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Leather — Chemical tests — Determination of pentachlorophenol content

Cuir — Essais chimiques — Détermination de la teneur 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.

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 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 Standardisation (CEN) Technical Committee CEN/TC 289, *Leather*, the secretariat of which is held by UNI, in accordance with the Agreement on technical co-operation 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 first edition of ISO 17070 cancels and replaces the first edition of CEN TS 14494:2003, which has been technically revised.

Introduction

This document is based on the English translation of *DIN 53313 Draft 1999* and describes a procedure where PCP is acetylated before the chromatographic detection and the amount of the detected PCP acetate is quantified via an internal standard correction.

Leather — Chemical tests — Determination of pentachlorophenol content

1 Scope

This International Standard specifies a method for determining the content of pentachlorophenol (PCP), its salts and esters in leather.

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

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

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

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

3 Principle

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

After extraction into *n*-hexane the PCP is acetylated by acetic anhydride and the PCP 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 correction made with an internal standard.

4 Apparatus

4.1 Gas chromatography with ECD or MSD.

4.2 Analytical balance, weighing to 0,1 mg.

4.3 Suitable apparatus designed for steam distillation.

4.4 Shaking machine.

4.5 Volumetric flasks, 500 ml, 50 ml.

4.6 Erlenmeyer (conical) flask, 100 ml.

4.7 Separating funnel, 250 ml, or **suitable vessel that allows separation of organic and aqueous phases**, that can be sealed for vigorous shaking.

4.8 Pasteur-pipette, **graduated pipette**, **suitable autopipette**.

4.9 Strainer with paper filter grade 4, diameter 125 mm.

5 Reagents

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

5.1 PCP solutions.

The concentration of pentachlorophenol can consist of free pentachlorophenol, its salts and esters.

5.1.1 Pentachlorophenol, 100 µg/ml in acetone.

5.1.2 PCP-acetate, 10 µg/ml in *n*-hexane.

5.1.3 PCP-acetate standard, 0,04 mg/l PCP-acetate (corresponds to 0,034 6 mg PCP/l) in *n*-hexane.

5.2 Tetrachloroguaiacol (TCG) (tetrachloro-*o*-methoxyphenol), 100 µg/ml acetone, marker and internal standard, melting point 118 °C to 119 °C.

5.3 Sulfuric acid, 1 mol/l.

5.4 *n*-Hexane, for residue analysis.

5.5 Potassium carbonate, K_2CO_3.

5.6 Acetic anhydride, $C_4H_6O_3$.

5.7 Anhydrous sodium sulfate.

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

5.9 Triethylamine.

5.10 Acetone.

6 Sampling and preparation of samples

If possible, sample in accordance with ISO 2418 and grind leather in accordance with ISO 4044. 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.

7 Procedures

7.1 Steam-distillation

Accurately weigh approximately 1,0 g of the leather sample into the distillation vessel (4.3). Add 20 ml 1 mol/l sulfuric acid (5.3) and 0,1 ml TCG stock-solution (5.2). Submit the content of the vessel to a steam distillation

by using a suitable steam distillation apparatus. Use a 500 ml volumetric flask (4.5) with 5 g K_2CO_3 (5.5) as a receiver.

Distill about 450 ml. Make up to volume with water.

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

7.2 Liquid-liquid-extraction and acetylation

7.2.1 Transfer 100 ml of the distillate obtained in 8.1 into a 250 ml separating funnel (4.7).

7.2.2 Add 20 ml *n*-hexane (5.4), 0,5 ml triethylamine (5.9) and 1,5 ml acetic anhydride (5.6) to the solution and shake for 30 minutes vigorously on a mechanical shaker (4.4).

The derivatization step is a two-phase reaction and depends very strongly on the intensity of shaking. Use a suitable mechanical shaker with a high shaking frequency (at least 200 cycles/min). Do not try to shake by hand because this will produce incorrect results. Pressure compensation should be carried out before fixing the funnel (4.7) to the mechanical shaker (4.4).

7.2.3 After phase separation, transfer the organic layer to a 100 ml conical flask (4.6) and shake the aqueous layer again with 20 ml *n*-hexane.

7.2.4 Dehydrate the combined *n*-hexane extracts, with anhydrous sodium sulfate (5.7) in a 100 ml conical flask (4.6) for approximately 10 min.

7.2.5 Afterwards, filter (4.9) the *n*-hexane extract quantitatively, washing with *n*-hexane into a 50 ml volumetric flask (4.5).

7.2.6 Make up to volume with *n*-hexane.

7.2.7 Analyse this solution by GC (4.1).

7.3 Preparation of calibration mixture for acetylated PCP and TCG

7.3.1 Derivatization of PCP and TCG standard for recovery rate

To calculate the recovery, prepare a PCP/TCG standard mixture like the sample.

Measure 100 µl of stock-solution (5.1.1) and 100 µl TCG (5.2) into the distillation vessel together with 20 ml sulfuric acid (5.3). Treat this solution in the same way as the sample.

The recovery rate shall be higher than 90 %.

7.3.2 PCP-acetate-standard (External standard)

In addition, analyse a PCP-acetate-standard (5.1.3) directly by gas-chromatography. The final concentration for the GC is 0,04 mg/l PCP-acetate.

This standard is included in the calculation.

7.3.3 Derivatization of TCG-standard

Acetylate 20 µl of the TCG-solution (5.2) in 30 ml of 0,1 mol/l K_2CO_3 in the same way as the sample and transfer the organic layer into a 50 ml volumetric flask (4.5).

Analyse a TCG-standard in the same way as the sample.