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.

Designation: D2868 – 17

Standard Test Method for Nitrogen Content (Kjeldahl) and Hide Substance Content of Leather, Wet Blue and Wet White[1]

This standard is issued under the fixed designation D2868; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of the nitrogen content of all types of leather, wet blue and wet white. The nitrogen content is used to calculate the hide substance (protein fiber) content of leather, wet blue and wet white.

NOTE 1—The original test method for leather was essentially a composite of Method 6441 of Federal Test Method Standard No. 311 and Method B5 of the American Leather Chemists Association.

NOTE 2—Melamine, if present in bonded leather, could give an artificially high value for the calculation of protein fiber.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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]

D2813 Practice for Sampling Leather for Physical and Chemical Tests

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 The specimen prepared according to an accepted procedure (see Note 3) is digested with acid in the presence of a catalyst to convert the nitrogen to ammonium ion. The ammonium ion formed is nonvolatile under these highly acid conditions.

NOTE 3—For leather use specimen prepared per Practice D2813. For wet blue and wet white, use specimen prepared per Practice D6659.

3.2 The acid mixture is then made alkaline and the ammonia liberated is distilled into either a boric acid solution which absorbs the ammonia, or a sulfuric acid solution which absorbs the ammonia.

3.3 When the boric acid solution is used, the amount of ammonia in the boric acid is then determined by back titration with standardized acid using a sharp color change indicator (green to purple) to determine the end point. When the sulfuric acid solution is used, the amount of ammonia in the sulfuric acid solution is then determined by back titration with standardized base using a sharp color change indicator (purple to green-blue) to determine the end point.

4. Significance and Use

4.1 The nitrogen content as determined by this test method is normally considered to be related to the amount of hide substance (protein fiber) present in the leather sample. A factor of 5.62 is normally used to calculate the hide substance from the nitrogen content.

4.1.1 The 5.62 factor represents the average result of many analyses of animal hides, but it cannot be considered to be accurate since it varies somewhat from hide to hide of the same type, from type of hide to type of hide, and also with the thickness of hide retained in the final leather (split thickness as compared to original hide thickness). As a result of these

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.06 on Chemical Analysis. This test method was developed in cooperation with the American Leather Chemists Assn. (Standard Method B5 – 1954).

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[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.

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variations, the true factor for any given leather may be expected to vary from 5.44 to 5.80 or about ±3 %.[3]

4.2 A given leather sample may contain nitrogenous substances other than hide substance (protein fiber) which will be analyzed for by this test method, such as resins, dyestuffs, etc., that contain nitrogen. Therefore, although this test method is fairly accurate for determining the nitrogen content of leather, its use for determining hide substance may result in large errors.

4.3 The hide substance value derived from this determination has a large bearing on other chemical determinations of a given leather. Any errors, such as those described in 4.1.1 and 4.2, will be carried over into these other analytical calculations.

5. Apparatus

5.1 *Kjeldahl Apparatus* consisting of:

5.1.1 *Kjeldahl Flask,* of 500 or 800-mL capacity for digestion of the sample.

5.1.2 *Heater,* (gas or electric) for the Kjeldahl flask with fume hood or other exhaust system.

5.1.3 *Distillation Apparatus,* consisting of an efficient vapor trap that can be sealed tightly in the top of the Kjeldahl flask and a condenser connected to the top of the trap. All elements of the distillation system shall be constructed of block tin, borosilicate glass, or other materials known not to react with hot ammonia vapor.

5.2 Semi-automated equipment (Kjeltec/micro-Kjeldahl) produce comparable results and may be substituted for Kjeldahl apparatus. See Precision and Bias (12.1 – 12.4).

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water, deionized water, or water of equal purity.

6.3 *Boric Acid Indicator Solution*—Dissolve 40 g of boric acid (H_3BO_3) (borax-free) in water, add 10 mL of mixed indicator solution (6.5) and dilute to 1 L.

6.4 *Catalyst Digestion Mixture*[5,6]— 20.0 g K_2SO_4 + 0.6 g $CuSO_4$ + 0.2 g pumice.

6.5 *Mixed Indicator Solution*[7]—Dissolve 0.060 g of methyl red indicator and 0.040 g of methylene blue indicator in 100 mL of 95 % ethyl alcohol.

6.6 *Sodium Hydroxide, Concentrated Solution* (450 g/L)—Dissolve 450 g of sodium hydroxide (NaOH) pellets (98 %) in water and dilute to 1 L.

6.7 *Sodium Hydroxide, Standard Solution* (0.1 N)—Dissolve 10 mL of the concentrated NaOH solution (6.6) in 1 L of boiled and cooled water. Determine the exact normality by titration against the standard sulfuric acid (6.10) using the mixed indicator (6.5) for the end point.

6.8 *Sucrose* ($C_{11}H_{22}O_{11}$).

6.9 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4), free from nitrogen.

6.10 *Sulfuric Acid, Standard* (0.3 N)—Dissolve 9 mL of concentrated H_2SO_4 (6.9) in water and dilute to 1 L. Determine the exact normality by titration against an equivalent solution of a primary standard such as anhydrous sodium carbonate or tris (hydroxymethyl) amino methane.

6.11 *Sulfuric Acid, Standard (0.5 N)*—Available commercially. Determine the exact normality by titration against an equivalent solution of a primary standard such as anhydrous sodium carbonate or tris (hydroxymethyl) amino methane.

6.12 *Sodium Hydroxide (0.5 N)*—Available commercially. Determine the exact normality by titration against a known solution of a primary standard such as potassium hydrogen phthalate.

6.13 When using semi-automatic equipment, follow the guidelines provided by the manufacturer.

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 manufacturer's label or SDS (Safety Data Sheet).

8. Standardization

8.1 *Blanks*—Run a blank determination substituting 1.0 g of sucrose in place of the leather specimen by the procedure shown in Section 9. Calculate the blank results, as shown in Section 9.3.

8.2 *Standard*—Tris (hydroxymethyl) amino methane can be used as an internal nitrogen standard for the method. Weigh out to 0.001 g approximately 1 g of tris (hydroxymethyl) amino methane and transfer to the Kjeldahl flask. Run this standard by the same procedure shown in Section 9. One gram of this reagent is equal to 0.1156 g of N_2 or 8.255 meq of N_2.

9. Procedure

9.1 *Procedure A – Kjeldahl Apparatus*

9.1.1 *Sample and Specimen:*

[3] Dahl, S., "Determination of Hide Substance in the Kjeldahl Method," in *Chemistry and Technology of Leather*, Vol 4, Reinhold Publishing Co., New York, NY, 1965.

[4] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[5] Dahl, S., and Oehler, R., "The Determination of Nitrogen in Leather by the Kjeldahl Method," JALCA, Vol 46, 1951, pp. 317–355.

[6] Available as a prepared catalyst mixture from some laboratory supply companies, for example, Alfie Packers, #20P.

[7] Available as prepared solution from some laboratory supply companies. Hach Bromcresol Green Methyl Red Indicator.