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Superseding J1704 OCT2000

Borate Ester Based Brake Fluids

1. **Scope**—This SAE Recommended Practice was prepared to provide engineers, designers, and manufacturers of motor vehicles with a set of minimum performance standards which covers the next level of performance properties above those of the present SAE J1703 brake fluid standard. These fluids are not intended for use under arctic conditions or in braking systems requiring the use of mineral oil based hydraulic fluid.

These fluids are designed for use in braking systems fitted with rubber cups and seals made from natural rubber (NR), styrene-butadiene rubber (SBR), or a terpolymer of ethylene, propylene, and a diene (EPDM). These fluids are not designed to operate in a pumped or a clutch braking system, especially those hydraulic systems requiring a mineral oil based fluid.

2. References

- 2.1 **Applicable Publications**—The following publications form a part of this specification to the extent specified herein. The latest issue of SAE publications shall apply.

- 2.1.1 SAE PUBLICATIONS—Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

SAE J527—Brazed Double Wall Low Carbon Steel Tubing
SAE J1703—Motor Vehicle Brake Fluid

- 2.1.2 ASTM PUBLICATIONS—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 91—Test Method for Precipitation Number of Lubricating Oils
ASTM D 395—Test Methods for Rubber Property—Compression Set
ASTM D 412—Test Methods for Rubber Properties in Tension
ASTM D 445—Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
ASTM D 664—Test Method for Neutralization Number of Potentiometric Titration
ASTM D 746—Test Method for Brittleness Temperature of Plastics and Elastomers by Impact
ASTM D 865—Test Method for Rubber—Deterioration by heating in Air (Test Tube Enclosure)
ASTM D 1120—Method of Test for Boiling Point of Engine Coolants
ASTM D 1209—Test Method for Color of Clear Liquids (Platinum-Cobalt Pigments)
ASTM D 1364—Test Method for Water in Volatile Solvents (Fischer Reagent Titration Method)
ASTM D 1415—Method of Test for International Hardness of Vulcanized Natural Rubber and Synthetic Rubbers

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SAE WEB ADDRESS:

ASTM D 1613—Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
 ASTM D 2240—Method of Test for Indentation Hardness of Rubber and Plastics by Means of a Durometer
 ASTM D 3182—Recommended Practice for Rubber-Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
 ASTM D 3185—Methods for Rubber-Evaluation of SBR (Styrene-Butadiene Rubber) including Mixtures with Oil
 ASTM E 1—Specification for ASTM Thermometers
 ASTM E 145—Specification for Gravity-Convection and Forced-Ventilation Ovens

2.2 Related Publications—The following publications are provided for information purposes only and are not a required part of this document.

2.2.1 ASTM PUBLICATIONS—Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 344—Method of Test for Relative Dry Hiding Power of Paints
 ASTM E 260—Standard Recommended Practice for General Gas Chromatography Procedure
 ASTM E 298—Evaluation of Benzoyl Peroxides

3. Materials—The quality of the materials used shall be such that the resulting product will conform to the requirements of these specifications and ensure uniformity of performance.

4. Requirements

4.1 Equilibrium Reflux Boiling Point (ERBP)—Brake fluid when tested by the procedure specified in 5.1 shall have an equilibrium reflux boiling point not less than 230 °C (446 °F).

4.2 Wet Equilibrium Reflux Boiling Point—Brake fluid when tested by the procedure specified in 5.2 shall have a wet equilibrium reflux boiling point not less than 155 °C (311 °F).

4.3 Viscosity—Brake fluid when tested by the procedure specified in 5.3 shall have the following kinematic viscosities:

4.3.1 At -40 °C (-40 °F)—Not more than 1800 mm²/s (1800 cSt)

4.3.2 At 100 °C (212 °F)—Not less than 1.5 mm²/s (1.5 cSt)

4.4 pH Value—Brake fluid when tested by the procedure specified in 5.4 shall have a pH value not less than 7.0 and not more than 11.5.

4.5 Fluid Stability

4.5.1 HIGH-TEMPERATURE STABILITY—When tested by the procedure specified in 5.5.1, the equilibrium reflux boiling point of the brake fluid shall not change by more than 5 °C (9 °F) increase or decrease.

4.5.2 CHEMICAL STABILITY—When tested by the procedure specified in 5.5.2 the test fluid mixture shall show no chemical reversion as evidenced by a change in recorded temperature of more than 5 °C (9 °F) increase or decrease.

4.6 Corrosion—See Table 1.**TABLE 1—CORROSION TEST STRIPS AND WEIGHT CHANGES**

Test Strips ⁽¹⁾	RM Number	Maximum Permissible Weight Change (mg/cm ² of surface area)
Tinned Iron	6A	0.2
Steel	7	0.2
Aluminum	8	0.1
Cast Iron	9	0.2
Brass	10	0.4
Copper	11	0.4

1. Obtainable from the Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

- 4.6.1 **DRY FLUID (AS RECEIVED)**—Brake fluid, when tested by the procedure specified in 5.6.1, shall not cause corrosion exceeding the limits shown in Table 1. The metal strip outside of the area where the strips are in contact shall neither be pitted nor roughened to an extent discernible to the naked eye, but staining or discoloration is permitted. The fluid at the end of the test shall show no jelling at 23 °C ± 5 °C (73.4 °F ± 9 °F). No crystalline-type deposit shall form and adhere to either the glass walls or the surface of the metal strips. The fluid shall not contain more than 0.10% sediment by volume.

The rubber specimens at the end of the test shall show no disintegration, as evidenced by blisters or sloughing indicated by carbon black separation on the surface of the rubber cup.

- 4.6.2 **BRAKE FLUID WITH WATER**—The brake fluid containing water, when tested by the procedure specified in 5.6.2, shall not cause corrosion exceeding the limits shown in Table 1. The metal strip outside of the area where the strips are in contact shall neither be pitted nor roughened to an extent discernible to the naked eye, but staining or discoloration is permitted. The fluid/water mixture at the end of the test shall show no jelling at 23 °C ± 5 °C (73.4 °F ± 9 °F). No crystalline-type deposit shall form and adhere to either the glass walls or the surface of the metal strips. The fluid/water mixture shall not contain more than 0.10% sediment by volume. The fluid/water mixture shall have a pH of not less than 7.0 and not more than 11.5.

