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Standard Specification for Recycled Glycol Base Engine Coolant Concentrate for Automobile and Light-Duty Service¹

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1. Scope

1.1 This specification covers the requirements for recycled ethylene glycol base engine coolant concentrate and recycled propylene glycol base engine coolant concentrate for use in automobiles and light-duty vehicles. This specification provides a procedure for evaluating engine coolant recycling technologies through the recycling of a reference synthetic used engine coolant. It also specifies the chemical, physical, and performance requirements for the recycled glycol base engine coolant concentrate that is produced from that procedure. When used at 40 to 70 volume % in water, it will function effectively during both winter and summer. This material is intended to provide protection against freezing, boiling, and corrosion in automobile or other light-duty service cooling systems.

NOTE 1—Committee D-15 has developed this specification using experience, knowledge, and research from the recycling of used aqueous base engine coolants and has not substantially studied the effects of recycling glycols from other sources or with excessive contaminant levels. Efforts are being made to produce a redistilled glycol specification that addresses recycling used aqueous glycol base engine coolants and glycols from other sources.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are approximate equivalents and provided 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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 512 Test Methods for Chloride Ion in Water²
- D 516 Test Methods for Sulfate Ion in Water²

¹ This specification is under the jurisdiction of ASTM Committee D-15 on Engine Coolants and is the direct responsibility of Subcommittee D15.15 on Recycled Engine Coolants.

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² *Annual Book of ASTM Standards*, Vol 11.01.

- D 1119 Test Method for Ash Content of Engine Coolants and Antitrusts³
- D 1120 Test Method for Boiling Point of Engine Coolants³
- D 1121 Test Method for Reserve Alkalinity of Engine Coolants and Antitrusts³
- D 1122 Test Method for Specific Gravity of Engine Coolant Concentrates and Engine Coolants by the Hydrometer³
- D 1123 Test Method for Water in Engine Coolant Concentrate by the Karl Fischer Reagent Method³
- D 1126 Test Method for Hardness in Water²
- D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants of Antitrusts for Testing Purposes³
- D 1177 Test Method for Freezing Point of Aqueous Engine Coolants³
- D 1193 Specification for Reagent Water²
- D 1287 Test Method for pH of Engine Coolants and Antitrusts³
- D 1293 Test Methods for pH of Water²
- D 1384 Test Method for Corrosion Test for Engine Coolants in Glassware³
- D 1881 Test Method for Foaming Tendencies of Engine Coolants in Glassware³
- D 1882 Test Method for Effect of Cooling System Chemical Solutions on Organic Finishes for Automotive Vehicles³
- D 1888 Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water⁴
- D 2570 Test Method for Simulated Service Corrosion Testing of Engine Coolants³
- D 2809 Test Method for Cavitation Corrosion Erosion-Corrosion Characteristics of Aluminum Pumps with Engine Coolants³
- D 2847 Practice for Testing Engine Coolants in Car and Light Truck Service³
- D 3306 Specification for Ethylene Glycol Base Engine Coolant for Automobile and Light-Duty Service³
- D 3634 Test Method for Trace Chloride Ion in Engine Coolants³

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ Discontinued. See *1991 Annual Book of ASTM Standards*, Vol 11.01.

