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Standard Test Method for Hexane Extraction of Leather[1]

This standard is issued under the fixed designation D 3495; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Keywords were added editorially in February 1994.

[1] This test method is under the jurisdiction of ASTM Committee D-31 on Leather and is the direct responsibility of Subcommittee D31.06 on Chemical Analysis—General Methods.

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1. Scope

1.1 This test method covers the quantitative extraction of all types of leather with hexane. This test method does not apply to wet blue.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 2813 Practice for Sampling Leather for Physical and Chemical Tests[2]

D 3790 Test Method for Volatile Matter (Moisture) of Leather by Oven Drying[2]

3. Significance and Use

3.1 This test method measures the amount of hexane-soluble lubricant present in all types of leather. Adequate lubrication prevents abrasion of leather fibers during flexing. This lubrication is generally obtained from the fat liquor added at the tannery. Some lubrication is also obtained from natural grease produced during the life of the animal.

4. Apparatus

4.1 *Analytical Balance*.

4.2 *Soxhlet Apparatus*, consisting of a boiling flask, extraction tube, and condenser.

4.3 *Forced Circulating Air Oven* , capable of maintaining the specified temperature.

4.4 *Electric Hot Plate*.

4.5 *Extraction Thimbles, fat-free* , cellulose, Alundum, or fritted.

4.6 *Absorbent Cotton*, fat-free.

4.7 *Steam Bath*.

5. Reagent

5.1 *Hexane*, ACS Reagent Grade conforming to the following requirements:

5.1.1 *Color (APHA)*—10 max.

5.1.2 *Density (g/mL)* at 25°C—0.687 max.

5.1.3 *Boiling Range*—1 to 95 mL, not more than 4.0°C.

5.1.4 *Residue After Evaporation*—0.001 % max.

5.1.5 *Acidity* (as CH_3COOH) —To pass test (limit 0.002 %).

5.1.6 *Sulfur Compounds* (as S)—0.005 % max.

5.1.7 *Thiophene*—To pass test.

NOTE 1—This reagent grade hexane is generally a mixture of several isomers of hexane (C_6H_{14}), predominantly *n*-hexane and methylcyclopentane (C_6H_{12}).

[2] *Annual Book of ASTM Standards*, Vol 15.04.

6. Test Specimens

6.1 The leather shall be sampled in accordance with Method D 2813. Leather test specimens shall be obtained from the composite sample prepared by random sampling, cutting, and mixing equal portions of leather representing the lot that is being analyzed. The well-mixed leather pieces shall be ground in a mill (Wiley or equal) having a No. 5 (4-mm) sieve. The ground leather that passes through this 4-mm sieve shall be mixed well and used as the composite sample.

7. Procedure

7.1 Determine the moisture content of the composite sample from which the ground leather for hexane extraction is taken in accordance with Test Method D 3790. Determine the weight of the ground leather taken from the composite sample for moisture content at the same time and under the same ambient conditions as the weight of the ground leather taken for hexane extraction.

7.2 Weigh 5 g of ground leather taken from the composite to the nearest 0.001 g and record this value as W_1. Loosely pack this material in an appropriately sized extraction thimble and cover with a pad of fat-free cotton. Place the loaded thimble in the Soxhlet extraction tube. Dry an extraction flask in an oven for 1 h at 100 ± 2°C, cool in a desiccator, and weigh to the nearest 0.001 g. Record this value as W_2. Fill the flask approximately two-thirds full with hexane, assemble the apparatus, circulate the water through the condenser, and heat the flask until the extraction of the leather has continued for a minimum of 50 cycles. If the Soxhlet drips continuously instead of cycling, extract the leather for a minimum of 5 h using maximum heat. At the end of the extraction period, remove the flask containing the extraction solvent and drive off the hexane. When 10 to 20 mL of hexane remain, heat gently on a steam bath until the odor of the solvent can no longer be detected. Facilitate the removal of the hexane by utilizing a vacuum or a gentle stream of filtered (oil- and water-free) air. After the hexane has been removed, dry the flask containing the extracted matter in a forced circulating air oven at 100 ± 2°C, for 2 h, cool to room temperature in a desiccator and weigh. Continue drying for successive 2-h periods until constant weight is obtained. When successive weighings vary by less than ± 0.005 g, consider the weight constant. Record this weight to the nearest 0.001 g as W_3. No more than three successive 2-h drying periods shall be allowed. If constant weight has not been obtained after the third drying, the results of the second drying period shall be repeated.

8. Calculation

8.1 Calculate the percentage of hexane-soluble matter, on a moisture-free basis, as follows:

$$\text{Hexane-soluble matter, \%} = \frac{W_3 - W_2}{W_1 \times \dfrac{(100 - \%\ moisture)}{100}} \times 100 \tag{1}$$

where:

W_1 = original weight of extracted leather,
W_2 = weight of extraction flask, and
W_3 = weight of extraction flask and hexane-soluble matter.

9. Report

9.1 Report the hexane-soluble matter in the leather as the average value obtained from the test results to nearest 0.01 %.

9.2 State that the results are calculated on a moisture-free basis.

10. Precision and Bias

10.1 *Interlaboratory Test Data*[3]—An interlaboratory test was run in 1972 in which each of 7 laboratories tested duplicate specimens from each of 15 different types of leather. All laboratories tested the following leathers: chamois, yellow lace, crust sheepskin, crust goatskin, vegetable-strap leather, chrome-retan side leather, vegetable-skirting leather, vegetable-retan side leather, latigo-lace leather, white-lace leather, suede sheepskin, horsehide (finished), white-side leather, chrome-split leather, and silicone-treated military leather. The components of variance for hexane-soluble matter expressed as standard deviations were calculated to be:

$$\text{Within-laboratory (single operator) component—0.14 percentage points} \tag{2}$$

$$\text{Between-laboratory component—0.41 percentage points} \tag{3}$$

10.2 *Precision*—For the components of variance reported in 9.1, two averages of two observations each of hexane soluble matter should be considered significantly different at the 95 % probability level if the difference equals or exceeds the following critical differences:

$$\text{Within-laboratory (single operator) precision 0.59 percentage points} \tag{4}$$

[3] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting Research Report ~~No.~~ RR: D-31-1003. ~~A copy is available from ASTM Headquarters, 1916 Race St., Philadelphia, PA 19103.~~

Between−laboratory precision 1.41 percentage points (see Note 3) (5)

NOTE 2—The critical differences were calculated using $t = 1.960$, which is based on infinite degrees of freedom.

NOTE 3—The calculated values of the critical differences should be considered to be a general statement particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained on randomized specimens from one sample of the material to be tested. The interlaboratory test showed that for certain leathers containing more than 18 % hexane-soluble matter the between-laboratory critical difference could be as high as 3 percentage points.

10.3 *Bias*—No justifiable statement on bias can be made, since the true value of hexane-soluble matter cannot be established by an accepted reference method.

11. Keywords

11.1 extractables; extraction; fat liquor; hexane—soluble lubricant; lubrication