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An American National Standard

Standard Specification for ASTM Reference Fluid for Coolant Tests[1]

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1. Scope

1.1 This specification covers a reference ethylene glycol-base test fluid to be used in providing base line data for ASTM coolant test procedures.

1.2 *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 501 Test Methods of Sampling and Chemical Analysis of Alkaline Detergents[2]

D 538 Specification for Trisodium Phosphate[2]

D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals[3]

D 1078 Test Method for Distillation Range of Volatile Organic Liquids[4]

D 1119 Test Method for Ash Content of Engine Coolants and Antirusts[3]

D 1120 Test Method for Boiling Point of Engine Coolants[3]

D 1121 Test Method for Reserve Alkalinity of Engine Coolant and Antirusts[3]

D 1122 Test Method for Density or Relative Density of Engine Coolant Concentrates and Engine Coolants by the Hydrometer[3]

D 1123 Test Method for Water in Engine Coolant Concentrate by the Karl Fischer Reagent Method[3]

D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes[3]

D 1177 Test Method for Freezing Point of Aqueous Engine Coolants[3]

D 1287 Test Method for pH of Engine Coolants and Antirusts[3]

D 1384 Test Method for Corrosion Test for Engine Coolants in Glassware[3]

D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products[4]

D 1881 Test Method for Foaming Tendencies of Engine Coolants in Glassware[3]

D 3634 Test Method for Trace Chloride Ion in Engine Coolants[3]

D 5827 Test Method for Analysis of Engine Coolant for Chloride and Other Anions by Ion Chromatography[3]

D 5931 Test Method for Density and Relative Density of Engine Coolant Concentrates and Aqueous Engine Coolants by Digital Density Meter[3]

E 202 Test Methods for Analysis of Ethylene Glycols and Propylene Glycols[3]

3. Chemical Composition Requirements

3.1 The reference test fluid concentrate shall be prepared to conform to the requirements as to chemical composition prescribed in Table 1.

4. Ingredient Requirements

4.1 The materials used to prepare the reference test fluid shall meet the requirements given in Annex A1-Annex A5.

5. Significance and Use

5.1 The data obtained for the reference test fluid are intended to be used by laboratory personnel to determine their capability to perform tests properly. If a particular determination does not fall within the prescribed limits, it has to be assumed that an error occurred in the application of the test procedure.

5.2 The coolant composition given in this specification is not intended to be a commercial product.

6. Chemical and Physical Requirements

6.1 The formulated reference test fluid concentrate shall conform to the requirements for physical and chemical properties prescribed in Table 2.

7. Performance Requirements

7.1 The formulated reference test fluid concentrate shall conform to the requirements for laboratory test performance

[1] This specification is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.01 on Reference Test Materials.

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[2] *Annual Book of ASTM Standards,* Vol 15.04.

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

[4] *Annual Book of ASTM Standards,* Vol 06.03.

TABLE 1 Chemical Composition Requirements

NOTE 1—The reference coolant shall be colored blue-green using Alizarine Cyanine Green G Extra 100 % added in the proportion of 0.3 g of dye/gal of coolant.

Ingredient	Mass %	lb/100 gal[A]	kg/m^3
Ethylene glycol	89.86	847.9	1016.0
Diethylene glycol	5.00	47.2	56.5
Sodium tetraborate, pentahydrate	3.06	28.9	34.6
Trisodium phosphate, dodecahydrate	0.30	2.8	3.4
Sodium mercaptobenzothiazole solution (50 mass % aqueous)	0.40	3.8	4.5
Pluronic L-61[B]			
Water[C]	0.02	0.2	0.2
	1.36	12.8	15.4

[A] Based on a test fluid relative density of 1.133 at 60/60°F (15.5/15.5°C).

[B] A nonionic polyol manufactured by BASF Corporation, 100 Cherry Hill Rd., Parsippany, NJ 07054.

[C] Calculated value; the total water content (water originally present in the base materials, added water, water of hydration, and water of reaction and quantitative interference by the reaction of the reagent (in Test Method D 1123) with the ingredients) should be adjusted to 4.0 ± 0.2 mass % as the final step in the preparation.

prescribed in Table 3.

8. Sampling

8.1 To obtain a sample of the concentrated reference test fluid from the storage container, allow the material to come to room temperature (not below 68°F (20°C)) and shake well before withdrawing the sample.

8.2 All aqueous solutions to be used for test purposes shall be prepared in accordance with Section 5 of Test Method D 1176.

