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An American National Standard

Standard Test Method for Compatibility of Supplemental Coolant Additives (SCAs) and Engine Coolant Concentrates[1]

This standard is issued under the fixed designation D 5828; 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.

INTRODUCTION

Supplemental coolant additives (SCAs) are used to impart special properties, usually resistance to cavitation corrosion, to engine coolants used in diesel engines with replaceable cylinder liner sleeves. Engines with this design require additives that are not normally found in commercial engine coolant concentrates.

1. Scope

1.1 This test method covers determination of the compatibility of commercial SCA and commercial ethylene and propylene glycol engine coolant concentrates. This test method focuses on the solubility of specific chemical species formed in the engine coolant. The short duration of the test (24 h), among other restrictions, makes the test method of limited use for sorting out a variety of chemical compatibility problems in which a component of the SCA may react with a component of the coolant additive package. The test as currently written also does not deal with the issue of hard water compatibility, in which a component of the coolant or SCA additive package reacts with the hardness (Ca and Mg) to form a precipitate.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are 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:*

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

D 1193 Specification for Reagent Water[3]

D 1796 Test Method for Water and Sediment in Fuel Oils by the Centrifuge Method (Laboratory Procedure)[4]

D 3585 Specification for ASTM Reference Fluid for Coolant Tests[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *engine coolant concentrate*—an undiluted ethylene or propylene glycol containing additives and only a small amount of water, usually less than 5 %.

3.1.2 *reference engine coolant concentrate*—a standard material prepared according to the formulary given in Annex A2 of this test method. This material should not be confused with reference coolant in accordance with Specification D 3585.

3.1.3 *reference supplemental coolant additive (SCA)*—a standard SCA prepared according to the formulary given in Annex A1 of this test method.

3.1.4 *supplemental coolant additive*—a liquid or solid material that is added to a coolant at a specified concentration.

4. Summary of Test Method

4.1 A mixture of engine coolant concentrate and deionized water containing approximately twice the recommended concentration of SCA is heated to 88°C (190°F) for 24 h. The solution is centrifuged after returning to ambient temperature, and the amount of insoluble material is determined volumetrically and compared to the amount of insolubles obtained with a mixture of standard reference SCA and reference engine coolant.

5. Significance and Use

5.1 This test was developed to mimic the formation of insolubles observed in some heavy-duty diesel cooling systems during the mid 1980s. It measures the compatibility of SCA and coolant concentrate solutions according to their tendency

[1] This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.11 on Coolants for Heavy Duty Engines.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

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

[4] *Annual Book of ASTM Standards*, Vol 05.01.

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

to form insolubles in service.[6] Such insoluble materials may accumulate within a cooling system, restrict heat transfer through radiator cores, and contribute to the damage of components such as water pumps.

6. Apparatus

6.1 *Two-pan General Laboratory Balance*, 1 to 2-kg capacity.

6.2 *Centrifuge Tube*, 100-mL capacity in accordance with Test Method D 1796.

6.3 *Centrifuge*, capable of maintaining 500 rcf, with trunnions and specimen holders suitable for the tube described in 6.2.

6.4 *Constant Temperature Oil Bath*, or equivalent, capable of maintaining the test temperature at 88°C (190°F), within ±1°C (2°F).

6.5 *Condenser Tube*, glass, approximately 5-mm outside diameter by 3-mm inside diameter by 300-mm long.

6.6 *Rubber Stoppers*, to fit the centrifuge tube with a single hole for the glass condenser tube.

6.7 *Rubber Stoppers*, as above but without a hole.

6.8 *Graduated Cylinder*, 100-mL capacity to deliver.

6.9 *Pipette*, to deliver volumes from 1 to 10 mL in 1-mL increments.

6.10 *Analytical Balance*, for preparing reference materials and capable of weighing within an accuracy of ±0.2 mg or better.

7. Reagents and Materials

7.1 *Coolant Concentrate*, and SCA for evaluation.

