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# INTERNATIONAL STANDARD



# 4925

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Road vehicles — Non-petroleum base brake fluid

*Véhicules routiers — Liquide de frein à base non pétrolière*

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## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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# Road vehicles — Non-petroleum base brake fluid

## 1 SCOPE

This International Standard lays down the characteristics and the test methods for the non-petroleum base brake fluid used in the hydraulic brake systems of road vehicles.

## 2 FIELD OF APPLICATION

The brake fluid described is for use in road vehicle hydraulic brake systems equipped with seals, cups or double-lipped type gland seals made from natural rubber (NR), styrene-butadiene rubber (SBR) and ethylene-propylene elastomer (EP); this brake fluid is not intended for use under arctic conditions.

## 3 REFERENCES

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 48, *Vulcanized rubbers — Determination of hardness (hardness between 30 and 85 IRHD).*

ISO/R 301, *Zinc alloy ingots.*

ISO/R 812, *Method of test for temperature limit of brittleness for vulcanized rubbers.*

ISO 815, *Vulcanized rubbers — Determination of compression set under constant deflection at normal and high temperatures.*

ISO 1250, *Mineral solvents for paints — White spirits and related hydrocarbon solvents.*

ISO 1817, *Vulcanized rubbers — Resistance to liquids — Methods of test.*

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity.*

ISO 4926, *Road vehicles — Hydraulic brake systems — Non-petroleum base reference fluids.*

ASTM D 91, *Test for precipitation number of lubricating oils.*

ASTM D 664, *Test for neutralization number by potentiometric titration.*

ASTM D 865, *Test for rubber deterioration by heating in a test tube.*

ASTM D 1120, *Test for boiling point of engine anti-freezes.*

ASTM D 1123, *Test for water in engine coolant concentrate by the iodine reagent method.*

ASTM D 3182, *Recommended practice for rubber — Materials, equipment and procedures for mixing standard compounds and preparing standard vulcanized sheets.*

ASTM D 3185, *Rubber — Evaluation of SBR (styrene-butadiene copolymers) including mixtures with oil.*

ASTM E 298, *Assay of organic peroxides.*

NOTE — The ASTM references will be replaced by ISO references when the latter become available.

## 4 MATERIALS

The quality of the materials used shall be such that the resulting product will conform to the requirements of this International Standard and ensure uniformity of performance. Colouring agents, if used, shall be such that the resulting colour will not be red or green. On visual inspection, the fluid shall be clear and free of suspended matter, dirt and sediment.

## 5 REQUIREMENTS

### 5.1 Equilibrium reflux boiling point (ERBP)<sup>2)</sup>

5.1.1 Brake fluid when tested by the procedure specified in 6.1.1 shall have an equilibrium reflux boiling point not less than 205 °C. (See also 6.1.4 and 6.1.5.)

### 5.1.2 Wet equilibrium reflux boiling point<sup>1)</sup>

Brake fluid when tested by the procedure specified in 6.1.6 shall have a wet equilibrium reflux boiling point not less than 140 °C.

1) Equilibrium reflux boiling point will be replaced by vapour lock measurements as soon as a suitable test method becomes available.

**5.2 Viscosity**

Brake fluid when tested by the procedure specified in 6.2 shall have the following kinematic viscosities :

**5.2.1 At -40 °C**

Not more than 1 500 mm<sup>2</sup>/s (1 500 cSt).

**5.2.2 At 100 °C**

Not less than 1,5 mm<sup>2</sup>/s (1,5 cSt).

**5.3 pH value**

Brake fluid when tested by the procedure specified in 6.3 shall have a pH value not less than 7,0 and not more than 11,5.

**5.4 Fluid stability**

**5.4.1 High-temperature stability**

When tested by the procedure specified in 6.4, the equilibrium reflux boiling point of the brake fluid shall not change by more than 3,0 °C plus 0,05 °C for each degree that the boiling point exceeds 225 °C.

**5.4.2 Chemical stability**

When tested by the procedure specified in 6.4.2, the test fluid mixture shall show no chemical reversion as evidenced by a decrease in recorded temperature of more than 2,0 °C.

**5.5 Corrosion**

Brake fluid when tested by the procedure specified in 6.5 shall not cause corrosion exceeding the limits shown in the table. The metal strips outside of the area where the strips are in contact shall be neither pitted nor roughened to an extent discernible to the naked eye, but staining or discoloration is permitted.

