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Methods of

Sampling and chemical testing of leather

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- British Leather Federation*
- British Leather Manufacturers' Research Association
- Department of Trade and Industry
- Ministry of Defence, Army Department*
- Ministry of Defence, Navy Department*
- Shoe and Allied Trades Research Association*
- Society of Leather Technologists and Chemists*

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Industrial Leathers Federation

This British Standard, having been approved by the Leather Industry Standards Committee, was published under the authority of the Executive Board on 12 March 1974

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The following BSI references relate to the work on this standard:
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Foreword

The 1959 edition of BS 1309 included methods derived from two sources, first, the Official Methods of the Society of Leather Trades' Chemists¹⁾ (SLTC), and secondly, work done by members of the Leather Panel of the Commonwealth Joint Services Committee. Since the publication of that edition the SLTC has adopted the methods of the International Union of Leather Technologists' and Chemists' Societies (IULTCS) and the present revision includes methods based upon those of the IULTCS. No IULTCS methods are available for the determinations of glucose and silicone and the corresponding methods given in this standard are based on those of the SLTC.

In comparison with the 1959 edition, a method for the determination of silicone has been added. Also, to the method included in this revision for the determination of substances (fats and other solubles) soluble in dichloromethane, has been added a suitable procedure for the determination of the free fatty acids present in the extract. One method, the determination of *p*-nitrophenol, has been omitted in this revision, since no method is currently adopted by the SLTC. On the adoption of a method by the SLTC, it is intended to include it in this standard. Similarly, the SLTC has not yet adopted a method for determination of zirconium. In the event of adoption by the SLTC of the IULTCS method, it is intended to include it in this standard.

This revision, in contrast to the 1959 edition, but in alignment with the current SLTC Methods, does not include a method for the determination of total ash (i.e. ash which is not sulphated). The current SLTC Methods define "total ash" to mean ash which has been sulphated. To avoid confusion, not least with the previous edition of this standard, it has been thought better not to follow the SLTC Methods in this matter of definition. Accordingly, the method described in this standard for determination of sulphated total ash is described as such.

This British Standard is not concerned with the physical testing of leather which is dealt with in BS 3144, "*Methods of sampling and physical testing of leather*".

Throughout this standard the units used are those of the International System of units (SI), or related units.

Clauses 2, 4, 6, 9 and 11 of this British Standard have, respectively, been superseded by the following British Standards: BS EN ISO 4044, BS EN ISO 4048, BS EN ISO 4047, BS EN ISO 4045 and BS EN ISO 5399.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 30, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

¹⁾ Now the Society of Leather Technologists and Chemists.

0 General instructions and recommendations

0.1 Scope

This British Standard describes methods for sampling from pieces of leather and for chemical testing of leather samples.

NOTE The titles of the British Standards referred to in this standard are listed on the inside back cover.

0.2 Sampling

Each of the analytical methods described in this standard is applicable to any piece of leather of sufficient mass irrespective of where in the hide or skin the leather originated. Although “1. Method of sampling” describes a procedure for taking samples from whole pieces of leather, in some cases the sample presented to the analyst is taken from other locations, or is of unknown origin and has an imperfectly recorded history. In practice, for example, analyses are required on manufactured leather goods or components. It is necessary when interpreting analytical results, or selecting specification limits, to take into account this factor and, where necessary, to make due allowance for any likely variation in properties over the extent of the original hide or skin.

0.3 Reagents

Unless otherwise stated, all reagents used should be of recognized reagent quality, and, except where stated otherwise, distilled or demineralized water, complying with BS 3978, should be used wherever water is required.

0.4 Duplication of tests

In some circumstances duplicate tests may be required. Agreement between duplicate results should be within the range stated under “Repeatability” for the corresponding method.

0.5 Expression of analytical results

A report of analysis should include the values for volatile matter and grease content of the sample, as determined by Methods 3 and 4 respectively.

Calculation of the equivalent values on the sample free from grease and volatile matter can, if desired, be made thus:

$$T_{(0,0)} = \frac{100 T_{(x,y)}}{100 - (x + y)}$$

where $T_{(0,0)}$ is the equivalent result on sample free from grease and volatile matter;

$T_{(x,y)}$ is the analytical result on sample with x % grease and y % volatile matter;

x is the percentage grease in the analysed sample, as determined by Method 4;

and y is the percentage of volatile matter in the analysed sample, as determined by Method 3.