7.2 *Reference SCA*, and coolant concentrate solutions (see Annex A1 and Annex A2).

7.3 *Deionized Water*, in accordance with Specification D 1193.

7.4 *Nichrome Wire*, or stainless steel wire.

7.5 *Filter Paper*, Whatman No. 4 or equivalent.

7.6 *Plastic Containers*, to store solutions. Polyethylene or polypropylene containers with screw caps are satisfactory.

8. Procedure

8.1 Compatibility testing of SCA shall be conducted using a ratio of 60 parts of coolant concentrate to 40 parts of a water-SCA mixture. The level of SCA in the total 60:40 mixture will be approximately twice the SCA manufacturer's recommended concentration.

8.2 Fill a 100-mL centrifuge tube to the 60-mL mark with coolant concentrate.

8.3 Determine the volume of water to be added based on the physical state and the recommended concentration of SCA to be evaluated. Add this volume of water to the centrifuge tube using a graduated cylinder. For example, if the SCA is a liquid to be added at the recommended concentration of 3 % by volume, twice the recommended concentration is 6 % or 6 mL. The volume of water to be added is 34 mL. This is 100 mL (volume of the centrifuge tube) less 60 mL (volume of coolant concentrate required) less 6 mL (volume of SCA required).

NOTE 1—Using hard water will greatly influence the amount of solubles formed. Testing the purity of the water with a conductivity meter is recommended.

NOTE 2—If the SCA is a solid, prepare a sufficient volume of a concentrated solution of the SCA in deionized water.

8.4 Pipette the required volume of SCA into the mixture of coolant concentrate and water. The sequence of mixing must be as follows: coolant concentrate, water, SCA solution. Cap with a solid rubber stopper and agitate thoroughly.

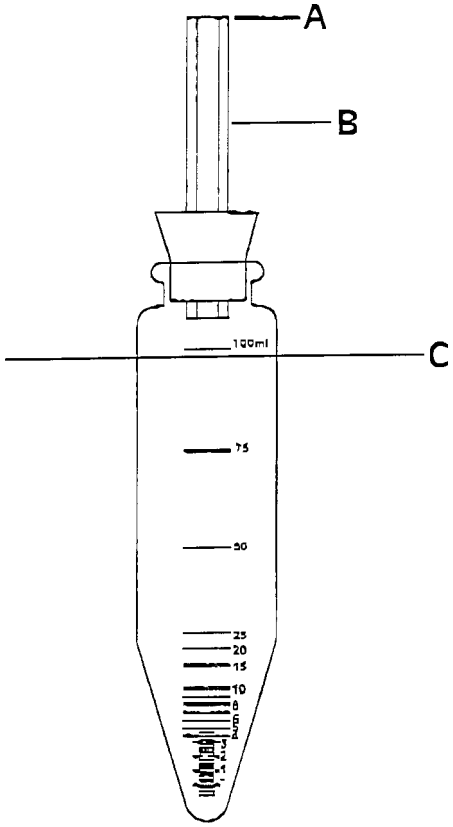
NOTE 3—Glycol and water mixtures exhibit a volume contraction due to the partial molal volume effect. The final volume of the mixture should be less than 100 mL, as indicated in Fig. 1.

8.5 In a similar manner, add 60 mL of reference coolant concentrate, 34 mL of deionized water, and 6 mL of reference SCA solution to a second 100-mL centrifuge tube, and agitate thoroughly. The reference coolant must be used within 30 days of preparation. Discard and prepare a new reference if any insoluble material is observed.

8.6 Replace the rubber stoppers with clean air condensers prepared by inserting a 300-mm (12-in.) length of glass tubing through a properly sized one-hole stopper.

8.7 Insert a length of dry Nichrome or stainless steel wire into the condenser past the bottom of the condenser tube. Immerse the centrifuge tube to the level of the solution in a constant temperature bath at 88°C (190°F) for 24 h (see Fig. 1).

NOTE 4—The purpose of the wire is to provide a means of directing condensate back to the centrifuge tube.