9. Mixing Procedure

9.1 Weigh the ingredients according to the batch size required.

9.2 Mix the ethylene and diethylene glycols.

9.3 Dissolve the sodium tetraborate in the glycol mixture using continuous agitation.

9.4 Dissolve the trisodium phosphate in the above solution.

9.5 Add the sodium mercaptobenzothiazole solution and continue agitating the mixture until it is homogeneous.

9.6 Slurry the dye into a convenient portion of the solution; then add the dye slurry to the formulation.

9.7 Add the Pluronic L-61 antifoam and mix thoroughly.

9.8 Determine the water content of the formula in accordance with Test Method D 1123 and adjust to 4.0 ± 0.2 mass % by the addition of distilled water.

10. Precision and Bias

10.1 For statements on the precision and bias of the various test methods for physical and chemical properties used in this specification, refer to the appropriate ASTM standards.

10.2 For statements on the precision and bias of the performance tests used in this specification, see the footnotes to Table 3.

10.3 It should be noted that the requirements listed in Table 2 on physical and chemical properties, and in Table 3 on performance, are based on the results of many different laboratories testing the same batch of reference test fluid.

11. Packaging and Storage

11.1 The test fluid concentrate may be affected by light. Therefore, after preparation, it should be packaged in opaque plastic containers or plastic lined metal cans.

11.2 Solutions prepared for testing should also be stored in opaque containers or kept in a cool, dark place to prevent the formation of flocculent precipitates.

11.3 Except when taking a sample, containers should be closed to avoid absorption of moisture from the air.

12. Keywords

12.1 chemical composition; engine coolant; performance requirements; specification; test fluid

TABLE 2 Physical and Chemical Requirements

Property	Requirements		ASTM Test Method
	min	max	
pH, concentrate	6.1	6.3	D 1287
33 volume % solution	7.7	8.0	
50 volume % solution	7.5	7.8	
Reserve alkalinity, mL	26.5	27.5	D 1121
Water content, weight %	3.8	4.2	D 1123
Freezing protection:			D 1177
Concentrate	−23°C (−10°F)	−25°C (−13°F)	
33 volume % solution	−18°C (0°F)	−19°C (−2°F)	
50 volume % solution	−36°C (−33°F)	−38°C (−36°F)	
Relative Density at 15.6°C	1.131	1.134	D 1122, D 5931
at 20°C	1.129	1.132	D 891
Boiling point, °C (°F)	330 (166)	340 (171)	D 1120
Ash, weight %	1.4	1.6	D 1119
Chloride, ppm	–	25	D 3634, D 5827

TABLE 3 Performance Requirements[A]

Test	Mass Loss, max, mg/Specimen[B]	ASTM Test Method
Corrosion in glassware		D 1384
Copper	5	
Solder	5	
Brass	5	
Steel	5	
Cast iron	5	
Aluminum	15	
Foaming	volume 75 mL, max[C] break time 5 s, max[D]	D 1881

[A] Average data for triplicate tests.

[B] The multilaboratory standard deviation has been found to be 1.8 mg for all metals that lose an average of less than 3 mg per specimen. Therefore, results of two properly conducted tests from two different laboratories on samples of the same lot of reference test fluid should not differ by more than 4.1 mg, provided the average loss is less than 3 mg per specimen. The multilaboratory standard deviation has been found to be 59 % of the obtained value on metals that lose an average of more than 3 mg per specimen. Therefore, results of two properly conducted tests from two different laboratories on samples of the same lot of reference test fluid should not differ by more than 167 %, provided the average loss is greater than 3 mg per specimen.

[C] The multilaboratory standard deviation for foam volume has been found to be 12.0 mL. Therefore, results of two properly conducted tests from two different laboratories on samples of the same lot of reference test fluid should not differ by more than 33.9 mL.

[D] The multilaboratory standard deviation for foam break time has been found to be 0.7 s. Therefore, results of two properly conducted tests from two different laboratories on samples of the same lot of reference test fluid should not differ by more than 2.0 s.

ANNEXES

(Mandatory Information)

A1. SPECIFICATION FOR ETHYLENE GLYCOL

Property	Requirements		ASTM Test Method
	min	max	
Relative Density at 15.6/15.6°C (60/60°F)	1.115	1.118	D 891[A]
20/20°C at (68/68°F)	1.113	1.116	
Distillation, 760 mm, °C (°F) initial boiling point	88	...	D 1078[A]
dry point	...	113	
Acidity (as mass % acetic acid)	...	0.01	D 1613[A]
Water, mass %	...	0.5	D 1123

Property	Requirements min	max	ASTM Test Method
Chloride, ppm	...	5	D 3634, D 5827

[A]These methods are referenced in Test Methods E 202.