1 Sampling

1.1 Introduction

This method is based on Method SLC.1 of the Society of Leather Technologists and Chemists and on Method IUC/2 of the International Union of Leather Technologists' and Chemists' Societies. It is applicable to skins and hides of either light or heavy leathers.

In regard to the location of pieces for chemical tests, this method is in agreement with International Standard ISO 2418, “Leather — Laboratory samples — Location and identification”, which is also based on Method IUC/2.

1.2 Location of pieces for chemical tests

1.2.1 General. Pieces selected for sampling shall be free from major defects, but slight defects, whether on the grain or the flesh side, are of no significance.

The sampling procedures described for skins, hides and sides (**1.2.2**), for bends and butts (**1.2.3**), for shoulders (**1.2.4**) and for bellies (**1.2.5**) are necessarily designed to allow concurrent physical and chemical testing, but the requirements for physical testing in accordance with BS 3144 shall always be given priority. In cases of dispute or arbitration, sampling for chemical testing shall be strictly as shown in Figure 1, Figure 2, Figure 3 and Figure 4 and as described in **1.2.2**, **1.2.3**, **1.2.4** and **1.2.5**, except that with very light leathers additional leather may be taken (if necessary to make up the mass required) from immediately adjacent to the prescribed areas (see Note 1). In other cases, when necessary to make up the mass required, the section to be taken for chemical tests may be enlarged or slightly displaced (see Notes 1 and 2).

In all cases where leather has been taken for chemical tests from positions other than those described in **1.2.2**, **1.2.3**, **1.2.4** and **1.2.5**, a note of this fact shall be made in the test report. Furthermore, in cases of dispute or arbitration, a sketch shall be included to demonstrate the position from which extra leather was taken.

NOTE 1 The following minimum mass of material is necessary for a chemical analysis of leather:

for vegetable and combination tanned leathers	100 g,
for mineral tanned leathers	60 g.

NOTE 2 Uncontaminated trimmings from the samples taken for physical tests may be used for the chemical analysis except in cases of dispute or arbitration, but if this is done, the fact shall be noted in the report.

1.2.2 Whole skins, sides and hides. Figure 1 represents a skin or hide with the head removed. B is the root of the tail. A is a point on the backbone such that $AC = 2AB$. AD is a line perpendicular to BC. F is the mid-point of AD, and AE is of length 50 mm. The lines GH and JK, whose mid-points are E and F respectively, are parallel to BC, and each is of length equal to EF. $2 HL = HK$.

Cut the pieces for chemical tests from the square HLMN (shaded in Figure 1). If necessary, cut material also from the corresponding place in the opposite half of the skin.

1.2.3 Bends (or butts). Figure 2 represents a bend. B is the root of the tail, and A is a point on the backbone BC equidistant from B and C. AD is a line perpendicular to BC. F is the mid-point of AD, and AE is of length 50 mm. The lines GH and JK, whose mid-points are E and F respectively, are parallel to BC, and each is of length equal to EF. $2 HL = HK$.

Cut the pieces for chemical tests from the square HLMN (shaded in Figure 2).

1.2.4 Shoulders. Figure 3 represents a side before the removal of belly and shoulder. P (Figure 3 and Figure 4) is the mid-point of RS. DC (Figure 3) is a line parallel to, and 20 mm distant from, RS, and PCB is a line through P parallel to the backbone. DA is a line parallel to, and 50 mm distant from, the backbone, and of length equal to half the length of DC. AB is parallel to DC. $2 AE = AB$.

Cut the pieces for chemical tests from the rectangle AEFG (shaded in Figure 3).

1.2.5 Bellies. Q (Figure 4) is the mid-point of the line TU. Cut two square pieces, each with side length of approximately 10 cm, from locations as close to point Q as possible, but avoiding inclusion of leather within 20 mm of line TU if this line existed as a cut edge prior to sampling. Arrange the area sampled for chemical tests (shaded squares VWXY in Figure 4) to be adjacent to and each side of the area reserved for physical tests.

