

Designation: D7736 - 12 (Reapproved 2018)

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

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 ( $\varepsilon$ ) 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.5 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>

D1176 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing PurposesD1193 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

## 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 ( $H_2SO_4$ ), 0.02 N in water.

<sup>&</sup>lt;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.

Current edition approved March 1, 2018. Published March 2018. Originally approved in 2011. Last previous edition approved in 2012 as D7736 - 12. DOI: 10.1520/D7736-12R18.

<sup>&</sup>lt;sup>2</sup> 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.