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

design and analysis of the data; the details are given in an ASTM Research Report.[3]

12.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

12.1.1.1 Repeatability limits are listed in Table 1 and Table 2.

12.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

12.1.3 The preceding terms (repeatability and reproducibility limit) are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statements 12.1 and 12.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. The repeatability limit and the reproducibility limit should be considered as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of the analytical results from seven laboratories, on one material under five different conditions. These conditions were described as the following:

| | |
|---|---|
| Condition A: | Wet Blue, as-is |
| Condition B: | Wet Blue dried at 100 ºC, for 1 h |
| Condition C: | Wet Blue dried at 100 ºC, for 2 h |
| Condition D: | Wet Blue air dried overnight, for 16 – 18 h |
| Condition E: | Wet Blue dried at 125 ºC, for 4 h |

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D31–1013.

12.4 To judge the equivalency of two test results, it is recommended to choose the condition closest in characteristics to the test condition.

12.5 A real world precision statement was determined through statistical examination[4] of 143 results from 9 laboratories, on 16 materials over nearly 2 years. Practice E691 was followed for the design and analysis of the data. The terms below (repeatability and reproducibility) are used as specified in Practice E177.

12.5.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.5.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

| Repeatability (r) | Reproducibility (R) |
|---|---|
| 0.48 | 1.98 |

12.6 The precision of this test method is based on an intralaboratoy study of ASTM WK40322, New Standard Test Method for the Determination of Oil and Grease in Wet Blue and Wet White, conducted between 2011 and 2013. Nine laboratories participated in this study, testing 16 Wet Blue samples. Every "test result" represents an individual determination. The laboratories were asked to report a single test result for 13 materials and duplicate test results for 3 materials. Except for the absence of replicate test results from all of the study materials, Practice E691 was followed for the design and analysis of the data; the details are give in ASTM research reports.[4]

12.6.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D31-1019 and RR:D31-1022. Contact ASTM Customer Service at service@astm.org.

**TABLE 1 Hexane Extraction (%)**

| Conditions | Average[A] $\bar{X}$ | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $s_R$ | Repeatability Limit $r$ | Reproducibility Limit $R$ |
|---|---|---|---|---|---|
| A | 0.683 | 0.199 | 0.057 | 0.203 | 0.161 |
| B | 0.592 | 0.114 | 0.058 | 0.121 | 0.162 |
| C | 0.574 | 0.104 | 0.056 | 0.111 | 0.157 |
| D | 0.620 | 0.206 | 0.080 | 0.213 | 0.223 |

[A] The average of the laboratories' calculated averages.