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IULTCS/IUC 4

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Leather — Chemical tests — Determination of matter soluble in dichloromethane and free fatty acid content

Cuir — Essais chimiques — Dosage des matières solubles dans le dichlorométhane et des acides gras libres

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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 4048/IUC 4 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, in collaboration with the Chemical Tests Commission of the International Union of Leather Technologists and Chemists Societies (IUC Commission, IULTCS), in accordance with the Agreement on technical co-operation between ISO and CEN (Vienna Agreement). It is based on IUC 4 originally published in *J. Soc. Leather Trades Chemists*, **49**, p. 10, 1965, and declared an official method of the IULTCS in 1965.

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 sampling and the testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

This second edition cancels and replaces the first edition (ISO 4048:1977), which has been technically revised.

Leather — Chemical tests — Determination of matter soluble in dichloromethane and free fatty acid content

1 Scope

This International Standard specifies a method for the determination of the substances in leather which are soluble in dichloromethane. This method is applicable to all types of leather.

Not all fatty and similar substances can be extracted from leather with organic solvents; they may be in part soluble and partly bound to the leather. On the other hand, the solvent can dissolve non-fatty substances, e.g. sulfur and impregnants, both of which cause difficulty in the determination of the acid value and saponification value of the fat.

This International Standard includes two techniques for extraction of the fatty substances: 1) extraction using the Soxhlet apparatus; and 2) extraction using a pressurized extraction system.

As the extraction is frequently done in conjunction with determination of the free fatty acid content of the leather, a suitable procedure for determination of the free fatty acids extracted by this method is included.

The apparatus and technique described in this method are also suitable for the extraction by solvents other than dichloromethane (although the temperature conditions may need to be varied for high pressure extraction).

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

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

ISO 4098, *Leather — Chemical tests — Determination of water-soluble matter, water-soluble inorganic matter and water-soluble organic matter*

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

3 Terms and definitions

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

3.1
extractable substances
fats and other soluble matter which can be extracted from leather with dichloromethane

3.2
free fatty acid content
fatty acid content of the extractable substances as determined by this method and expressed as oleic acid

4 Principle

The prepared leather is extracted with dichloromethane. The solvent is evaporated from the extract which is then dried at 102 °C. Subsequent analysis can then be performed on the resulting extract to determine the free fatty acid content of the leather.

5 Reagents

During analysis, use only reagents of a recognized analytical grade.

5.1 Determination of substances soluble in dichloromethane.

5.1.1 Dichloromethane, boiling point 38 °C to 40 °C, freshly distilled and kept in a dark flask over calcium oxide. Dichloromethane that has been allowed to stand for a long time should be tested as follows for the presence of any hydrochloric acid which may have formed.

— Shake 10 ml of dichloromethane with 1 ml of 0,1 mol/l silver nitrate solution.

— If the silver nitrate solution becomes turbid, the dichloromethane should be redistilled and kept in a dark flask over calcium oxide.

WARNING — Dichloromethane has toxic properties and should be used with caution. The supplier's handling instructions should be followed.

5.2 Determination of free fatty acid content of the leather.

5.2.1 Mixed solvent, a mixture of equal volumes of diethyl ether and 95 % (volume fraction) ethanol, neutralized with 0,1 mol/l sodium hydroxide (phenolphthalein is used as the indicator).

If, for any purpose, other solvents are used, the solvent or solvents used shall be stated in the test report.

5.2.2 Sodium hydroxide, 0,1 mol/l standard solution.

5.2.3 Phenolphthalein indicator solution, 10 g/l prepared in 95 % (volume fraction) ethanol.

In determinations on dark coloured solutions, observation of the end point of the titration may be facilitated either by the substitution of thymolphthalein or alkali blue 6B for phenolphthalein. Alternatively, 1 ml of a 0,1 % solution of methylene blue can be added to each 100 ml of the phenolphthalein solution before titration. Phenolphthalein should be used as the preferred option if possible.

NOTE Dichloromethane which has been used for this analysis can be recovered and reused after distillation.

6 Apparatus

6.1 Soxhlet extraction apparatus, including an extraction flask of suitable capacity and a condenser, or **pressurized extraction system**, including extraction flask of suitable capacity.

6.2 Filter paper thimbles, of suitable sizes, or suitable **glass filter bells**.

6.3 Oven, capable of being maintained at (102 ± 2) °C.

6.4 Analytical balance, capable of weighing to an accuracy of 0,1 mg.

6.5 Desiccator, suitable for cooling the extraction vessels.

6.6 Glass wool or **cotton wad**.

If a cotton wad is used, this shall be pre-extracted in dichloromethane (5.1.1).

7 Sampling and preparation of the samples

If possible, sample in accordance with ISO 2418. If sampling in accordance to ISO 2418 is not possible, then details about sampling shall be given in the test report. Grind the leather in accordance with ISO 4044.

Samples shall be analysed in duplicate.

8 Procedure

8.1 General

Weigh accurately using the analytical balance (6.4) (10 ± 0,1) g of the prepared sample and press evenly into the filter paper thimble or glass bell (6.2). Cover the leather with a thin layer of glass wool or cotton wad (6.6).

