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IULTCS/IUC 25

Second edition
2015-02-15

**Leather — Chemical tests —
Determination of tetrachlorophenol-,
trichlorophenol-, dichlorophenol-,
monochlorophenol-isomers and
pentachlorophenol content**

*Cuir — Essais chimiques — Détermination de la teneur en
isomères de monochlorophénol, dichlorophénol, trichlorophénol,
tétrachlorophénol et en pentachlorophénol*

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 17070 was prepared by the Chemical Test Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS) in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

IULTCS, originally formed in 1897, is a world-wide organization of professional leather societies to further the advancement of leather science and technology. IULTCS has three Commissions, which are responsible for establishing international methods for the sampling and testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

This second edition of ISO 17070 cancels and replaces the first edition (ISO 17070:2006), which has been technically revised. ISO 17070:2006 previously replaced CEN/TS 14494.

The following changes have been made:

- the additional chlorinated phenol substances tetrachlorophenol-, trichlorophenol-, dichlorophenol-, and monochlorophenol-isomers are included;
- a new [Clause 3](#) has been inserted to list the chlorophenol abbreviations;
- the examples of chromatographic conditions, previously in 7.4, have been moved to a new [Annex A](#) (the previous [Annex A](#) becomes [Annex B](#)).

[Annexes A](#) and [B](#) are informative.

Introduction

This International Standard describes a procedure where the chlorinated phenols (CP) are acetylated before the chromatographic detection and the amount of the detected chlorinated phenyl acetate is quantified via an internal standard correction.

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Leather — Chemical tests — Determination of tetrachlorophenol-, trichlorophenol-, dichlorophenol-, monochlorophenol-isomers and pentachlorophenol content

1 Scope

This International Standard specifies a method for determining the content of tetrachlorophenol-, trichlorophenol-, dichlorophenol-, monochlorophenol-isomers, and pentachlorophenol, its salts, and esters in leather.

NOTE Bromophenol isomers can also be determined using this method.

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 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

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

ISO 4044, *Leather — Chemical tests — Preparation of chemical test samples*

ISO 4684, *Leather — Chemical tests — Determination of volatile matter*

3 Abbreviations

The following abbreviations are used for chlorinated phenols in this International Standard:

CP	chlorinated phenols
DiCP	dichlorophenol
MoCP	monochlorophenol
PCP	pentachlorophenol
TCG	tetrachloroguaiacol (tetrachloro- <i>o</i> -methoxyphenol)
TeCP	tetrachlorophenol
TriCP	trichlorophenol

4 Principle

First of all, the leather sample is submitted to steam-distillation.

After extraction into *n*-hexane, the chlorinated phenols (CP) are acetylated by acetic anhydride and the chlorinated phenyl acetates are analysed by gas-chromatography with an electron capture detector (ECD) or mass selective detector (MSD). Quantification is performed by an external standard and a correction made with an internal standard.

5 Apparatus

- 5.1 **Gas chromatography (GC)**, with ECD or MSD detector.
- 5.2 **Analytical balance**, weighing to an accuracy of 0,1 mg.
- 5.3 **Suitable apparatus designed for steam distillation.**
- 5.4 **Shaking machine**, capable of at least 200 cycles per minute.
- 5.5 **Volumetric flasks**, 500 ml and 50 ml.
- 5.6 **Erlenmeyer (conical) flask**, 100 ml.
- 5.7 **Separating funnel**, 250 ml, or **suitable vessel that allows separation of organic and aqueous phases**, that can be sealed for vigorous shaking.
- 5.8 **Pasteur-pipettes, graduated pipettes, suitable auto-pipettes.**
- 5.9 **Strainer with paper filter, grade 4**, diameter 125 mm.

6 Reagents

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Unless otherwise specified, analytical grade chemicals should be used. Water shall be distilled or deionized, Grade 3 in accordance with ISO 3696.

6.1 Chlorinated phenol mix

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A mix of the chlorinated phenols which contains the following isomers at a concentration of 100 µg/ml in acetone.

2-Chlorophenol	CAS ¹⁾ -Number: 95-57-8
3-Chlorophenol	CAS-Number: 108-43-0
4-Chlorophenol	CAS-Number: 106-48-9
2,3-Dichlorophenol	CAS-Number: 576-24-9
2,4-Dichlorophenol	CAS-Number: 120-83-2
2,5-Dichlorophenol	CAS-Number: 583-78-8
2,6-Dichlorophenol	CAS-Number: 87-65-0
3,4-Dichlorophenol	CAS-Number: 95-77-2
3,5-Dichlorophenol	CAS-Number: 591-35-5
2,3,4-Trichlorophenol	CAS-Number: 15950-66-0
2,3,5-Trichlorophenol	CAS-Number: 933-78-8
2,3,6-Trichlorophenol	CAS-Number: 933-75-5

1) CAS Chemical Abstracts Service.

2,4,5-Trichlorophenol	CAS-Number: 95-95-4
2,4,6-Trichlorophenol	CAS-Number: 88-06-2
3,4,5-Trichlorophenol	CAS-Number: 609-19-8
2,3,4,5-Tetrachlorophenol	CAS-Number: 4901-51-3
2,3,4,6-Tetrachlorophenol	CAS-Number: 58-90-2
2,3,5,6-Tetrachlorophenol	CAS-Number: 935-95-5
Pentachlorophenol	CAS-Number: 87-86-5

NOTE This chlorinated phenol mix is available from laboratory chemical suppliers.

6.2 Tetrachloroguaiacol (TCG) (tetrachloro-*o*-methoxyphenol), at a concentration of 100 µg/ml in acetone (internal standard), melting point 118 °C to 119 °C.

6.3 Sulfuric acid, 1 mol/l.

6.4 *n*-hexane, for residue analysis.

6.5 Potassium carbonate, K₂CO₃.

6.6 Acetic anhydride, C₄H₆O₃.

6.7 Anhydrous sodium sulphate.

6.8 Distilled water, in accordance with Grade 3 of ISO 3696.

6.9 Triethylamine.

6.10 Acetone.

7 Sampling and preparation of samples

If possible, sample in accordance with ISO 2418. Cut the leather sample into small pieces or grind the leather in accordance with ISO 4044. The dimensions of the pieces shall not be larger than 2 mm to 3 mm. If sampling in accordance with ISO 2418 is not possible (e.g. leathers from finished products like shoes, garments), details about sampling shall be given together with the test report.

8 Procedure

8.1 Steam-distillation

Accurately weigh approximately 1,0 g of the leather sample into the distillation vessel (5.3). Add 20 ml of 1 mol/l sulfuric acid (6.3) and 100 µl of the TCG stock solution (6.2). Submit the contents of the vessel to a steam distillation by using a suitable steam distillation apparatus. Use a 500 ml volumetric flask (5.5) with 5 g K₂CO₃ (6.5) to collect the distillate.

Distill about 450 ml. Make up to volume (500 ml) with distilled water (6.8).

In the case of extreme foaming, the heat source should be reduced.