Designation: D7674 – 14a (Reapproved 2021)

Standard Test Method for Hexane/Petroleum Ether Extract in Wet Blue and Wet White[1]

This standard is issued under the fixed designation D7674; 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.

1. Scope

1.1 This test method covers the quantitative extraction of all types of wet blue and wet white with hexane or petroleum ether.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 7.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3495 Test Method for Hexane Extraction of Leather

D6658 Test Method for Volatile Matter (Moisture) of Wet Blue by Oven Drying

D6659 Practice for Sampling and Preparation of Wet Blue and Wet White for Physical and Chemical Tests

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 A specimen is analyzed as received in wet state, diced; or pre-dried at the determined setting then ground prior to analysis. The prepared specimen is extracted with solvent. Another specimen from the same sample is also analyzed for moisture content in accordance with Test Method D6658. Following completion of the extraction process, the extract is dried, then cooled and weighed. The extract is reported as extractables on a moisture-free basis.

4. Significance and Use

4.1 This test method measures the amount of solvent-soluble (hexane or petroleum ether) materials in wet blue and wet white.

5. Apparatus

5.1 *Analytical Balance.*

5.2 *Extraction Apparatus*—Soxhlet, consisting of a boiling flask, extraction tube, and condenser. Alternate Extraction Apparatus: Soxtec-type system consisting of an extraction unit and a control unit.

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

5.4 *Electric Hot Plate* (or steam bath).

5.5 *Extraction Thimbles,* fat-free: cellulose, Alundum, glass microfiber, or fritted glass.

5.6 *Absorbent Cotton,* fat-free, or glass wool.

6. Reagents and Materials

6.1 *Hexane,* ACS Reagent Grade, or

6.2 *Petroleum Ether,* ACS Reagent Grade.

7. Hazards

7.1 All reagents and chemicals should be handled with care. Before using any chemical, read and follow all safety precautions and instructions on the manufacturers' label or MSDS (Material Safety Data Sheet).

8. Sampling

8.1 The wet blue or wet white shall be sampled in accordance with Test Method D6659.

9. Procedure

NOTE 1—Two sample conditions are listed below. Both sample conditions produce acceptable results (See Precision and Bias section).

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.02 on Wet Blue.

Current edition approved Sept. 1, 2021. Published October 2021. Originally approved in 2010 as E7674-10. Last previous edition approved in 2014 as E7674-14a. DOI: 10.1520/D7674-14AR21.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

9.1 *Condition A*—As received in wet state, diced (prepared per Test Method D6659 Method A).

9.1.1 Weigh out specimen for both moisture and hexane/petroleum ether extraction at the same time. For the hexane/petroleum ether extraction, weigh an 8-10 g specimen to the nearest 0.001g and record this value as W1. Loosely pack the material in an appropriately sized extraction thimble and cover with a pad of fat-free cotton or glass wool. Proceed with extraction using either the Soxhlet apparatus or the Soxtec-type apparatus.

9.1.2 Determine the moisture content of the prepared sample from which the specimen for extraction is taken (9.1.1) in accordance with Test Method D6658.

NOTE 2—The cubed specimen weighed out for extraction may be air-dried overnight, prior to extraction.

9.2 *Condition B*—Oven or air dried, ground (prepared per Test Method D6659 Method B).

9.2.1 Weigh a 4-5 g specimen to the nearest 0.001 g and record this value as W1. Loosely pack the material in an appropriately sized extraction thimble and cover with a pad of fat-free cotton or glass wool. Proceed with extraction using either the Soxhlet apparatus or the Soxtec-type apparatus.

9.2.2 Determine the residual moisture content of the prepared sample from which the specimen for extraction is taken in accordance with Test Method D6658.

9.3 *Soxhlet Apparatus*—Place the loaded thimble in the Soxhlet extraction tube. Dry an extraction flask in an oven for at least 1 h at 100 ± 2 °C (212 ± 3.6 °F) to remove residual moisture. Cool in a desiccator, and weigh to the nearest 0.001 g. Record this value as W2. Fill the flask approximately two-thirds full with hexane or petroleum ether, assemble the apparatus, circulate the water through the condenser, and heat the flask until the extraction of the sample has continued for a minimum of 50 cycles. If the Soxhlet drips continuously instead of cycling, extract the sample for a minimum of 5 h at that setting. At the end of the extraction period, remove the flask containing the extraction solvent and drive off the solvent. When 10 to 20 mL of solvent remain, heat gently on a steam bath until the odor of the solvent can no longer be detected. Facilitate removal of the solvent by utilizing a vacuum or a gentle stream of filtered (oil and water-free) air. After the solvent has been removed, dry the flask containing the extracted matter in a forced circulating air oven at 100 ± 2 °C (212 ± 3.6 °F) for 1 h. Cool to room temperature in a desiccator and weigh. Continue drying for successive 1-h periods at 100 ± 2 °C (212 ± 3.6 °F) 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 W3. If constant weight has not been obtained after the third drying, record that weight as the final weight.

9.4 *Soxtec-Type Apparatus*—Dry an extraction cup in an oven for at least 1 h at 100 ± 2 °C (212 ± 3.6 °F) or 25-30 min at 125 ± 1 °C (257 ± 1.8 °F) to remove residual moisture. Cool in a desiccator, and weigh to the nearest 0.001g. Record this value as W2. Circulate water through the condensers. Turn on the service unit and set the temperature control at 90 ± 1 °C (194 ± 1.8 °F). Fill the cup approximately two-thirds full with petroleum ether or hexane. Place the loaded thimble in the Soxtec-type apparatus. Extract the sample by using the Soxtec-type boiling cycle for 45-50 min, followed by a rinse cycle of 45-50 min. After the rinse cycle, close the condenser and collect the solvent for 10-15 min. Open the Evaporation valve, press the Air button and pull air through the cups for 10-15 min. Close the Evaporation valve and release the extraction cups with the safety catch. Dry the cups in a forced air circulating oven at 100 ± 2 °C (212 ± 3.6 °F) for 60-65 min or 25-30 min at 125 ± 1 °C (257 ± 1.8 °F). Cool the cups for 30-35 min (or to room temperature) in a desiccator. Continue drying for successive 15-min 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 W3. If constant weight has not been obtained after the third drying, record that weight as the final weight.

10. Calculation of Results

10.1 Calculate the percentage of hexane (or petroleum ether) extract, on a moisture-free basis, as follows:

Hexane (or petroleum ether) extract =

$$\frac{W3 - W2}{W1 \times \frac{(100 - \%\ \text{moisture})}{100}} \times 100 \qquad (1)$$

where:

W1	=	weight of specimen, wet blue or wet white,
W2	=	weight of extraction flask,
W3	=	weight of extraction flask and hexane (or petroleum ether) extract, and
% moisture	=	moisture content of the sample from which the specimen was taken.

11. Report

11.1 Report the hexane (or petroleum ether) extract in the wet blue or wet white as the average value obtained from the test results to the nearest 0.01 %.

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

11.3 Report condition of the specimen (that is, Test Method D6659 Method A, or Test Method D6659 Method B).

11.4 Report extraction apparatus used.

12. Precision and Bias

12.1 The precision of this test method is based on an interlaboratory study of WK15217, New Test Method for Hexane/Petroleum Ether Extract in Wet Blue or Fats and Oils in Wet Blue, conducted in 2007. Seven laboratories tested the same material under five different test conditions using both an ether and a hexane extraction. Every "test result" represents an individual determination. Each laboratory was asked to submit two replicate test results, from a single operator, for each analysis and condition. Except for the limited number of reporting laboratories, Practice E691 was followed for the