NOTE 1—(*A*) nichrome wire, (*B*) condenser tube, and (*C*) immersion level.

FIG. 1 Apparatus Assembly

[6] Hercamp and Hudgens, "Silicate Gelation in Heavy-Duty Engine Cooling Systems," Paper No. 852327, *Society of Automotive Engineers*, December 1985.

8.8 At the end of the heating period, remove the centrifuge tube and solution from the bath, and cool to room temperature (allow at least 1 h). Remove the air condenser stopper, and replace with a solid rubber stopper. Using a two-pan balance, balance the centrifuge tube, stopper, and sample against a centrifuge tube (with stopper) containing any suitable liquid.

NOTE 5—The centrifuge tube may be balanced against another centrifuge tube containing another sample. A few drops of a solution of 60 % reagent grade ethylene glycol and 40 % deionized water may be added to one of the tubes to equalize the mass of the tubes.

8.9 Centrifuge for 30 min at 500 rcf. Calculate the speed of the centrifugal rotation in r/min as follows:

$$\text{r/min} = 299\sqrt{\text{rcf}/r} \quad (1)$$

where:

r = radius in centimetres of the circle of rotation described by the tips of the tubes when in a rotating position, and
rcf = 500.

The following may be used as a simple means of checking the calculation:

Radius, cm (in.)	r/min
20.3 (8)	1480
25.4(10)	1325
36.5(12)	1210

NOTE 6—The following equation may be used when r is measured in inches:

$$\text{r/min} = 188\sqrt{\text{rcf}/r} \quad (2)$$

NOTE 7—When operating a centrifuge, tubes must be balanced properly and used in pairs to distribute the centrifugal forces evenly.

8.10 At the end of the centrifuging, stop the centrifuge and immediately read the volume of solids in the bottom of the tube, estimating to the nearest part of a millilitre as appropriate.

8.11 Clean the centrifuge tubes using the following procedure:

8.11.1 Shake the tubes to loosen the insoluble materials, and discard liquid and insolubles into a suitable container. Fill the tubes partially with tap water, and repeat as necessary.

NOTE 8—The insolubles may be loosened with the aid of an ultrasonic cleaner.

8.11.2 Rinse the inside of the tubes with 1:1 HCl solution, and discard washings.

8.11.3 Clean the tubes with a solution of detergent and water, brushing as needed.

8.11.4 Rinse the tubes three times with warm tap water.

8.11.5 Rinse once with acetone or isopropyl alcohol to remove water, and air dry.

9. Calculation and Interpretation of Results

9.1 The tendency to form insolubles for the SCA-coolant concentrate combination under evaluation is expressed in relation to the tendency to form insolubles for the reference SCA in a diluted reference coolant concentrate.

9.2 Calculate the ratio of the volumes of insolubles formed as follows:

$$\text{ratio} = A/B \quad (3)$$

where:

A = volume of insolubles formed in the test solution, and
B = volume of insolubles formed in the reference solution.

10. Report

10.1 Report the volume insolubles in millilitres for the test mixture, the volume insolubles in millilitres for the reference mixture, and the ratio calculated in 9.2.

11. Precision and Bias

11.1 *Precision*—The precision of this test method as determined by an interlaboratory round robin[7] performed in accordance with Practice E 691 has been determined to be dependent upon the test level and may be estimated as follows:

Repeatability (r) = $0.12341X$
Reproducibility (R) = $0.31641X$

where X = average of two results being compared.

11.1.1 In the long run, the average of two results determined on identical materials by the same operator following this procedure in the same laboratory using the same equipment on the same day will exceed the repeatability only once in twenty times. Similarly, the average of two results determined by two different laboratories on random samples of the same batch of identical materials will exceed the reproducibility only once in twenty times.

11.2 Table 1 shows the range of ratios determined in the round robin. The volume of insolubles measured on the reference materials was in the range from 0.30 to 0.80 mL.

11.3 *Bias*—No information is available on the bias of the procedure in this test method since the compatibility of all materials is determined in relation to a set of standard reference materials.

