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Leather — Physical and mechanical tests — Determination of surface coating thickness

Cuir — Essais physiques et mécaniques — Détermination de l'épaisseur du revêtement de surface

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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 17186 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 289, *Leather*, in collaboration with the Physical Test Commission of the International Union of Leather Technologists and Chemists Societies (IUP Commission, IULTCS), 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 cancels and replaces the first edition (ISO 17186:2002), which has been technically revised, particularly in Clause 6 with the addition of alternative methods of measurement and analysis.

Leather — Physical and mechanical tests — Determination of surface coating thickness

1 Scope

This International Standard specifies a method for determining the thickness of the surface coating applied to leather when measured under zero compression. It is applicable to all types of 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 2418, *Leather — Chemical, physical and mechanical and fastness tests — Sampling location*

ISO 2419, *Leather — Physical and mechanical tests — Sample preparation and conditioning*

3 Principle

A section of leather is taken perpendicular to the coated surface. The thickness of the surface coating is measured using a microscope and expressed both as a thickness and as a percentage of the total thickness.

4 Apparatus

4.1 Light microscope or scanning electron microscope, which can be operated with an object-field size of 1 mm × 1 mm, or lower, and is

— fitted with a camera-image unit connected to a computer (resolution of at least 500 × 500 pixels), or

— fitted with a photographical unit, or

— equipped with an eyepiece with a graduated scale (at least 100 scale marks).

NOTE 100 scale marks correspond to a scaling of 10 µm in the object-field dimension for an object-field size of 1 mm × 1 mm.

For measurement of coating thicknesses of less than 50 µm, an object-field size of 0,4 mm × 0,4 mm or lower has to be used. For measurement of values of less than 15 µm, a scanning electron microscope with a suitable object-field dimension should be used.

4.2 Razor blade.

4.3 Grid or **similar calibration device**, reading to at least 10 µm and suitable for use in the light microscope or scanning electron microscope.

4.4 Coating unit, using sputter or evaporation, including a suitable element or alloy (e.g. gold) for coating, if a scanning electron microscope is used.

4.5 Specimen stubs, suitable for a scanning electron microscope.

5 Sampling and sample preparation

5.1 Sample in accordance with ISO 2418. Condition in accordance with ISO 2419.

5.2 Cut three test pieces of approximately 10 mm × 10 mm. If there is a requirement for more than two hides or skins to be tested in one batch, then only one test piece need be taken from each hide or skin, provided that the overall total is not less than three pieces. Section each of the three test pieces as described in 5.3.

5.3 Place the cutting edge of the razor blade (4.2) on the flesh side, with the blade perpendicular to the leather surface, and press the blade through the whole thickness of the leather, ensuring that the blade remains vertical. It is recommended that a new razor blade be used for each section.

If using a scanning electron microscope, stick a section prepared as described above onto a specimen stub (4.5) with the cut surface uppermost. Coat the stub and section in the coating unit (4.4) so that a sufficient image quality can be obtained.

6 Procedure

6.1 General descriptions

6.1.1 Principles of measurement

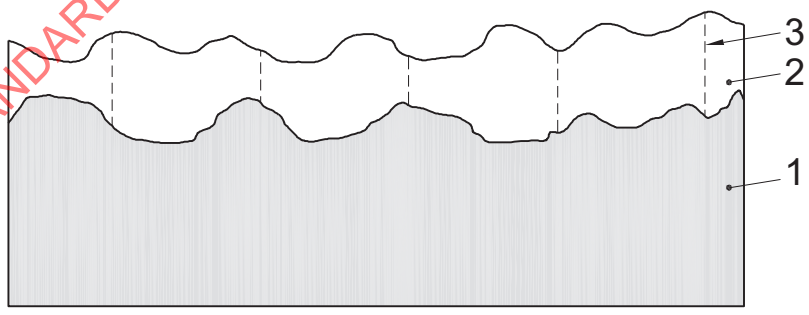
This International Standard describes two basic principles to measure the surface coating thickness.

Method A is based on the measurement of a number of values on equidistant positions (see Figure 1). It enables the determination of a standard deviation as well as a confidence interval.

Method B is based on a measurement using parallel lines (see Figure 2). A statistical evaluation of the results is not possible due to the fact that the operator has to position the graduation of the grid between peaks and troughs manually. This process already includes a calculation of the average.