TABLE — Corrosion test strips and mass changes

Test strips*	Maximum permissible mass change mg/cm <sup>2</sup> of surface area
Tinned iron	0,2
Steel	0,2
Aluminium	0,1
Cast iron	0,2
Brass	0,4
Copper	0,4
Zinc	0,4

\* See annex B.

The fluid/water mixture at the end of the test shall show no jelling at 23 ± 5 °C. No crystalline-type deposit shall form and adhere to either the glass jar 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 cups 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 rubber cup shall not decrease by more than 15 IRHD, the base diameter shall not increase by more than 1,4 mm and volume increase shall not be greater than 16 %.

**5.6 Fluidity and appearance at low temperatures**

**5.6.1 At -40 °C**

When brake fluid is tested by the procedure specified in 6.6.1, the black contrast lines on a hiding power chart shall be clearly discernible when viewed through the fluid in the sample bottle. The fluid shall show no stratification or sedimentation and upon inversion of the sample bottle, the air bubble shall travel to the top of the fluid in not more than 10 s.

**5.6.2 At -50 °C**

When brake fluid is tested by the procedure specified in 6.6.2, the black contrast lines on a hiding power chart shall be clearly discernible when viewed through the fluid in the sample bottle. The fluid shall show no stratification or sedimentation and upon inversion of the sample bottle, the air bubble shall travel to the top of the fluid in not more than 35 s.

**5.7 Evaporation**

When brake fluid is tested by the procedure specified in 6.7, loss by evaporation shall not exceed 80 % by mass. Residue from the brake fluid after evaporation shall contain no precipitate that remains gritty or abrasive when rubbed with the fingertip. Residue shall have a pour point below - 5 °C.

**5.8 Water tolerance**

**5.8.1 At -40 °C**

When brake fluid is tested by the procedure specified in 6.8.1, 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.

**5.8.2 At 60 °C**

When brake fluid is tested by the procedure specified in 6.8.2, 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.

## 5.9 Compatibility

### 5.9.1 At $-40^{\circ}\text{C}$

When brake fluid is tested by the procedure specified in 6.9.1, 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.

### 5.9.2 At $60^{\circ}\text{C}$

When brake fluid is tested by the procedure specified in 6.9.2, the fluid shall show no stratification, and sedimentation shall not exceed 0,05 % by volume after centrifuging.

## 5.10 Resistance to oxidation

Brake fluid when tested by the procedure specified in 6.10 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 aluminium strips shall not change in mass by more than 0,05 mg/cm<sup>2</sup> and the cast iron strips shall not change in mass by more than 0,3 mg/cm<sup>2</sup>.

## 5.11 Effect on rubber

5.11.1 Styrene-butadiene rubber brake cups subjected to brake fluid as specified in 6.11.1 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. The increase in the diameter of the base of the cups shall not be less than 0,15 mm, or more than 1,4 mm. Volume increase shall not be less than 1 % or greater than 16 %.

5.11.2 Styrene-butadiene rubber brake cups subjected to brake fluid as specified in 6.11.2 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. The increase in the diameter of the base of the cups shall not be less than 0,15 mm, or more than 1,4 mm. Volume increase shall not be less than 1 % or greater than 16 %.

5.11.3 Natural rubber brake cups subjected to brake fluid as specified in 6.11.3 shall show no increase in hardness, shall not decrease in hardness by more than 10 IRHD and shall show no disintegration as evidenced by excessive blisters, or sloughing indicated by carbon black separation on the surface of the rubber cup. The increase in the diameter of the base of the cups shall not be less than 0,15 mm, or more than 1,4 mm. Volume increase shall not be less than 1 % or greater than 16 %.

## 5.12 Simulated service performance

Brake fluid when tested by the procedure specified in 6.12 shall meet the following performance requirements :

5.12.1 Metal parts shall not show corrosion as evidenced by pitting to an extent discernible to the naked eye, but staining or discoloration is permitted.

5.12.2 The initial diameter of any cylinder or piston shall not change by more than 0,13 mm during test.

5.12.3 Rubber cups shall not decrease in hardness by more than 15 IHRD and shall not be in an unsatisfactory operating condition as evidenced by excessive amounts of scoring, scuffing, blistering, cracking, chipping (heel abrasion), or change in shape from original appearance.