- D 4327 Test Method for Anions in Water by Chemically/Suppressed Ion Chromatography²
- D 4340 Test Method for Corrosion of Cast Aluminum Alloys in Engine Coolants Under Heat-Rejecting Conditions³
- D 4656 Specification for Prediluted Aqueous Ethylene Glycol Base Engine Coolant (50 Volume % Minimum) for Automobile and Light-Duty Service³
- D 4725 Terminology for Engine Coolants³
- D 5216 Specification for Propylene Glycol Base Engine Coolant for Automobile and Light-Duty Service³
- D 5827 Test Method for Determination of Chloride in Engine Coolant by Ion Chromatography³
- D 6129 Test Method for Silicon in Engine Coolant Concentrates by Atomic Absorption Spectroscopy³
- D 6130 Test Method for Determination of Silicon and Other Elements in Engine Coolant by Inductively Coupled Plasma-Atomic Emission Spectroscopy³
- D 6208 Test Method for Galvanostatic Measurement of Pitting Potential of Aluminum and Its Alloys³
- D 6257 Specification for Prediluted Aqueous Propylene Glycol Base Engine Coolant (50 Volume % Minimum) for Automobile and Light-Duty Service³
- D 6471 Specification for Recycled Prediluted Aqueous Glycol Base Engine Coolant (50 Volume % Minimum) for Automobile and Light-Duty Service³

2.2 Other Documents:

- SAE HS40 Maintenance for Automotive Engine Cooling System⁵
- GM 6043M —Automotive Engine Coolant Concentrate—Ethylene Glycol Type⁶
- GM 1825M —Automotive Engine Coolant Concentrate—Ethylene Glycol; §3.11 Storage Stability and Compatibility⁷

3. Reagents and Materials

3.1 *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 such specifications are available⁸. 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.

3.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

⁵ SAE Handbook, available from Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

⁶ Applicable rewrite available in Annex A2.

⁷ Applicable rewrite available in Annex A3.

⁸ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

4. Procedure

4.1 In order to properly evaluate a coolant recycling technology, a reference synthetic used coolant (Annex A1) is to be used as the feed stock. The reference synthetic used coolant shall be prepared in accordance with Annex A1. An appropriately sized batch of reference synthetic used coolant is to be prepared and used to purge the recycling process components and yield enough consistent product to complete the testing specified within this specification

4.2 Prepare the coolant recycling equipment or technology in accordance with the manufacturer’s directions and recommendations.

4.3 Vigorously stir or mix the reference synthetic used coolant using appropriate vessels and mixing equipment immediately prior to and during the processing through the coolant recycling process.

4.4 Recycle the reference synthetic used coolant according to the manufacturer’s directions and recommendations.

4.5 Evaluate the recycled coolant according to the requirements listed in this specification.

5. General Requirements

5.1 The recycled glycol base engine coolant concentrate shall consist essentially of ethylene glycol or propylene glycol. It also shall contain suitable corrosion inhibitors, a foam suppressor, and sufficient water to dissolve the additives.

5.1.1 Recycled ethylene glycol base engine coolant concentrates may be packaged and should be able to be poured at temperatures as low as –18°C (0°F). Other glycols such as propylene and diethylene glycol may be included up to a maximum of 15 % if the chemical and physical properties in Table 1 are met.

5.1.2 Recycled propylene glycol base engine coolant concentrates may contain glycols other than propylene glycol up to 1 % provided the chemical and physical properties of Table 1 are met..

5.2 The recycled glycol base engine coolant concentrate shall conform to the chemical and physical properties in Table 1. If the recycled glycol base engine coolant concentrate

TABLE 1 Physical and Chemical Requirements

Property	Ethylene Glycol Base Specific Values	Propylene Glycol Base Specific Values	ASTM Test Method
Relative density, 15.5°C (60/60°F)	1.110 to 1.145	1.030 to 1.065	D 1122
Freezing point, 50 volume % in distilled water, C ()	–37 (–34) max or lower	–32 (–26) max or lower	D 1177
Boiling point ^a , undiluted, °C (°F) 50 volume % in distilled water	163 (325) min	152 (305) min	D 1120
	107.8 (226) min	104 (219) min	D 1120
Effect on automotive finish	no effect	no effect	D 1882
Ash content, mass %	5 max	5 max	D 1119
pH, 50 volume % in distilled water	7.5 to 11.0	7.5 to 11.0	D 1287
Chloride, ppm	25 max	25 max	D 3634, D 5827
Sulfate, ppm	100 max	100 max	D 516, D 4327
Water, mass %	5 max	5 max	D 1123
Reserve alkalinity, mL	Report ^b	Report ^b	D 1121

^aSome precipitate may be observed at the end of the test. This should not be cause for rejection.

