



Designation: D2619 – 95 (Reapproved 2002)^{e1}

Standard Test Method for Hydrolytic Stability of Hydraulic Fluids (Beverage Bottle Method)¹

This standard is issued under the fixed designation D2619; 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.

^{e1} NOTE—Warning notes were placed in the text editorially in May 2002.

1. Scope

1.1 This test method² covers the determination of the hydrolytic stability of petroleum or synthetic-base hydraulic fluids.

NOTE 1—Water-base or water-emulsion fluids can be evaluated by this test method but are run “as is.” Additional water is not added to the 100-g sample.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are provided for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in 3.1, 6.1, 6.3, 6.4, and [Annex A1](#).

2. Referenced Documents

2.1 ASTM Standards:³

[D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids \(and Calculation of Dynamic Viscosity\)](#)³

[D974 Test Method for Acid and Base Number by Color-Indicator Titration](#)

3. Summary of Test Method

3.1 The sample of 75 g of fluid plus 25 g of water and a copper test specimen are sealed in a pressure-type beverage bottle. The bottle is rotated, end for end, for 48 h in an oven at 93°C (200°F). Layers are separated, and insolubles are weighed. Weight change of copper is measured. Viscosity and

acid number changes of fluid and acidity of water layer are determined. (**Warning**—In addition to other precautions, because this test method involves the use of a glass bottle that may contain approximately 200 kPa (2 atm) of air and water vapor at temperatures up to 93°C, a full face shield and heavy woven fabric gloves should be worn when handling or working with the heated and sealed sample container.)

4. Significance and Use

4.1 This method differentiates the relative stability of hydraulic fluids in the presence of water under the conditions of the test. Hydrolytically unstable hydraulic fluids form acidic and insoluble contaminants which can cause hydraulic system malfunctions due to corrosion, valve sticking, or change in viscosity of the fluid. The degree of correlation between this test and service performance has not been fully determined.

5. Apparatus

5.1 *Air Oven*, convection, adjusted to $93 \pm 0.5^\circ\text{C}$ ($200 \pm 1^\circ\text{F}$).⁴

5.2 *Pressure-Type Beverage Bottles*,⁵ 200 mL (7-oz).

5.3 *Capping Press*, for bottles.

5.4 *Rotating Mechanism*, for holding bottles and rotating end over end at 5 rpm in oven.

5.5 *Centrifuge*, 1500 rpm.

5.6 *Centrifuge Tubes*, cone-shaped, 100-mL.

5.7 *Filtration Assembly*,⁶ stainless screen/glass, membrane type 47-mm diameter.

5.8 *Typewriter Brush*.

5.9 *Separatory Funnel*, 125-mL.

5.10 *Microscope*, for 20 $\times$ magnification.

5.11 *Balance*, sensitive to 0.2 mg.

5.12 *Caps*, for sealing bottles.

5.13 *Inert Seal*,⁷ for cap gasket.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.N0.08 on Thermal Stability.

Current edition approved Jan. 15, 1995. Published March 1995. Originally published as D2619 – 67. Last previous edition D2619 – 88 (1994) ^{e1}. DOI: 10.1520/D2619-95R02E01.

² This test method is a modification of Federal Test Method Standard No. 791a, Method 3457 for Hydrolytic Stability.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ An acceptable commercial unit is available from Falex Corp. 1020 Airpark Dr., Sugar Grove, IL 60554.

⁵ A 200-mL (7-oz) glass Coca Cola trademarked bottle has proven satisfactory. Bottles can be obtained from beverage distributors.

⁶ A Millipore XX10 047 30 or equivalent has proven satisfactory.

⁷ A 0.127-mm (0.005-in.) thick fluorocarbon seal has proven satisfactory.



5.14 *Membrane Type Filter*,⁸ cellulosic, 5-µm porosity, 47-mm diameter.

6. Reagents and Materials

6.1 *n*-Heptane. (**Warning**—Flammable, harmful if inhaled, skin irritant on repeated contact, aspiration hazard; see A1.1.)

6.2 Phenolphthalein, 1 % alcoholic solution.

6.3 Potassium Hydroxide (KOH), 0.1 *N* aqueous solution standardized to within 0.0005 *N*. (**Warning**—Caustic.)

6.4 1,1,1-Trichloroethane. (**Warning**—Harmful if inhaled, high concentrations may cause unconsciousness or death; contact may cause skin irritations and dermatitis, may produce toxic vapors if burned, eye irritant; see A1.2.)

