

Designation: D2603 – 01 (Reapproved 2007)

Test Method for Sonic Shear Stability of Polymer-Containing Oils¹

This standard is issued under the fixed designation D2603; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

- 1.1 This test method covers the evaluation of the shear stability of an oil containing polymer in terms of the permanent loss in viscosity that results from irradiating a sample of the oil in a sonic oscillator. This test method can be useful in predicting the continuity of this property in an oil where no change is made in the base stock or the polymer. It is not intended that this test method serve to predict the performance of polymer-containing oils in service.
- 1.2 Evidence has been presented that correlation between the shear degradation results obtained by means of sonic oscillation and those obtained in mechanical devices can be poor. This is especially true in the case of automotive engines. Further evidence indicates that the sonic technique may rate different families of polymers in a different order than mechanical devices.^{2,3}
- 1.3 Because of these limitations, the committee under whose jurisdiction this test method falls has developed an alternative shear test method using a diesel injector nozzle, Test Method D3945. While that test method has found some utility in the evaluation of crankcase oils, the stress imparted to the sample has been found to be insufficient to shear polymers of the shear-resistant type found in aircraft hydraulic fluids.
- 1.4 This test method is used for polymeric additive specifications, especially in the hydraulic fluid market.
- 1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for informational purposes only.
- 1.6 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:⁴

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

D3945 Test Methods for Shear Stability of Polymer-Containing Fluids Using a Diesel Injector Nozzle⁵
D6022 Practice for Calculation of Permanent Shear Stability Index

3. Summary of Test Method

3.1 A convenient volume of polymer-containing oil is irradiated in a sonic oscillator for a period of time and the changes in viscosity are determined by Test Method D445. Standard reference fluids containing either a readily sheared or shear-resistant polymer are run frequently to ensure that the equipment imparts a controlled amount of sonic energy to the sample.

Note 1—The conditions to obtain the data for the precision statement were a 30-mL sample, 10 min, and at 0°C.

4. Significance and Use

4.1 This test method permits the evaluation of shear stability with minimum interference from thermal and oxidative factors which may be present in some applications. Within the limitations expressed in the scope of this test method, it has been successfully applied to hydraulic fluids, transmission fluids, tractor fluids, and other fluids of similar applications. It has been found applicable to fluids containing both readily sheared and shear-resistant polymers. Correlation with performance in the case of automotive engine applications has, to date, not been established.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07.0B on High Temperature Rheology of Non-Newtonian Fluids.

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² The Effects of Polymer Degradation on Flow Properties of Fluids and Lubricants, ASTM STP 382, ASTM, 1965. Available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428, www.astm.org.

³ Shear Stability of Multigrade Crankcase Oil, ASTM DS 49, ASTM, 1973. Available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428, www.astm.org.

⁴ 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

⁵ Withdrawn.

5. Apparatus

- 5.1 Sonic Shear Unit, fixed frequency oscillator and sonic horn.
- 5.2 Auxiliary Equipment—To facilitate uniform performance, the following auxiliary equipment is recommended:
- 5.2.1 Cooling Bath or Ice Bath—To maintain a desired temperature such as 0° C.
 - 5.2.2 Griffin 50 mL Beaker, borosilicate glass.
- 5.2.3 *Sonic-Insulated Box*—To enclose the sonic horn to reduce the ambient noise level produced by the sonic shear unit.
- 5.3 *Viscometer*—Any viscometer and bath meeting the requirements of Test Method D445.

6. Reference Fluids

6.1 The primary reference fluid is ASTM Reference Fluid A,⁶ a petroleum oil containing a polymer capable of being broken down by turbulence at high rates of shear. This oil has the following typical properties:

	Petroleum Base	Reference Fluid A
Viscosity at 100°C, mm ² /s (cSt)	5.4	10.8
Viscosity at 40°C, mm ² /s (cSt)	32	58

6.2 A second reference fluid is ASTM Reference Fluid B,⁶ a petroleum oil containing a polymer capable of being broken down by turbulence at high rates of shear. This oil has a viscosity of about 13.6 mm²/s (cSt) at 40°C.

7. Calibration of Apparatus

- 7.1 The reference fluid provides a practical way to define the performance (severity level) of a sonic oscillator unit so that satisfactory comparison can be made between tests run on different days in the same unit and between tests run with different units.
- 7.2 The decrease in viscosity observed for a given polymer-containing oil on irradiation in an oscillator unit depends on a number of factors; these include sample volume, irradiation time, and oscillator power setting. These parameters can be varied in order to increase or decrease severity of test exposure. Tuning of the oscillator-horn combination is also required in order to assure efficiency of energy coupling between the two units. The procedure described in 7.3 and 7.4 is recommended for establishing a reproducible performance level for a given unit.
- 7.3 Introduce 30 mL of a reference fluid into the 50-mL Griffin beaker. Immerse the beaker in ice water or in another constant temperature bath until sample fluid level is below the liquid level in the bath. The beaker shall be positioned in a vertical position in the bath. Secure beaker in this position and condition the sample for 10 min before commencing exposure. Immerse the sonic horn into sample fluid until tip is exactly 10 mm below surface of fluid (see Fig. 1). The horn shall be positioned in a vertical position in the fluid and centered in the beaker (a weighted ringstand may be used to support the horn

