Designation: D7603 – 13

Standard Test Method for Determination of Storage Stability and Compatibility in Automotive Gear Oils[1]

This standard is issued under the fixed designation D7603; 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 covers the determination of storage stability characteristics and the compatibility of automotive gear lubricants when blended with reference lubricants. The purpose of the test is to determine if performance-enhancing additives separate out under defined conditions.

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]

D235 Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)

D1193 Specification for Reagent Water

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D5760 Specification for Performance of Manual Transmission Gear Lubricants

E542 Practice for Calibration of Laboratory Volumetric Apparatus

E1272 Specification for Laboratory Glass Graduated Cylinders

2.2 *SAE Standards:*[3]

J2360 Lubricating Oil, Gear Multipurpose (Metric) Military Use

2.3 *Federal Test Method Standard:*[4]

FED-STD-791/3440.1 Storage Solubility Characteristics of Universal Gear Lubricants

FED-STD-791/3440.2 Compatibility Characteristics of Universal Gear Lubricants

3. Terminology

3.1 *Definitions:*

3.1.1 *calibrate, v*—to determine the indication or output of a measuring device (for example, thermometer, manometer, engine) with respect to that of a standard.

3.1.2 *candidate oil, n*—an oil that is intended to have the performance characteristics necessary to satisfy a specification and is to be tested against that specification. **D4175**

3.1.3 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison.

3.1.3.1 *Discussion*—Reference oils are used to calibrate testing facilities, to compare the performance of other oils, or to evaluate other materials (such as seals) that interact with oils. **D4175**

3.1.4 *test oil, n*—any oil subjected to evaluation in an established procedure. **D4175**

4. Summary of Test Method

4.1 Separation of the performance-enhancing additives in a test oil during storage is determined by heating it to 120 °C, storing at room temperature for 30 days, and making a qualitative observation as to whether any material has separated out.

4.1.1 An optional, non-mandatory test method is also described in Appendix X1 and Appendix X2 for quantifying the percent of the additive that separated out during storage of the test oil.

4.2 The compatibility of a test oil is determined by blending it with different reference oils, heating to 120 °C, storing at room temperature for 30 days, and making a qualitative observation as to whether any material has separated out.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.B0.01 on Passenger Car Engine Oils.

Current edition approved June 15, 2013. Published July 2013. Originally approved in 2010. Last previous edition approved in 2010 as D7603 – 10. DOI:10.1520/D7603-13.

[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.

[3] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

[4] Available online at www.assistdocs.com (search for FED-STD-791 in the document ID field or from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

***A Summary of Changes section appears at the end of this standard**

4.2.1 An optional, non-mandatory test method is also described in Appendix X1 and Appendix X2 for quantifying the percent of the additive that separated out of the test oil/reference oil mixture due to a compatibility problem between these two oils.

5. Significance and Use

5.1 To avoid equipment failure, a gear oil should remain a homogeneous liquid and the performance-enhancing additives should not separate out when the oil is stored for an extended period of time.

5.2 In addition, because different oils are often mixed when topping off, gear oils from different manufacturers, or containing different base fluids or performance-enhancing additives should be completely miscible and compatible with each other. Any incompatibility of such mixtures can also result in equipment failure if gelation or additive dropout occurs.

5.3 The test procedures described in this test method are designed to evaluate the performance of gear oils in each of the above circumstances.

5.4 This test method is based on the separate test methods FED-STD-791/3440.1 and FED-STD-791/3440.2. Minor changes have been made to the FED test methods to provide a coherent unified procedure. These changes do not significantly alter the test procedures. This test method has, therefore, potential for use as an alternative to the FED test methods in gear oil specifications such as SAE J2360 and Specification D5760.