^bAgreed value between the supplier and the customer.

contains chloride, or sulfate levels greater than the requirements specified in Table 1, fleet testing requirements described in Section 6 shall be met. If the chloride, or sulfate requirements, or both, in Table 1 are met, fleet testing is not required.

NOTE 2—The intention of conducting fleet testing is to verify that the effects of residual contaminants left in the recycled glycol base engine coolant are neutralized through real world application in a fleet test. Due to the fact that there are not enough industry field test data available to support setting hard specification limits, the fleet test requirements in Section 6 specify a protocol and limits based on comparing the recycled glycol base engine coolant to an industry standard reference engine coolant that is known to adequately protect modern day cooling systems from corrosion.

5.3 The recycled glycol base engine coolant concentrate shall conform to the general requirements in Table 2.

5.4 The recycled glycol base engine coolant concentrate shall be in accordance with the performance requirements in Table 3.

5.5 When preparing solutions for actual service, use municipal (treated) water, or low mineral content well water. If such water is not available, then use deionized (demineralized) or distilled water. This practice will minimize the formation of hard water scale and avoid the introduction of aggressive contaminants, such as chlorides and sulfates, that can increase the corrosion rate of aluminum and iron (see Appendix X1 for additional information).

5.6 The product, when installed in accordance with the recommendations of the manufacturer of the vehicle or the coolant recycling process, and those on the product label, shall be suitable for use in a properly maintained cooling system (see Appendix X1) in normal passenger car service for a minimum of one year without adversely affecting fluid flow and heat transfer.

6. Fleet Testing Requirements

6.1 *Engine Coolants*—Reference engine coolant concentrate (see Annex A2) and recycled engine coolant concentrate shall be used to conduct the fleet testing.

6.2 *Vehicles*—A minimum of fourteen vehicles is recommended to start the test: seven containing reference engine coolant concentrate (control vehicles) and seven containing recycled engine coolant concentrate (test vehicles). A minimum of five control vehicles and five test vehicles shall finish the test. Starting with fourteen vehicles allows for vehicles to drop out of the test for various reasons, that is, mechanical failure, coolant contamination, etc.

6.2.1 *Vehicle Selection*—All vehicle cooling systems should be inspected prior to selection. Vehicles with corroded cooling systems should not be used for testing purposes. Vehicle selection shall be made as follows:

6.2.1.1 Control vehicles and test vehicles shall be matched pairs and be new or have similar low mileage accumulation.

6.2.1.2 Same year, make, and model.

TABLE 2 General Requirements

Property	Specified Values	ASTM Test Method
Color	Distinctive	—
Effect on nonmetals	No adverse affect	—

TABLE 3 Performance Requirements

Property	Specific Values	ASTM Test Method
Corrosion in glassware; weight loss, mg/specimen		D 1384
Copper	10 max	
Solder	30 max	
Brass	10 max	
Steel	10 max	
Cast iron	10 max	
Cast aluminum	30 max	
Simulated service test; weight loss, mg/specimen		D 2570
Copper	20 max	
Solder	60 max	
Brass	20 max	
Steel	20 max	
Cast iron	20 max	
Cast aluminum	60 max	
Corrosion of cast aluminum alloys at heat-rejecting surfaces; weight loss, mg/cm ² /week	1.0 max	D 4340
Foaming		D 1881
Volume, mL	150 max	
Break time, s	5 max	
Cavitation-Erosion rating for pitting, cavitation, or Erosion of the water pump, rating	8 min	D 2809
Aluminum galvanostatic pitting potential, V v SHE	−0.40 min	D 6208
Fleet Test	see 6.8 and appropriate subsections	

6.2.1.3 Similar mileage and cooling system conditions.

6.2.1.4 Same cooling system and power train configuration.

6.2.1.5 Aluminum head(s), aluminum radiator, aluminum heater core, and aluminum water pump (housing).

6.2.1.6 Cast iron block (optional).

NOTE 3—Vehicles equipped with cast iron heads may be tested in addition to those specified above. A minimum of five control vehicles and five test vehicles, equipped with aluminum heads, shall complete the test and be included in the conclusive analysis report.

