



Standard Test Method for Oxidation and Thermal Stability Characteristics of Gear Oils Using Universal Glassware¹

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^{E1} NOTE—Added research report to existing footnote for Section 13 editorially in December 2008.

1. Scope

1.1 This test method covers the determination of the oxidation characteristics of extreme pressure and non-extreme pressure gear oils and includes the quantitative determination of total sludge, viscosity change, and oil loss.

NOTE 1—While the round-robin tests used ISO VG 220 extreme pressure gear oils for developing precision data, the test method can be extended to other viscosity grades and to non-extreme pressure gear oils. Refer to Classification D2422 for viscosity grades.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are 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.*

2. Referenced Documents

2.1 ASTM Standards:²

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D2422 Classification of Industrial Fluid Lubricants by Viscosity System

D2893 Test Methods for Oxidation Characteristics of Extreme-Pressure Lubrication Oils

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4871 Guide for Universal Oxidation/Thermal Stability Test Apparatus

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09.0D on Oxidation of Lubricants.

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² 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.

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *adherent sludge, n*—sludge that is formed on the walls of a container and is not easily removed.

3.1.2 *aliquot, n*—portion of sample being tested that is a representative portion of the whole.

3.1.3 *extreme pressure gear oil, n*—gear oil that contains chemical additives, such as sulfur and phosphorus compounds, which produce a protective film on the metal surface to provide anti-scuffing and anti-scoring properties.

3.1.4 *filterable sludge, n*—sludge that is formed in the oil.

3.1.5 *non-extreme pressure gear oil, n, n*—gear oil that contains no extreme pressure additives.

3.1.6 *oxidation, n*—the process by which oxygen chemically reacts with materials.

3.1.7 *sludge, n—in gear oils*, a precipitate that sometimes forms as the oil ages or oxidizes.

3.1.8 *universal glassware, n*—the glassware that is described in the universal oxidation thermal stability test. Refer to Guide D4871.

4. Summary of Test Method

4.1 The viscosity of the gear oil being tested is determined. A 100-g aliquot of the oil in a weighed apparatus is subjected to a temperature of 120°C for 312 h while dry air is passed through the aliquot at 3 L/h.

4.2 At the end of the stress period, the aliquot is cooled to room temperature. The apparatus is reweighed to determine oil loss. Filterable sludge is recovered by vacuum filtration using a 2.8- μ m glass fiber filter medium. The viscosity of the filtered oil is determined. Sludge adhering to the oxidation cell and associated glassware is rinsed with heptane and the washings passed through the same filter used to filter the filterable sludge. The filter is dried in an oven to a constant weight to determine the total filterable sludge.

4.3 The apparatus is dried and weighed to determine the amount of adherent sludge. The sum of the filterable sludge and adherent sludge is reported as total sludge. The percentage change in viscosity and percent oil loss are also reported.

5. Significance and Use

5.1 Degradation of gear oils by oxidation or thermal breakdown, or both, can result in sludge buildup and render the oil unsuitable for further use as a lubricant.

5.2 This is the only test method that employs glassware to measure the amount of sludge produced during oxidation and thermal degradation. This test method is a modification of Test Method **D2893** which measures the viscosity increase and precipitation number of the oil stressed at 95°C, but does not measure the amount of sludge formed.

5.3 This test method can be used to evaluate the oxidation/thermal stability of gear oils. However, the test results may not correlate with the performance of gear oils in field service.

6. Apparatus

6.1 *Heating Bath or Block*,³ thermostatically controlled, capable of maintaining the oil sample in the oxidation cell at a uniform temperature of 120 ± 1°C and large enough to hold a minimum of two oxidation cells and sufficiently deep to allow approximately 120 mm of the test tubes to extend above the heating liquid or block. The heating block is further described in Test Method **D4871**.

6.2 *Oxidation Cell*,³ consists of borosilicate glass; a 38-mm inside diameter and a 300 ± 5-mm length is required.

NOTE 2—While the round-robin test used the oxidation cell from a specific equipment manufacturer in determining the precision statement, the test method permits the use of other oxidation cells that meet the requirements of 6.2.