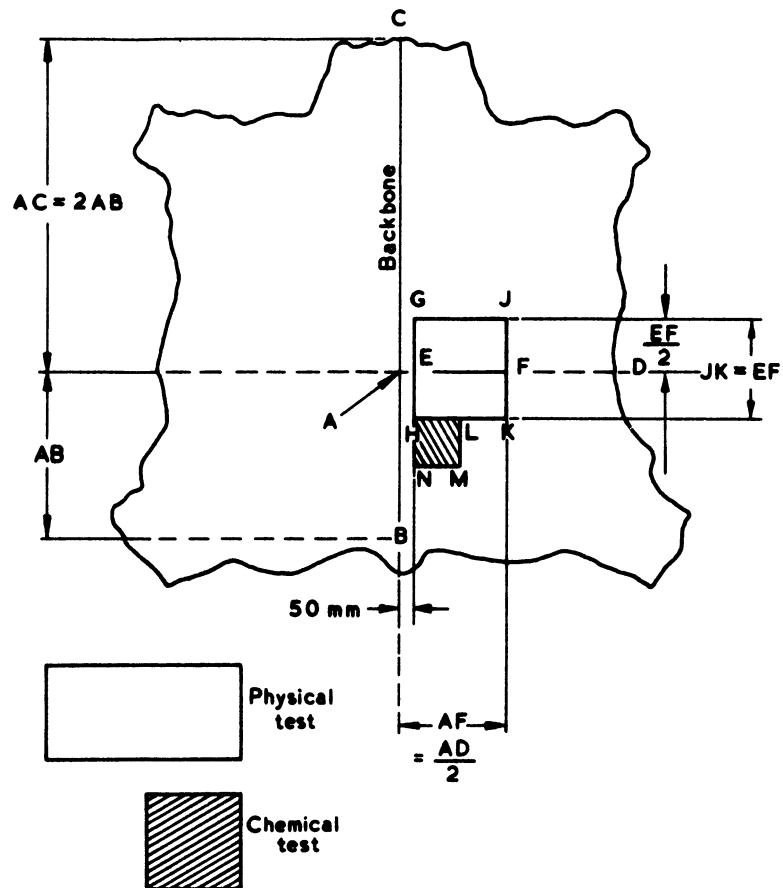


Figure 1 — Sampling location for whole skins, sides, and hides

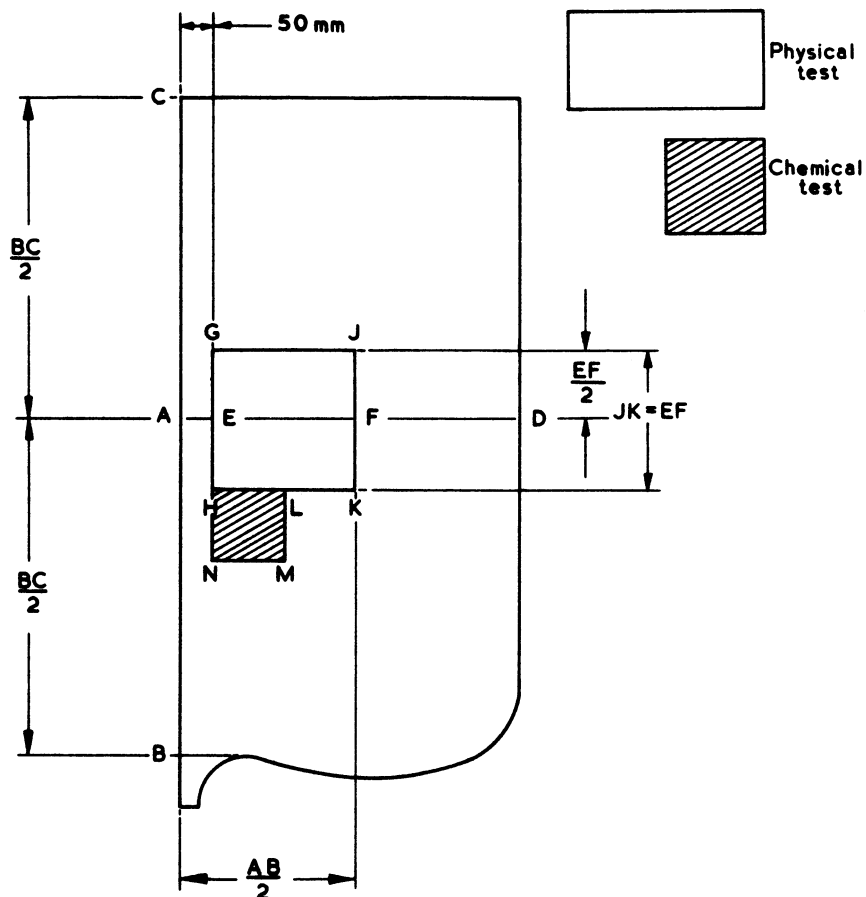


Figure 2 — Sampling location for bends (or butts)