6.5 Copper Strip (QQ-C-576A), 16-22 B and S gage, 13 by 51 mm.

6.6 Steel Wool, grade 1-medium fine.

6.7 Litmus Paper.

7. Procedure

7.1 Fill the pressure beverage bottle with distilled water and allow to stand overnight. Drain and rinse, with distilled water, but do not dry.

7.2 Weigh 75 g of test fluid and 25 g of distilled water to 0.5 g into the beverage bottle.

7.3 Polish the copper test specimen to a clean surface with the steel wool and wash with *n*-heptane. (**Warning**—see 6.1). Dry and weigh to 0.2 mg. Immediately immerse the copper specimen in the test fluids in the beverage bottle. Avoid specimen contact by handling the cleaned copper test strip with cotton gloves or filter paper.

7.4 Prepare a disk of the inert seal⁶ and place in a new bottle cap. Seal the bottle using the cap with the gasket.

7.5 Place the bottle in the rotating mechanism in the oven adjusted to 93 ± 0.5°C (200 ± 1°F). Allow to rotate, end for end, at 5 rpm for 48 h.

7.6 Remove the bottle and place on an insulated surface until cool.

7.7 Open the bottle, empty the contents into a 100-mL, cone-shaped centrifuge tube, and centrifuge for 10 min at 1500 rpm. Decant the separated water and emulsion layers and set aside. (A pipet can be used as an alternative method to remove the water layer without centrifuging, provided a clear water separation results by sample settling.)

7.8 Filter the fluid oil layer through a membrane filter weighed to 0.2 mg. Transfer the fluid layer to a 125-mL separatory funnel. Rinse the filter flask with 25 mL of distilled water and add to the separatory funnel. Repeat the water washes of the oil in the separatory funnel until the washings are neutral to litmus. Save the combined water washings (Note 2). Dry the filtered, washed fluid by vacuum dehydration when in contact with anhydrous sodium sulfate (Note 3). Filter the fluid through filter paper to remove the sodium sulfate solids.

NOTE 2—If the fluid sample is heavier than water, drain the fluid from

the separatory funnel, remove the water wash, and return the fluid to the separatory funnel for repeated water washes.

NOTE 3—Mechanical stirring for 1 h with the anhydrous sodium sulfate dries the fluid efficiently. This is done prior to vacuum dehydration.

7.9 Determine the viscosity of the filtered fluid in accordance with Test Method D445. Compare the result with viscosity of the original fluid sample and calculate the percentage viscosity change at 40°C (104°F).

7.10 Determine the total acid number of the filtered fluid in accordance with Test Method D974. Acidity for the filtered fluid is compared to that of the original fluid and the acid number change recorded.

7.11 Filter the water phase and the emulsion layer (which usually contains the bulk of the insoluble material) through the membrane filter. Rinse the copper test specimen, pipet, centrifuge tube, beaker, and beverage bottle with distilled water and *n*-heptane. Filter these washes through the membrane. Segregate the water wash. Then wash with 50 mL of *n*-heptane. Dry the membrane in a 60°C (140°F) oven and weigh. Calculate the percentage of insolubles in the 75-g sample.

7.12 Combine all the water portion and washes. Determine total acidity by adding 1.0 mL of phenolphthalein solution and titrating rapidly with 0.1 *N* KOH solution to the appearance of pink phenolphthalein end point which persists for 15 s. Calculate the water layer acidity as follows:

$$\text{Total Acidity, mg KOH} = [(A - B)N] \times 56.1 \quad (1)$$

where:

A = millilitres of KOH solution required for titration of the sample,

B = millilitres of KOH solution required for titration of the blank, and

N = normality of KOH solution.

7.13 Wash the copper specimen with warm *n*-heptane, followed by warm 1,1,1-trichloroethane. (**Warning**—See 6.4.) Brush with a short bristled typewriter-type brush while washing in both solvents. Dry and weigh and examine under a 20× microscope. Report appearance and weight loss in milligrams per square centimetre.

8. Report

8.1 The report shall include the following:

8.1.1 Acid number change of fluid in milligrams of KOH per gram,

8.1.2 Total acidity of water in milligrams of KOH,

8.1.3 Weight change of copper strip in milligrams per square centimetre, and

8.1.4 Appearance of strip under 20× magnification.

9. Precision and Bias

9.1 The precision as determined by statistical examination of interlaboratory result is as follows:

9.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in twenty, where:

$\bar{X}$ = the average of the two test results.

⁸ A Millipore SMWP 04700 or (5.0-µm porosity) or equivalent has proven satisfactory. If there is a test fluid filter material incompatibility a fluorocarbon membrane may be used. A Millipore FSLW 04700 (3.0-µm or equivalent porosity) has proven satisfactory for example with phosphate ester fluids.