The rubber test specimens at the end of the test shall show no disintegration, as evidenced by blisters or sloughing indicated by carbon black separation on the surface of the rubber cup. The hardness of the SBR cup shall not decrease by more than 15 IRHD and the hardness of the EPDM rubber specimen shall not decrease by more than 10 IRHD. The SBR rubber cup shall not decrease in volume and shall not increase in volume by more than 16%. The EPDM rubber specimen shall not decrease in volume and shall not increase in volume by more than 10%.

4.7 Fluidity and Appearance at Low Temperatures

- 4.7.1 **AT -40 °C (-40 °F)**—When brake fluid is tested by the procedure specified in 5.7.1, the fluid shall show no stratification, sedimentation, or crystallization. Upon inversion of the sample bottle, the air bubble shall travel to the top of the fluid in not more than 10 s. Cloudiness is permissible, but on warming to room temperature 23 °C ± 5 °C (73.4 °F ± 9 °F), this fluid shall regain its original uniformity, appearance, and clarity.
- 4.7.2 **AT -50 °C (-58 °F)**—When brake fluid is tested by the procedure specified in 5.7.2, the fluid shall show no stratification, sedimentation, or crystallization. Upon inversion of the sample bottle, the air bubble shall travel to the top of the fluid in not more than 35 s. Cloudiness is permissible, but on warming to room temperature 23 °C ± 5 °C (73.4 °F ± 9 °F), the fluid shall regain its original uniformity, appearance, and clarity.

4.8 Water Tolerance

- 4.8.1 At -40°C (-40°F)—When the humidified brake fluid is tested by the procedure specified in 5.8.1, the black contrast lines on a hiding power chart shall be clearly discernible when viewed through the fluid in the centrifuge tube. The fluid shall show no stratification or sedimentation. Upon inversion of the centrifuge tube, the air bubble shall travel to the top of the fluid in not more than 10 s.
- 4.8.2 At 60°C (140°F)—When brake fluid is tested by the procedure specified in 5.8.2, the fluid shall show no stratification, and sedimentation shall not exceed 0.05% by volume after centrifuging when fluid is tested for qualification, or shall not exceed 0.15% by volume for a commercial packaged fluid.

4.9 Compatibility

- 4.9.1 At -40°C (-40°F)—When brake fluid is tested by the procedure specified in 5.9.1, the black contrast lines on a hiding power chart shall be clearly discernible when viewed through the fluid in the centrifuge tube. The fluid shall show no stratification or sedimentation.
- 4.9.2 At 60°C (140°F)—When brake fluid is tested by the procedure specified in 5.9.2, the fluid shall show no stratification, and sedimentation shall not exceed 0.05% by volume after centrifuging.

- 4.10 **Resistance to Oxidation**—When the humidified brake fluid is tested by the procedure specified in 5.10, it shall not cause the metal strips outside the areas in contact with the tinfoil to be pitted or roughened to an extent discernible to the naked eye, but staining or discoloration is permitted. No more than a trace of gum shall be deposited on the test strips outside of the areas in contact with the tinfoil. The aluminum strips shall not change in mass by more than 0.05 mg/cm^2 and the cast iron strips shall not change in mass by more than 0.3 mg/cm^2 .

4.11 Effect on Rubber

- 4.11.1 Rubber brake cups (RM-3a) subjected to brake fluid as specified in 5.11.1 shall show no increase in hardness, shall not decrease in hardness by more than 10 IRHD, and shall show no disintegration as evidenced by blisters or sloughing indicated by carbon black separation on the surface of the rubber cup. Volume increase shall not be less than 1% or greater than 16%.
- 4.11.2 Rubber brake cups (RM-3a) subjected to brake fluid as specified in 5.11.2 shall show no increase in hardness, shall not decrease in hardness by more than 15 IRHD and shall show no disintegration as evidenced by blisters or sloughing indicated by carbon black separation on the surface of the rubber cup. Volume increase shall not be less than 1% or greater than 16%.
- 4.11.3 Rubber slab stock (RM-69) subjected to brake fluid, as specified in 5.11.3, shall show no increase in hardness, shall not decrease in hardness by more than 10 IRHD, and shall show no disintegration as evidenced by blisters or sloughing indicated by carbon black separation on the surface of the test specimens. The test specimens shall not decrease in volume and the increase in volume shall not exceed 10%.
- 4.11.4 Rubber slab stock (RM-69) subjected to brake fluid, as specified in 5.11.4, shall show no increase in hardness, shall not decrease in hardness by more than 15 IRHD, and shall show no disintegration as evidenced by blisters or sloughing indicated by carbon black separation on the surface of the test specimens. The test specimens shall not decrease in volume and the increase in volume shall not exceed 10%.

5. Test Procedures

5.1 Equilibrium Reflux Boiling Point—Determine the equilibrium reflux boiling point of the fluid by ASTM D 1120 with the following exceptions:

5.1.1 APPARATUS

5.1.1.1 *4.4 Thermometer*—ASTM E 1, 76 mm immersion, calibrated. Use ASTM 3C or 3F thermometer. For fluids boiling below 300 °C, ASTM 2C or 2F thermometer may be used.

5.1.1.2 *4.5 Heat Source*—Use a suitable variac-controlled 100 mL heating mantle designed to fit the flask, capable of supplying the heat required to conform to the specified heating and reflux rates. (Supplier: GLAS COL Apparatus Co., Terre Haute, IN. Serial number: 135464. 230 W, 135 V [max.])

5.1.1.3 *Boiling Point Stones RM-75*

5.1.1.4 *Preparation of Apparatus*—6.4 Thoroughly clean and dry all glassware before use. Attach the flask to the condenser. Place the mantle under the flask and support it with a suitable ring clamp and laboratory-type stand, holding the whole assembly in place by a clamp.

NOTE— Place the whole assembly in an area free from drafts or other types of sudden temperature changes.

