Designation: D7922 – 14

Standard Test Method for Determination of Glycol for In-Service Engine Oils by Gas Chromatography[1]

This standard is issued under the fixed designation D7922; 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 glycol based antifreeze for in-service engine oil by derivative headspace/gas chromatography.

1.2 Sample is derivatized in-situ directly in a headspace sampling vial prior to vapor phase extraction and injection into a gas chromatograph.

1.3 The chemistry of the derivatization is unique to the detection of the molecules of ethylene glycol and 1,2-propylene glycol. 1,3-propylene glycol could also be detected but is not used in any known anti-freeze at this time. Other coolant analyses are beyond the scope of this test method.

1.4 The derivatization process does not affect glycol breakdown products such as glycolate and formate and hence the presence of these compounds in the oil will not be quantified.

1.5 The test method concentration range is from 50 µg/g to 1000 µg/g. Lower levels are possible by method modifications. Higher levels are possible through sample dilution.

1.6 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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]

D4291 Test Method for Trace Ethylene Glycol in Used Engine Oil

E355 Practice for Gas Chromatography Terms and Relationships

E594 Practice for Testing Flame Ionization Detectors Used in Gas or Supercritical Fluid Chromatography

E1510 Practice for Installing Fused Silica Open Tubular Capillary Columns in Gas Chromatographs

3. Terminology

3.1 *Definitions:*

3.1.1 This test method makes reference to common gas chromatographic procedures, terms, and relationships. Detailed definitions of these can be found in Practices E355 and E594.

3.1.2 *antifreeze, n*—antifreeze is typically a dilution of ethylene glycol and possibly other glycols, and additives, in water to act as a machine coolant. 1,2-propanediol is found in some antifreeze formulations.

3.1.3 *derivitization reagent, n*—a saturated solution of phenylboronic acid (PBA) in solvent. Acetone and 2,2-dimethoxypropane have been used successfully. Gentle warming at 50 °C will hasten dissolution. Solution is stable for three months at room temperature if kept away from moisture.

3.1.4 *glycol, n*—the amount, expressed as a percentage, of glycol found in the in-service lubricating oil. The most common glycol formulated into antifreeze is ethylene glycol (CAS# 107-21-1) with some antifreeze also containing 1,2-propanediol also known as propylene glycol (CAS# 57-55-6). Another glycol such as 1,3-propanediol (CAS# 504-63-2) is detected by this test method but is not commonly used in antifreeze formulations.

3.1.5 *glycols, n*—the summed amount of individual glycols found in the in-service lubricating oil.

3.1.6 *in-service oil, n*—lubricating oil that is present in a machine that has been at operating temperature for at least 1 h.

4. Summary of Test Method

4.1 A representative aliquot of in-service engine oil is introduced into a headspace sampling vial along with a derivatizing agent. The headspace vial is heated to volatilize the derivatized glycol into the vapor phase. Using a small aliquot of sample and derivatization reagent approaches a total vaporization technique to minimize partition coefficient differences in in-service oil samples. A representative aliquot of the

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.04.0L on Gas Chromatography Methods.

Current edition approved July 15, 2014. Published September 2014. DOI: 10.1520/D7922-14.

[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.

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States