Designation: D 6472 – 03

Standard Specification for Recycled Glycol Base Engine Coolant Concentrate for Automobile and Light-Duty Service[1]

This standard is issued under the fixed designation D 6472; 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 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 D15 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:* [2]

D 512 Test Methods for Chloride Ion in Water

D 516 Test Method for Sulfate Ion in Water

D 1119 Test Method for Ash Content of Engine Coolants and Antitrusts

D 1120 Test Method for Boiling Point of Engine Coolants

D 1121 Test Method for Reserve Alkalinity of Engine Coolants and Antitrusts

D 1122 Test Method for Specific Gravity of Engine Coolant Concentrates and Engine Coolants by the Hydrometer

D 1123 Test Method for Water in Engine Coolant Concentrate by the Karl Fischer Reagent Method

D 1126 Test Method for Hardness in Water

D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants of Antirusts for Testing Purposes

D 1177 Test Method for Freezing Point of Aqueous Engine Coolants

D 1193 Specification for Reagent Water

D 1287 Test Method for pH of Engine Coolants and Antirusts

D 1293 Test Methods for pH of Water

D 1384 Test Method for Corrosion Test for Engine Coolants in Glassware

D 1881 Test Method for Foaming Tendencies of Engine Coolants in Glassware

D 1882 Test Method for Effect of Cooling System Chemical Solutions on Organic Finishes for Automotive Vehicles

D 1888 Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water[3]

D 2570 Test Method for Simulated Service Corrosion Testing of Engine Coolants

D 2809 Test Method for Cavitation Corrosion Erosion-Corrosion Characteristics of Aluminum Pumps with Engine Coolants

D 2847 Practice for Testing Engine Coolants in Car and Light Truck Service

D 3306 Specification for Ethylene Glycol Base Engine Coolant for Automobile and Light-Duty Service

D 3634 Test Method for Trace Chloride Ion in Engine Coolants

D 4327 Test Method for Anions in Water by Chemically

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

Current edition approved Nov. 1, 2003. Published January 2004. Originally approved in 2000. Last previous edition approved in 2000 as D 6472–00.

[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.

[3] Discontinued. See *1991 Annual Book of ASTM Standards*, Vol 11.01.

Suppressed Ion Chromatography

D 4340 Test Method for Corrosion of Cast Aluminum Alloys in Engine Coolants Under Heat-Rejecting Conditions

D 4656 Specification for Prediluted Aqueous Ethylene Glycol Base Engine Coolant (50 Volume % Minimum) for Automobile and Light-Duty Service

D 4725 Terminology for Engine Coolants

D 5216 Specification for Propylene Glycol Base Engine Coolant for Automobile and Light-Duty Service

D 5827 Test Method for Determination of Chloride in Engine Coolant by Ion Chromatography

D 6129 Test Method for Silicon in Engine Coolant Concentrates by Atomic Absorption Spectroscopy

D 6130 Test Method for Determination of Silicon and Other Elements in Engine Coolant by Inductively Coupled Plasma-Atomic Emission Spectroscopy

D 6208 Test Method for Galvanostatic Measurement of Pitting Potential of Aluminum and Its Alloys

D 6257 Specification for Prediluted Aqueous Propylene Glycol Base Engine Coolant (50 Volume % Minimum) for Automobile and Light-Duty Service

D 6471 Specification for Recycled Prediluted Aqueous Glycol Base Engine Coolant (50 Volume % Minimum) for Automobile and Light-Duty Service

2.2 *Other Documents:*

SAE HS40 Maintenance for Automotive Engine Cooling System[4]

GM 6043M —Automotive Engine Coolant Concentrate—Ethylene Glycol Type[5]

GM 1825M —Automotive Engine Coolant Concentrate—Ethylene Glycol; §3.11 Storage Stability and Compatibility[6]

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 available[7]. 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.