6.2.2 *Vehicle Setup*—All vehicles shall be set up in accordance with Practice D 2847 unless otherwise specified within this specification.

6.2.2.1 All vehicles shall be set up with a new aluminum head(s), aluminum radiator, aluminum water pump, thermostat, hoses, belts and radiator/cooling system cap. Installation of new heater cores is recommended. New aluminum heads and radiators are required, as they will be sectioned for evaluation at the end of the test.

6.2.2.2 Each vehicle shall be set up with six coupon bundles in the bypass heater circuit coupon bundle capsule(s). Once the test has been started, no additional or replacement corrosion coupon bundles can be added to any of the vehicle cooling systems. The coupon bundle capsule(s) should be arranged in the cooling system to minimize the amount of coolant spillage when bundles are removed throughout the testing period. More than one coupon bundle capsule may be used in the bypass heater circuit in series if the vehicle design does not accommodate the incorporation of a single capsule.

NOTE 4—Additional coupon bundles may be added to the cooling system prior to starting the test for informational purposes.

6.3 *Reference Coolant Solution*—Prepare the reference coolant solution with reference engine coolant concentrate and corrosive water described in Test Method D 1384. The glycol concentration should give a freeze point of $-37 \pm 1^\circ\text{C}$ ($-34 \pm 2^\circ\text{F}$) for a 50 volume % ethylene glycol base reference coolant solution or a freeze point of $-32 \pm 1^\circ\text{C}$ ($-26 \pm 2^\circ\text{F}$) for a 50 volume % propylene glycol base reference coolant solution. Enough reference coolant solution should be prepared to allow for additions to the vehicle cooling systems throughout the test duration and for individual vehicle test restarts.

6.4 *Test Coolant Solution*—Prepare the test coolant solution with recycled engine coolant concentrate and corrosive water described in Test Method D 1384. The glycol concentration should give a freeze point of $-37 \pm 1^\circ\text{C}$ ($-34 \pm 2^\circ\text{F}$) for a 50 volume % ethylene glycol base test coolant solution or a freeze point of $-32 \pm 1^\circ\text{C}$ ($-26 \pm 2^\circ\text{F}$) for a 50 volume % propylene glycol base test coolant solution. Enough test coolant solution should be prepared to allow for additions to the vehicle cooling systems throughout the test duration and for individual vehicle test restarts.

6.5 *Vehicle Test Parameters:*

6.5.1 *Preparation and Testing*—All vehicles shall be tested in accordance with Practice D 2847, unless otherwise specified within this specification.

6.5.2 *Duration*—One year, 48,280 km (30,000 miles) minimum, and 4,828 km (3,000 miles) per month maximum.

6.5.3 *Driving Conditions*—All vehicles shall undergo similar driving conditions. A minimum of 40 % of the test duration shall be city driving (stop and go).

6.5.4 *Duty Cycle*—All vehicles shall be turned off and allowed to cool for a minimum of 8 h per day.

6.5.5 *Coolant Solution Additions*—Coolant solution additions are not to exceed 10 volume % of the vehicle cooling system capacity within the fleet test duration for top-off or to replace lost coolant solution. Coolant solution used to top-off the cooling system shall be from the same batch that was initially prepared and put into that vehicle.

6.5.6 *Number of Vehicles to Finish*—A minimum of five control vehicles containing the reference coolant solution and a minimum of five test vehicles containing the test coolant solution shall finish the fleet test and have valid results.

