Designation: D2619 – 09

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.

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-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 warning statements are given in 3.1, 6.1, 6.3, 6.9 and Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D130 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 A copper test specimen and 75 g of test fluid plus 25 g of water (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 the weight change of the copper specimen is measured. The acid number change of the fluid and acidity of the 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 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.

5. Apparatus

5.1 *Air Oven*, convection, adjusted to 93 ± 0.5 °C (200 ± 1 °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 r/min in oven.

5.5 *Büchner Funnel and Filter Flask.*

5.6 *Water Aspirator.*

5.7 *Typewriter Brush.*

5.8 *Separatory Funnel*, 125-mL.

5.9 *Balance*, sensitive to 0.2 mg.

5.10 *Caps*, for sealing bottles.

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.)

[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 Dec. 1, 2009. Published February 2010. Originally approved in 1967. Last previous edition approved in 2002 as D2619–95(2002) $^{\epsilon 1}$. DOI: 10.1520/D2619-09.

[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* volume information, refer to the standard's Document Summary page on the ASTM website.

[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,[1] which you may attend.

[5] Bottles can be obtained from beverage distributors.

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

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

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

7.1 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). 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—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. 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:

$$\text{Total Acidity, mg KOH} = [(A - B)N] \times 56{,}100 \text{ mg / Eq}(1 \text{ L} / 1000 \text{ mL}) \quad (1)$$

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.

7.12 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.

$$F = (C - D) / E \quad (2)$$

where:

C = final weight of copper specimen, mg,
D = initial weight of copper specimen, mg,
E = surface area of copper specimen, cm^2, and
F = weight change, mg/cm^2.

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, or if this could not be determined because no separation occurred, a remark to this effect.

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

8.1.4 Appearance of strip as per the instructions in Test Method D130.

9. Precision and Bias [6]

9.1 The precision of this test method is based on an interlaboratory study of D2619-95 (Note 4), Standard Test Method for Hydrolytic Stability of Hydraulic Fluids, conducted in 2006. Each of six laboratories tested five different materials. Every "test result" represents an individual determination. For H_2O Acidity and weight change of the copper strip

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1676.

TABLE 1 Δ (delta) Cu (mg/cm^2)

Fluid	Average, Δ $\overline{X}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
1	–0.0219	0.0156	0.0180	0.0438	0.0505
2	–0.0742	0.0438	0.0438	0.1226	0.1226
3	–0.5283	0.0748	0.2051	0.2093	0.5741
4	–3.6439	0.6490	0.8274	1.8172	2.3166
5	–0.0161	0.0157	0.0193	0.0441	0.0540

TABLE 2 H_2O Acidity (mg KOH)

Fluid	Average, $\overline{X}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
1	2.3267	0.5684	1.1103	1.5916	3.1088
2	0.3458	0.2156	0.4694	0.6038	1.3143
3	15.2358	1.4479	3.6158	4.0542	10.1244
4	0.6025	0.3282	0.5146	0.9190	1.4409
5	0.6533	0.2929	0.4413	0.8202	1.2355

TABLE 3 Δ (delta) TAN (mg KOH/g oil)

Fluid	Average, $\overline{X}$	Repeatability Standard Deviation, s_r	Reproducibility Standard Deviation, s_R	Repeatability Limit, r	Reproducibility Limit, R
1	–0.0030	0.0750	0.0750	0.2100	0.2100
2	0.0130	0.0708	0.0895	0.1983	0.2505
3	0.4460	0.1403	0.1403	0.3927	0.3927
4	0.5155	0.0904	0.1095	0.2530	0.3066
5	0.0220	0.0479	0.0639	0.1342	0.1788

(Δ Cu, mg/cm^2), five laboratories obtained two replicate test results from each of two operators for every material, while one laboratory obtained just two replicate test results (from one operator) for each material. For Δ TAN, four laboratories obtained two replicate test results from each of two operators for every material, while one laboratory obtained just two replicate test results (from one operator) for each material (Note 5).

NOTE 4—The purpose of the 1,1,1-trichloroethane solvent is to thoroughly remove phosphate ester fluids from the copper strips; however none of the participating labs routinely test phosphate esters, and as a result used only *n*-heptane for cleaning the strips in this study. Furthermore, none of the round robin test fluids was phosphate ester-based. Therefore, this precision statement cannot necessarily be extrapolated to phosphate ester fluids or to procedures using 1,1,1-trichloroethane solvent.

NOTE 5—The data used to generate Tables 1-3 are available from ASTM International Headquarters and may be obtained by requesting RR: D02–1676.