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)	163 (325) min	152 (305) min	D 1120
50 volume % in distilled water	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 5827
Water, mass %	5 max	5 max	D 1123
Reserve alkalinity, mL	Report[B]	Report[B]	D 1121

[A] Some precipitate may be observed at the end of the test. This should not be cause for rejection.

[B] Agreed value between the supplier and the customer.

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

[5] Applicable rewrite available in Annex A2.

[6] Applicable rewrite available in Annex A3.

[7] *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.

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± 1°C (−34 ± 2°F) for a 50 volume % ethylene glycol base reference coolant solution or a freeze point of −32 ± 1°C (−26 ± 2°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 ± 1°C (−34 ± 2°F) for a 50 volume % ethylene glycol base test coolant solution or a freeze point of −32 ± 1°C (−26 ± 2°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

weight losses or gains from the individual control vehicle average coupon weight losses or gains. Also, report the test vehicle group average coupon weight losses or gains from the individual test vehicle average coupon weight losses or gains. Conduct statistical analysis on the coupon weight losses or gains for the fleet test and report the results at the 95th percentile confidence level. The T-test and F-test may be used to conduct statistical analysis on the fleet test results.

6.7.3.2 Sample the coolant solutions from each test vehicle (60 mL [2 oz] minimum) and conduct coolant sample 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.

6.7.3.3 Evaluate each of the control and test vehicle cooling system components in accordance with Practice D 2847. Additional to Practice 2847, the cooling system components of each finishing vehicle, unless otherwise specified, shall be photographed and evaluated as described in the following subsections.

(a) Radiator—Section each radiator, visually evaluate and report on the tube and tube-end deposits, erosion/corrosion, tube pitting, and header crevice corrosion.

(b) Water Pump—Disassemble each water pump and rate the pump impeller and housings in accordance with Test Method D 2809. Average the ratings for the test vehicles and report the result. Average the ratings for the control vehicles and report the result.

(c) Cylinder Head(s)—Section each head from a minimum of three control vehicles and three test vehicles near an exhaust port and a valve bridge. The sectioning of each head shall be in the same location for comparison evaluation purposes. Evaluate each head for deposits, pitting, corrosion, and discoloration on the coolant side of the exhaust valve ports and valve bridge areas. Also, evaluate each head for general corrosion. In this context, corrosion means pitting, etching, copper plating, metal surface phenomena, erosion, cavitation, or crevice corrosion.

6.8 *Fleet Test Performance Specifications*:

6.8.1 *Metal Coupons*—For each metal test coupon type, the test vehicle group average coupon weight loss shall be less than or equal to the maximum weight losses specified in the following subsections or shall be less than or equal to the average coupon weight losses obtained from the control vehicle group, whichever is greater for each coupon type (see Note 7).

6.8.1.1 *Copper Weight Loss*—20 mg/coupon max.

6.8.1.2 *Solder Weight Loss*—40 mg/coupon max.

6.8.1.3 *Brass Weight Loss*—20 mg/coupon max.

6.8.1.4 *Steel Weight Loss*—20 mg/coupon max.

6.8.1.5 *Cast Iron Weight Loss*—20 mg/coupon max.

6.8.1.6 *Cast Aluminum Weight Loss*—40 mg/coupon max.

NOTE 7—If the average weight loss for one or more coupon types in the control vehicle group is higher that the maximums specified in 6.8.1.1-6.8.1.6, then the average weight loss obtained for the control vehicle group for that metal coupon or coupons takes precedence and becomes the maximum allowed weight loss for that metal coupon type. Otherwise, the maximums specified in 6.8.1.1-6.8.1.6 apply.

6.8.2 *Radiator*—The radiators from the test vehicle group shall exhibit similar or less deposit formation in the radiator tubes and at the tube ends, erosion/corrosion, and header crevice corrosion than radiators from the control vehicle group.

6.8.3 *Water Pump Cavitation/Erosion Rating*—The average rating for test vehicle water pumps shall be a minimum of 8 or shall be equal to or greater than the obtained average control vehicle water pump rating.