12. Keywords

12.1 engine coolants; supplemental coolant additives (SCA)

[7] Supporting data have been filed at ASTM Headquarters and may be obtained by requesting RR: D 15–1019.

TABLE 1 Range of Ratios

Material	Average	Repeatability (r)	Reproducibility (R)	95 Percent Limits Any Two Results	
				Repeatability	Reproducibility
C1:S1	0.00	0	0	0	0
C1:S2	5.65	1.114	2.320	3.12	6.50
C2:S1	6.52	0.803	1.916	2.25	5.36
C2:S2	11.71	1.243	3.544	3.48	9.92

ANNEXES

(Mandatory Information)

A1. PREPARATION OF REFERENCE SCA MANDATORY INFORMATION

A1.1 *Components*—All solid components shall be certified ACS grade chemicals, except where otherwise listed in Table A1.1. They shall be used in the proportions listed in Table A1.1. Sodium hydroxide solution may be a certified high-purity reagent or prepared from ACS grade chemical and Specification D 1193 Type IV water.

A1.2 *Blending Procedure:*

TABLE A1.1 Components

Component	Mass, %
1. Water, deionized	20.00
2. 50 % sodium hydroxide solution	5.15
3. Sodium tetraborate pentahydrate ($Na_2B_4O_7$, $5H_2O$, technical grade)	10.00
4. Water, deionized	57.35
5. Sodium metasilicate pentahydrate (Na_2SiO_3, $5H_2O$, technical grade)	3.50
6. Sodium nitrite (Na_2NO_2)	4.00
	100.00

A1.2.1 Referring to Table A1.1, add Items 1, 2, and 3, mix, and heat to 66°C (150°F).

A1.2.2 Discontinue heating when all of the sodium tetraborate is dissolved (in approximately 10 to 20 min). Add Items 4 and 5 (Table A1.1), and mix until all of the sodium metasilicate is dissolved.

A1.2.3 Add Item 6, and mix 5 min or until dissolved.

A1.2.4 Cool to ambient temperature, and filter using a Whatman No. 4 or other suitable filter medium. Store in a sealed plastic container.

A1.3 *Usage*—The recommended concentration for usage of the reference SCA is 3 % by volume. The pH of a 3 % solution of reference SCA in deionized water should be 10.5 to 11.0.

A2. PREPARATION OF REFERENCE COOLANT CONCENTRATE

A2.1 *Components*—All components shall be used in the proportions listed in Table A2.1. All solid components shall be reagent grade chemicals, unless otherwise indicated in Table A2.1. The ethylene glycol shall be a technical or antifreeze grade material.

TABLE A2.1 Components

Component	Mass, %
1. Ethylene glycol	95.78
2. Sodium tetraborate pentahydrate ($Na_2B_4O_7{\cdot}5H_2O$, technical grade)	1.00
3. Water, distilled, deionized	2.00
4. Sodium nitrate ($NaNO_3$)	0.20
5. Sodium metasilicate pentahydrate ($Na_2SiO_3{\cdot}5H_2O$)	0.15
6. Sodium orthophosphate ($Na_3PO_4{\cdot}12H_2O$)	0.45
7. Sodium hydroxide (NaOH)	0.20
8. Sodium tolyltriazole (technical grade, 50 % water solution)	0.20
9. Pluronic L-61 (commercial grade)	0.02
	100.00

A2.2 *Blending Procedure:*

A2.2.1 Referring to Table A2.1, dissolve the sodium tetraborate pentahydrate in the ethylene glycol with stirring.

A2.2.2 Add the water in a separate container, and add the sodium nitrate, sodium metasilicate pentahydrate, sodium orthophosphate dodecahydrate, and sodium hydroxide with mild heating to assist dissolution.

A2.2.3 Add the aqueous solution slowly to the glycol-borate solution with stirring.

A2.2.4 Finally add the sodium tolyltriazole solution and the Pluronic L-61 solution, in that order.