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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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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html

This document was prepared by the Chemical Tests 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 co-operation between ISO and CEN (Vienna Agreement).

It is based on IUC 4 published in *J. Soc. Leather Tech. Chem.*, **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 the sampling and the testing of leather. ISO recognizes IULTCS as an international standardizing body for the preparation of test methods for leather.

This third edition cancels and replaces the second edition (ISO 4048:2008), which has been technically revised as follows:

- [5.1](#) text for dichloromethane has been simplified;
- [Clause 7](#) has been revised to allow analysis of a single sample, for example when too little sample is available;
- [8.1](#) has been revised to allow a smaller sample for determining only dichloromethane soluble substances;
- [8.2.1](#) and [8.3](#) have been revised to define a drying time of at least 6 h, and the last paragraph of [8.3](#) has been deleted;
- previous [subclauses 9.1](#) and [9.2](#) have been moved to a new informative [Annex B](#);
- [Clause 9](#) has been separated into [9.1](#) and [9.2](#) and modified to allow for presenting results for a single sample;

- [Clause 10 d\)](#) has been revised to allow for the changes in [Clause 9](#).

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

1 Scope

This document 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, for example sulfur and impregnants, both of which cause difficulty in the determination of the acid value and saponification value of the fat.

This document 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 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.

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

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

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.

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

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

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* (3.1) 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, CAS No. 75-09-2.

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

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 reason, 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.

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, or any other validated equipment giving equivalent results to Soxhlet can be used.

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, or something equivalent.

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

7 Sampling and preparation of the samples

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 should be analysed in duplicate, if it is possible.

8 Procedure

8.1 General

Weigh accurately using the analytical balance (6.4) ($10 \pm 0,1$) g or ($5 \pm 0,1$) g (if only soluble matter is determined) of the prepared leather 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, or equivalent (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).

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 cases of dispute, the Soxhlet extraction shall be used. A comparison of results obtained using both techniques is shown in Annex A and Annex B.

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). After at least 30 changes of solvent, distil the dichloromethane from the flask containing the extract (see 8.2.3).

Dry the extract for at least 6 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.

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.

After removal of the solvent, the extracted leather can be used for determination of water soluble substances in accordance with ISO 4098[1].

8.3 Extraction using a pressurized extraction system

Place the sample prepared in 8.1 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 at least 6 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.

8.4 Determination of free fatty acid content

Add 40 ml of mixed solvent (5.2.1) to the flask containing the weighed extract obtained from 8.2 or 8.3. 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 of the matter extractable in dichloromethane

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 (in accordance with ISO 4684), in percent.

The result of the matter extractable in dichloromethane is either:

— the mean value of the two individual determinations;

or

— the value for a single sample.

The figures shall be given to one decimal place.

9.2 Calculation of the free fatty acid content

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;

F see [9.1](#).

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

The result of the free fatty acid content is either:

— the mean value of the two individual determinations;

or

— the value for a single sample.

The figures shall be given to one decimal place.

10 Test report

The test report shall include the following details:

- a) reference to this document (i.e. ISO 4048);
- b) complete identification of the sample;
- c) the characteristics of the solvent(s) used;
- d) the matter extractable in dichloromethane and/or the free fatty acid content as measured in [9.1](#) and [9.2](#);
- e) details of any deviations from the test procedure or circumstances that may have affected the results;
- f) details of the extraction procedure used.

Annex A (informative)

Comparison of five leather samples

A comparison has been carried out of five leather samples to determine if differences are observed between the pressurized extraction system and standard Soxhlet extraction.

Results are illustrated in [Table A.1](#) and are an average of triplicate analysis.

Table A.1 — Comparison data on five different leather samples

Sample	Soxhlet		Pressurized solvent extraction	
	Grease removed %	Standard deviation	Grease removed %	Standard deviation
Tan	5,4	0,2	5,3	0,2
Beige	3,3	0,1	3,2	0,3
Light blue	10,0	0,2	9,4	0,1
Dark blue	12,3	0,2	12,0	0,2
Black	9,6	0,1	9,1	0,2