6.3 *Air Delivery Tube*,³ a borosilicate glass tube having an inside diameter of 5 mm and a minimum length of 320 mm, with the lower tip cut at a 45° angle.

NOTE 3—The oxidation cell and delivery tube are further described in Test Method **D4871**.

6.4 *Flowmeters*,³ one for each oxidation cell, capable of measuring an air flow of 3 L/h ± 0.5 L/h.

6.5 *Air Dryer*—Before being supplied to the flowmeters, the air shall be passed through a drying tower packed with indicating grade anhydrous calcium sulfate or equivalent. The quantity of desiccant should be sufficient to last for the entire test. It is recommended that the drying tower be filled with fresh desiccant prior to the test.

6.6 *Filter*, glass fiber, 2.8-μm porosity, 47 mm in diameter.

6.7 *Balance*, electronic, top-loading, capable of weighing to the nearest centigram (0.01 g) and having the capacity to weigh up to 2000 g.

6.8 *Filter Holder*, 47 mm, consisting of a borosilicate glass funnel and a funnel base with a coarse-grade fritted glass filter support or stainless steel screen support such that the filter can be clamped between the ground glass sealing surfaces of the funnel and its base by means of a metal clamp.

³ The sole source of supply of the apparatus (universal glassware and heating bath with flowmeters as a complete unit) known to the committee at this time is Falex Corp., 1020 Airpark Drive, 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.

6.9 *Oven*, explosive-proof, capable of heating from 50 to 60°C and of a sufficient size to hold oxidation cells.

6.10 *Thermometer*, ASTM solvent distillation thermometer having a range from 98 to 152°C and conforming to the requirement for Thermometer 41C in accordance with Specification **E1**.

6.11 *Vacuum Source*, to provide pressure reduction to 100 ± 5 mm Hg absolute pressure.

7. Reagents and Materials

7.1 *Air Supply*, dried air, oil free, at constant pressure to permit 3 L/h air flow through the system. House air supply or pressurized air cylinders can be used.

7.2 *Calcium Sulfate Desiccant*, Anhydrous, indicating grade (desiccant that changes color when it nears saturation with water). Desiccants equivalent to calcium sulfate can be used.

7.3 *Heptane*, minimum purity—99.75 %. (**Warning**—Heptane is flammable and a health hazard.)

8. Sampling

8.1 Samples for this test method can come from tanks, drums, small containers, or operating equipment. Therefore, use the applicable apparatus and techniques described in Practice **D4057** to obtain suitable samples.

8.2 Special precautions to preserve the integrity of a sample will not normally be required. It is good practice to avoid undue exposure of samples to heat, sunlight, or strong direct light. Visibly heterogeneous samples should not be used.

8.3 It is recommended that a 200-mL representative be obtained. To ensure the aliquot being tested is representative of the sample, agitation; for example, stirring or shaking of the oil prior to obtaining an aliquot, is recommended.

9. Preparation of Apparatus

9.1 *Cleaning Glassware*:

9.1.1 Clean new glassware by washing with a hot detergent solution (using a bristle brush) and rinse thoroughly with tap water. When any visible deposits remain, soaking with a hot detergent solution can be helpful. After final cleaning by soaking with a suitable cleaning solution rinse thoroughly with tap water and then distilled water, and allow to dry at room temperature or in an oven.

9.1.2 Used glassware should be cleaned immediately following the end of a test. When additional cleaning is necessary, use a non-chromic acid containing cleaning solution.

9.2 *Heating Block or Bath*—Ensure that the heating block or bath is able to heat the oxidation cell at the control temperature of 120°C.

9.3 *Flowmeter*—Ensure that the flowmeter is capable of delivering the desired flow rate of 3.0 L of air per hour.

10. Procedure

10.1 Using Test Method **D445**, determine the viscosity of the oil at 100°C. Record as V_2 .

10.2 Adjust the heating bath or block to a temperature high enough to maintain the oil in the desired number of oxidation cells at the required temperature of 120 ± 1°C.

