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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.

~~ε[1] NOTE—Warning notes were placed in the text editorially in May 2002.~~

1. Scope

1.1 This test method[2] covers the determination of the hydrolytic stability of petroleum or synthetic-based 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—Water-based or water-emulsion fluids can be evaluated by this test method, but they are run "as is." Additional water is not added to the 100-g sample. In these cases, the person requesting the test needs to let the test operator know that water is present.

1.2 The values stated in SI units are to be regarded as the standard. The English 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~~warning statements are given in 3.1, 6.1, 6.3, ~~6.4~~6.9~~,~~ 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]

130 Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test

D974 Test Method for Acid and Base Number by Color-Indicator Titration

3. Summary of Test Method

3.1 ~~The sample of~~ A copper test specimen and 75 g of test fluid plus 25 g of water ~~and a copper test specimen~~ (or 100 g of a water-containing fluid) 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~~ the weight change of the copper specimen is measured. ~~Viscosity and~~ The acid number ~~changes~~change of the fluid and acidity of the water layer are determined. (~~Warning—~~ ~~*In*~~**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 test 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 method and service performance has not been fully determined.

[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.

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

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* ~~, Vol 05.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

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5. Apparatus

5.1 *Air Oven*, convection, adjusted to 93 ± 0.5 °C (200 ± 1 °F).[4]

5.2 ~~*Pressure-Type Beverage Bottles*, 200 mL (7-oz).~~ 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~~r/min in oven.

5.5 ~~*Centrifuge*, 1500 rpm.~~ Büchner Funnel and Filter Flask.

5.6 ~~*Centrifuge Tubes*, cone-shaped, 100-mL.~~ Water Aspirator.

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

~~5.8~~*Typewriter Brush*.

~~5.9~~

5.8 *Separatory Funnel*, 125-mL.

~~5.10~~*Microscope*~~, for 20× magnification.~~

~~5.11~~

5.9 *Balance*, sensitive to 0.2 mg.

~~5.12~~

5.10 *Caps*, for sealing bottles.

~~5.13~~*Inert Seal,*~~for cap gasket.~~

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

5.11 *Inert Seal,* for cap gasket, 0.127-mm (0.005-in.) thick fluorocarbon seal.

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. (~~**Warning—**Caustic.)

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

~~6.6~~6.5 *Steel Wool*, grade 1-medium fine.

~~6.7~~

6.6 *Litmus Paper*.

6.7 *Filter Paper, Whatman No. 41*.

6.8 *Anhydrous Sodium Sulfate* (Na_2SO_4).

6.9 *1,1,1-Trichloroethane* (optional–for use when the test fluid is a phosphate ester). (**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.)

7. Procedure

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

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

~~7.3Polish 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.~~

~~7.4Prepare a disk of the inert seal[6] and place in a new bottle cap. Seal the bottle using the cap with the gasket.~~

~~7.5Place 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.~~

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

~~7.7Open 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.)~~

~~7.8Filter 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~~

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

[4] The sole source of supply of the apparatus known to the committee at this time is Falex Corp. 1020 Airpark Dr., Sugar Grove, IL 60554. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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

[5] Bottles can be obtained from beverage distributors.

~~separatory funnel until the washings are neutral to litmus. Save the combined water washings (~~

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

7.2 Determine the total acid number of the test fluid in accordance with Test Method D974.

7.3 Weigh 75 g of test fluid and 25 g of distilled water (or in the case of water-containing fluids, 100 g of the test fluid) to 0.5 g into the beverage bottle.

7.4 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 fluid in the beverage bottle. Avoid specimen contact by handling the cleaned copper test strip with cotton gloves or filter paper.

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

7.6 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 r/min for 48 h.

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

7.8 Open the bottle and decant the contents (except for the copper specimen) into a 125 mL separatory funnel. Allow the layers to separate and remove the aqueous layer (Note 2~~). Dry the filtered, washed fluid by vacuum dehydration when in contact with anhydrous sodium sulfate (~~). Wash the oil layer with 25 mL portions of distilled water, repeating until the washings are neutral to litmus paper. Save the combined water washings. Dry the washed fluid with anhydrous sodium sulfate or by vacuum dehydration (Note 3), or both. 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.~~ 2—For water-containing fluids, there will be no separation, and so this step should be bypassed. Certain other fluids may emulsify with water and not separate during this step. In either of these cases, no determination of water acidity will be conducted and a remark should be inserted into the test report to this effect. 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.~~

~~7.9Determine 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).~~

~~7.10Determine 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.~~

~~7.11Filter 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.~~

~~7.12Combine 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:~~ 3—Mechanical stirring for 1 h with the anhydrous sodium sulfate dries the fluid efficiently. Add sufficient sodium sulfate with swirling until it no longer forms clumps in the fluid.

7.9 Determine the total acid number of the filtered fluid in accordance with Test Method D974. The acid number of the filtered fluid is compared to that of the original fluid (determined in 7.2) and the change recorded.

7.10 Rinse the copper test specimen and beverage bottle with distilled water and *n*-heptane into the combined water washes and then return to the separatory funnel. Separate the layers and wash the aqueous phase with one 50 mL portion of *n*-heptane.

7.11 Transfer the water layer to an Ehrlenmeyer flask. Determine total acidity by adding 1.0 mL of phenolphthalein solution and titrating rapidly with 0.1 *N* KOH solution to the appearance of a pink phenolphthalein end point which persists for 15 s. Calculate the water layer acidity as follows:

~~Total Acidity, mg KOH (1)~~

~~Total Acidity, mg KOH (1)~~

$$= A - BN \times 56{,}100 \text{ mg / Eq } 1 \text{ L / } 1000 \text{ mL}$$

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.

~~7.13Wash 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.~~

7.12 Wash the copper specimen with warm *n*-heptane, followed by warm 1,1,1-trichloroethane (if using). (**Warning—**see 6.9.) Brush with a short bristled typewriter-type brush while washing. Dry and weigh. Report weight change in milligrams per square centimetre and appearance as determined using the ASTM Copper Strip Corrosion Standard, following the interpretation guidelines in Test Method D130, Section 11.