6.8.4 *Cylinder Heads*—The cylinder heads from the test vehicle group shall exhibit similar or lesser amounts of deposits, pitting, and corrosion on the coolant side of the exhaust ports and valve bridges than the heads from the control vehicle group. They shall also exhibit similar or less general corrosion in the head cooling system passageways than that generated in the heads from the control vehicle group. Report any head surface discoloration on the coolant side of the exhaust ports and valve bridges.

6.9 *Fleet Testing Coordinator*—Either the fleet test managing company, or coordinator name, or both, and contact information shall be reported and included with fleet testing results and documentation.

NOTE 8—Inclusion of either the fleet test managing company, or coordinator information, or both, provides a means for the customer to verify and validate test results provided by the supplier. Generally, it is the supplier's responsibility to locate qualified parties to coordinate the fleet test and conduct evaluations; however, this may be negotiated and agreed upon between the customer and supplier.

6.10 *Fleet Test Cooling System Component Evaluation*—The cooling system components that require evaluation (radiators, water pumps, and heads) within Section 6 shall be evaluated and reported on by an independent laboratory with automotive and ASTM coolant testing and evaluation experience.

6.11 *Fleet Test Cooling System Component Storage*—The cooling system components that are evaluated in this fleet test protocol shall be kept for a minimum of one year for reference purposes. The components shall be kept in a suitable location or environment that prevents or minimizes component deterioration or degradation beyond that obtained from testing.

7. Other Requirements

7.1 The recycled glycol base engine coolant concentrate shall visually provide a similar or smaller amount of precipitate than the reference coolant concentrate (see Annex A2) in the test method for storage and stability and compatibility (see Annex A3). The comparison evaluation of the reference coolant concentrate versus the recycled coolant concentrate specified within this section takes precedence over limits or specifications detailed in Annex A3.

NOTE 9—When conducting testing for purposes of approval by a customer, generally it is recommended to have the testing conducted or supervised by an independent laboratory where reference materials are produced by a qualified laboratory or manufacturer.

8. Keywords

8.1 glycol base; light-duty engine coolant; recycled engine coolant concentrate; reference engine coolant concentrate; reference synthetic used coolant

ANNEXES

(Mandatory Information)

A1. REFERENCE SYNTHETIC USED COOLANT

A1.1 The reference synthetic used coolant described in the following is predilute and represents a synthetic used coolant feedstock. An equivalent used coolant may be used in evaluation of a coolant recycling process; however, it shall meet the requirements specified in A1.3.

A1.2 *Preparation of the Reference Synthetic Used Coolant*—The reference synthetic used coolant is to be prepared by blending the components listed in Table A1.1 in an appropriately sized stainless steel or plastic vessel with aggressive mixing or agitation capabilities. Blending vessels, tanks, mixing equipment, transfer pumps, hoses, and lines shall be clean, free of contaminants, and rinsed with deionized or distilled water prior to blending and use.

A1.2.1 Blend an appropriately sized batch with all the components listed in Table A1.1 in the order that the components are listed, with the exception of the SAE 5W30 engine oil.

A1.2.2 Once all of the components listed in Table A1.1 have been blended together, with the exception of the SAE 5W30 engine oil, adjust the pH of the solution to 8.0 ± 0.2 with sodium hydroxide (50 weight % aqueous sodium hydroxide may be used).

A1.2.3 Add the SAE 5W30 engine oil to the blend and vigorously mix. Complete emulsification of the engine oil is not necessary, and it is expected that oil will rise to the surface of the final blend.

A1.3 Blended batches of reference synthetic used coolant shall be tested for ingredient presence and quality by conforming to the parameters in Table A1.2 after being filtered through a 0.7-μ filter.