Dry the extraction flask (see 6.1) with four glass beads in it by heating for 30 min at (102 ± 2) °C. Weigh after cooling in a desiccator (6.5).

NOTE If determination of free fatty acids is not being carried out, boiling chips may be used as an alternative to glass beads.

Two techniques for extraction of the fatty substances are described: 1) extraction using the Soxhlet apparatus (see 8.2); and 2) extraction using a pressurized extraction system (see 8.3). In the case of dispute, the Soxhlet extraction shall be used.

8.2 Extraction using the Soxhlet apparatus

8.2.1 Place the sample prepared in 8.1 into the extraction apparatus and begin continuous extraction with the dichloromethane (see 8.2.2); then after at least 30 changes of solvent, distil the dichloromethane from the flask containing the extract (see 8.2.3).

Dry the extract for 4 h in the oven (6.3), maintained at (102 ± 2) °C (if drops of water are visible before drying, add 1 ml to 2 ml of ethanol). Weigh after cooling for 30 min in a desiccator.

Repeat the drying, cooling and weighing operations, but with drying periods of 1 h, until either the further loss in mass does not exceed 0,01 g, or the total drying time equals 8 h (see 8.2.3).

8.2.2 Dichloromethane (5.1.1) can also dissolve non-fatty materials from the leather, for example sulfur. (The presence of sulfur is recognizable by a yellow precipitate in the flask.) As sulfur causes difficulty, it can be removed in the following way.

— Dissolve the extract in the smallest possible quantity of diethyl ether and filter through a little cotton wad (6.6) into a previously weighed flask.

— After thoroughly washing out the cotton wad filter with ether, remove the ether from the extract in the flask by distillation over a hot water bath from which any flame has previously been removed.

— If the sulfur should precipitate again, repeat the procedure.

— After the diethyl ether has been distilled off, dry the flask and residue and weigh.

8.2.3 The extract may be used for analysis, for example to determine acid and saponification values of the fats, or to determine the free fatty acid content of the leather.

NOTE After removal of the solvent, the extracted leather can be used for determination of water soluble substances in accordance with ISO 4098.

8.3 Extraction using a pressurized extraction system

Place the sample prepared in 8.1 above in the extraction apparatus, and fill with dichloromethane as appropriate. The sample should be boiled at 180 °C for 40 min. Following this, sufficient dichloromethane should be distilled from the flask containing the extract to ensure that the extraction thimble is clear of the solvent. Extraction should then continue for a further 40 min with the distilled dichloromethane percolating through the leather sample and collecting in the extraction vessel below. Finally, the remainder of the dichloromethane should be distilled from the flask containing the extract (see 8.2.2 and 8.2.3).

Dry the extract for 4 h in the oven (6.3), maintained at (102 ± 2) °C (if drops of water are visible before drying, add 1 ml to 2 ml of ethanol). Weigh after cooling for 30 min in a desiccator.

Repeat the drying, cooling and weighing operations, but with drying periods of 1 h until either the further loss in mass does not exceed 0,01 g, or the total drying time equals 8 h (see 8.2.3).

8.4 Determination of free fatty acid content

To the flask containing the weighed extract obtained from 8.2 or 8.3, add 40 ml of mixed solvent (5.2.1). Add 0,5 ml of phenolphthalein indicator solution (5.2.3) to the flask.

Dissolve the extract completely by rotating the flask, warming if necessary. Cool, and rapidly titrate the solution with 0,1 mol/l sodium hydroxide solution (5.2.2), shaking vigorously during the titration, until a slight but definite colour change, persisting for 15 s, shows that the end point has been reached.

9 Expression of results

9.1 Calculation

The matter extractable in dichloromethane is given, as a percentage by mass on dry matter, by the formula:

$$\frac{m_1}{m_0} \times 100 \times F$$

where

m_0 is the mass, in grams, of the test sample;

m_1 is the mass, in grams, of the extract;

and

$$F = \frac{100}{100 - w}$$

where w is the mass fraction of the volatile matter (based on ISO 4684), in percent.

The free fatty acid content, expressed as oleic acid, $C_{18}H_{34}O_2$, is given, as a percentage by mass, by the formula:

$$\frac{V}{m_0} \times 2{,}82 \times F$$

where

V is the volume, in millilitres, of the 0,1 mol/l sodium hydroxide solution used in the titration;

m_0 is the mass, in grams, of the test sample;

and 1 ml of 0,1 mol/l sodium hydroxide (5.2.2) will titrate 0,028 2 g of oleic acid.

9.2 Repeatability

The results of duplicate determinations carried out by the same operator in the same laboratory should not differ by more than 0,2 %, calculated on the original mass of the leather.

9.3 Reproducibility

The results of two determinations carried out by different operators in different laboratories on the same sample should not differ by more than 0,5 %, calculated on the original mass of the leather.

10 Test report

The test report shall include the following details:

a) reference to this International Standard (ISO 4048);

b) complete identification of the sample;

c) the characteristics of the solvent(s) used;

d) the mean value of the results obtained, to one decimal place;

e) details of any deviations from the test procedure or circumstances that may have affected the results;

f) details of the extraction procedure used.