



Standard Test Methods for Detecting Glycol-Base Antifreeze in Used Lubricating Oils¹

This standard is issued under the fixed designation D 2982; 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 These test methods describe the qualitative determination of glycol-base antifreeze in used lubricating oils (mineral base) by two procedures, one using reagents in tablet form and the other using laboratory shelf reagents. Principally the test methods detect ethylene glycol but will also detect other 1,2-glycols that may be present.

1.1.1 When a positive result is obtained and a sample of the unused oil is available, the unused oil is also tested and used as a reference.

NOTE 1—Since the inception of this test method (1971), there have been many changes in base stock technology and additive technology. Therefore, when available, the new, unused oil, or a sample of the same used oil, known to not contain antifreeze, is tested as a reference.

1.2 The tablet procedure (Procedure A) is sensitive to about 100 mg/kg and the shelf reagent procedure (Procedure B) to about 300 mg/kg of ethylene glycol.

1.3 Carbohydrates such as sugars and sugar-containing substances are sometimes used for sabotage purposes. If the presence of these substances is suspected, Procedure A contains a modification to remove these interferences.

1.4 Both procedures are adaptable to field kit use, and brief descriptions for converting to field kit form are given in Annex A1.

1.4.1 Commercial field testing kits are available.²

1.5 The results obtained by this method are qualitative expressions. However, for the preparation of reagents and in the procedures, acceptable SI units are to be regarded as the standard.

1.6 *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:

¹ These test methods are under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and are the direct responsibility of Subcommittee D02.06 on Analysis of Lubricants.

Current edition approved Dec. 10, 1998. Published February 1999. Originally published as D 2982 – 71 T. Last previous edition D 2982 – 93.

² The Gly-Tek Test Kit is available from the Nelco Co., 1047 McKnight Rd., S., St. Paul, MN 55119, and in Canada, it is available from Metro Tech Preventative Maintenance Ltd., 112-5621, 11th St., N.E., Calgary, AB, Canada T2E 6Z7. Test instructions are provided with the kit.

D 95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation³

D 1193 Specification for Reagent Water⁴

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products⁵

D 4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants⁵

D 4177 Practice for Automatic Sampling of Petroleum⁵

3. Terminology

3.1 Definitions:

3.1.1 *glycol-base antifreeze, n*— in engine coolants, ethylene or propylene glycol commonly used in admixture with water and additives to lower the coolant freezing point.

3.1.2 *used oil, n*—any oil that has been in a piece of equipment (for example, an engine, gear box, transformer, or turbine) whether operated or not.

3.1.2.1 *Discussion*—In the development of this test method, the used oil is a mineral lubricating oil from an engine crankcase.

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4. Summary of Test Methods

4.1 The ethylene glycol is extracted from the sample with an acid solution and oxidized to formaldehyde with periodic acid which is detected colorimetrically with decolorized fuchsin.

5. Significance and Use

5.1 Leakage of glycol-base antifreeze into the crankcase is serious because the coolant tends to interfere with the lubricant and its ability to lubricate; it also promotes sludging. Ethylene glycol present in the coolant can increase varnish deposit formation in the crankcase as a result of glycol oxidation and the interaction between glycol and lubricant. Furthermore, because glycol is a higher boiling material than water, it will tend to stay longer in the crankcase oil than water. Lubricant displacement, sludging, and deposit formation all lead to engine malfunction and possible seizure.

5.2 These tests are designed to detect glycol-base coolant contamination even at low levels because early detection enables corrective measures to be taken to prevent leaking coolant from accumulating and seriously damaging the engine.

5.3 These test methods are also significant because the

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Annual Book of ASTM Standards*, Vol 05.02.

reagents can be packaged as a field kit, and the procedure can be followed at the site where there is a concern.

6. Interferences

6.1 The reactions are not specific to ethylene glycol; other 1,2-glycols and many carbohydrates will give a positive test.

6.2 Hexylene glycol and methoxy glycol, which are often used as gasoline anti-icing additives, do not interfere when present in gasoline-diluted used oils.

