



Designation: ~~D7637~~—~~10~~ D7637 – 10 (Reapproved 2015)

Standard Test Method for Determination of Glycerin Assay by Titration (Sodium Meta Periodate)¹

This standard is issued under the fixed designation D7637; 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 provides the quantitative determination of glycerin by the titrimetric method. It is applicable to all glycerin or glycerin/water mixtures >75 %.

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 may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult the established and appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautions are listed in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[D4725 Terminology for Engine Coolants and Related Fluids](#)

[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](#)

2.2 *Other Standards:*

[The United States Pharmacopoeia 31 Glycerin Monograph – Assay Method](#)³

[AOCS Official Method EA 6-94 \(Replaces AOCS EA 6-51\), Determination of Crude Glycerin, Titrimetric Method](#)⁴

[BS 5711: Part 3, 1979 Sampling and Test for Glycerol - Determination of Glycerol Content](#)⁵

3. Terminology

3.1 *Definitions:*

3.1.1 *glycerin*—propane-1,2,3-triol, $C_3H_5(OH)_3$ (also known as glycerine), 1,2,3-propanetriol, 1,2,3-trihydroxypropane, glyceritol, glycylic alcohol, glycerol. CAS #56-81-5.

3.1.2 *SMP*—Sodium Meta Periodate.

3.1.3 For other definitions of terms used in this specification, refer to Terminology [D4725](#).

4. Significance and Use

4.1 This test method is based on the cold oxidation of the glycerol by sodium metaperiodate in a strong acidic medium. Formaldehyde and formic acid are produced in this reaction and the latter is used to measure the glycerol content by titration with standard sodium hydroxide solution, to a pH 8.1 ± 0.1 . The glycerol content is expressed as a percentage (m/m).

¹ This test method is under the jurisdiction of ASTM Committee [D15](#) on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee [D15.93](#) on Research and Long Range Planning.

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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.

³ Available from U.S. Pharmacopoeia (USP), 12601 Twinbrook Pkwy., Parkway, Rockville, MD 20852-1790, [http://www.usp.org](#).

⁴ Available from AOCS—American Oil Chemists' Society (AOCS), 2710 S. Boulder, Urbana, IL 61802-6996, [http://www.aocs.org](#).

⁵ Available from British Standards Institute (BSI), 389 Chiswick High Rd., London W4 4AL, U.K., [http://www.bsi-global.com](#) [http://www.bsigroup.com](#).



4.2 *Interferences*—The glycerin sample should be free of organic compounds with more than two hydroxyl groups on adjacent carbon atoms (for example, sugars, glycols). These types of compounds may produce formic acid which interferes with the determination of the glycerol content.

5. Apparatus

5.1 *Burette*—50 mL “Type A” unit that can deliver 30 drops per mL and calibrated to meet the NIST standards.

NOTE 1—It is critical that all glassware be “Type A” and thoroughly cleaned before use as dirty glassware will impact the analysis results.

5.2 *pH meter*, fitted with glass electrode.

NOTE 2—The pH meter should be standardized with a minimum of two buffer solutions at 10.0 and 7.0. (See 7.12.)

5.3 *Stirrer*—preference should be given to a magnetic stirrer. In this case, check that it does not influence the pH meter.

6. Purity of Reagents and Water

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. 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 indicated otherwise, references to water shall be understood to mean Type II reagent water as defined in Specification **D1193**.

NOTE 3—The presence of carbon dioxide can introduce errors; therefore it is advisable to prepare 500 mL of water by boiling for 5 min and cooling to room temperature. Check the pH of the distilled water before use. The pH should be 7.0 ± 0.1 . If not, adjust the pH with 0.1 N sodium hydroxide (NaOH) or 0.1 N hydrogen chloride (HCl), whichever is appropriate. *Store the freshly boiled and neutralized water in a tightly closed container.*

7. Reagents

7.1 Sodium Metaperiodate, NaIO_4 , acidic solution—Dissolve 60 g of sodium metaperiodate in sufficient water containing 120 mL of 0.1 N sulfuric acid to make 1000 mL. *Do not heat to dissolve the periodate.* If the solution is not clear, pass it through a sintered-glass filter. Store the solution in a glass-stoppered, light-resistant container.

7.2 Hydrochloric Acid, aqueous solution, approximately 0.1 N.

7.3 Sodium Hydroxide, aqueous solution, approximately 0.05 N.

7.4 Sodium Hydroxide, 0.1 N, Standardized—Dissolve four grams of sodium hydroxide (NaOH) in water and dilute to one liter. Standardize accurately to ± 0.0001 N. The standardized sodium hydroxide solution should be protected against evaporation and absorption of carbon dioxide (CO_2) from the air.

7.5 Ethylene glycol (1,2-ethanediol, free from glycerol) solution in water. 1:1 (v/v).

7.6 Sulfuric Acid Solution, aqueous solution, approximately 0.1 N.

7.7 Sulfuric Acid Solution, aqueous solution, approximately 0.2 N.

7.8 Sodium Thiosulfate, 0.1 N, aqueous solution.

7.9 Potassium Iodide, aqueous solution—Dissolve 16.5 g of potassium iodide in water to make 100 mL. Store tightly capped in a light-resistant container.

7.10 Starch Solution—Mix 1g of soluble starch with sufficient cold water to make a thin paste. Add 200 mL of boiling water, and boil for 1 min with continuous stirring. Cool, and use only the clear solution.

NOTE 4—It is recommended that freshly prepared starch solution be used to ensure a sharper endpoint. Commercially available, stabilized starch indicator solution may be also used in place of freshly prepared starch.

7.11 Bromothymol blue indicator solution—Dissolve 100 mg of bromothymol blue in 100 mL of water to make 100 mL of indicator solution.

7.12 pH Buffers (7.0 ± 0.01 and 10.0 ± 0.01 at 25°C) needed for pH meter standardization.

7.13 Freshly boiled water, or equivalent. (See 6.2.)

8. Precautions

8.1 Sulfuric acid and sodium hydroxide solutions are corrosive. Avoid eye and skin contact.