5.12.4 The base diameter of the rubber cups shall not increase by more than 0,9 mm.

5.12.5 The average lip diameter interference set of all the rubber cups in the test shall not be greater than 65 %.

5.12.6 During any period of 24 000 strokes the volume loss of fluid shall be not more than 36 ml.

5.12.7 The cylinder pistons shall not seize or function improperly throughout the test.

5.12.8 During the last 100 strokes at the end of the test, the volume loss of fluid shall not be more than 36 ml.

5.12.9 The fluid at the end of the test shall not be in an unsatisfactory operating condition as evidenced by sludging, jelling, or abrasive grittiness, and sedimentation shall not exceed 1,5 % by volume after centrifuging.

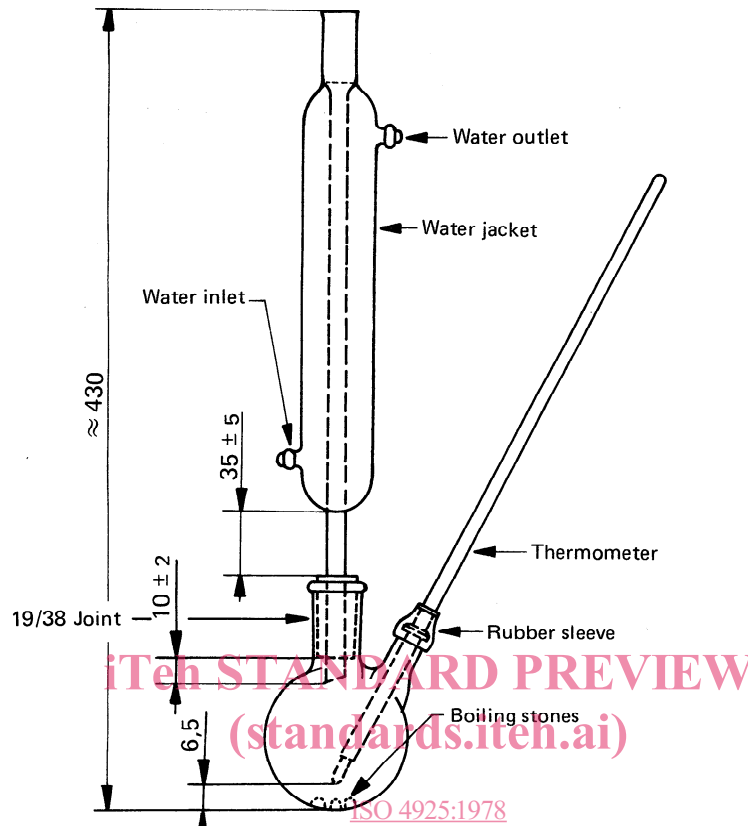
5.12.10 No more than a trace of gum shall be deposited on brake cylinder walls or other metal parts during test. The brake cylinders shall be free of deposits which are abrasive or which cannot be removed when rubbed with a cloth wetted with ethanol.

## 6 TEST METHODS

### 6.1 Equilibrium reflux boiling point (see figures 1 and 2)

6.1.1 Determine the equilibrium reflux boiling point of the fluid by ASTM D 1120, with the following exceptions in the apparatus used :

- thermometer, 76 mm immersion, calibrated;
- heat source : Use either a suitable variac-controlled heating mantle designed to fit the flask, or an electric heater with rheostat heat control. The heat source shall be capable of supplying the heat required to conform to the specified heating and reflux rates.



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FIGURE 1 – Boiling point test apparatus