6.6 *Vehicle Exclusion Criteria*—Criteria for vehicle exclusion from the fleet test evaluation are:

6.6.1 If the fleet testing requirements are not followed.

6.6.2 If cooling system leakage exceeds 10 volume % or cooling system failure occurs.

6.6.3 If the vehicle becomes disabled, that is, accident, engine failure, vehicle becomes inoperable, etc.

6.6.4 If the cooling system becomes contaminated with oil.

6.6.5 If the cooling system is topped off or the coolant solution was replaced with a coolant solution volume greater than 10 volume % of the vehicle cooling system capacity.

6.6.6 Coolant solutions or chemicals other than those prepared for that vehicle are added to the cooling system.

6.6.7 If the coolant solution has to be drained or removed for major vehicle repairs, for example, milling or warped heads, engine overhaul, etc.

6.6.8 If the vehicle coolant solution properties significantly change, that is, cooling system contamination, coolant solution dilution, addition of cooling system additives, etc.

NOTE 5—If a vehicle falls out of the fleet testing requirements, that same vehicle can be rebuilt and restarted providing the fleet is not more than 8,047 km (5,000 miles) into the test. Replacement or additional matched vehicles conforming to the requirements specified previously may be added to the fleet test provided the original fleet is not more than 8,047 km (5,000 miles) into the test. Vehicles that are run together in a fleet test should be started at the same time so that all the vehicles in the test experience similar environmental and driving conditions.

6.7 *Fleet Test Evaluation:*

6.7.1 *Beginning of Test*—Sample coolant solutions from each vehicle (60 mL [2 oz]) and conduct coolant analysis. The coolant analysis for each vehicle shall include pH, reserve alkalinity, glycol weight %, corrosion inhibitor content, contaminants (chloride, sulfate, etc.), and corrosion metal levels. Analysis for glycol degradation products is optional. The reference and test coolant solutions in the vehicles shall have a glycol concentration near 50 volume %. Report the results.

6.7.2 *Middle of Test (24,140 km [15,000 miles])*—Conduct mid-test coolant and coupon bundle analysis as follows:

6.7.2.1 Remove two coupon bundles from each vehicle cooling system, clean the coupons and measure weight losses or gains and report the average weight losses or gains for each coupon type.

6.7.2.2 Sample the coolant solutions from each vehicle (60 mL [2 oz]) and conduct coolant analysis. The coolant analysis for each vehicle shall include pH, reserve alkalinity, glycol weight %, corrosion inhibitor content, contaminants (chloride, sulfate, etc.), and corrosion metal levels. Analysis for glycol degradation products is optional. The reference and test coolant solutions in the vehicles shall have a glycol concentration near 50 volume %. Report the results.

6.7.2.3 Coolant samples and extra coupon bundles placed in the cooling system for information purposes may be removed at intervals more frequently than that specified within this specification but shall not be more frequent than 8,047 km (5,000 miles) intervals. If excessive amounts of coolant solution are removed from control or test vehicle cooling systems for analysis purposes or otherwise, the maximum allowable amount of coolant to top-off cooling systems may be exceeded.

NOTE 6—The intended application of conducting mid-test analysis is to determine the status of the fleet test. If the reference or test coolant solutions are performing outside the expected performance levels, vehicles can be withdrawn from the test, saving considerable amounts of time and expenses.

6.7.3 *End of Test (48,280 km [30,000 miles])*—Conduct end of test coolant, coupon, and vehicle component analysis on all vehicles that finish the fleet test as follows:

6.7.3.1 Remove the remaining four coupon bundles from each finishing vehicle cooling system, clean the coupons, measure the weight losses or gains and report the results as described in Practice D 2847. Results shall be reported on all finishing vehicles with a minimum of five vehicles being from the control vehicle group and five from the test vehicle group. Report the average weight loss or gain for each coupon type for each vehicle. Report the control vehicle group average coupon