A1.4 Reference Synthetic Used Coolant conforming to the above specifications is suitable for use to evaluate a coolant recycling process. To evaluate a coolant recycling process or technology, the reference synthetic used coolant shall be recycled within 30 days from when it was made and shall be thoroughly agitated immediately prior to recycling.

NOTE A1.1—The reference synthetic used coolant has been, and is currently being, used by industry to evaluate coolant recycling processes. This coolant significantly exceeds corrosion weight loss specification limits when tested according to D 1384 and D 4340. A research report presenting the test data will be generated after the specification has been adopted by ASTM. When the research report as been generated, this note will be revised.

TABLE A1.1 Reference Synthetic Used Coolant Components[A]

Material	Ethylene Glycol Base Weight %	Propylene Glycol Base Weight %
Glycol[B]	52.5250	50.7236
Water[C]	47.0972	48.8986
Sodium tetraborate ($Na_2B_4O_7 \cdot 5H_2O$)	0.0140	
Sodium sulfate (Na_2SO_4)	0.0419	
Glycolic acid ($C_2H_4O_3$)	0.1100	
Formic acid (CH_2O_2)	0.0300	
Acetic acid, glacial ($C_2H_4O_2$)	0.0100	
Sodium chloride (NaCl)	0.0324	
Sodium nitrate ($NaNO_3$)	0.0110	
Copper chloride ($CuCl_2 \cdot 2H_2O$)	0.0009	
Iron sulfate ($FeSO_4 \cdot 7H_2O$)	0.0050	
Aluminum nitrate ($Al(NO_3)_3 \cdot 9H_2O$)	0.0028	
Lead nitrate ($Pb(NO_3)_2$)	0.0008	
Silicate stabilizer (Dow Corning Q1-6083)[D]	0.0100	
Sodium metasilicate ($Na_2SiO_3 \cdot 5H_2O$)	0.0140	
Surfactant (BASF Pluronic L61)[D]	0.0100	
SAE 5W30 Engine Oil[E]	0.0850	

[A] Materials listed are to be ACS reagent grade, or equivalent, unless otherwise specified. Materials may be substituted for alternative species materials provided the ionic concentrations of important are maintained. Table A1.2 lists the ionic species minimums and maximums allowed.

[B] A high quality technical grade ethylene or propylene glycol shall be used.

[C] Water quality shall conform to Specification D 1193 Type IV water requirements.

[D] Chemical equivalents may be used.

[E] A commercially available SAE 5W30 engine oil may be used. The engine oil concentration represents oil contamination in used coolants and was arrived at by surveying coolant recyclers in the industry.

TABLE A1.2 Reference Synthetic Used Coolant Property Requirements

Property or Material	Ethylene Glycol Base Parameter	Propylene Glycol Base Parameter	Suggested Analysis Method
Chloride (Cl^-)	200 mg/L min.		D 5827
Sulfate (SO_4^{-2})	300 mg/L min.		D 5827
Glycolic acid	1100 mg/L min.		D 5827
Formic acid	300 mg/L min.		D 5827
Acetic acid	100 mg/L min.		D 5827
Tetraborate ($B_4O_7^{-2}$)	300 mg/L max.		D 5827, D 6129, D 6130
Nitrate (NO_3^-)	400 mg/L max.		D 5827
Silicate (SiO_3^{-2})	50 mg/L max.		D 5827, D 6129, D 6130
Iron (Fe)	10 mg/L min.		D 6129, D 6130
Copper (Cu)	3 mg/L min.		D 6129, D 6130
Aluminum (Al)	2 mg/L min		D 6129, D 6130
Lead (Pb)	5 mg/L min.		D 6129, D 6130
pH	8.0±0.2 max.		D 1287
Visual	may have some cloudiness or phase separation due to the oil. No solids precipitation is permitted.		visual
Freeze point	-37°C (-34°F) or lower	-32°C (-26°F) or lower	D 1177

A2. REFERENCE ENGINE COOLANT CONCENTRATE—ETHYLENE GLYCOL FOR LIGHT DUTY SERVICE[8]

A2.1 Scope

A2.1.1 This specification covers a reference ethylene glycol type engine coolant intended to protect automotive engine cooling systems from corrosion and freezing. The requirements are intended to insure that coolants give satisfactory performance when used at recommended concentrations. This specification also provides a coolant concentrate that is to be used as reference coolant in performance testing listed earlier in this specification.