10.3 Place and weigh a 1000-mL beaker on the balance. Zero the tare of the beaker and place the oxidation cell in the

beaker. Insert the air delivery tube in the cell. The angled tip of the air delivery tube should rest on the bottom of the oxidation cell. Obtain the weight of the cell and delivery tube. Record as B_1 .

10.4 Slowly pour 100.00 ± 0.1 g of oil sample into the oxidation cell which contains the air delivery tube. Obtain the weight of the oil sample, air delivery tube, and oxidation cell, A_2 .

10.5 Place the oxidation cell containing the oil sample and air delivery tube in the heating block or bath.

10.6 Connect the air delivery tube to the air source from the flowmeter. Adjust the flow of dried air to 3 ± 0.5 L/h.

10.7 Monitor the temperature of the oil sample and adjust the heating block/bath thermostat, if necessary. Observe the temperature of the oil sample twice during the first hour, then once every hour for at least 3 h, making the necessary adjustment each time until a constant temperature of $120 \pm 1^\circ\text{C}$ is read twice in succession. Check the air flow and temperature at least twice a day and adjust the control flow, when necessary.

10.8 At the end of 312 h (13 days), remove the oxidation cell containing the oxidized oil and delivery tube from the bath or block, and allow the oxidized oil to cool to room temperature. When an oil bath is used, rinse the outside of the oxidation cell with heptane and wipe dry. Place the cell and its contents in a tared 1000-mL beaker and obtain the weight of the oil sample, oxidation cell, and delivery tube as in 10.4. Record this weight, A_1 .

10.9 Weigh a 2.8- μm glass fiber filter to the nearest 0.01 g and record, F_1 . Using the filter holder apparatus, vacuum filter the cooled oxidized oil through the glass filter. Determine the viscosity at 100°C of the filtered oil by Test Method D445. Record the viscosity result, V_1 .

NOTE 4—The viscosity and sludge determinations for the oxidized oil should be done within four days after removing the cell from the heating bath or block. It has been found that extending storage time beyond four days can give variable test results.

10.10 Wash the oxidation cell and air delivery tube with heptane and filter the washings through the same filter. Continue washing until the heptane is no longer colored by the oxidized oil. Draw air through the filter until the precipitate appears dry. Place the filter in an operating hood for 0.5 h to evaporate heptane. Transfer the filter to a 50 to 60°C explosive-proof oven for 0.5 h. Let cool to room temperature and weigh, F_2 .

10.11 Allow heptane to evaporate from the oxidation cell and then place the cell in an explosive-proof oven at 50 to 60°C for 1 to 2 h. Dry the air delivery tube by passing air through the tube. Let the oxidation cell cool to room temperature. Weigh the oxidation cell and air delivery tube, B_2 .

11. Calculations

11.1 Determine the amount of sludge, viscosity change, and oil loss by the following equations.

11.1.1 Filterable sludge on filter, g.

$$D_2 = F_2 - F_1 \quad (1)$$

where:

D_2 = amount of sludge on filter pad, g,
 F_2 = weight of filter pad and sludge, g, and
 F_1 = weight of filter, g.

11.1.2 Adherent sludge on oxidation cell and air delivery tube, g.

$$D_1 = B_2 - B_1 \quad (2)$$

where:

D_1 = amount of sludge on oxidation cell and air delivery tube, g,
 B_2 = weight of oxidation cell and air delivery tube, solvent washed and dried, g, and
 B_1 = original weight of oxidation cell and air delivery tube, g.

11.1.3 Total sludge, g.

$$D_3 = D_1 + D_2 \quad (3)$$

where:

D_3 = total amount of sludge, g.

11.1.4 Viscosity at 100°C increase, mm^2/s , %

$$V_3 = \frac{V_1 - V_2}{V_2} \times 100 \quad (4)$$

where:

V_3 = viscosity increase, %,
 V_2 = original viscosity of oil sample, and
 V_1 = viscosity of oxidized oil sample.