5.1.2 PROCEDURE—7.1 When everything is in readiness, turn on the condenser water and apply heat to the flask at such a rate that the fluid is refluxing in 10 min $\pm$ 2 min at a rate in excess of 1 drop/s. Immediately adjust heat input to obtain a specified equilibrium reflux rate of 1 to 2 drops/s over the next 5 min $\pm$ 2 min period. Maintain a timed and constant equilibrium reflux rate of 1 to 2 drops/s for an additional 2 min; record the average value of four temperature readings taken at 30 s intervals as the equilibrium reflux boiling point.

5.1.2.1 Report the boiling point to the nearest degree Celsius. Duplicate runs which agree within 3 °C are acceptable for averaging (95% confidence level).

5.2 Wet Equilibrium Reflux Boiling Point—Humidify the fluid and determine the boiling point.

5.2.1 HUMIDIFICATION PROCEDURE—Lubricate the ground-glass joint of a 250 mm ID bowl-form desiccator having matched tubulated glass cover and fitted with a No. 8 rubber stopper. Pour 450 mL $\pm$ 10 mL of distilled water into the dessicator and insert a perforated porcelain plate (Coors No. 60456 or equivalent). Immediately place one open RM-49 corrosion test jar containing 350 mL $\pm$ 5 mL of the test brake fluid into the dessicator. Place a second open RM-49 corrosion test jar containing 350 mL $\pm$ 5 mL of TEGME (triethylene glycol monomethyl ether; brake fluid grade - Appendix E) (RM-71) into the same desiccator. The water content of the TEGME control fluid at the start of exposure shall have been adjusted to 0.50% $\pm$ 0.05% by weight (Karl Fischer analysis or equivalent). Replace desiccator cover and insert at once into an ASTM E 145, Type II A, forced ventilation oven set at 50 °C $\pm$ 1 °C (122 °F $\pm$ 1.8 °F).

Periodically, during oven humidification, remove the rubber stopper from the desiccator and, using a long needle hypodermic syringe, quickly sample the control fluid and determine its water content. When the water content of the control fluid has reached 3.70% $\pm$ 0.05% by weight, remove the dessicator from the oven and seal the test jar promptly using a screw-cap lid (RM-63). Allow the sealed jar to cool for 60 to 90 min at 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F).

5.2.2 WET EQUILIBRIUM REFLUX BOILING POINT—Humidify the fluid as described in 5.2.1 and determine the boiling point as described in 5.1.

5.3 Viscosity—Determine the kinematic viscosity of the fluid by ASTM D 445.

- 5.3.1 Report the viscosity to the nearest mm^2/s (centistoke). Duplicate runs which agree within 1.2% relative are acceptable for averaging (95% confidence level).

5.4 pH Value—Mix the fluid with an equal volume of an 50% ethanol/50% distilled water mixture neutralized to a pH of 7. Determine the pH of the resulting solution electrometrically at $23\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ ($73.4\text{ }^{\circ}\text{F} \pm 9\text{ }^{\circ}\text{F}$) using a pH meter equipped with a calibrated full range (0 to 14) glass electrode and a calomel reference electrode, as specified in ASTM D 664.**5.5 Fluid Stability**

- 5.5.1 HIGH TEMPERATURE STABILITY—Heat a new sample of the original test brake fluid to a temperature of $185\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ($365\text{ }^{\circ}\text{F} \pm 3.6\text{ }^{\circ}\text{F}$) by the procedure specified in 5.1 and maintain at that temperature for 2 h. Then determine the boiling point of this brake fluid as specified in 5.1. The difference between this observed boiling point and that previously determined in 5.1 shall be considered as the change in boiling point of the brake fluid.
- 5.5.2 CHEMICAL STABILITY—Mix 30 mL of brake fluid with 30 mL of SAE Compatibility Fluid described in Appendix B (RM-66-05). Determine the equilibrium reflux boiling point of this fluid mixture by use of the test apparatus specified in 5.1, applying heat to the flask at such a rate that the fluid is refluxing in $10\text{ min} \pm 2\text{ min}$ at a rate in excess of 1 drop/s. The reflux rate shall not exceed 5 drops/s. Record the maximum fluid temperature observed during the first minute after the fluid begins refluxing at a rate in excess of 1 drop/s. Over the next $15\text{ min} \pm 1\text{ min}$, adjust and maintain the rate of reflux to 1 to 2 drops/s. Maintain a timed and constant equilibrium reflux rate of 1 to 2 drops/s for an additional 2 min; record the average value of four temperature readings taken at 30 s intervals as the final equilibrium reflux boiling point. Chemical reversion is evidenced by the decrease in temperature between the maximum fluid temperature recorded and the final equilibrium reflux boiling point.

5.6 Corrosion

- 5.6.1 DRY FLUID (AS RECEIVED)—Prepare two sets of strips from each of the metals listed in Table 1, each strip having a surface area of $25\text{ cm}^2 \pm 5\text{ cm}^2$ (approximately 8 cm long, 1.3 cm wide, and not more than 0.6 cm thick). Drill a hole between 4 and 5 mm in diameter and about 6 mm from one end of each strip. With the exception of the tinned iron strips, clean the strips by abrading them on all surface areas with 320A (RM-29) or P400 waterproof carborundum paper and isopropanol or ethanol until all surface scratches, cuts, and pits are removed from the strips, using a new piece of carborundum paper for each different type of metal. Wash the strips, including the tinned iron, with isopropanol or ethanol and dry the strips with a clean lint-free cloth and place strips in a desiccator containing desiccant maintained at $23\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ ($73.4\text{ }^{\circ}\text{F} \pm 9\text{ }^{\circ}\text{F}$) for at least 1 h.

Handle the strips with clean forceps after polishing to avoid fingerprint contamination.

Weigh each strip to the nearest 0.1 mg and assemble each set of strips on an uncoated steel bolt (RM-61) in the order tinned iron, steel, aluminum, cast iron, brass, and copper, so that the strips are in electrolytic contact. Bend the strips, other than cast iron, so that there is a separation of at least 3 mm between adjacent strips for a distance of about 6 cm from the free end of the strips. (Immerse strip assemblies in isopropanol or ethanol to eliminate fingerprints and then handle only with clean forceps.)