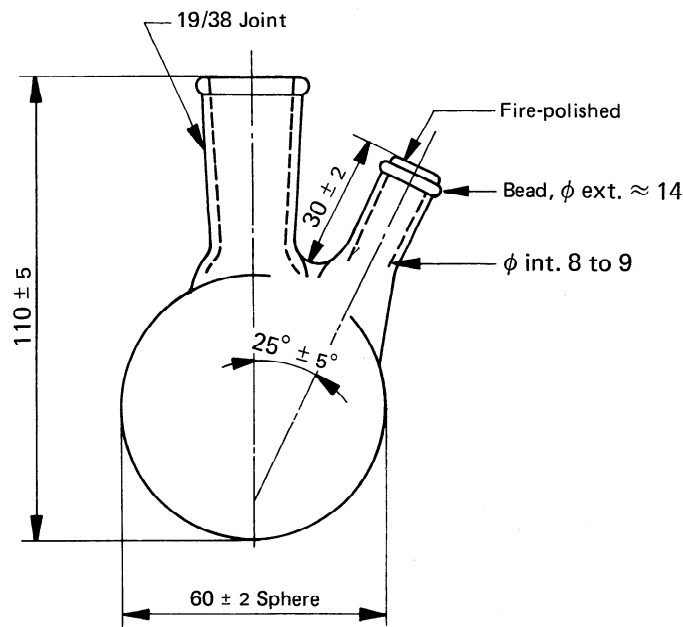


FIGURE 2 – Detail of 100 ml short-neck flask



### 6.1.2 Preparation of apparatus

Thoroughly clean and dry all glassware before use. Attach the flask to the condenser. When using a heating mantle, 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. When using a rheostat-controlled heater, centre a standard porcelain or hard asbestos refractory, having a suitable diameter opening (32 to 38 mm), over the heating element of the electric heater and mount the flask on the refractory so that direct heat is applied to the flask only through the opening in the refractory.

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

### 6.1.3 Procedure

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  $10 \pm 2$  min at a rate in excess of 1 drop/s. The reflux rate shall not exceed 5 drops/s. Immediately adjust the heat input to obtain a specified equilibrium reflux rate of 1 to 2 drops/s over the next  $5 \pm 2$  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.

### 6.1.4 205 and 232 °C fluids

Report the boiling point to the nearest degree Celsius. Duplicate runs which agree within 1 °C are acceptable for averaging (95 % confidence level).

#### 6.1.4.1 REPEATABILITY (single analyst)

The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0,4 °C at 72 degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 1,5 °C.

#### 6.1.4.2 REPRODUCIBILITY (multilaboratory)

The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 1,8 °C at 17 degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 5 °C.

### 6.1.5 288 °C fluid

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

#### 6.1.5.1 REPEATABILITY (single analyst)

The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 1,3 °C at 34 degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 4 °C.

#### 6.1.5.2 REPRODUCIBILITY (multilaboratory)

The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 3,5 °C at 15 degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 10,5 °C.

### 6.1.6 Wet equilibrium reflux boiling point

#### 6.1.6.1 APPARATUS

**6.1.6.1.1 Corrosion test jars<sup>1)</sup>**. Four corrosion test jars or equivalent screw-top, straight-sided, round glass jars each having a capacity of about 475 ml and approximate inner dimensions of 100 mm height by 75 mm diameter, with matching lids having new, clean inserts providing water-vapour proof seals.

**6.1.6.1.2 Desiccator and cover.** Four bowl-form glass desiccators, 250 mm inside diameter, having matching tubulated covers fitted with No. 8 rubber stoppers (see figure 3).

**6.1.6.1.3 Desiccator plate.** Four 230 mm diameter, perforated porcelain desiccator plates, without feet, glazed on one side.

**6.1.6.2** Determine the wet ERBP of a brake fluid by running duplicate samples according to the following procedure (see figure 3).

A 100 ml sample of the brake fluid is humidified under controlled conditions; 100 ml of compatibility fluid (see ISO 4926) is used to establish the end point for humidification. After humidification, the water content and ERBP of the brake fluid are determined.

Lubricate the ground glass joint of the desiccator. Load each desiccator with  $450 \pm 25$  g of ammonium sulphate and add  $125 \pm 10$  ml of distilled water. The surface of the salt slurry shall lie within  $45 \pm 7$  mm of the top surface of the desiccator plate. Place the desiccators in an area with temperature controlled at  $23 \pm 2$  °C throughout the humidification procedure. Leave the desiccators with the slurry and allow to condition with the covers on and stoppers in place at least 12 h before use. Use a fresh charge of salt slurry for each test.

1) Suitable corrosion test jars and tinned steel lids may be obtained from the Society of Automotive Engineers, Inc., 400 Commonwealth Drive, Warrendale, Pa. 15096 U.S.A.