Method A should be preferred.

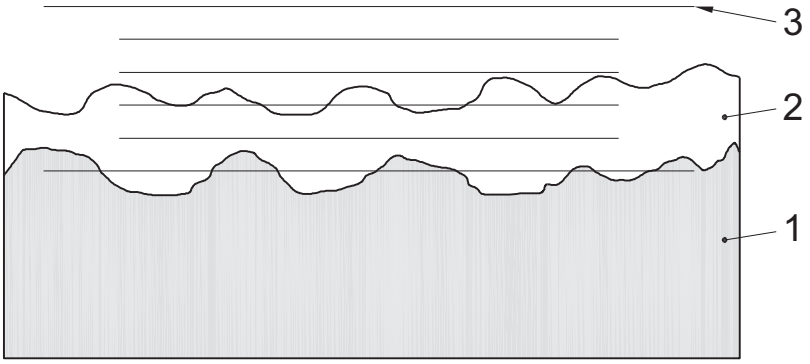
Methods A and B can be used for light microscopes as well as for scanning electron microscopes. The working distance and the acceleration voltage of the scanning electron microscope shall be the same for calibration and measurement of the sample.



Key

1 leather
2 coating
3 equidistant single measurement

Figure 1 — Principle of method A



Key

1 leather
2 coating
3 graduated scale or parallel lines

Figure 2 — Principle of method B

6.1.2 Magnification and calibration

Due to the variation in the typical thickness of surface coating (e.g. 10 µm to 200 µm) and the total thickness of leather (e.g. 800 µm to 3 000 µm), it is usually necessary to use different magnifications of the microscope.

For each case, select a suitable magnification, which allows a reliable measurement of the thickness.

Calibrate the measurement system following the detailed instructions in 6.2 and 6.3 for each magnification used.

6.2 Method A — Based on the measurement of single values on equidistant positions

6.2.1 Measurement using measuring marks of a suitable computer program as analysis unit of the microscope

6.2.1.1 Calibrate the measuring system of the microscope (4.1) using the grid (4.3). Follow the instructions in the manual for the system used.

6.2.1.2 Place one section prepared in 5.3 under the microscope. Start the measurement at a random point of the cross-section and adjust this point in the middle of the field of vision. According to the software of the measuring system, set the starting point of the measurement in the coating/leather boundary.

6.2.1.3 Then set the measuring point on the surface of the leather, at which the adjustment has to be executed, perpendicular to the run of the coating boundary (see Figure 1). Note the indicated distance.

6.2.1.4 Move the sample parallel to the surface by moving the microscope table over a defined distance of one field of vision or a fix offset (i.e. 50 µm to 500 µm) so that a new measuring point is located in the middle of the field of vision. The distance between two measurements shall be larger than the coating thickness. Measure the coating thickness at this point as described in 6.2.1.3.

6.2.1.5 Continue the measurement in the manner described in 6.2.1.2 to 6.2.1.4, until at least six measurements have been carried out.

6.2.1.6 Convert all readings obtained to micrometres using the calibration described in 6.2.1.1.

6.2.1.7 Measure the total thickness using the procedure described in 6.2.1.2 to 6.2.1.6, aligning the measurement point to the flesh side of the leather.

6.2.1.8 Repeat 6.2.1.2 to 6.2.1.7 for the two remaining sections.

6.2.1.9 Calculate the arithmetic mean of all single values of thickness and total thickness. Express the coating thickness to the nearest 5 µm for a coating thickness of 50 µm or higher, and to the nearest 2 µm for a coating thickness less than 50 µm. Express the total thickness to the nearest 10 µm.

If required, calculate the standard deviation as follows:

$$s = \sqrt{\frac{\sum (t_i - t)^2}{n - 1}}$$

where

s is the standard deviation;

t_i is the single value of the coating thickness;

t is the mean of the coating thickness;

n is the number of single measurements.

6.2.2 Measurement using a light microscope fitted with a graduated scale

6.2.2.1 Calibrate the microscope with the graduated scale (4.1) using the grid (4.3).

6.2.2.2 Place one section prepared in 5.3 under the microscope. Start the measurement at a random point of the cross-section and adjust this point in the middle of the field of vision. Position the section such that the crosswire or one of the major graduations is aligned with the coating/leather boundary exactly at this point of the cross-section.