6. Apparatus

6.1 *Centrifuge Tubes*—Cone shaped, with a capacity of 100 mL, conforming to the dimensions given in Fig. 1, and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1, shall be clear and distinct. Constrict the mouth of each tube for closure with a solvent-resistant rubber stopper. Scale error tolerances and the smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air free water at 20 °C when reading the bottom of the shaded meniscus. Verify the accuracy of the graduation marks, in accordance with Practice E542, using equipment traceable through the National Institute of Standards and Technology (NIST) (www.nist.gov) or other national standards. Tubes conforming to these requirements are commercially available.

TABLE 1 Minimum Graduation Requirements and Maximum Calibration Tolerances for the Centrifuge Tubes

Range, mL	Subdivision, mL	Volume Tolerance, mL
0 to 0.1	0.05	± 0.02
> 0.1 to 0.3	0.05	± 0.03
> 0.3 to 0.5	0.05	± 0.05
> 0.5 to 1.0	0.10	± 0.05
> 1.0 to 2.0	0.10	± 0.10
> 2.0 to 3.0	0.20	± 0.10
> 3.0 to 5.0	0.5	± 0.20
> 5.0 to 10	1	± 0.50
> 10 to 25	15	± 1.00
> 25 to 100	25	± 1.00

6.2 *Centrifuge:*

6.2.1 Use a centrifuge meeting all safety requirements for normal use and capable of spinning two or more filled centrifuge tubes at a speed that can be controlled to give a minimum relative centrifugal force, rcf, of 407 at the tips of the tubes.

6.2.2 A centrifuge with a diameter of swing (measured between the tips of opposite tubes when in the rotating position) of 407 mm ± 13 mm achieves this condition at a rotation speed of 1500 r/min ± 25 r/min.

6.2.2.1 If the available centrifuge does not conform dimensionally to that described in 6.2.2, determine the rotation speed necessary to produce an rcf of 407 for other diameters of swing from the following equation:

$$r/min = 1500\sqrt{407/d} \quad (1)$$

where:

d = the diameter, in millimetres, of the swing measured between the tips of opposite tubes when in the rotating position.

6.2.3 Construct the revolving head, trunnion rings, and trunnion cups, including the cushions, to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. Enclose the centrifuge by a metal shield or case strong enough to contain flying debris in the event a tube breaks or the centrifuge malfunctions.

6.3 *Mass Balance*—Having a minimum indication resolution of 1 mg.

6.4 *Beaker*—Made from heat-resistant glass and having a capacity of 400 mL.

6.5 *Desiccator*—Capable of holding several centrifuge tubes in a dry condition.

6.6 *Forced Circulation Oven*—Capable of being controlled at 105 °C ± 3 °C and 120 °C ± 1 °C.

6.7 *Graduate Cylinders:*

6.7.1 *For Measuring Volume of Test Oil for the Storage Stability Test*—Having a capacity of 250 mL.

6.7.2 *For Measuring Volume of Reference and Test Oils for the Compatibility Test*—Conforming to Specification E1272, Class A and having a capacity of 250 mL with graduation marks of 2 mL.

6.8 *Mechanical Stirrer*—Capable of stirring the contents of a beaker with a capacity of 400 mL at approximately 200 r/min.

7. Reagents and Materials

7.1 *Reference Oils for Compatibility Testing*—Use the six reference oils available from the ASTM Test Monitoring Center (TMC)[5] as a 6-pack and identified as *SSCT Fluid (6-pack)*.

7.2 *Solvent*—Use only mineral spirits meeting Specification D235, Type II, Class C for aromatic content (0 % to 2 %

[5] ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206–4489, www.standards.astmtmc.cmu.edu.