Use two SBR cups (RM-3a) and two 25.4 mm x 25.4 mm (1 in x 1 in) EPDM rubber slab stock (RM-69) test specimens as described in Appendix C and Appendix D, respectively.

Obtain two straight-sided round glass jars, having a capacity of approximately 475 mL and inner dimensions of approximately 100 mm in height and 75 mm in diameter (RM-49). To the RM-49 corrosion test jar, apply four wrappings of 19 mm (3/4 in) Teflon tape around the jar threads allowing a 3 mm (1/8 in) height above the top of the jar. Place one SBR cup (RM-3a) with lip edge facing up, in each of the two glass jars. Use only tinned steel lids vented with a hole $0.8 \text{ mm} \pm 0.1 \text{ mm}$ in diameter (RM-64).

Insert a metal strip assembly inside each cup with the bolted end in contact with the concavity of the cup and the free end extending upward in the jar. Place one EPDM rubber slab stock (RM-69) test specimen flat on the bottom of the test jar.

Add 400 mL of fluid to cover the metal strip assembly in each jar. Tighten the lid and place the jars in an oven maintained at $100 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ ($212 \text{ }^{\circ}\text{F} \pm 3.6 \text{ }^{\circ}\text{F}$) for $120 \text{ h} \pm 2 \text{ h}$. Allow the jars to cool at $23 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ ($73.4 \text{ }^{\circ}\text{F} \pm 9 \text{ }^{\circ}\text{F}$) for 60 to 90 min. Immediately following the cooling period, remove the metal strips from the jars by use of a forceps, removing loose adhering sediment by agitation of the metal strip assembly in the fluid in the jar. Examine test strips and test jars for adhering crystalline deposit, disassemble the metal strips, removing adhering fluid by flushing with water, and clean individual strips by wiping with a cloth wetted with isopropanol or ethanol. Examine the strips for evidence of corrosion and pitting. Place strips in a desiccator containing a desiccant maintained at $23^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ ($73.4 \text{ }^{\circ}\text{F} \pm 9 \text{ }^{\circ}\text{F}$) for at least 1 h. Weigh each strip to the nearest 0.1 mg.

Determine the difference in weight of each metal strip and divide the difference by the total surface area of the metal strip measured in square centimeters. Average the measured quantities of the duplicates. In the event of a marginal pass on inspection, or of a failure in only one of the duplicates, another set of duplicate test samples shall be run. Both repeat samples must meet all the requirements of 4.6.

Immediately following the cooling period, remove the rubber cups from the jars by use of a forceps, removing loose adhering sediment by agitation of the cup in the fluid in the jar.

Rinse the rubber specimens in isopropanol or ethanol and air dry cups. Examine the rubber specimens for evidence of sloughing, blisters, and other forms of disintegration.

Examine the fluid in the jars for jelling. Agitate the fluid in the jars to suspend and uniformly disperse sediment and transfer a 100 mL portion of this fluid to an ASTM cone-shaped centrifuge tube and determine percent sediment as described in 5.2 of ASTM D 91.

Measure the pH value of the corrosion test fluid by the procedure specified in 5.4.

5.6.2 CORROSION TEST ON BRAKE FLUID WITH WATER—Same test procedure as 5.6.1 except the test is performed on a brake fluid containing 5% by volume water.

Prepare two sets of strips from each of the metals listed in Table 1 (see Appendix A), each strip having a surface area of $25 \text{ cm}^2 \pm 5 \text{ cm}^2$ (approximately 8 cm long, 1.3 cm wide, and not more than 0.6 cm thick). Drill a hole between 4 and 5 mm in diameter and about 6 mm from one end of each strip. With the exception of the tinned iron strips, clean the strips by abrading them on all surface areas with 320A or P400 waterproof carborundum paper (RM-29) and isopropanol or ethanol until all surface scratches, cuts, and pits are removed from the strips, using a new piece of carborundum paper for each different type of metal. Wash the strips, including the tinned iron, with isopropanol or ethanol and dry the strips with a clean lint-free cloth and place strips in a desiccator containing desiccant maintained at $23 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ ($73.4 \text{ }^{\circ}\text{F} \pm 9 \text{ }^{\circ}\text{F}$) for at least 1 h.

Handle the strips with clean forceps after polishing to avoid fingerprint contamination.

Weigh each strip to the nearest 0.1 mg and assemble each set of strips on an uncoated steel bolt (RM-61) in the order tinned iron, steel, aluminum, cast iron, brass, and copper, so that the strips are in electrolytic contact. Bend the strips, other than cast iron, so that there is a separation of at least 3 mm between adjacent strips for a distance of about 6 cm from the free end of the strips. (Immerse strip assemblies in isopropanol or ethanol to eliminate fingerprints and then handle only with clean forceps.)

Use two SBR cups (RM-3a) and two 25.4 mm x 25.4 mm (1 in x 1 in) EPDM rubber slab stock (RM-69) test specimens as described in Appendix C and Appendix D, respectively.

Determine the weight of the cups and rubber slab stock in air (m_1) to the nearest 1 mg then determine the apparent weight of the cup and slab stock immersed in distilled water at room temperature (m_2). Quickly dip each specimen in alcohol and then blot dry with filter paper free of lint and foreign matter. Measure the hardness of each specimen by the procedure specified in ASTM D 1415 using the Standards Tester.

NOTE— ASTM D 2240 may be used for quality control and routine tests when a type A durometer is equipped with a fixture for keeping the plane of the pressure foot on the durometer parallel to the plane of the cup face during measurement.

Obtain two straight-sided round glass jars, having a capacity of approximately 475 mL and inner dimensions of approximately 100 mm in height and 75 mm in diameter (RM-49). To the RM-49 corrosion test jar, apply four wrappings of 19 mm (3/4 in) Teflon tape around the jar threads allowing a 3 mm (1/8 in) height above the top of the jar. Place one SBR cup (RM-3a) with lip edge facing up, in each of the two glass jars. Use only tinned steel lids vented with a hole $0.8 \text{ mm} \pm 0.1 \text{ mm}$ in diameter (RM-64).

