Designation: D2893 – 04 (Reapproved 2014)$^{\varepsilon 1}$

Standard Test Methods for Oxidation Characteristics of Extreme-Pressure Lubrication Oils[1]

This standard is issued under the fixed designation D2893; 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—Subsection 6.1 was updated editorially in July 2014.

1. Scope

1.1 These test methods (A and B) cover the determination of the oxidation characteristics of extreme-pressure fluid lubricants, gear oils, or mineral oils.

NOTE 1—The changes in the lubricant resulting from these test methods are not always necessarily associated with oxidation of the lubricant. Some changes may be due to thermal degradation.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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]

D91 Test Method for Precipitation Number of Lubricating Oils

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

D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Summary of Test Method

3.1 The oil sample is subjected to a temperature of 95°C (Test Method A) or 121°C (Test Method B) in the presence of dry air for 312 h.

3.2 The oil is then tested for precipitation number and increase in kinematic viscosity.

4. Significance and Use

4.1 These test methods have been widely used to measure the oxidation stability of extreme pressure lubricating fluids, gear oils, and mineral oils.

5. Apparatus

5.1 *Heating Bath or Block,* thermostatically controlled, capable of maintaining the oil sample in the test tube at a temperature of 95 ± 0.2°C (Test Method A), or 121 ± 1.0°C (Test Method B) and large enough to hold the desired number of oxidation cells immersed in the heating bath or block to a depth of approximately 350 mm. The liquid heating bath shall be fitted with a suitable stirring device to provide a uniform temperature throughout the bath.

5.2 *Test Tubes,* of borosilicate glass, 41 ± 0.5 mm inside diameter and 600 mm in length are required, each fitted with a slotted cork (Note 2) stopper into which shall be inserted a glass air delivery tube of 4 to 5 mm of inside diameter. The length of the air delivery tube shall be such that one end reaches to within 6 mm of the bottom of the tube and the other end projects 60 to 80 mm from the cork stopper.

NOTE 2—New corks should be used for each run.

5.3 *Flowmeter,* one to each test tube, capable of measuring an air flow of 10 L/h with an accuracy of ±0.5 L/h.

5.4 *Thermometer*—ASTM Solvent Distillation Thermometer having a range from 76 to 126°C and conforming to the requirement for Thermometer 40C as prescribed in Specification E1. Alternatively, calibrated thermocouples may be used.

5.5 *Air Supply*—Oil-free, dried air at constant pressure shall be supplied to each flowmeter.

5.6 *Air Dryer*—Before being supplied to the flowmeters, the air shall be passed through a drying tower packed with indicating grade of anhydrous calcium sulfate or equivalent. The quantity of dessicant should be sufficient to last for the entire test.

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[1] These test methods are under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand 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.

6. Preparation of Apparatus

6.1 *Cleaning of Oxidation Cells*—Clean glassware with a suitable cleaning solution. (**Warning**—Chromic acid (see Note 3) causes severe burns. A recognized carcinogen. Strong oxidizer, contact with other material may cause fire. Hygroscopic.)

NOTE 3—While other suitable cleaning solutions are now available, the round robin used glassware cleaned with chromic acid. Other cleaning solutions such as NoChromix and Micro Clean have been found suitable. In a referee situation, glassware shall be cleaned by a cleaning solution satisfactory to all parties involved.

7. Procedure

7.1 Adjust the heating bath to a temperature high enough to maintain the oil in the desired number of oxidation cells at the required temperature of 95 ± 0.2°C (Test Method A) or 121 ± 1.0°C (Test Method B). Determine the viscosity at 100°C by Test Method D445/IP 71 and the precipitation number by Test Method D91, on each sample.

7.2 Pour 300 mL of each oil sample into a test tube and immerse the test tube in the heating bath so that the heating medium is at least 50 mm above the level of the oil sample. Place the corks and air delivery tubes in the test tubes making sure that the lower ends of the tubes are within 6 mm of the bottoms of the test tubes.

7.3 Connect the air delivery tubes to the dried air supply through the flowmeters. Adjust the flow of air to 10 ± 0.5 L/h. Check the temperature of the oil samples and the rate of air flow every hour and make necessary adjustments. Once the oil samples have reached the desired temperature of 95 ± 0.2°C (Test Method A) or 121 ± 1.0°C (Test Method B), initiate the start of the test.

NOTE 4—When using multi-cell baths, one way of checking the temperature of the oil samples can be to use a dummy cell in the bath, similar to the way it is used in Test Method D943.

7.4 Maintain the air flow and bath or block temperature constant, checking them periodically for the duration of the test.

7.5 Remove the test tubes from the bath or block 312 ± 1 h (13 days) after the start of the test. Mix each oil sample thoroughly and test them for viscosity at 100°C by Test Method D445/IP 71 and precipitation number by Test Method D91.