Take the reading of the scale graduation corresponding to the outer surface of the coating at the measuring point.

6.2.2.3 Move the sample parallel to the surface by moving the microscope table over a defined distance on one field of vision or a fix offset (i.e. 50 µm to 500 µm) so that a new measuring point is located in the middle of the field of vision. The distance between two measurements shall be larger than the coating thickness. Measure the coating thickness at this point as described in 6.2.2.2.

6.2.2.4 Continue the measurement in the manner described in 6.2.2.2 and 6.2.2.3, until at least six measurements have been carried out.

6.2.2.5 Convert all the readings obtained to micrometres using the calibration described in 6.2.2.1.

6.2.2.6 Measure the total thickness using the procedure described in 6.2.2.2 to 6.2.2.5, aligning the graduated scale to the flesh side of the leather.

6.2.2.7 Repeat 6.2.2.2 to 6.2.2.6 for the two remaining sections.

6.2.2.8 Calculate the arithmetic mean of all single values of coating thickness and total thickness. Express the coating thickness to the nearest 5 µm for a coating thickness of 50 µm or higher, and to the nearest 2 µm for a coating thickness less than 50 µm. Express the total thickness to the nearest 10 µm.

If required, calculate the standard deviation as described in 6.2.1.9.

6.3 Method B — Based on a measurement using parallel lines

6.3.1 Measurement using measuring lines of a suitable computer program as analysis unit of the microscope

6.3.1.1 Calibrate the measuring system of the microscope (4.1) using the grid (4.3). Follow the instructions in the manual for the system used.

6.3.1.2 Place one section prepared in 5.3 under the microscope. Position the section such that the measuring line of the computer program is aligned with the coating/leather boundary. If the coating/leather boundary undulates, position the measuring line of the computer program midway between the peaks and troughs, as shown in Figure 2. Following the function of the computer-based program, realign the measuring line in the same way to the outer surface of the coating and note the indicated distance.

6.3.1.3 Measure the total thickness at the same point by positioning the section such that the measuring line of the computer program is aligned with the flesh side of the leather. Then align the measuring line to the outer surface of the coating and note the indicated distance.

6.3.1.4 Convert the readings obtained in accordance with 6.3.1.2 to micrometres using the calibration described in 6.3.1.1.

6.3.1.5 Repeat 6.3.1.2 to 6.3.1.4 for the two remaining sections.

6.3.1.6 Calculate the arithmetic mean of the three coating thicknesses and the three total thicknesses. Express the coating thickness to the nearest 5 µm for a coating thickness of 50 µm or higher, and to the nearest 2 µm for a coating thickness less than 50 µm. Express the total thickness to the nearest 10 µm.

6.3.2 Measurement using a light microscope fitted with a graduated scale

6.3.2.1 Calibrate the microscope with the graduated scale (4.1) using the grid (4.3).

6.3.2.2 Place one section prepared in 5.3 under the microscope. Position the section such that the crosswire or one of the major graduations is aligned with the coating/leather boundary. If the coating/leather boundary undulates, position the crosswire or major graduation midway between the peaks and troughs, as shown in Figure 2.

Take the reading of the scale graduation corresponding to the outer surface of the coating.

6.3.2.3 Measure the total thickness at the same point by positioning the section such that the crosswire or one of the major graduations is aligned with the flesh side of the leather. Take the reading of the scale graduation corresponding to the outer surface of the coating.

6.3.2.4 Convert the readings obtained in accordance with 6.3.2.2 and 6.3.2.3 to micrometres using the calibration described in 6.3.2.1.

6.3.2.5 Repeat 6.3.2.2 to 6.3.2.4 for the two remaining sections.

6.3.2.6 Calculate the arithmetic mean of the three coating thicknesses and the three total thicknesses. Express the coating thickness to the nearest 5 µm for a coating thickness of 50 µm or higher, and to the nearest 2 µm for a coating thickness less than 50 µm. Express the total thickness to the nearest 10 µm.

6.3.3 Measurement using a microscope fitted with a means of obtaining photographs

6.3.3.1 Place the grid (4.3) under the microscope, photograph it and obtain a print.

6.3.3.2 Place one section prepared in 5.3 under the microscope, photograph it at the same magnification as used in 6.3.3.1 and obtain a print.