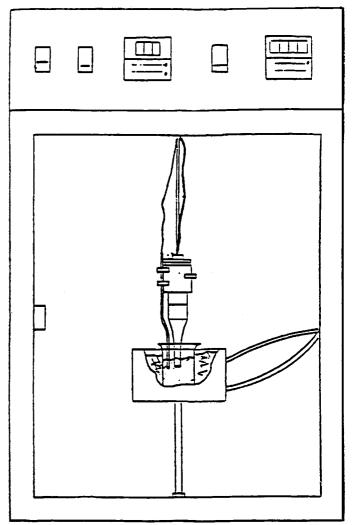


FIG. 1 Schematic of Sonic Probe Type Apparatus, Support Systems, Cabinet and Power Supply

in this position). Irradiate the fluid for 10 min at a preselected power setting. Experience has shown that with some instruments this may require readjustment of the controls during irradiation to maintain the preselected power. Determine the decrease in reference fluid viscosity. Repeat as necessary to determine the proper power setting to produce a viscosity change at 40° C of 15.0% (± 0.5 %). Use this power setting for subsequent test runs. Daily recalibration of the apparatus is required because the power setting required to produce a stated viscosity loss will probably vary from day to day. Experience with a given apparatus set will determine whether more frequent calibration is required. Calibration in this manner will assure that repeatable severity levels are established for any predetermined set of test parameters.

7.4 This procedure can be used to establish severity levels appropriate for the requirements of a variety of applications. Once the conditions for a given severity level have been established, it is possible to compare the shear stability of fluids at one or more irradiation times. If calibration at conditions other than 15.0 % \pm 0.5 % decrease of reference fluid viscosity at 40°C are employed, that fact shall be included in the test report.

⁶ This fluid can be obtained from RohMax USA, Inc., 723 Electronic Dr., Horsham, PA 19044-2228.

Note 2—For interlaboratory comparisons, the laboratories should define a common severity level in terms of a specified viscosity loss of the reference fluid occurring in a given irradiation time with a given volume of sample. For example, "the reference fluid should undergo a viscosity loss of 15 % when a 30 mL sample is treated for 10 min."

Alternatively, two or more laboratories can define equivalent operating conditions by a curve of percent viscosity loss of a reference fluid as a function of irradiation time. Power setting and sample size are varied until each laboratory finds a combination that satisfies two or more points on the reference curve. This procedure provides a comparison of the shear stability of an unknown fluid with that of the reference fluid over a range of irradiation times.

8. Procedure

8.1 Clean the sonic horn with a suitable solvent and calibrate the apparatus as described in 7.3 and 7.4. Introduce 30 mL of the sample into a clean 50-mL Griffin beaker and immerse in the constant temperature bath at the desired temperature (0°C unless otherwise specified). The beaker must be in a vertical position. Allow the sample to equilibrate for 10 min. Immerse the sonic horn in the fluid as described in 7.3. Irradiate the sample at exactly the same energy level and for the same time as determined in 7.3 and 7.4. Upon completion of irradiation, remove the sample and clean the sonic horn with a suitable solvent.

8.2 Determine the viscosity of the sample by Test Method D445 before and after irradiation.

9. Calculation

9.1 Calculate the percentage loss of viscosity as follows:

Viscosity loss,
$$\% = [(V_0 - V_f)/V_0] \times 100$$
 (1)

where:

 V_0 = viscosity of oil before irradiation, mm²/s (cSt), and

 V_f = viscosity of oil after irradiation, mm 2 /s (cSt).

The permanent shear stability index (PSSI) may also be calculated in accordance with Practice D6022, provided the base fluid viscosity is known.

10. Report

10.1 Report the result for the sample and the specified reference fluid as the percentage loss of viscosity at 40°C in 10 min. If other irradiation times/temperatures for determination of viscosity or reference fluids are employed, the report shall indicate those facts.

11. Precision and Bias ⁷

- 11.1 Repeatability—The difference between two independent 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 1.6 percentage loss units only in one case in twenty.
- 11.2 Reproducibility—The difference between two independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed 3.3 percentage loss units only in one case in twenty.
- 11.3 *Bias*—The procedures in this test method have no bias because the results can be defined only in terms of the test method.

12. Keywords

12.1 shear stability; sonic shear test; viscosity stable

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⁷ Supporting data are available from ASTM Headquarters. Request D02-1264.