



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

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^{ε1} NOTE—Editorial changes were made throughout in November 2001.

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.*

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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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.

2. Referenced Documents

2.1 ASTM Standards:

- 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.

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.

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.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**—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 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**—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*.⁷ (**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 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.

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.

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.

10.3 Sodium Sulfite/Pararosaniline Hydrochloride Tablet⁷ (Warning—Toxic. Hazardous.)

10.3.1 Composition of Tablet:

Ingredient	Amount	Function
Sodium sulfite (Caution —Toxic. Hazardous.)	1.05 ± 0.03 g	reducing agent
Pararosaniline hydrochloride (Caution —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) ⁸	0.01 g	binder
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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 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 **Caution**—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 D 95 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 manufacture can be confirmed by carrying out the test on new or preferably on the used oil known not to be contaminated by glycol-base antifreeze as the concentration of these compounds tends to diminish in use. Allow for such interference when reporting results.

13.5 When a positive test result is obtained, perform the test on the same oil, unused, or the same oil, used and known to contain no antifreeze. A difference in the color formation, especially a bluish tint, between the blank and the used oil indicates a positive result. A positive result cannot be confirmed if there is no difference between the color formation of the blank and used oil.

13.6 A light red (13.2) or a deep red (13.3) may also be an indication of a positive test. However this can be confirmed by testing the color formation in the same oil, unused, or on the same used oil which is known to contain no antifreeze. A difference in the color formation, especially a bluish tint, between the blank and the used oil indicates a positive result. A positive result cannot be confirmed if there is no difference between the color formation of the blank and used oil.

NOTE 3—The Gly-Tek test kit² is provided with a color chart. This chart uses only varying intensities of the purple color.

PROCEDURE B—USING SHELF REAGENTS

14. Apparatus

14.1 *Test Bottle*—A 250-mL stoppered, graduated cylinder, or a 250-mL jar, approximately 57 mm in outside diameter, 127 mm tall,⁹ marked at 50-, 100-, and 175-mL volume levels.

15. Reagents and Materials

15.1 For purity and quality of reagents, see 7.1

15.2 *Calcium chloride* (CaCl₂). (**Warning**—Toxic. Hazardous.)

15.3 *Methanol* (CH₃OH). (**Warning**—Toxic. Flammable.)

15.4 *Hydrochloric acid*, concentrated (relative density 1.19).

⁹ Dominion No. 101-2673 or equivalent has been found satisfactory for this purpose.

15.5 *Potassium Periodate* (KIO_4), powder. (**Warning**—Toxic. Hazardous.)

15.6 *Fuschin (Rosaniline Hydrochloride)*. (**Warning**—Toxic. Hazardous.)

15.7 *Pararosaniline Hydrochloride*. (**Warning**—Toxic. Hazardous.)

15.8 *Sodium Bisulfite* (NaHSO_3). (**Warning**—Toxic. Hazardous.)

15.9 *Acid-Salt Solution Solvent*—Using a graduated cylinder, add 100 mL of hydrochloric acid to 500 mL of water. When this has cooled down below 25°C (room temperature or lower), add 400 mL of methanol using a graduated cylinder; mix thoroughly. (**Warning**—Mixing hydrochloric acid with water generates heat. Always add the acid to the water and add it slowly. Never add the water to the acid.)

15.10 *Acid-Salt Solution*—Dissolve 10 ± 0.1 g of calcium chloride in the solvent prepared in 15.9.

15.11 *Schiff's Reagent Solvent*—Using graduated cylinders add 20 mL of concentrated hydrochloric acid (relative density 1.19) to 800 mL of water (**Warning**—Mixing hydrochloric acid with water generates heat. Always add the acid to the water and add it slowly. Never add the water to the acid.) Mix thoroughly.

15.12 *Schiff's Reagent*—Into the solvent prepared in 15.11, dissolve 1.0 ± 0.1 g of basic fuschin or pararosaniline or a mixture of both and 18 ± 0.1 g of sodium bisulfite (NaHSO_3) and dilute the solution to 1000 mL. Allow the solution to stand for a few hours or overnight before using. Store in an amber bottle away from direct sunlight.

15.13 *Toluene*. (**Warning**—Toxic. Flammable.) *Naphtha* (**Warning**—Toxic. Flammable) is a suitable alternative. See 16.3.