[8] GM 6043M *Automotive Engine Coolant Concentrate—Ethylene Glycol Type*, General Motors Engineering Standards, Revision: December, 1984.

TABLE A2.1 Reference Coolant Concentrate Formulation[A]

Material	Weight %
Ethylene Glycol[B]	95.53
Sodium Nitrate ($NaNO_3$)	0.10
Sodium molybdate (Na_2MoO_4-$2H_2O$)	0.20
Sodium tetraborate (Na_2B_4O $5H_2O$)	0.40
Sodium silicate (Liquid SiO_3), SiO_2/Na_2O weight ratio of 3.22	0.30
Phosphoric acid (85 % H_3PO_4)[C]	0.15
Sodium mercaptobenzothiazole (50 % solution)	0.50
Sodium tolytriazole (50 % solution)	0.20
Sodium hydroxide	0.235
Water (Type II, Specification D 1193)	2.30
Silicate sabilizer (Dow Corning Q1 Specification-6083)[D]	0.06
Surfactant (BASF Pluronic L61)[D]	0.02
Green dye[E]	0.005

[A] The values specified for chemical additives are intended as target values, as a subsequent analysis of a formulated product should provide values that are equal to or greater than those shown. Sodium hydroxide is used to adjust pH and the concentration may have to be varied to satisfy the pH requirements of the specification. The stabilizer improves storage stability of the silicate, allowing sufficient time between additions to obtain complete mixing.

[B] A high quality technical grade ethylene or propylene glycol shall be used. A maximum of 15 % of the ethylene glycol can be replaced with other glycols, provided the formulated product satisfies the requirements under A2.3. A maximum of 1.0 % of the propylene glycol can be replaced with other glycols, provided the formulated product satisfies the requirements under A2.3.

[C] Alternatively, sodium phosphate dibasic and tribasic may be used with equivalent PO_4 content and appropriate adjustment of sodium hydroxide and water additions.

[D] Chemical equivalents may be used.

[E] 0.002 % Fluorescein (Uranine-C) and 0.003 % Alizarine Cyanone Green B Extra (Acid Green 25).

TABLE A2.2 Reference Coolant Concentrate Properties

Property	Acceptable Range	ASTM Test Method
Relative density 15.5°C (60/60°F)	1.120 to 1.130	D 1122
Reserve alkalinity, mL	10.0 min	D 1121
Chloride, ppm	25 max	D 3634
Freezing point,°C (°F)		D 1177
At 50.0 volume % in distilled H_2O	-37 (-34) or lower	
pH at 50.0 volume % in distilled H_2O	10.0 to 11.0	D 1287[A]

[A] The pH is subject to a slight change with time. As the concentrate ages, the pH measured immediately after dilution of a sample may be higher than it was when the concentrate was first made; however, the pH of a diluted sample may decrease with elapsed time after dilution.

A2.2 Formulation

A2.2.1 The coolant concentrate shall be prepared by mixing the materials in the weight percent indicated in Table A2.1.

A2.2.2 *Stability*—The reference coolant concentrate shall be used within one year of being made and should be mixed prior to use if left standing for long periods of time.

A2.3 Properties

A2.3.1 The coolant concentrate, when formulated as specified, shall meet the following properties as determined by methods listed in Table A2.2.

A2.3.2 Sampling shall be performed in accordance Test Method D 1176.

