-30-1
26	3,4,5-Trichlorotoluene	21472-86-6
27	2,3,4,5-Tetrachlorotoluene	1006-32-2
28	2,3,4,6-Tetrachlorotoluene	875-40-1
29	2,3,5,6-Tetrachlorotoluene	1006-31-1
30	Pentachlorotoluene	877-11-2
	α-chlorinated toluenes	
31	α -Chlorotoluene	100-44-7
32	α,α -Dichlorotoluene	98-87-3
33	α,α,α -Trichlorotoluene	98-07-7

Substance number	Substance	CAS RN®
34	$\alpha,\alpha,2,6$ -Tetrachlorotoluene	81-19-6
35	$\alpha,\alpha,\alpha,2$ -Tetrachlorotoluene	2136-89-2
36	$\alpha,\alpha,\alpha,4$ -Tetrachlorotoluene	5216-25-1
	Internal standard	
37	2,4,5,6-Tetrachloro-m-xylene	877-09-8

5.3 Standard solutions

5.3.1 Target compound stock solutions

Based on its tasks, a laboratory shall decide which substances from Table 1 need to be determined. Based on this decision, standard stock solutions for each substance (e.g., $c = 100 \mu\text{g/ml}$ for each substance) shall be available either as commercially available certified mixes or individual components in solution or as self-prepared individual or mixed standard stock solutions of each substance (Table 1) in dichloromethane (5.1).

EXAMPLE For standard stock solutions with concentrations of $100 \mu\text{g/ml}$, weigh 25,0 mg of each substance (Table 1) into a 250 ml volumetric flask, fill it up to the mark with dichloromethane (5.1.1) and mix thoroughly to dissolve the substance completely.

5.4.2 Target compound working solution

Prepare a mixed working solution containing all required substances in a suitable concentration for your GC-MSD system (e.g., $c = 10 \mu\text{g/ml}$).

5.4.3 Internal standard solution

Prepare an IS solution of a suitable concentration for your GC-MSD system (e.g., $c = 1 \mu\text{g/ml}$) by dissolving 2,4,5,6-Tetrachloro-m-Xylene (Table 1, item 37) in dichloromethane (5.1).

6 Apparatus

The usual laboratory apparatus and laboratory glassware, according to EN ISO 4787, shall be used, in addition to the following:

- 6.1 **Analytical balance**, with a precision of at least 0,1 mg.
- 6.2 **Glass vials** with tight closure, e.g., 40 ml.
- 6.3 **Ultrasonic bath**.
- 6.4 **Polytetrafluoroethylene (PTFE) filter**, at maximum 0,45 μm pore size.
- 6.5 **GC vial with cap**, e.g., 2 ml
- 6.6 **Gas chromatograph (GC)**, coupled with single-quadrupole mass spectrometer (MS) or triple-quadrupole mass spectrometer (MS/MS).

7 Sampling

7.1 Preparation of test specimens

The test specimen shall consist of a single material type (fibres, yarns, fabrics, coated fabrics or plastics). Up to three test specimens (of equal mass) of the same material type can be tested together taking into consideration the limits of quantification. Since different colours can contain different

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concentrations of chlorobenzenes, chlorotoluenes, and α -chlorinated toluenes, each coloured material shall be considered as one test specimen. Multicoloured material with small pattern shall only be tested as a single test specimen.

Cut the test specimen into pieces of about 0,3 cm to 0,5 cm edge length and store them in a sealed glass vial (6.2) until further processing.

8 Procedure**8.1 Extraction**

Weigh ($2,0 \pm 0,1$) g of the specimen (7.1) in a glass vial (6.2), record the mass to the nearest 10 mg, add 20 ml internal standard solution (5.4.3) appropriate for your GC-MSD (6.6) system, and seal the vial. If less than 2 g of test specimen is available, the test specimen mass shall be more than 0,5 g, and the volume of the internal standard solution (5.4.3) shall be reduced proportionately. Extract the test specimen for (30 ± 1) min in an ultrasonic bath (6.3) initially at laboratory ambient temperature.

After the extraction, let the solution cool down to nearly room temperature before opening the vial. Filter the solution through a PTFE filter (6.4). Transfer an aliquot of the extract into a GC-MS vial (6.5) and seal with a cap.

8.2 Determination by GC-MS or GC-MS/MS**8.2.1 Preparation of calibration solutions**

Prepare at least 5 calibration solutions of a mixture of the required substances listed in Table 1, including internal standard, from the working solutions 5.4.2, in dichloromethane (5.1), at suitable concentrations for the analysis. Each solution shall contain the internal standard in a concentration that matches the internal standard concentration in the extract (8.1).

8.2.2 Determination by GC-MS or GC-MS/MS

Determine the compounds listed in table 1 extracted in 8.1 by GC-MS or GC MS/MS (6.6) as required.

Examples of chromatographic conditions GC-MS and GC-MS/MS are given in Annex C.

It is known that particular substances used for textile finishing may degrade at temperatures above 150 °C in a GC inlet and release α -chlorinated toluenes (see Annex A). Take quality measures to avoid false positive results (Annex A). Examples of suitable instrumental parameters to avoid the formation of α -chlorinated toluenes are shown in Annex A and Annex B.

9 Expression of results**9.1 Calculation**

The concentrations of chlorobenzenes, chlorotoluenes and α -chlorinated toluenes arise as a mass fraction in $\mu\text{g/ml}$ from the following Formula 1:

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

where

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

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

c_{sample} is the mass concentration of the substance in the extract ($\mu\text{g/ml}$);

c_{IS} is the mass concentration of the internal standard in the extract ($\mu\text{g/ml}$);

m is the slope of the calibration graph.

The concentrations of the analytes are calculated as mass fraction w in mg/kg using the following Formula 2:

$$w = \frac{(C_{\text{sample}} \times V)}{E} \quad (2)$$

where

w is the mass fraction (mg/kg);

V is the extraction volume (ml);

E is the initial mass (g).

9.2 Sum of chlorobenzenes and chlorotoluenes

In certain cases, the sum of different chlorobenzenes and chlorotoluenes (chlorinated organic carriers) is requested as a final result. Only chlorobenzenes and chlorotoluenes that have been clearly identified shall be included in the sum.

The results of the identified chlorobenzenes and chlorotoluenes (as obtained in 9.1) are added to give the result of the sum. If the result for a single chlorobenzene or chlorotoluene is lower than $0,3 \text{ mg/kg}$ (9.3), this result is considered as zero and shall not be included in the sum.

9.3 Reliability of the method

This method is able to determine the concentrations of the substances listed in Table 1 with a limit of quantification (LOQ) of $0,3 \text{ mg/kg}$ or lower.

For the reliability of the method, see Annex C.

10 Test report

The test report shall include at least the following particulars:

- a) reference to this document (including year of publication);
- b) identification of the submitted sample;
- c) description of the sampling of individual components;
- d) date of test;
- e) mass fraction for each chlorobenzene, chlorotoluene and α -chlorinated toluene, in mg/kg (9.1);
- f) if requested, sum of certain chlorobenzenes and chlorotoluenes, in mg/kg (9.2);
- g) any deviation from the given procedure;
- h) any unusual features observed.