16. Procedure

16.1 Bring sample and reagents to a temperature of at least 21°C and preferably 27°C.

16.2 Fill the test jar with acid-salt solution to the 50-mL mark. Add 0.20 ± 0.01 g of potassium periodate and dissolve by swirling the jar.

16.3 Shake the sample well (Section 8) and pour it into the jar to the 100-mL mark. Close the jar with the screw cap and shake the contents vigorously for about 30 s. Allow to stand for several minutes until the water and oil layers have separated. If the oil and water layers have not separated within 5 min, add 50 mL of naphtha or other appropriate glycol-free solvent immiscible with water and shake. This will reduce the viscosity of the oil and standing another 5 min or less should allow the layers to separate.

16.4 Add Schiff's reagent, filling the jar to the 125- or 175-mL mark if solvent was added, swirl to disperse the reagent, and allow the mixture to stand undisturbed.

16.5 Observe the color of the water layer.

17. Observations and Interpretations

17.1 Disregard any color appearing after the test has stood for 10 min. (16.4).

17.2 When a definite pink, red purple, or violet color appears at the oil-water interface within 10 min. of standing, perform a blank test on the same new, unused oil or, preferably, on the same used oil which is known to contain no antifreeze.

17.3 When the color at the oil-water interface is a definite purple or violet color and the blank (17.2) does not give the same color, regard the color as a positive test.

NOTE 4—When there are several used oil samples to be tested consecutively and all are from the same new oil, only one blank test need be performed.

17.4 When a red or definite pink color appears at the oil-water interface, antifreeze may or may not be present. It depends on the intensity of the color relative to the blank.

17.5 When sugar or sugar-containing materials are suspected to be present or when the steps outlined in 17.3 and 17.4 cannot provide a definite answer, perform a water test as outlined in Test Method D 95 using 20 mL of the test sample and 80 mL of toluene. Take the water (glycol) - toluene extract from the condenser trap and proceed as outlined in 16.3 and 16.4. The appearance of a purple or violet color within 10 min. indicates the presence of antifreeze in the original oil sample.

18. Report

18.1 Report the results as glycol-base antifreeze in used oil as positive, trace, or negative, Test Methods D 2982, Procedure A, or, positive or negative, Test Methods D 2982, Procedure B.

18.2 When a sample for the reference test is not available, and the test result is positive, report the positive test and indicate that a reference test was not performed.

19. Precision and Bias

19.1 No statement is made about either the precision or bias of these test methods for measuring glycol-base antifreeze since the result merely states whether there is conformance to the criteria for success specified in the procedure.

20. Keywords

20.1 glycol-base antifreeze; glycol-base coolant; used lubricating oil

ANNEX**(Mandatory Information)****A1. FIELD TEST KITS****A1.1 Adaptability of Procedures A and B**

A1.1.1 Both procedures are adaptable for field kit use.

A1.1.2 A kit using Procedure A can be prepared for about 15 tests by storing the acid solution in a 1-L acid-resistant bottle and the toluene in a 250-mL toluene-resistant bottle in the kit. Instead of two glass-stoppered cylinders, use two tall-form 125-mL sample bottles with calibration marks for acid solution, toluene, and oil. The cap can be used for measuring approximate quantities of oil under test. The test may be carried out in only one 125-mL bottle so long as the ratio of acid solution to toluene to test oil of 60 mL:16 mL:4 mL is retained in conjunction with whole tablets.

A1.1.3 The kit size for Procedure A can be reduced by using a smaller test bottle and half quantities of reagents and tablets

to carry out a test. A reduction to quarter-size quantities too is possible, provided a still smaller sample bottle or vial, tall-form, is used with quarter-size reagents and tablets. **Caution**—Cutting the tablets specified in Procedure A into quarters has to be done with care as the sodium sulfite/pararosaniline hydrochloride tablet may crumble. A tall-form sample bottle is essential to facilitate the separation of the oil/water layers.

A1.1.4 Procedure B can be adapted to field use more or less as described in Sections 14-17. The 0.2 g of potassium periodate needed for each test would have to be weighed beforehand and stored in vials in the kit because a balance is not likely to be readily available in the field. The sample and reagents should be at a temperature of 21°C or higher, preferably 27°C when carrying out the test.

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