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.

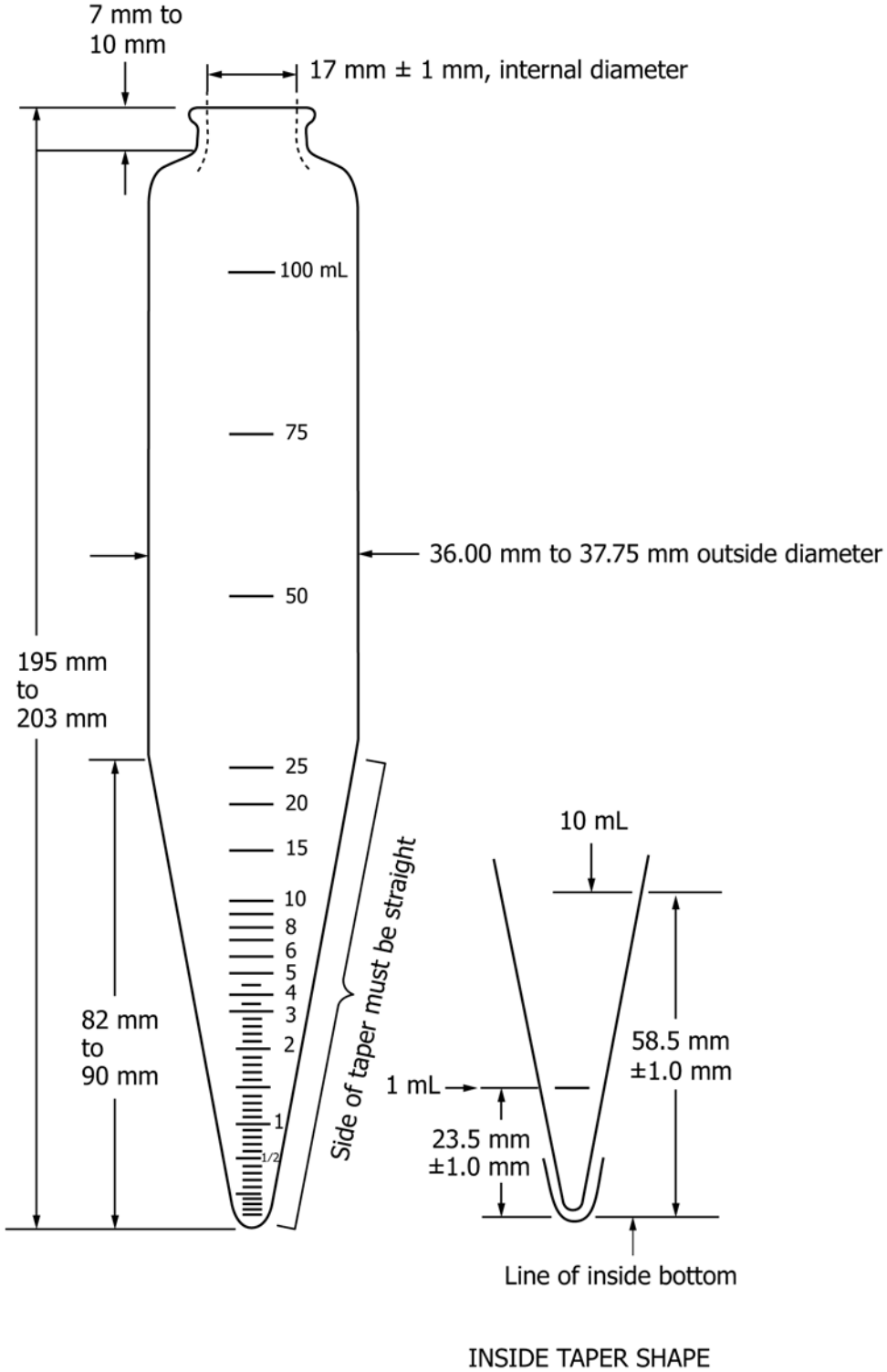

FIG. 1 Cone-Shaped Centrifuge Tube

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.

7.4 *Acetone*—Reagent grade or better. (**Warning—** Extremely flammable. Vapors may cause flash fire.)

7.5 *Liquid Detergent*—Liquinox,[6] a concentrated, anionic liquid product. Use neat.

8. Procedure

8.1 *Storage Stability Testing:*

8.1.1 Perform duplicate storage stability tests (that is, use two centrifuge tubes) for each test oil.

8.1.2 *Preparation of the Centrifuge Tubes, Graduated Cylinders, and Beakers:*

8.1.2.1 Clean each piece of glassware as follows:

(1) Rinse with solvent.

(2) Clean with Liquinox[6] cleaning solution.

(3) Rinse with distilled water.

(4) Rinse with acetone (see Warning in 7.4).

8.1.2.2 Dry the cleaned glassware by laying flat in an oven maintained at 105 °C ± 3 °C for at least 30 min.

8.1.2.3 Remove from the oven and allow to cool to room temperature. Cool the centrifuge tubes in a desiccator.

8.1.2.4 Number each centrifuge tube and measure its mass to the nearest 1 mg. (This information is required if it is desired to quantify the amount of residues; see 8.1.6.2 and 8.2.9.5). Store the tubes in the desiccator until required.

8.1.3 *Preparation of the Test Oil:*

8.1.3.1 Thoroughly shake the test oil prior to sampling.

8.1.3.2 Using a clean, dry, graduated cylinder (see 6.7.1), pour 220 mL of the test oil into a clean, dry beaker (400 mL) and heat in an oven controlled at 120 °C ± 1 °C for 20 min ± 1 min.