8. Calculation

8.1 Calculate the kinematic viscosity increase as follows:

$$\text{Viscosity increase, \%} = \left[(B - A)/A\right] \times 100 \tag{1}$$

where:

A = kinematic viscosity on original sample, and
B = kinematic viscosity after oxidation.

9. Report

9.1 On the original sample, and on the oxidized sample at the termination of test, report the precipitation number determined in accordance with Test Method D91.

9.2 Report the percent increase in viscosity at 100°C as determined in Section 8.

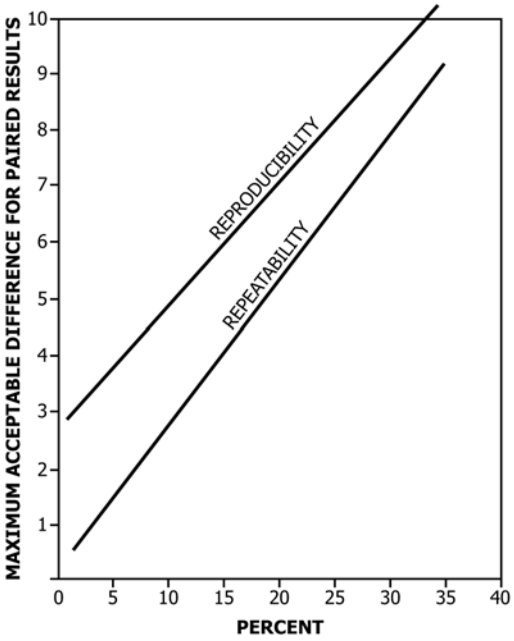

FIG. 1 Precision Data, Viscosity Increase

10. Precision and Bias (Test Method A)[3]

10.1 The precision of this test method is not known to have been obtained in accordance with currently accepted guidelines (for example, in Committee D02 Research Report RR:D02-1007).

10.2 *Viscosity Increase:*

10.2.1 *Repeatability*—Duplicate results by the same operator shall be considered suspect if they differ by more than the maximum acceptable difference for repeatability as shown in Fig. 1.

10.2.2 *Reproducibility*—The results submitted by each of two laboratories shall be considered suspect if they differ by more than the maximum acceptable difference for reproducibility as shown in Fig. 1.

10.3 *Precipitation Number, Increase:*

10.3.1 *Repeatability*—Duplicate results by the same operator shall be considered suspect if they differ by more than the maximum acceptable difference for repeatability as shown in Fig. 2.

10.3.2 *Reproducibility*—The results submitted by each of two laboratories shall be considered suspect if they differ by more than the maximum acceptable difference for reproducibility as shown in Fig. 2.

10.4 *Bias*—The procedure in this test method has no bias because the value of these changes can only be defined in terms of a test method.

11. Precision and Bias (Test Method B)[4]

11.1 *Viscosity Increase:*

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[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1150.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1539.

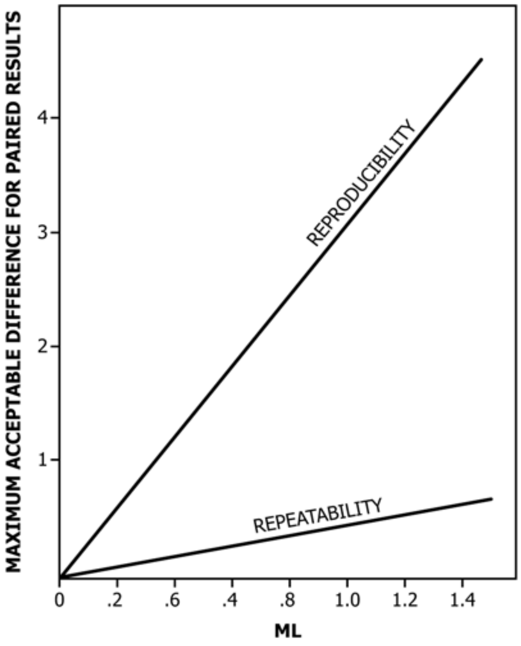

FIG. 2 Precision Data, Precipitation Number Increase

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

$$\text{Repeatability} = 0.30X \qquad (2)$$

where:

X = the mean value.

11.1.2 *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:

$$\text{Reproducibility} = 1.1X \qquad (3)$$

where:

X = the mean value.

NOTE 5—This precision statement was prepared with data on six oils tested by six cooperators. The oils covered values of 0-20 % viscosity increase.

11.2 The precision for the precipitation number was not determined.

11.3 *Bias*—The procedure in this test method has no bias, because the value of these changes can only be defined in terms of a test method.

12. Keywords

12.1 extreme pressure gear oils; oxidation testing—petroleum; stability—oxidation; stability—thermal

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