9.1.1 *Repeatability*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

9.1.2 *Reproducibility*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

9.1.3 Any judgment in accordance with these two statements would have an approximate 95% probability of being correct.

9.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

9.3 The precision statement was determined through statistical examination of 310 results, from six laboratories, on five materials. These five fluids were the following:

Fluid 1	A passing ashless formulation in mineral oil
Fluid 2	A passing zinc dithiophosphate-containing formulation in mineral oil
Fluid 3	A failing ashless formulation in mineral oil
Fluid 4	A failing zinc dithiophosphate-containing formulation in mineral oil
Fluid 5	A passing zinc dithiophosphate-containing formulation in synthetic base oils (poly-alpha-olefin and complex ester)

9.3.1 To judge the equivalency of two test results, it is recommended to choose the fluid closest in characteristics to the test fluid.

9.4 The precision statement from the 1995 round robin is included in Appendix X1. Also included is a comparison of those results with the latest precision statement.

10. Keywords

10.1 beverage bottle; copper corrosion; hydraulic fluid; hydrolytic stability

ANNEX

(Mandatory Information)

A1. WARNING STATEMENTS

A1.1 *n*-Heptane

Keep away from heat, sparks, and open flame.

Keep container closed.

Use with adequate ventilation.

Avoid prolonged breathing of vapor or spray mist.

Avoid prolonged or repeated skin contact.

A1.2 1,1,1-Trichloroethane

Avoid prolonged or repeated breathing of vapor or spray mist.

Use only with adequate ventilation.

Eye irritation and dizziness are indications of overexposure.

Do not take internally. Swallowing may cause injury, illness or death.

Avoid prolonged or repeated contact with skin.

Do not get in eyes.

APPENDIX

(Nonmandatory Information)

X1. REPRODUCIBILITY INFORMATION

X1.1 Precision Statement from D2619–95 (Conducted Using 1,1,1–Trichloroethylene Solvent)

X1.1.1 Table X1.1 shows recommended precision quantities from interlaboratory study of hydrolytic stability test method.

X1.2 Comparison of Precision Statements from D2619–95 and D2619–09

X1.2.1 Table X1.2 compares the reproducibility for the change in the weight of the copper specimen obtained in the current study with those obtained from the previous round robin. Reproducibility has generally gotten worse, except in the case of Fluid 4, where it improved.

X1.2.2 Table X1.3 compares the reproducibility for the absolute values of the change in TAN obtained in the current study with those obtained from the previous round robin. Reproducibility seems to vary from fluid to fluid much less since the previous study, although it has gotten worse, except in the case of Fluids 3 and 4.

X1.2.3 Table X1.4 compares the reproducibility for the water acidity values obtained in the current study with those obtained from the previous round robin. For this measurement, reproducibility worsened for Fluids 2, 4 and 5; stayed the same for Fluid 1 and improved for Fluid 3.

TABLE X1.1 Recommended Precision Quantities from Interlaboratory Study of Hydrolytic Stability Test Method[A]

Property	Repeatability, r	Reproducibility, R
Copper corrosion, mg/cm^2	0.3 $\overline{X}$	0.9 $\overline{X}$
Acid number change, mg KOH/g oil	0.8 $\overline{X}$	1.9 $\overline{X}$
Total acidity of water layer, mg KOH	0.8 $\overline{X}$	1.3 $\overline{X}$

[A] $\overline{X}$ denotes mean value.

TABLE X1.2 Δ (delta) Cu (mg/cm^2)

Fluid	Absolute Value Average, Δ $\overline{X}$	Reproducibility as Calculated Using Previous Study, (0.9 $\overline{X}$)	Current Reproducibility Limit, R
1	0.0219	0.0197	0.0505
2	0.0742	0.0668	0.1226
3	0.5283	0.4755	0.5741
4	3.6439	3.2795	2.3166
5	0.0161	0.0145	0.0540

TABLE X1.3 Δ (delta) TAN (mg KOH/g oil)

Fluid	Absolute Value of Average, $\overline{X}$	Reproducibility as Calculated Using Previous Study, (1.9 $\overline{X}$)	Current Reproducibility Limit, R
1	0.0030	0.0057	0.2100
2	0.0130	0.0247	0.2505
3	0.4460	0.8474	0.3927
4	0.5155	0.9795	0.3066
5	0.0220	0.0418	0.1788

TABLE X1.4 H_2O Acidity (mg KOH)

Fluid	Average, $\overline{X}$	Reproducibility as Calculated Using Previous Study, (1.3 $\overline{X}$)	Current Reproducibility Limit, R
1	2.3267	3.0247	3.1088
2	0.3458	0.4495	1.3143
3	15.2358	19.8065	10.1244
4	0.6025	0.7833	1.4409
5	0.6533	0.8493	1.2355