Insert a metal strip assembly inside each cup with the bolted end in contact with the concavity of the cup and the free end extending upward in the jar. Place one EPDM rubber slab stock (RM-69) test specimen flat on the bottom of the test jar.

Mix 760 mL of fluid with 40 mL of distilled water.

Add 400 mL of the mixture to cover the metal strip assembly in each jar. Tighten the lid and place the jars in an oven maintained at $100 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ ($212 \text{ }^\circ\text{F} \pm 3.6 \text{ }^\circ\text{F}$) for $120 \text{ h} \pm 2 \text{ h}$. Allow the jars to cool at $23 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$ ($73.4 \text{ }^\circ\text{F} \pm 9 \text{ }^\circ\text{F}$) for 60 to 90 min. Immediately following the cooling period, remove the metal strips from the jars by use of a forceps, removing loose adhering sediment by agitation of the metal strip assembly in the fluid in the jar. Examine test strips and test jars for adhering crystalline deposit, disassemble the metal strips, removing adhering fluid by flushing with water, and clean individual strips by wiping with a cloth wetted with isopropanol or ethanol. Examine the strips for evidence of corrosion and pitting. Place strips in a desiccator containing a desiccant maintained at $23 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$ ($73.4 \text{ }^\circ\text{F} \pm 9 \text{ }^\circ\text{F}$) for at least 1 h. Weigh each strip to the nearest 0.1 mg.

Determine the difference in weight of each metal strip and divide the difference by the total surface area of the metal strip measured in square centimeters. Average the measured quantities of the duplicates. In the event of a marginal pass on inspection, or of a failure in only one of the duplicates, another set of duplicate test samples shall be run. Both repeat samples must meet all the requirements of 4.6.

Immediately following the cooling period, remove the rubber cups from the jars by use of a forceps, removing loose adhering sediment by agitation of the cup in the fluid in the jar.

Rinse the rubber specimens in isopropanol or ethanol and air dry cups. Examine the rubber specimens for evidence of sloughing, blisters, and other forms of disintegration.

Within 15 min after removal from the fluid, weigh each specimen in air (m_3), again to the nearest milligram, then reweigh immersed in room temperature distilled water (m_4) to determine the volume change after hot fluid immersion.

Measure the hardness of each specimen. Volume changes shall be reported as a percentage of the original volume, calculated as follows:

$$\% \text{ change in volume} = \frac{(m_3 - m_4) - (m_1 - m_2)}{(m_1 - m_2)} \times 100 \quad (\text{Eq. 1})$$

where:

m_1 = the initial mass in grams in air

m_2 = the initial mass in grams in water

m_3 = the mass in grams in air after immersion in test fluid

m_4 = the apparent mass in grams in water after test

Examine the fluid in the jars for jelling. Agitate the fluid in the jars to suspend and uniformly disperse sediment and transfer a 100 mL portion of this fluid to an ASTM cone-shaped centrifuge tube and determine percent sediment as described in 5.2 of ASTM D 91.

Measure the pH value of the corrosion test fluid by the procedure specified in 5.4.

5.7 Fluidity and Appearance at Low Temperatures

- 5.7.1 AT -40°C (-40°F)—Place 100 mL of fluid in a glass sample bottle (RM-59a) having a capacity of approximately 125 mL, an outside diameter of $37 \text{ mm} \pm 0.5 \text{ mm}$ and an overall height of $165 \text{ mm} \pm 2.5 \text{ mm}$. Stopper the bottle with cork and place in a cold bath maintained at $-40^\circ\text{C} \pm 2^\circ\text{C}$ ($-40^\circ\text{F} \pm 3.6^\circ\text{F}$) for $144 \text{ h} \pm 4 \text{ h}$. Remove the bottle from the bath, quickly wipe the bottle with a clean, lint-free cloth saturated with isopropanol or ethanol, and examine the fluid for evidence of stratification, sediment, or crystals. Invert the bottle and determine the number of seconds required for the air bubble to travel to the top of the fluid. Allow the fluid to warm to room temperature, $23^\circ\text{C} \pm 5^\circ\text{C}$ ($73.4^\circ\text{F} \pm 9^\circ\text{F}$); if necessary, allow to stand for as long as 4 h. Examine the fluid for clarity and appearance by comparing it to a sample of the original test fluid in an identical container.
- 5.7.2 AT -50°C (-58°F)—Place 100 mL of fluid in a glass sample bottle (RM-59a) having a capacity of approximately 125 mL, an outside diameter of $37 \text{ mm} \pm 0.5 \text{ mm}$ and an overall height of $165 \text{ mm} \pm 2.5 \text{ mm}$. Stopper the bottle with a cork and place in a cold bath maintained at $-50^\circ\text{C} \pm 2^\circ\text{C}$ ($-58^\circ\text{F} \pm 3.6^\circ\text{F}$) for $6 \text{ h} \pm 0.2 \text{ h}$. Remove the bottle from the bath, quickly wipe the bottle with a clean, lint-free cloth saturated with isopropanol or ethanol, and examine the fluid for evidence of stratification, sediment, or crystals. Invert the bottle and determine the number of seconds required for the air bubble to travel to the top of the fluid. Allow the fluid to warm to room temperature, $23^\circ\text{C} \pm 5^\circ\text{C}$ ($73.4^\circ\text{F} \pm 9^\circ\text{F}$); if necessary, allow to stand for as long as 4 h. Examine the fluid for clarity and appearance by comparing it to a sample of the original test fluid in an identical container.

5.8 Water Tolerance

- 5.8.1 AT -40°C (-40°F)—Pour 100 mL of fluid which has been humidified according to 5.2.1 into an ASTM cone-shaped centrifuge tube described in 3(a) in ASTM D 91. Stopper the tube with a cork and place in a cold bath maintained at $-40^\circ\text{C} \pm 2^\circ\text{C}$ ($-40^\circ\text{F} \pm 3.6^\circ\text{F}$) for $22 \text{ h} \pm 2 \text{ h}$. Remove the centrifuge tube from the bath, quickly wipe the tube with a clean, lint-free cloth saturated with isopropanol, determine the transparency of the fluid by placing the tube against a hiding power test chart (RM-28) and observing the clarity of the contrast lines on the chart when viewed through the fluid. Examine the fluid for evidence of stratification and sedimentation. Invert the tube and determine the number of seconds required for the air bubble to travel to the top of the fluid. (The air bubble shall be considered to have reached the top of the fluid when the top of the bubble reaches the 2 mL graduation of the centrifuge tube.)

