



Designation: D7736 – 11

Standard Test Method for Determination of Acids and Glycol Esters in Ethylene Glycol¹

This standard is issued under the fixed designation D7736; 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 determination of free acids and glycol esters in ethylene glycol by titration.

1.2 This test method is for used on ethylene glycol used for the manufacture of engine coolant. It can not be used on formulated engine coolant. The inhibitors will interfere with the determination.

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

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1176 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes

D1193 Specification for Reagent Water

D4725 Terminology for Engine Coolants

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology **D4725**.

4. Summary of Test Method

4.1 This test method is used to determine the acid and ester content of ethylene glycol by titration. The sample is titrated to the phenolphthalein end point with 0.02 N NaOH to determine the acidity. Then a known amount of base is added and the

sample is heated at 100°C to hydrolyze the esters. It is then back-titrated with 0.02 N sulfuric acid to determine the ester content.

5. Significance and Use

5.1 The presence of acids or glycol esters in the ethylene glycol used to produce engine coolant is undesirable. Under conditions in an engine cooling system, the esters can hydrolyze to form glycol and an acid. The acid will react with the corrosion inhibitors, thereby reducing the useful life of the coolant. This method can determine the amount of acid and glycol ester present in the ethylene glycol.

6. Interferences

6.1 This test method is based on a color change titration. Glycol with a strong color could interfere with the results of this method.

6.2 High pH recycled glycols streams will affect the results of this test method.

7. Apparatus

7.1 *250 mL Borosilicate Glass Bottles*, with screw caps.

8. Reagents and Materials

8.1 *Phenolphthalein Solution*—Dissolve 0.5 g of phenolphthalein in methanol or ethanol and dilute to 100 mL.

8.2 *Sodium Hydroxide (NaOH)*, 0.02 N in water.

8.3 *Sulfuric Acid (H₂SO₄)*, 0.02 N in water.

8.4 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II or III of Specification **D1193**.

8.5 *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.³ Other grades may be

¹ This test method is under the jurisdiction of ASTM Committee **D15** on Engine Coolants and is the direct responsibility of Subcommittee **D15.04** on Chemical Properties.

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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 *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 *Annual 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.