11.1.5 Oil loss, g.

$$C_3 = A_2 - A_1 \quad (5)$$

where:

C_3 = oil loss, g,
 A_2 = original combined weight of oil sample, oxidation cell, and air delivery tube, g, and
 A_1 = combined weight of oxidized oil, oxidation cell, and air delivery tube, g.

12. Report

12.1 Report the amount of filterable sludge on the filter to the nearest 0.01 g.

12.2 Report the amount of adherent sludge on the oxidation tubes to the nearest 0.01 g.

12.3 Report the total amount of sludge to the nearest 0.01 g or to the nearest 0.01 %.

12.4 Report the viscosity increase in percent.

12.5 Report oil loss to the nearest 0.01 g or percent.

13. Precision and Bias⁴

13.1 *Precision*—The precision of this test method as determined by the statistical examination of the inter-laboratory test results is as follows.

13.1.1 *Total Sludge*:

13.1.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials

⁴ Supporting data have been filed at ASTM International Headquarters and can be obtained by requesting D02-1403 and D02-1150.

would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$\text{Repeatability} = 0.42 \sqrt{x} = g (\%) \quad (6)$$

where:

x = found mean value

13.1.1.2 *Reproducibility*—The difference between two single and independent operators working in different laboratories on identical material would in the long run, exceed the following values only in one case in twenty:

$$\text{Reproducibility} = 1.4 \sqrt{x} \text{ g}/100 \text{ g} (\%) \quad (7)$$

where:

x = found mean value.

13.1.2 *Viscosity Increase:*

13.1.2.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would in the long run, in the normal and correct operation of the test method, exceed the following values only in one in twenty:

$$\text{Repeatability} = 0.058 (x)^2 = \% \quad (8)$$

where:

x = found mean value.

13.1.2.2 *Reproducibility*—The difference between two single and independent results obtained by different operators in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty:

$$\text{Reproducibility} = 0.14 (x)^2 = \% \quad (9)$$

where:

x = found mean value.

13.1.3 The precision for filterable sludge, adherent sludge, and oil loss were not determined.

13.2 *Bias*—There is no accepted reference material suitable for determining the bias for this test method; therefore, bias has not been determined.

13.2.1 The round-robin test used to determine the precision and bias consisted of eleven laboratories, five gear oil samples, and duplicate testing per sample per laboratory.

14. Keywords

14.1 extreme pressure gear oils; gear oil; lubricants; non-extreme pressure gear oils; oxidation; sludge; universal oxidation/thermal stability test glassware; universal oxidation test; viscosity increase

APPENDIX

(Nonmandatory Information)

X1. SAMPLE CALCULATION SHEET

X1.1 *Oil Loss Calculation:*

Before Test—

A_1 Mass of Test Tube + Air Tube + Oil Sample = 291.69 g

After Test—

A_2 Mass of Test Tube + Air Tube + Oil Sample = 290.19 g

$$\text{Oil Loss } (A_1 - A_2) = 1.50 \text{ g} (\%) \quad (\text{X1.1})$$

X1.2 *Calculation of Adherent Sludge on Tubes:*

B_2 Mass of Oxidation Cell + Air Tube, After Test = 193.35 g

B_1 Original Mass of Oxidation Cell and Air Tube = 191.69 g

D_1 Mass of Adherent Sludge on Tubes ($B_2 - B_1$) = 1.66 g (%)

X1.3 *Calculation of Filterable Sludge on Filter:*

F_2 Mass of Filter + Filterable Sludge = 0.38 g

F_1 Mass of Filter = 0.07 g

D_2 Mass of Filterable Sludge ($F_2 - F_1$) = 0.31 g (%)

X1.4 *Calculation of Total Sludge per 100 g Oil:*

D_1 Mass of Adherent Sludge on Tubes + D_2

Mass of Filterable Sludge = 1.97 g (%)

X1.5 *Calculation of Viscosity Increase:*

V_2 Viscosity Before Test = 17.74 mm²/s

V_1 Viscosity After Test = 18.18 mm²/s

$$\text{Viscosity Increase, \%} = \frac{V_1 - V_2}{V_2} \times 100 = 3.04 \% \quad (\text{X1.2})$$

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