- 5.8.2 At 60 °C (140 °F)—Place the centrifuge tube from 5.8.1 in an oven maintained at 60 °C $\pm$ 2 °C (140 °F $\pm$ 3.6 °F) for 22 h $\pm$ 2 h. Remove the tube from the oven and immediately examine the contents for evidence of stratification. Determine percentage sediment by volume as described in 5.2 of ASTM D 91.

5.9 Compatibility

- 5.9.1 At -40 °C (-40 °F)—Mix 50 mL of fluid with 50 mL of SAE Compatibility Fluid described in Appendix B (RM 66-05) and pour this mixture into an ASTM cone-shaped centrifuge tube described in 3(a) in ASTM D 91 and stopper with a cork. Place centrifuge tube for 22 h $\pm$ 2 h in a bath maintained at -40 °C $\pm$ 2 °C (-40 °F $\pm$ 3.6 °F). Remove the centrifuge tube from the bath, quickly wipe the tube with a clean, lint-free cloth saturated with isopropanol, determine the transparency of the fluid by placing the tube against a hiding power test chart¹ (RM-28) and observing the clarity of the contrast lines on the chart when viewed through the fluid. Examine the fluid for stratification and sedimentation.
- 5.9.2 At 60 °C (140 °F)—Place the centrifuge tube mentioned in 5.9.1 in an oven maintained at 60 °C $\pm$ 2 °C (140 °F $\pm$ 3.6 °F) for 22 h $\pm$ 2 h. Remove the tube from the oven and immediately examine the contents for evidence of stratification. Determine percentage sediment by volume as described in 5.2 of ASTM D 91.

- 5.10 Resistance to Oxidation**—Prepare two sets of aluminum and cast iron test strips (as listed in Table 1) by the procedure specified in 5.6. Weigh each strip to the nearest 0.1 mg and assemble a strip of each metal on an uncoated steel bolt (RM-62), separating the strips at each end with a piece of tinfoil² (RM-27) (99.5% tin, 0.5% lead, max.) approximately 12 mm square and between 0.02 and 0.06 mm in thickness.

Place 30 mL $\pm$ 1 mL of fluid in a small glass bottle approximately 120 mL in capacity. Add 60 mg $\pm$ 2 mg of reagent grade benzoyl peroxide and 1.5 mL $\pm$ 0.05 mL distilled water to the bottle. Stopper the bottle and place the bottle in an oven at 70 °C $\pm$ 2 °C (158 °F $\pm$ 3.6 °F) for 20 min $\pm$ 10 min, shaking every 15 min to effect solution of the peroxide. Remove the bottle from the oven, do not disturb the stopper, and cool in air at room temperature 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F) for 2 h.

Place approximately 1/8 section of a standard SBR cup described in Appendix C (RM-3a) in the bottom of each of two test tubes about 22 mm in diameter and 175 mm in length. Add 10 mL of prepared test fluid to each test tube. Place a metal strip assembly in each tube with the end of the strips resting on the rubber, the solution covering about one-half the length of the strips, and the bolted end remaining out of the solution. Stopper the tubes with corks and store upright 22 h $\pm$ 2 h at 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F). Loosen the stoppers and place the tubes for 168 h $\pm$ 2 h in an oven maintained at 70 °C $\pm$ 2 °C (158 °F $\pm$ 3.6 °F). After the heating period, remove and disassemble the metal strips. Examine the strips for gum deposits. Wipe the strips with a cloth saturated with isopropanol and examine for pitting or roughening of surface. Place strips in a desiccator containing a desiccant maintained at 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F) for at least 1 h. Weigh each strip to the nearest 0.1 mg.

Determine corrosion loss by dividing the difference in weight of each metal strip by the total surface area of each metal strip measured in square centimeters. Average the measured values of the duplicates. In the event of a marginal pass on inspection, or of a failure in only one of the duplicates, another set of duplicate test samples shall be run. Both repeat samples must meet all the requirements of 4.10.

1. A suitable hiding power chart as described in ASTM D 344, Method of Test for Relative Dry Hiding Power of Paints, published by the American Society for Testing and Materials, or in Method 4112 of Federal Test Method Standard No. 141, is obtainable from the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096-0001.

2. Obtainable from the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096-0001.

5.11 Effect on Rubber—For test procedures 5.11.1, 5.11.2, 5.11.3, and 5.11.4, determine the volume of each specimen in the following manner:

Weigh the specimen in air (m_1) to the nearest milligram and then weigh the specimen immersed in distilled water at room temperature (m_2) containing no more than 0.2% of a suitable wetting agent. Pluronic L-61 (BASF Wyandotte) or equivalent has been found to be acceptable.

5.11.1 TEST AT 70 °C (158 °F)—Place two SBR cups (RM-3a) in a straight-sided round glass jar (RM-51) having a capacity of approximately 250 mL and inner dimensions of approximately 125 mm height and 50 mm diameter, and a tinned steel lid (RM-52a). Add 75 mL of fluid to the jar and heat 70 h $\pm$ 2 h at 70 °C $\pm$ 2 °C (158 °F $\pm$ 3.6 °F). Allow the jar to cool at 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F) for 60 to 90 min. Remove the rubber specimens from the jar, wash quickly with isopropanol or ethanol, and air-dry the cups. Examine the cups for disintegration as evidenced by blisters or sloughing.

Within 15 min after removal from the fluid, weigh each specimen in air (m_3), again to the nearest milligram, then reweigh immersed in room temperature distilled water (m_4) to determine the volume change after hot fluid immersion.