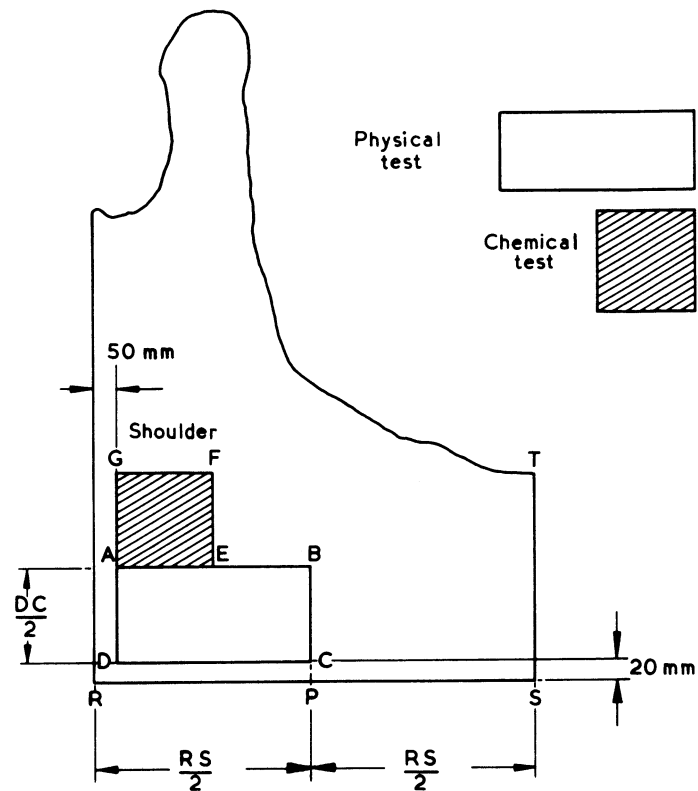


Figure 3 — Sampling location for shoulders

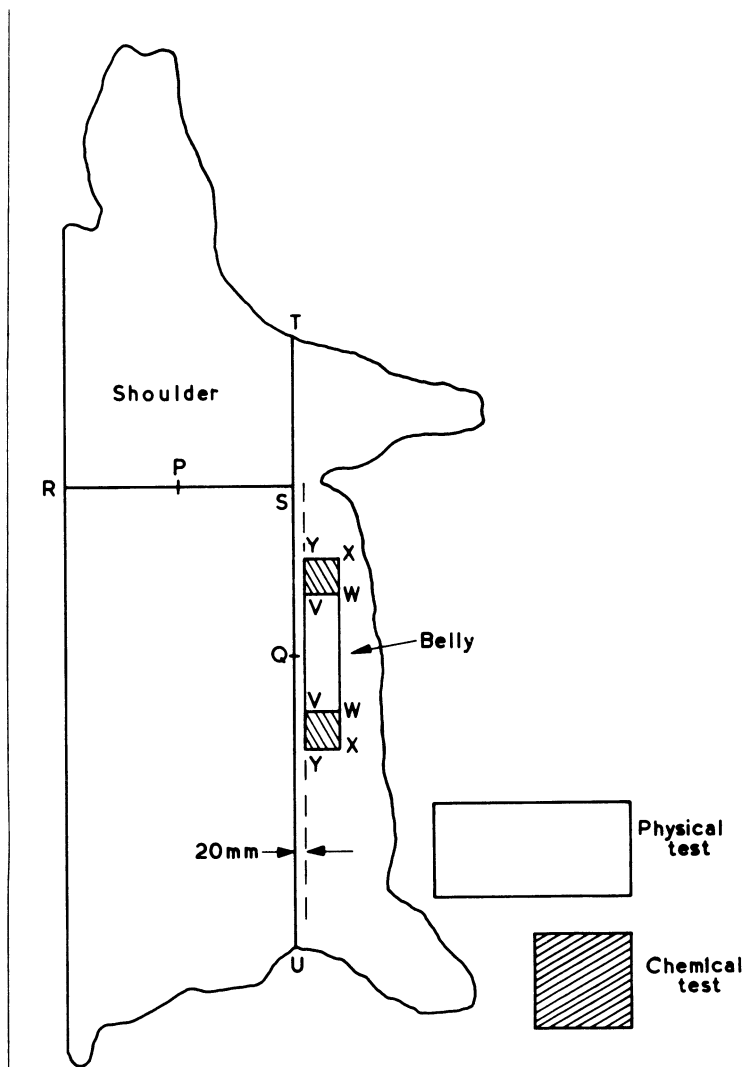


Figure 4 — Sampling locations for bellies

1.3 Number of pieces to be sampled and analysed

Sample three pieces (skins, hides, sides, butts, bends, shoulders, or bellies) and carry out at least a duplicate analysis on an average sample. If for any reason this procedure cannot be followed the relevant details shall be noted in the test report.

1.4 Sample report

The report shall include:

- a. a reference to the method for sampling, i.e. "Method 1 of BS 1309";
- b. details of any deviation from the specified general procedure, in particular, with respect to:
 - 1) inclusion of leather from positions other than those specified in 1.2.2, 1.2.3, 1.2.4, or 1.2.5;
 - 2) inclusion of trimmings from samples taken for physical tests;
 - 3) the number of pieces sampled, where this is not in accordance with the procedure described in 1.3.
- c. identification details of the sample.

3 Determination of volatile matter

3.1 Introduction

This method is based on Method SLC.3 of the Society of Leather Technologists and Chemists and Method IUC/5 of the International Union of Leather Technologists' and Chemists' Societies. It is applicable to all types of leather.

The exact determination of the moisture content in leather by this method is not possible, as at elevated temperature other volatile substances escape and tannins and fats can be oxidized. Some absorbed water may be left in the leather after drying.

For a rapid indication of the volatile matter in pelts and wet leather, the water can be determined by the distillation method, using a hydrocarbon solvent (e.g. xylene) in a Dean and Stark apparatus (see BS 756), and taking 20 g of leather powder.

3.2 Definition

For the purposes of this method the following definition applies.

Volatile matter. Loss of mass by leather when dried to constant mass at $102 \pm 2^\circ\text{C}$, as described in this method.

3.3 Apparatus

