



Designation: D2982 – 07

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

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1. Scope*

1.1 These test methods cover 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 Glycol-based coolant leaks into crankcases may not be detected or may result in a low bias using these test methods if the glycol has degraded or been thermally or otherwise oxidized. The conditions in crankcases may be such that contaminant glycols are oxidized or degraded to a degree to which the color indicator reaction does not occur or is biased enough so as to not trigger the color change. Other test methods for the detection of coolants or coolant additives in lubricating oils should be used if the results from these test methods alone are inconclusive or questionable.

1.4 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.

1.5 Both procedures are adaptable to field kit use, and brief descriptions for converting to field kit form are given in [Annex A1](#).

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

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1.5.1 Commercial field testing kits are available.^{2,3}

1.6 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.

1.7 *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*:⁴

D95 Test Method for Water in Petroleum Products and Bituminous Materials by Distillation

D1193 Specification for Reagent Water

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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.

² The sole source of supply of the apparatus known to the committee at this time is the Gly-Tek Test Kit available from the Nelco Co., 1047 McKnight Rd., S., St. Paul, MN 55119. In Canada, it is available from Metro Tech Preventative Maintenance Ltd., 112-5621, 11th St., N.E., Calgary, AB, Canada T2E 6Z7.

³ If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁴ 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.

*A Summary of Changes section appears at the end of this standard

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 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.⁵ 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 **D1193**.

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**—These solvents are toxic and flammable.) Use a volume of solvent sufficient to provide a fluid sample for the test.

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. (**Warning**—Avoid a top or dipstick sample because the glycol portion, if present, can be missed.)

8.5 Where applicable, Practice **D4057** (manual sampling) and Practice **D4177** (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**.

⁵ *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.

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**—Corrosive. Causes severe burns. 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*.^{3,6} (**Warning**—Toxic. Hazardous.)

10.2.1 Composition of Tablet:

Ingredient	Amount	Function
Potassium periodate (Warning —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) ^{3,7}	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 2**). 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.

NOTE 2—Exercise care in the preparation of the tablets to assure that they are not so brittle that they will crumble in handling or not so compact that they will not readily dissolve in the acid solution.

10.3 *Sodium Sulfite/Pararosaniline Hydrochloride Tablet*.^{3,6} (**Warning**—Toxic. Hazardous.)

10.3.1 Composition of Tablet:

Ingredient	Amount	Function
Sodium sulfite (Warning —Toxic. Hazardous.)	1.05 ± 0.03 g	reducing agent
Pararosaniline hydrochloride (Warning —Toxic. Hazardous.)	0.01 ± 0.001 g	indicator
Sodium bicarbonate	0.42 ± 0.01 g	diffusing agent
Magnesium stearate	0.01 g	lubricant
Polyvinylpyrrolidone (5 mass % dissolved in isopropyl alcohol) ^{3,7}	0.01 g	binder
Total weight	1.50 ± 0.04 g	

10.3.2 *Preparation of Tablet*—Mix and grind together the sodium sulfite, sodium bicarbonate, and the pararosaniline hydrochloride. Pass the mixture through a 180- μ m (80-mesh) sieve, dampen with the polyvinylpyrrolidone in alcohol, and pass through a 425- μ m (40-mesh) sieve. Dry for 4 h at 380°C and overnight at room temperature. Pass through a 500- μ m (30-mesh) sieve. Sprinkle with magnesium stearate and mix. Press into tablet form not to exceed 13 mm in diameter. The tablet should withstand reasonable handling and be readily

⁶ The sole source of supply of the apparatus known to the committee at this time is potassium periodate and sodium sulfite/pararosaniline hydrochloride tablets 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.

⁷ The sole source of supply of the apparatus known to the committee at this time is Polyvinylpyrrolidone available as "Plasdone" from Antara Chemicals Division of General Aniline and Film Corp., New York, NY.

soluble in the acid solution (**Note 2**). Store in an amber glass bottle with plastic cap at room temperature or lower and out of the direct rays of the sun. In this way, the tablets will remain stable for at least five years.

10.4 *Toluene*—(**Warning** —Toxic. Flammable.)

11. Preparation of Apparatus

11.1 Rinse the cylinder first with toluene to remove the used oil/toluene mixture and then with hot water. Dry.

11.2 **Warning**—Do not use soaps or detergents for cleaning because they can leave residues that can interfere with the separation of the aqueous layer.

12. Procedure

12.1 Bring the sample to room temperature and shake well to ensure even distribution of any glycol throughout the sample (Section 8). Use only reagents at room temperature (above 18°C).

12.2 Pour toluene into a 100-mL glass-stoppered cylinder to the 80-mL mark. Add the well-mixed oil sample to the 100-mL mark. Stopper the cylinder and mix well.

12.3 Pour acid solution into another 100-mL glass-stoppered cylinder to the 60-mL mark. Drop a potassium periodate tablet into the acid solution and allow it to dissolve. Bring to the 80-mL mark with the well-mixed toluene/oil mixture from the other cylinder. Shake vigorously for 1 min, being careful to avoid leakage of liquid from around the stopper. Remove the stopper and let the cylinder stand for 10 min.

12.4 Drop in a sulfite/pararosaniline tablet and wait for 30 min. Upon addition of the tablet, effervescence commences, and a deep brownish color develops in the aqueous layer in about 1 min and then disappears.

12.5 After the disappearance of the brown color, observe the formation of color in the aqueous layer.

13. Observations and Interpretations

13.1 Regard a yellow or pale green coloration that persists for at least 30 min as a negative test result.

13.2 Regard a pale, dirty green color that gradually turns to purple in 30 min as a trace test result. This trace quantity is in the range of 100 to 300 mg/kg.

13.3 Regard a purple color, which may intensify upon standing for 30 min, as a positive test result. When the purple color appears within a few minutes, more than 1 % glycol is present.

13.4 When the test result is positive or trace, and sugar or sugar-containing materials are suspected of being present, instead of performing the test directly on the toluene/oil mixture (12.2), do a water test (Test Method D95 first on the 100 mL of this mixture, transfer the condensed trap contents to the acid/periodate solution in the glass-stoppered cylinder (12.3), and carry out the rest of the antifreeze test. Discard the oil/solvent residue from the water test that would contain any sugar. The presence or absence of interfering compounds from