**6.1.6.3** Pour  $100 \pm 1$  ml of the brake fluid into a corrosion test jar. Promptly place the jar into a desiccator. Prepare duplicate test samples, and two duplicate specimens of the compatibility fluid. Adjust the water content of the compatibility fluid to  $0,50 \pm 0,05$  % by mass at the start of the test. At intervals remove the rubber stopper in the top of each desiccator containing compatibility fluid. Using a long-needled hypodermic syringe, take a sample of not more than 2 ml from each jar and determine its water content. Remove no more than 10 ml of fluid from each reference sample during the humidification procedure. When the water content of the reference fluid reaches  $3,50 \pm 0,05$  % by mass (average of the duplicates), remove the two test fluid specimens from their desiccators and promptly cap each jar tightly. Determine their ERBP's in accordance with 6.1.1 to 6.1.3. If the two ERBP's agree within  $4^\circ\text{C}$ , average them to determine the wet ERBP; otherwise, repeat and average the four individual ERBP's as the wet ERBP of the brake fluid. Make all water content measurements according to ASTM D 1123-73.

**6.2 Viscosity**

**6.2.1** Determine the kinematic viscosity of the fluid by ISO 3104.

**6.2.2** Report the viscosity to the nearest  $1 \text{ mm}^2/\text{s}$  at  $-40^\circ\text{C}$  and to the nearest  $0,01 \text{ mm}^2/\text{s}$  at  $+100^\circ\text{C}$ . Duplicate runs which agree within 1,2 % relative are acceptable for averaging (95 % confidence level).

**6.2.2.1 REPEATABILITY** (single analyst)

The coefficient of variation of results (each the average of duplicates) obtained by the same analyst on different days has been estimated to be 0,4 % at 47 degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 1,2 %.

**6.2.2.2 REPRODUCIBILITY** (multilaboratory)

The coefficient of variation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 1,0 % at 15 degrees of freedom. Two such values should be considered suspect (95 % confidence level) if they differ by more than 3,0 %.

**6.3 pH value**

Mix the fluid with an equal volume of an 80 %/20 % (V/V) ethanol/distilled water mixture neutralized to a pH of 7,0. Determine the pH of the resulting solution electrometrically at  $23 \pm 5^\circ\text{C}$ , 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.

Prior to its use, adjust the pH of the ethanol/water mixture to 7,0 at  $23 \pm 5^\circ\text{C}$  using 0,1 N sodium hydroxide solution. If more than 4,0 ml of sodium hydroxide solution is required for neutralization, discard the mixture. All reagents used should be of a recognized analytical grade.

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Dimensions in millimetres

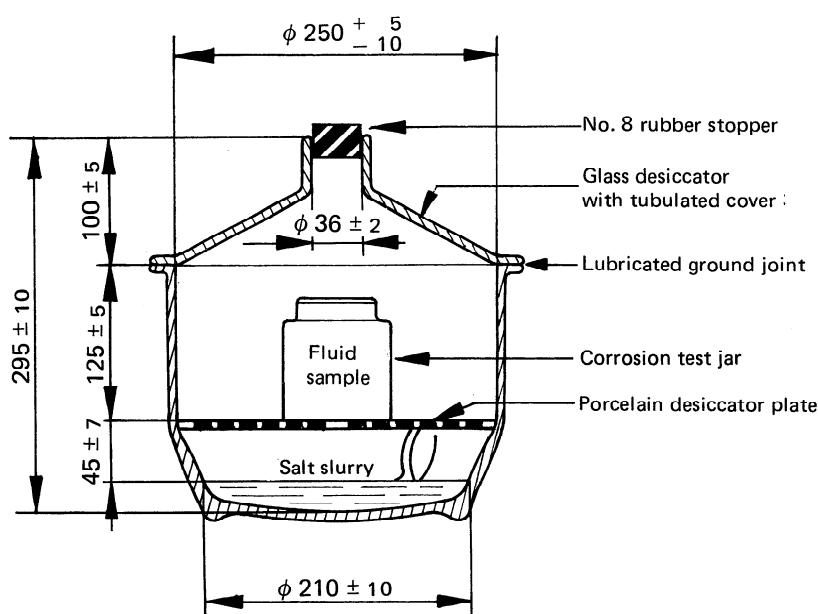


FIGURE 3 – Humidification apparatus

## 6.4 Fluid stability

### 6.4.1 High-temperature stability

Heat a new sample of the original test brake fluid to a temperature of  $185 \pm 2^\circ\text{C}$  by the procedure specified in 6.1 and maintain at that temperature for 2 h. Then determine the boiling point of this brake fluid as specified in 6.1. The difference between this observed boiling point and that previously determined in 6.1 shall be considered as the change in boiling point of the brake fluid.