Usual laboratory apparatus is required and, in particular, the following.

a. Flat, shallow weighing vessels with ground glass stoppers, or *flat open dishes*.

NOTE Small weighing vessels with ground glass stoppers allow more accurate work than do open dishes.

b. Oven, capable of being maintained at $102 \pm 2^\circ\text{C}$, complying with the requirements of BS 2648.

3.4 Procedure (see 3.5.1)

Sample in accordance with Method 1. Grind in accordance with Method 2. Weigh, to the nearest 0.001 g, approximately 3 g of the sample into a tared weighing vessel and dry at $102 \pm 2^\circ\text{C}$ for 5 hours. Cool the vessel and contents for 30 min in a desiccator (see 3.5.2) and weigh. Repeat the drying, cooling and weighing but with drying times of 1 hour until either the further loss in mass does not exceed 3 mg (i.e. 0.1 % of the sample mass), or the total drying time equals 8 hours.

Record the final mass of sample and weighing vessel, and calculate the dried mass of sample.

3.5 Notes on the procedure

3.5.1 For leathers which contain large quantities of oxidizable fats further useful information may be gained by carrying out the following procedure.

Determine the fats and other solubles by Method 4. Determine the dry mass of the resultant dichloromethane-extracted leather by drying in accordance with Method 3. Calculate the total loss of mass expressed as a percentage of the mass of the prepared sample taken, and subtract the percentage extractable substances, thus giving the percentage volatile matter.

If this special procedure is used, full details should be included in the test report.

3.5.2 When working with open dishes, not more than one dish should be put into a small desiccator to cool at any one time, and not more than two dishes should be put into a large desiccator.

3.6 Expression of results

Calculate the following percentage.

$$\text{Volatile matter, percentage by mass} = \frac{100 (M_1 - M_2)}{M_1}$$

where M_1 is the mass of sample before drying

and M_2 is the mass of sample after drying.