NOTE A2.1—Propylene glycol may be substituted for the ethylene glycol in the reference engine coolant concentrate. A maximum of 1 % of

the propylene glycol can be replaced with other glycols. If propylene glycol is substituted for ethylene glycol, the acceptable range for relative density is 1.030 to 1.065 and the freezing point requirement is -32°C (-26°F) or lower. All other property requirements shall be met.

A3. TEST METHOD FOR STORAGE STABILITY AND COMPATIBILITY[9]

A3.1 Samples of the coolant concentrate shall show no separation or precipitation when diluted with a synthetic hard water and tested as follows: Prepare the hard water by adding 275 mg of $CaCl_2$ to one liter of the synthetic hard water described in Test Method D 1384. Mix 100 mL of the coolant concentrate plus 100 mL of the synthetic hard water (room temperature) in a 250 mL beaker, and allow to stand in the dark 24 h. Make a second mixture, as above, heat to 82°C (179.6°F) and allow to cool to room temperature and to stand in the dark 24 h. Slight cloudiness is permitted, but an excessive amount of precipitate is considered to interfere with bulk storage and use of the mixtures.

A3.2 Compatibility with other mixtures shall be similarly determined at the request of the purchaser.

A3.3 Allow an undiluted sample of the coolant to stand for 24 h. Any separation into phases shall disqualify the engine coolant concentrate for factory-fill but does not prevent its use as a package item, provided the package states that it should be shaken before adding to the cooling system. Some minor separation, not sufficient to form a continuous film over the entire surface, is to be considered normal.

NOTE A3.1—This test method is an exact quotation from the General Motors engine coolant concentrate engineering standards and has not been modified from its original form for copyright and usage permission purposes. Evaluation of the reference engine coolant versus the recycled engine coolant is specified in 7.1, which takes precedence over the limits and specifications detailed within this annex.

[9] GM 1825M *Automotive Engine Coolant Antifreeze Concentrate—Ethylene Glycol*, General Motors Engineering Standards, Revision: April, 1985.

APPENDIXES

(Nonmandatory Information)

X1. COOLING SYSTEM MAINTENANCE

X1.1 *Filling the Cooling System*:

X1.1.1 Before installing engine coolant, the cooling system should be inspected and necessary service work completed.

X1.1.2 Cooling system fill should consist of coolant concentrate and water.

X1.1.3 When preparing solutions, the water should be of such quality that it does not contain excessive solids, hardness salts, sulfates, or chlorides. In the absence of specific recommendation from the engine or vehicle manufacture, see Table X1.1. Contact you local water department, the responsible government agency, or submit a water sample for analyses, if there is a question on water quality.

X1.1.4 The recommended coolant concentration is 40 to 70 volume %.

X1.2 *Essential Spring and Fall Cooling System Service*: [10]

X1.2.1 Check coolant concentration. (See

X1.2.2 Check coolant level and condition. Replace coolant at service intervals recommended by engine manufacturer, vehicle manufacturer, or designated service organization. Follow recommended practices.

X1.2.3 Pressure test system for leaks (preferably when cold).

X1.2.4 Test pressure cap and inspect radiator filler neck.

X1.2.5 Inspect hoses and tighten hose connections if necessary.

X1.2.6 Inspect drive belts and check for proper tension.

X1.2.7 Test thermostat if the engine is running too hot or too cold. Replace with a thermostat recommended by the manufacturer.

X1.3 Premix recycled coolant concentrate and water before adding to the cooling system.

X1.4 When preparing additions or when replacing the coolant in the engine system, use only clean, low mineral content water or coolant flush equipment that is able to properly collect the used coolant. ASTM MNL 6[11] provides suggestions for proper water quality limits. Additions to the cooling system should be done with 50/50 engine coolant concentrate complying with this specification, Specification

[10] *Engine Cooling System Care*, Chemical Specialties Manufacturers Association (CSMA), Washington, DC., Ninth revision, 1984. Available from CSMA, 1913 I St., NW, Washington, DC 20006.