A2. SPECIFICATION FOR DIETHYLENE GLYCOL

Property	Requirements min	max	ASTM Test Method
Relative Density at 60/60°F (15.6/15.6°C)	1.119	1.122	D 891[A]
68/68°F at (20/20°C)	1.117	1.120	
Distillation, 760 mm, °C initial boiling point	240	...	D 1078[A]
dry point	...	250	
Acidity (as mass % acetic acid)	...	0.005	D 1613[A]
Water, mass %	...	0.20	D 1123
Chloride, ppm	...	5	D 3634, D 5827

[A]These methods are referenced in Test Methods E 202.

A3. SPECIFICATION FOR SODIUM TETRABORATE, PENTAHYDRATE[A]

Chemical Analysis[A]:	min	max
Sodium oxide (Na_2O), mass %	21.3	21.7
Boron trioxide (B_2O_3), mass %	47.8	48.7
Water of crystallization, mass %	28.5	30.9
Equivalent anhydrous borax ($Na_2B_4O_7$), mass %	69.1	70.5
Chloride	...	0.05

[A]See Test Methods D 501.

A4. SPECIFICATION FOR TRISODIUM PHOSPHATE DODECAHYDRATE[A]

Chemical Requirements[A]:	min	max
Phosphoric anhydride (P_2O_5), mass %	18.1	...
Total alkalinity to methyl orange as Na_2O, mass %	16.0	19.0
Chloride	...	0.1

[A]See Test Methods D 501 and Specification D 538.

A5. SPECIFICATION FOR SODIUM MERCAPTOBENZOTHIAZOLE SOLUTION (50 %)[A]

Chemical Requirements[A]:	min	max
Sodium MBT content, mass %	49.0	51.0
Free alkalinity as NaOH, mass %	...	0.5
Chloride, mass %	...	0.1

[A]See Annex A6.

A6. GRAVIMETRIC DETERMINATION OF SODIUM MERCAPTOBENZOTHIAZOLE

A6.1 Reagents

A6.1.1 *Ammonium Hydroxide* (*relative density 0.90*)—Concentrated ammonium hyroxide (NH_4OH.

A6.1.2 *Silver Nitrate Solution* (*16 g/L*)— Dissolve 0.4 g of silver nitrate crystals in 25 mL of distilled water.

A6.1.3 *Hydrochloric Acid* (*1 + 1*)—Mix 1 volume of HCl (relative density 1.19) with 1 volume of water.

A6.2 Procedure

A6.2.1 Weigh into a 250-mL beaker 0.5 to 0.6 g of the sodium mercaptobenzothiazole solution and record the weight to the nearest 0.1 mg.

A6.2.2 Add 10 mL of concentrated ammonium hydroxide to the beaker and dilute the contents to 150 mL with distilled water.

A6.2.3 Add the 25 mL of silver nitrate solution to the ammoniacal solution while stirring.

A6.2.4 Place the beaker and contents in an oven at 60°C and allow the mixture to digest for 15 min.

A6.2.5 Remove and allow to cool to room temperature. (The elapsed time between precipitation and the start of filtration should be a minimum of 1 h.)

A6.2.6 Prepare a medium-frit Gooch crucible by placing in an oven for 15 min at 110°C. Remove, place in desiccator to cool to room temperature, and then record the mass of the crucible.

A6.2.7 Filter the precipitated silver mercaptobenzothiazole through the fritted crucible and wash with distilled water until no fumes of ammonia remain and the rinse liquid does not form a silver chloride precipitate with hydrochloric acid (1 + 1).

A6.2.8 Dry the crucible and precipitate to constant weight in an oven at 110°C (a minimum of 2 h drying).

A6.2.9 Record the weight of silver mercaptobenzothiazole to the nearest 0.1 mg.

A6.3 Calculation

A6.3.1 Calculate the percentage of sodium mercaptobenzothiazole (NaMBT) in solution as follows:

$$\text{NaMBT, mass \%} = (A \times 189.23 \times 100)/(B \times 274.1) \quad \text{(A6.1)}$$

where:

A = grams of dried precipitate, and
B = grams of sample used.