Measure the hardness of each specimen. Volume changes shall be reported as a percentage of the original volume, calculated as follows:

$$\% \text{ change in volume} = \frac{(m_3 - m_4) - (m_1 - m_2)}{(m_1 - m_2)} \times 100 \quad (\text{Eq. 2})$$

where:

m_1 = the initial mass in grams in air

m_2 = the apparent initial mass in grams in water

m_3 = the mass in grams in air after immersion in test fluid

m_4 = the apparent mass in grams in water after test

5.11.2 TEST AT 120 °C (248 °F)—Place two standard SBR cups (RM-3a) in a straight-sided round glass jar (RM-51) having a capacity of approximately 250 mL and inner dimensions of approximately 125 mm height and 50 mm diameter, and a tinned steel lid (RM-52a). Add 75 mL of fluid to the jar and heat for 70 h $\pm$ 2 h at 120 °C $\pm$ 2 °C (248 °F $\pm$ 3.6 °F). Allow the jar to cool at 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F) for 60 to 90 min. Remove the cups from the jars, wash quickly with isopropanol or ethanol, and air-dry the cups. Examine the cups for disintegration as evidenced by blisters or sloughing.

Determine the volume change as in 5.11.1. Measure the volume change and hardness of each cup within 15 min after removal from the fluid.

5.11.3 TEST AT 70 °C (158 °F)—Place two 25.4 mm x 25.4 mm (1 in x 1 in) standard EPDM (RM-69) test specimens in a straight-sided round glass jar (RM-51) having a capacity of approximately 250 mL and inner dimensions of approximately 125 mm height and 50 mm diameter, and a tinned steel lid. Add 75 mL of fluid to the jar and heat for 70 h $\pm$ 2 h at 70 °C $\pm$ 2 °C (158 °F $\pm$ 3.6 °F). Allow the jar to cool at 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F) for 60 to 90 min. Remove the specimens from the jar, wash quickly with isopropanol or ethanol, and air dry. Examine the specimens for disintegration as evidenced by blisters or sloughing. Determine the volume change as in 5.11.1. Measure the volume change and hardness of each specimen within 15 min after removal from the fluid.

5.11.4 TEST AT 120 °C (248 °F)—Place two 25.4 mm x 25.4 mm (1 in x 1 in) standard EPDM (RM-69) test specimens in a straight-sided round glass jar (RM-51) having a capacity of approximately 250 mL and inner dimensions of approximately 125 mm height and 50 mm diameter, and a tinned steel lid (RM-52a). Add 75 mL of fluid to the jar and heat for 70 h $\pm$ 2 h at 120 °C $\pm$ 2 °C (248 °F $\pm$ 3.6 °F). Allow the jar to cool to 23 °C $\pm$ 5 °C (73.4 °F $\pm$ 9 °F) for 60 to 90 min. Remove the specimens from the jar, wash quickly with isopropanol or ethanol, and air dry. Examine the specimens for disintegration as evidenced by blisters or sloughing. Determine the volume change as in 5.11.1

Measure the volume change and hardness of each specimen within 15 min after removal from the fluid.

6. Notes

6.1 **Marginal Indicia**—The change bar (I) located in the left margin is for the convenience of the user in locating areas where technical revisions have been made to the previous issue of the report. An (R) symbol to the left of the document title indicates a complete revision of the report.

PREPARED BY THE SAE BRAKE FLUIDS STANDARDS COMMITTEE

APPENDIX A

STANDARD CORROSION TEST STRIPS

A.1 See Table A1.

TABLE A1—STANDARD CORROSION TEST STRIPS⁽¹⁾⁽²⁾

Corrosion Test Strip	Material Specification	General Material Data	Dimensions	Surface Requirements
Tinned Iron RM-6a	ASTM A 624, Federal Specification QQ-T-425A	SR tin plate electrolytic, bright No. 25, type MR Temper 3, base weight 85 lb Ferrostand and DOS oil	Approx. 8 cm long; 1.3 cm wide Thickness: As purchased Surface Area: $25 \text{ cm}^2 \pm 5 \text{ cm}^2$	As sheared. Clean and uniform tinning.
Steel RM-7	SAE 1018	Low carbon sheet Cold rolled Hardness: 40 to 72 RB	Approx. 8 cm long; 1.3 cm wide Thickness: Approx. 0.2 cm Surface Area: $25 \text{ cm}^2 \pm 5 \text{ cm}^2$	Edges machined to remove shearing marks. Clean uniform surfaces.
Aluminum RM-8	SAE AA2024	Wrought aluminum alloy Temper T3 Hardness: 75 RB typical	Approx. 8 cm long; 1.3 cm wide Thickness: Approx. 0.2 cm Surface Area: $25 \text{ cm}^2 \pm 5 \text{ cm}^2$	Edges machined to remove shearing marks. Clean uniform surfaces.
Cast Iron RM-9	SAE G3000	Soft automotive cast iron. Must be free from shrinkage cavities, porosity, or any other defects detrimental to specification use of the material. Hardness: 86 to 98 RB	Approx. 8 cm long; 1.3 cm wide Thickness: Approx. 0.4 cm Surface Area: $25 \text{ cm}^2 \pm 5 \text{ cm}^2$	Surface grind 4 sides to dimension using a well-dressed No. 80 Alundum wheel. Clean uniform surfaces.
Brass RM-10	SAE CA260	Wrought alloy—yellow brass Rolled sheet or strip; half hard temper Hardness: 57 to 74 RB	Approx. 8 cm long; 1.3 cm wide Thickness: Approx. 0.2 cm Surface Area: $25 \text{ cm}^2 \pm 5 \text{ cm}^2$	Edges machined to remove shearing marks. Clean uniform surfaces.
Copper RM-11	SAE CA114	Cold rolled copper sheet or strip Half-hard temper Hardness: 35 to 56 RB	Approx. 8 cm long; 1.3 cm wide Thickness: Approx. 0.2 cm Surface Area: $25 \text{ cm}^2 \pm 5 \text{ cm}^2$	Edges machined to remove shearing marks. Clean uniform surfaces.

1. Drill hole between 4 and 5 mm in a diameter and approximately 6 mm from one end of each strip. Holes to be clean and free from burrs. Hardness ranges are commercially for the designated metals. Hardness is not specified for the tinned iron because it is not considered a practical requirement.
2. Obtainable from the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096-0001.