6.3 Oil oxidation products present do not interfere with the test.

6.4 Some new oils can contain small amounts of glycol derivatives as part of their makeup and thus give a positive test. These oils, after use, invariably give a negative or trace reaction as the glycol derivatives are slowly destroyed under conditions of use in the engine.

6.5 A modification is described in Procedure A for removing interferences caused by carbohydrates such as sugars and sugar-containing substances that are sometimes used for sabotage purposes.

7. Purity of Reagents

7.1 *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.⁶ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water conforming to Type III of Specification D 1193.

8. Sampling

8.1 Ethylene glycol is immiscible with and heavier than mineral lubricating oil; hence, it will tend to settle. Do not take a sample that is too large to shake vigorously in the laboratory because vigorous shaking is required before conducting the test.

8.2 If the sample delivered is too large to be shaken vigorously, then draw the sample to be tested from a low point in the container.

8.3 Under some circumstances ethylene glycol will emulsify with the oil to form a sludge. If the sample to be tested is a sludge, then dilute the sample with a solvent, such as naphtha or toluene. (**Warning**—See Note 2.) Use a volume of solvent sufficient to provide a fluid sample for the test.

NOTE 2—**Warning:** These solvents are toxic and flammable.

8.4 When drawing a sample directly from an engine or machine, ensure that the sample is representative by drawing it

just after the engine or machine has been shut down. If the engine or machine has seized, or it has not seized but is not to be turned over, draw the sample from a low point so as to sample the settled glycol if present.

NOTE 3—**Caution:** Avoid a top or dipstick sample because the glycol portion, if present, can be missed.

8.5 Where applicable, Practice D 4057 (manual sampling) and Practice D 4177 (automatic sampling) will provide useful direction for obtaining consistent and representative samples. Consistent and representative sampling is especially important when the lubricant is in equipment which is still operational and other tests on the sample are also required.

PROCEDURE A—USING TABLETS

9. Apparatus

9.1 *Graduated Cylinder*, glass-stoppered, 100 mL, with 1-mL graduations (two required).

9.2 *Tablet Press*—See 10.2.

10. Reagents and Materials

10.1 *Acid Solution* (12 volume %)—Add and mix slowly with caution 12 volumes of concentrated sulfuric acid (H₂SO₄, relative density 1.84) into 88 volumes of water. (**Warning**—See Note 4 and Note 5.)

NOTE 4—**Warning:** Corrosive. Causes severe burns.

NOTE 5—**Precaution:** Mixing sulfuric acid with water generates heat. Always add the acid to the water and add it slowly. Never add the water to the acid.

10.2 *Potassium Periodate Tablet*.⁷ (**Warning**—See Note 6.)

NOTE 6—**Warning:** Toxic. Hazardous.

10.2.1 *Composition of Tablet:*

Ingredient	Amount	Function
Potassium periodate (Caution —Toxic. Hazardous.)	0.3 ± 0.01 g	oxidizing agent
Ammonium chloride	0.9 ± 0.03 g	diluent
Sodium bicarbonate	0.3 ± 0.01 g	diffusing agent
Polyvinylpyrrolidone (5 mass % dissolved in isopropyl alcohol) ⁸	0.01 g	binder
Total weight	1.5 ± 0.05 g	

10.2.2 *Preparation of Tablet*—Bind chemicals into tablet form not to exceed 13 mm in diameter. The tablet should withstand reasonable handling and be readily soluble in the acid solution (Note 7). Store in amber glass bottles with a plastic cap at room temperature and out of direct rays of the sun. In this way the tablet will remain stable for at least five years.

⁷ Suitable potassium periodate and sodium sulfite/pararosaniline hydrochloride tablets are available from Accurate Manufacturing Chemicals Inc., PO Box 26, Cote St., Lue Station, Montreal, Quebec, Canada H4V 1H8 and from Technical Products Inc., PO Box 11428, Baltimore, MD 21239.

⁸ Polyvinylpyrrolidone is available as "Plasdone" from Antara Chemicals Division of General Aniline and Film Corp., New York, NY.

⁶ *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K. and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.