



Designation: D7638 – 10 (Reapproved 2021)

## Standard Test Method for Determination of Fatty Acids and Esters in Glycerin<sup>1</sup>

This standard is issued under the fixed designation D7638; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method provides the quantitative determination of the fatty acid and ester content in purified glycerin by the titrimetric method.

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 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. Specific precautions are listed in Section 8.*

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:*<sup>2</sup>

[D1193 Specification for Reagent Water](#)

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

2.2 *Other Standard:*

[The United States Pharmacopoeia 31 Glycerin Monograph – Fatty Acids and Esters](#)<sup>3</sup>

### 3. Terminology

3.1 *Definitions:*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

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

<sup>3</sup> Available from U.S. Pharmacopoeial Convention (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, <http://www.usp.org>.

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, glycol alcohol. CAS #56-81-5

3.1.2 *FA&E*—fatty acid and esters

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

### 4. Significance and Use

4.1 Any residual fatty acid and esters (FA&E) should be present only at very low levels in purified glycerine.

4.2 This procedure requires the addition of a measured volume of sodium hydroxide, in excess of the amount actually needed to react with FA&E in the glycerine sample. After boiling, the excess sodium hydroxide is back titrated with standardized hydrochloric acid. The quantity of the substance being titrated is calculated as the difference between the volumes of the sodium hydroxide solution originally added, corrected by means of a blank titration, and that was consumed by the titrant in the back titration.

NOTE 1—The standardized solutions for sodium hydroxide and hydrochloric acid are commercially available.

### 5. Apparatus

5.1 *Standard Type A Glassware.*

5.2 *Erlenmeyer flask*, 200 mL to 250 mL, alkali-resistant with a standard tapered 24/40 ground glass neck joint, or equivalent.

5.3 *Microburette* having a capacity of 5 mL, graduated to 0.02 mL and calibrated to meet the National Institute of Standards and Technology (NIST) specification. An automatic titrator with a minimum capacity of 5 mL can also be used.

5.4 *Pipette*, standard or automatic that can accurately deliver  $5\text{ mL} \pm 0.01\text{ mL}$ .

5.5 *Air reflux condenser* with standard taper ground glass joint which fits the Erlenmeyer flask in 5.1. Minimum length, 550 mm.

### 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



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NOTE 1—The standardized solutions for sodium hydroxide and hydrochloric acid are commercially available.

### 5. Apparatus

5.1 *Standard Type A Glassware.*

5.2 *Erlenmeyer flask*, 200 mL to 250 mL, alkali-resistant with a standard tapered 24/40 ground glass neck joint, or equivalent.

5.3 *Microburette* having a capacity of 5 mL, graduated to 0.02 mL and calibrated to meet the National Institute of Standards and Technology (NIST) specification. An automatic titrator with a minimum capacity of 5 mL can also be used.

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**TABLE 1 Fatty Acids and Esters in Glycerin (%)**

Material	Average	Std Dev	Repeatability Std Dev	Reproducibility Std Dev	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	$S\bar{x}$	$S_r$	$S_R$	$r$	$R$
A	0.30	0.07	0.004	0.068	0.012	0.189
B	0.54	0.05	0.009	0.047	0.025	0.131

## 11. Report

11.1 Report the Fatty Acid and Ester content of the sample to the nearest 0.01 mL of NaOH consumed per 50 g of sample.

## 12. Precision and Bias<sup>5</sup>

12.1 *Precision*—See [Table 1](#).

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D15-1026.

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.

## 13. Keywords

13.1 antifreeze; engine coolant; esters; ethylene; fatty acids; glycerin; glycerine; glycerol; titrimetric

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