### 6.4.2 Chemical stability

Mix 30 ml of brake fluid with 30 ml of compatibility fluid (see ISO 4926).

Determine the equilibrium reflux boiling point of this fluid mixture by use of the test apparatus specified in 6.1, applying heat to the flask at such a rate that the fluid is refluxing in  $10 \pm 2$  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  $15 \pm 1$  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.

## 6.5 Corrosion

Prepare two sets of strips from each of the metals listed in the table in 5.5, each strip having a surface area of  $25 \pm 5$  cm<sup>2</sup> (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 surfaces with 320 A silicon carbide paper and ISO 1250 white spirit until all surface scratches, cuts and pits are removed from the strips, using a new piece of silicon carbide paper for each different type of metal. With the exception of the tinned iron strips, polish the strips with 00 grade (very fine) steel wool, using a new piece of steel wool for each strip. Wash the strips, including the tinned iron, with 95 % ethanol, dry them with a clean, lint-free cloth and place them in a desiccator containing desiccant maintained at  $23 \pm 5^\circ\text{C}$  for at least 1 h. Handle the strips with clean forceps after polishing, to avoid fingerprint contamination.

Determine the mass of each strip to the nearest 0,1 mg and assemble each set of strips on an uncoated steel cotter pin or bolt in the order tinned iron, steel, aluminium, cast iron, brass, copper and zinc, so that the strips are in electrical contact. Bend the strips, except the cast iron, so that there is a separation of approximately 10 mm between two adjacent strips at their free ends. Immerse the strip assemblies in 95 % ethanol containing 5 % by volume of distilled water to eliminate fingerprints and then handle only with clean forceps. (See Annex E.)

Measure the base diameter of two standard SBR cups, described in annex A (figure 7), using an optical comparator or micrometer, to the nearest 0,02 mm along the centreline of the ISO and rubber type identifications and at right angles to this centreline. Take the measurements at least 0,4 mm and not more than 2,4 mm above the bottom edge and parallel to the base of the cup. Discard any cup if the two measured diameters differ by more than 0,08 mm. Take the average of the measurements on each cup. Determine the hardness of each cup thus supported by the procedure specified in ISO 48. If this International Standard cannot be used, another procedure may be selected, possibly using a rubber anvil (see figure 9a). Determine the volume change by the method given in 6.11.1

Place one rubber cup, with lip edge facing up, in each of two straight-sided round glass jars having a capacity of approximately 475 ml and inner dimensions of approximately 100 mm height and 75 mm diameter. Use only tinned steel lids vented with a hole  $0,8 \pm 0,1$  mm in diameter.

Insert a metal strip assembly inside each cup with the pinned end in contact with the concavity of the cup and the free end extending upward in the jar. Mix 760 ml of brake fluid with 40 ml of distilled water.

Add a sufficient amount of the mixture to cover the metal strip assembly in each jar to a depth of approximately 10 mm above the tops of the strips. Tighten the lids and place the jars in a gravity convection oven maintained at  $100 \pm 2^\circ\text{C}$  for  $120 \pm 2$  h. Allow the jars to cool at  $23 \pm 5^\circ\text{C}$  for 60 to 90 min. Immediately following the cooling period, remove the metal strips from the jars by use of forceps, removing loose adhering sediment by agitation of the metal strip assembly in the fluid in the jar. Examine the test strips and test jars for adhering crystalline deposit, disassemble the metal strips, remove adhering fluid by flushing with water and clean individual strips by wiping with a cloth wetted with 95 % ethanol. Examine the strips for evidence of corrosion and pitting. Place the strips in a desiccator containing a desiccant maintained at  $23 \pm 5^\circ\text{C}$  for at least 1 h. Determine the mass of each strip to the nearest 0,1 mg. Determine the difference in mass of each metal strip and divide the difference by the total surface area of the metal strip measured in square centimetres. 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 5.5.

Immediately following the cooling period, remove the rubber cups from the jars by use of forceps, removing loose adhering sediment by agitation of the cup in the fluid in the jar. Rinse the cups in 95 % ethanol and air-dry them. Examine the cups for evidence of sloughing, blisters, and other forms of disintegration. Measure the base diameter, hardness and volume of each cup within 15 min after removal from the fluid.

Examine the fluid/water mixture in the jars for presence of gel. Agitate the fluid in the jars to suspend and uniformly disperse sediment, transfer a 100 ml portion of this fluid