



## Standard Test Method for Trace Ethylene Glycol in Used Engine Oil<sup>1</sup>

This standard is issued under the fixed designation D 4291; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method provides for the determination of ethylene glycol as a contaminant in used engine oil. This test method is designed to quantitate ethylene glycol in the range from 5 to 200 mass ppm.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.* For specific hazard statements see Notes 2-5.

NOTE 1—A qualitative determination of glycol-base antifreeze is provided in Test Method D 2982. Procedure A is sensitive to about 100 ppm.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

D 2982 Test Methods for Detecting Glycol-Base Antifreeze in Used Lubricating Oils<sup>3</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>4</sup>

### 3. Summary of Test Method

3.1 The sample of oil is extracted with water and the analysis is performed on the water extract. A reproducible volume of the extract is injected into a gas chromatograph using on-column injection and the eluting compounds are detected by a flame ionization detector. The ethylene glycol peak area is determined and compared with areas obtained from the injection of freshly prepared known standards.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis and D02.06 on Analysis of Lubricants.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 05.02.

### 4. Significance and Use

4.1 Leakage of aqueous engine coolant into the crank case weakens the ability of the oil to lubricate. If ethylene glycol is present, it promotes varnish and deposit formation. This test method is designed for early detection to prevent coolant from accumulating and seriously damaging the engine.

### 5. Apparatus

5.1 *Gas Chromatograph*—Any gas chromatograph equipped with the following:

5.1.1 *Flame Ionization Detector*, capable of operating continuously at a temperature equivalent to the maximum column temperature employed, and connected to the column so as to avoid any cold spots.

5.1.2 *Sample Inlet System*, providing for on-column injection and capable of operating continuously at a temperature equivalent to the maximum column temperature employed.

5.2 *Recorder*—Recording potentiometer with a full-scale response time of 2 s or less may be used.

5.3 *Column*—1.2-m (4-ft) by 6.4-mm (1/4-in.) copper tube packed with 5 mass % Carbowax 20-M liquid phase on 30/60 mesh Chromosorb T solid support.

5.4 *Integrator*—Manual, mechanical, or electronic integration is required to determine the peak area. However, best precision and automated operation can be achieved with electronic integration.

5.5 *Centrifuge*—RCF 600 minimum and centrifuge tubes with stoppers.

5.6 *Syringe*—A microsyringe, 10  $\mu$ L is needed for sample introduction.

5.7 *Pasteur Pipets*.

5.8 *Vials*, 2 mL, with crimped septum caps.

### 6. Reagents and Materials

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