TABLE X1.1 Suggested Water Quality Limits[A]

Property	Specific Values	ASTM Test Method
Total solids, ppm (grains/gal)	340 (20) max	D 1888
Total hardness, ppm (grains/gal)	170 (10) max	D 1126
Chloride, ppm (grains/gal)	40 (2.4) max	D 512, D 4327
Sulfate, ppm (grains/gal)	100 (5.9) max	D 516, D 4327
pH	5.5 to 9.0	D 1293

[A] Adopted from a survey by the Committee D-15 Water Quality Task Force.

[11] ASTM Manual Series: MNL 6 *Engine Coolants and Cooling System Chemicals*, 1989, ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.

D 3306 or D 5216 and water, meeting at least the requirements outlined in this appendix or prediluted aqueous glycol base engine coolant complying with Specifications D 4656, D 6257, or D 6471. (**Warning**—Do not remove the radiator pressure cap when the engine is hot. The cooling system will likely be under pressure. When the engine has cooled, carefully turn the cap to the first notch to vent the system pressure or use a cooling system pressure relieving tool, then remove. If the coolant overflows when the cap is vented, immediately re-tighten and permit the system to cool further.)

X1.5 Additional information can be found in ASTM MNL 6.

X1.6 Other sources of information are SAE-HS40 and *Engine Cooling System Care*.[10]

X2. DETERMINATION OF FREEZE POINT

X2.1 If propylene glycol (PG) base coolants and ethylene glycol (EG) base coolants are mixed in a cooling system, problems may result when attempting to determine freezing point in the field. The hydrometers used in North America are calibrated to the higher relative density of ethylene glycol base coolants. These hydrometers cannot be used to determine the freeze point of propylene glycol base engine coolants or mixtures of PG and EG coolants. Using this type of hydrometer to determine the freeze point of a PG base coolant is likely to result in a high coolant to water mix ratio (80/20), which in turn may cause engine and cooling system problems. A hydrometer specifically calibrated to the relative density of propylene glycol shall be used to determine the freezing point of propylene glycol base coolants. A convenient and preferred means of determining the freeze points for PG coolants or mixtures of PG and EG coolants is by refractometer. Test strips may also be used. Table X2.1 lists methods for determining the freeze point of propylene glycol base engine coolants when used either alone or when mixed with ethylene glycol base coolants. A refractometer provides an accurate method for measuring freezing points. Dip-and-read test strips will provide only an approximation of freeze points.

TABLE X2.1 Methods for Determining Freezing Points

Method	Base Glycol		
	Ethylene	Propylene	Mixture
Hydrometer	yes[A]	yes[B]	no
Refractometer	yes	yes[C]	yes[D]
Test Strips	yes	yes	yes

[A] Conventional field service hydrometers calibrated for use with ethylene glycol.
[B] Hydrometer specifically calibrated for use with PG coolants.
[C] Shall be a refractometer with a propylene glycol freeze point scale.
[D] Approximate freeze point determinations can be made for mixtures of EG and PG base coolants by calculating the average of readings on each scale. The freeze point determined by this method will be within ±4°C (±7°F), regardless of whether the coolant is all EG, PG, or a mixture of both.

X2.2 It is recommended that propylene glycol base coolant containers be labeled with an appropriate cautionary statement to alert the user to the differences described in X2.1. It is also recommended that a peel-off label be attached to the filler-neck of the cooling system to advise the user that the system has been charged with a propylene glycol base coolant.

X3. RECYCLED COOLANT ADVISORY

X3.1 This recycled coolant concentrate specification is based on performance experience developed in the industry from the recycling of used engine coolants. Recycled engine coolant concentrates originating from sources other than light, medium, heavy-duty vehicles, and mixtures thereof, as used coolant streams, have not been fully studied.

X3.2 Committee D-15 is in the process of developing specifications for redistilled glycols from used engine coolant and non-engine coolant sources to be used in engine coolant formulation and application.