APPENDIX B

SAE RM-66-05 COMPATIBILITY FLUID³

B.1 This fluid is a blend of six proprietary polyglycol brake fluids of fixed composition, in equal parts by volume. The six fluids selected comprise five factory-fill and one aftermarket fluid, as follows:

- a. Clariant Safebrake 9 M (DOT 4)
- b. Delco Supreme 11
- c. DOW 1000
- d. Dow HD 50-4
- e. Toyota BF2500H
- f. Wagner 21 B

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3. Obtainable from the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096-0001.

APPENDIX C

STANDARD STYRENE-BUTADIENE RUBBER (SBR) BRAKE CUPS FOR TESTING
SAE MOTOR VEHICLE BRAKE FLUIDS

C.1 Formulation of Rubber Compound—See Table C1.

TABLE C1—FORMULATION OF RUBBER COMPOUND

Ingredient ⁽¹⁾	Parts by Weight
SBR type 1503 ⁽²⁾	100
Oil furnace black (NBS 378)	40
Zinc Oxide (NBS 370)	5
Sulfur (NBS 371)	0.25
Stearic acid (NBS 372)	1
n-tertiary butyl-2-benzothiazole sulfenamide (NBS 384)	1
Symmetrical-dibetanophthyl-p-phenylene diamine	1.5
Dicumyl peroxide (40% on precipitated CaCO ₃) ⁽³⁾	4.5
Total	153.25

1. The ingredients labeled (NBS xxx) must have properties identical with those supplied by the National Institute of Standards and Technology.
2. Philgreen 1503 has been found suitable.
3. Use only within 90 d of manufacture and store at temperature below 27 °C (80 °F).

C.2 Procedure for Mixing Rubber Compound—The rubber compound shall be mixed in accordance with the procedure given in ASTM D 3185 for Formula 2B.

C.3 Properties of Rubber Compound—Vulcanizates cured for 12 min at 180 °C (356 °F) by the procedure described in ASTM D 3182 shall meet the requirements in Table C2.

TABLE C2—PROPERTIES OF RUBBER COMPOUND

Property	Requirement	ASTM Method
Hardness	63 ± 3	D 1415 or D 2240
Tensile Strength	17.5 MPa (2500 lbf/in ² , min)	D 412
Ultimate Elongation	350% min	D 412
Tensile Strength after 70 h at 125 °C (257 °F)	30% decrease, max	D 865
Ultimate Elongation after 70 h at 125 °C (257 °F)	50% decrease, max	D 865
Hardness after 70 h at 125 °C (257 °F)	0 to 10 increase	D 865
Compression set after 22 h at 125 °C (257 °F)	15 to 20%	D 395 (Method B)
Brittleness temperature	−40 °C (−40 °F), max	D 746

C.4 Brake Cups Prepared from Rubber Compound—Brake cups⁴ shall be prepared from the rubber compound by vulcanization under the conditions required to obtain the properties given in Section C.3. The dimensions of the cups shall be suitable for the brake cylinders used to determine stroking test procedure in 5.12. Cups may be used for testing brake fluids within 60 months from date of manufacture when stored at temperatures under 23 °C (73 °F), out of sunlight (storage in dark preferred), and adequately protected from contaminants. Rubber cups should not be stored under any strain and different parts should be stored separately to avoid migration of constituents. Lastly, rubber cups should be stored away from sources of ozone.

After removal of cups from storage, they shall be conditioned base down on a flat surface for at least 12 h at room temperature in order to allow cups to reach their true configuration before measurement.

4. Obtainable from the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, PA 15096-0001.

APPENDIX D

STANDARD ETHYLENE, PROPYLENE, DIENE (EPDM)
TERPOLYMER RUBBER SLABSTOCK (RM-69)

D.1 Formulation of Rubber Compound—See Table D1.

TABLE D1—FORMULATION OF RUBBER COMPOUND

Ingredient ⁽¹⁾	Parts by Weight
EPDM Type (Nordel 1320) ⁽²⁾	100
Zinc Oxide (NBS 370)	5
Oil Furnace Black (NBS 378)	43
Polymerized 1,2-dihydro-2,2,4-trimethylquinoline	2
Dicumyl peroxide (40% on precipitated CaCO ₃) ⁽³⁾	10
Total	160

1. The ingredients labeled (NBS xxx) must have properties identical with those supplied by the National Institute of Standards Technology (NIST) formerly the National Bureau of Standards.
2. E.I. DuPont Nordel EPDM 1320.
3. Use only within 90 days of manufacture and store at a temperature below 27 °C.

D.2 Procedure for Mixing Rubber Compound—The rubber compound shall be mixed in accordance with the procedures given in ASTM D 3182.

D.3 Properties of Rubber Compound—Vulcanizates cured for 25 min at 175 °C by the procedure described in ASTM D 3182 shall meet the following requirements as in Table D2:

TABLE D2—PROPERTIES OF RUBBER COMPOUND

Property	Requirement	ASTM Method
Hardness, IRHD	70 ± 3	D 1415
Tensile Strength, min	13.8 MPa	D 412
Ultimate Elongation, min	225%	D 412
Tensile Strength, decrease after 22 h at 175 °C, max.	15%	D 865
Ultimate Elongation, decrease after 22 h at 175 °C, max	30%	D 865
Hardness, increase after 22 h at 175 °C	0 to 10	D 865
Compression set after 22 h at 175 °C	20% max	D 395 (Method B)
Brittleness Temperature, max	−54 °C	D 746

D.4 Slabstock Prepared from Rubber compound—Test slabs approximately 50 x 150 x 1.9 mm shall be prepared from the rubber compound by vulcanization under the conditions stated Section D.3. These slabs may be used in testing brake fluids within 60 months from their date of manufacture, when stored in the dark at ambient temperatures not exceeding 38 °C and adequately protected from atmospheric or other contaminants.

When stored at other than 23 °C ± 5 °C, the material shall be allowed to stabilize at laboratory temperature prior to measurements.