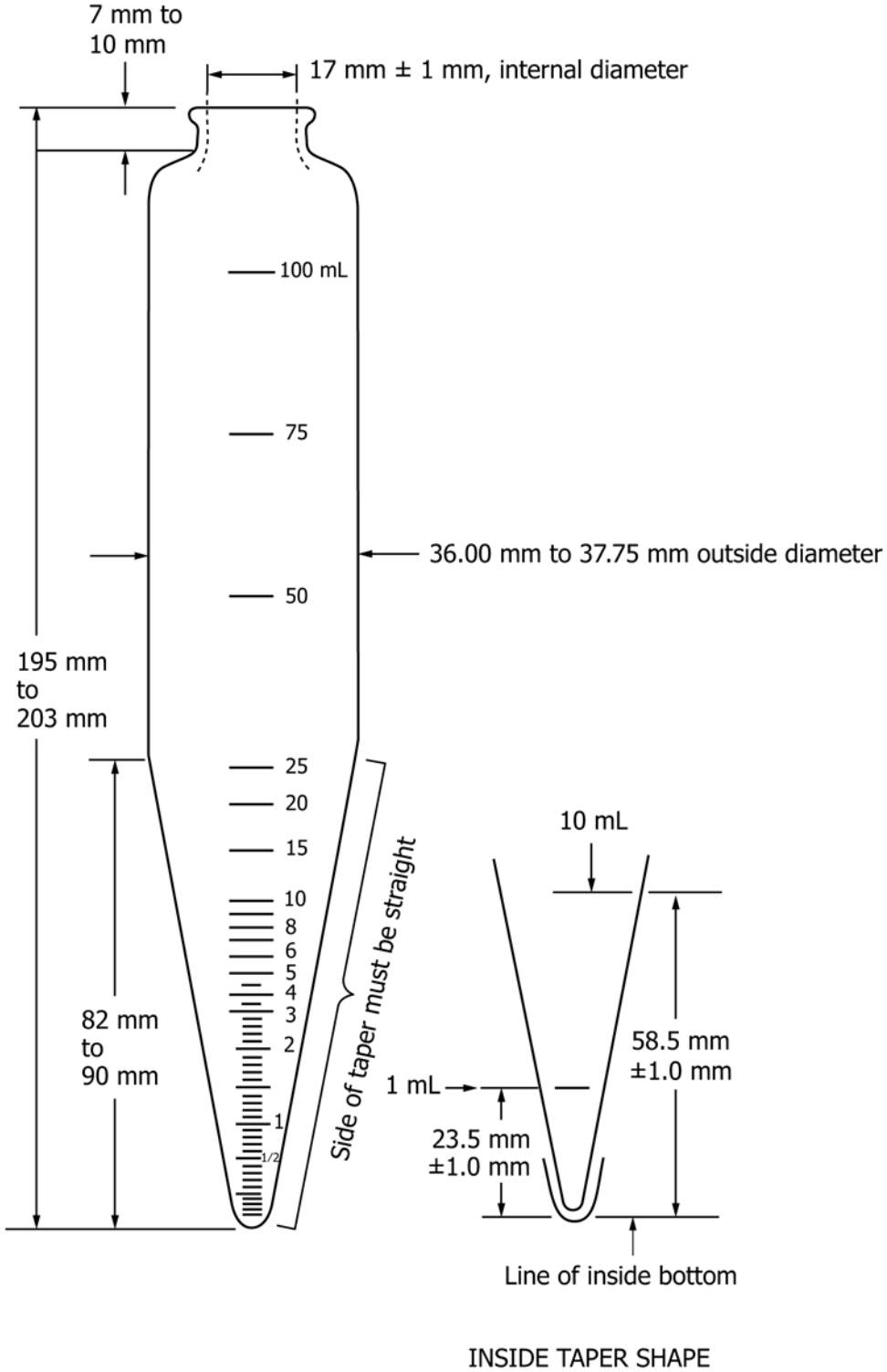

FIG. 1 Cone-Shaped Centrifuge Tube

volume), flash point ≥ 61 °C and color (not darker than +25 on Saybolt scale or 25 on Pt-Co scale). Obtain a Certificate of Analysis for each batch of mineral spirits from the supplier. (**Warning—**Combustible. Health hazard.)

7.3 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets that defined as Type IV of Specification D1193.