8.1.3.3 Remove the beaker from the oven and allow to cool to 25 °C ± 3 °C. Fill each centrifuge tube with test oil to the mark at 100 mL. Stopper the tubes.

8.1.4 *Storing the Test Oil*—Store the centrifuge tubes for 30 days ± 1 day. Place the tubes in an upright position in a darkened area (such as a drawer or cupboard) in a room whose temperature is controlled at 25 °C ± 3 °C.

8.1.5 *Centrifuging*—Transfer the tubes to the centrifuge, taking care not to disturb any material that may have separated from the oil. Operate the centrifuge for 30 min ± 1 min at a speed to give a rcf of 407 at the tips of the tubes (see 6.2).

8.1.6 After centrifuging, remove the tubes from the centrifuge and check each tube visually to determine if solid or liquid material has separated out.

8.1.6.1 If there is no visual evidence of separated material in the centrifuge tubes, stop the storage stability procedure, record that the storage stability test resulted in zero residue, and report as detailed in 9.1.1.

8.1.6.2 If solid or liquid material has separated out, record that the storage stability test resulted in residue, and report as detailed in 9.1.2. If it is desired to quantify the amount of residue, proceed as directed in Appendix X1 and Appendix X2.

8.2 *Compatibility Testing*—In general, when a mixture of a test oil and reference oil is stored, residue can originate because of incompatibility between the test oil and reference oil, or from a storage stability problem associated with the test and reference oils themselves, or both. In the specific case of the reference oils *SSCT Fluid (6-pack)*, the situation is simplified because they do not normally produce storage stability residues.

NOTE 1—In principle, therefore, any residue observed in the compatibility test involving these reference oils arises either from incompatibility between the test oil and the reference oil or from storage stability problems from the test oil alone. In practice, however, there is a possibility that the reference oil(s) alone might produce residue during storage. For instance, the reference oil(s) might have deteriorated during storage, or have become contaminated, or the test might have been carried out incorrectly. To cover such eventualities, the procedure requires that in the event a residue is observed in the compatibility test with a test oil, the reference oil(s) involved are subjected to the storage stability test with a view to eliminating them as the source of residue.

8.2.1 If desired, the compatibility testing may be carried out in parallel with the storage stability testing described in 8.1.

8.2.2 Perform duplicate tests (that is, use two centrifuge tubes).

8.2.3 *Screen the Test Oil for Storage Stability*—Subject the test oil alone (that is, not in combination with the reference oils) to the storage stability test as described in 8.1.

8.2.4 *Preparation of the Centrifuge Tubes, Graduated Cylinders, and Beakers*—Proceed as directed in 8.1.2.

8.2.5 *Preparation of the Test Oil/Reference Oil Mixtures:*

8.2.5.1 Thoroughly shake the test and reference oils prior to sampling.

8.2.5.2 For each reference oil, use separate graduated cylinders (see 6.7.2) to pour 110 mL ± 1 mL each of the reference oil and the test oil into a clean, dry beaker (400 mL). Use the same graduated cylinder each time the test oil is measured, but a clean, dry, graduated cylinder for each of the reference oils. Heat the beakers containing the reference oil/test oil mixtures in an oven at 120 °C ± 1 °C for 20 min ± 1 min.

8.2.5.3 Remove the beakers from the oven and stir the contents while still hot for 5 min, using a mechanical stirrer operated at approximately 200 r/min.

8.2.5.4 Allow to cool to 25 °C ± 3 °C. Fill each centrifuge tube with reference oil/test oil mixture to the mark at 100 mL. Stopper the tubes.

8.2.6 Store and centrifuge the test oil/reference oil mixtures as directed in 8.1.4 and 8.1.5, respectively.

8.2.7 After centrifuging, remove the tubes from the centrifuge and check each tube visually to determine if solid or liquid material has separated out.

8.2.8 *No Residue Observed*—If there is no visual evidence of separated material in the centrifuge tubes, stop the compatibility procedure, record that the test oil produced zero residue in the compatibility test, and report as detailed in 9.2.1.

8.2.9 *Residue Observed*—If solid or liquid material has separated out from one or more of the reference oil/test oil mixtures, proceed as follows:

8.2.9.1 *Check the Operational Validity of the Tests*—This check is to eliminate operational problems as the source of the residue. Items to check include possible contamination of the

[6] The sole source of supply of the detergent known to the committee at this time is Alconox, 30 Glenn St., White Plains, NY, www.alconox.com. Trademark of Alconox, 30 Glenn St., White Plains, NY, www.alconox.com. 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.

test equipment, insufficient cleaning of equipment, and inadvertent use of a wrong oil in place of a reference oil. Rectify any obvious problems.

8.2.9.2 *Check the Storage Stability of the Reference Oil(s)*—For each test oil/reference oil mixture giving a residue, subject the reference oil(s) involved alone (that is, not mixed with test oil) to the storage stability test as described in 8.1. This is to check that the residue does not arise from any storage stability issues with the reference oil(s) themselves (see Note 1). (There is no requirement to check the reference oils for those test oil/reference oil mixtures not giving residues.)

If there are no obvious operational problems and if any of the reference oils alone produce residue in the storage stability test request the Surveillance Panel responsible for this test method to investigate. Proceed to 8.2.9.3 only after it has been confirmed that none of the reference oils alone is responsible for producing residues in the storage stability test.

8.2.9.3 Repeat the compatibility test, as described in 8.2.4 to 8.2.7, for each test oil/reference oil mixture giving residue.

8.2.9.4 *No Residue Observed*—Proceed as described in 8.2.8.

8.2.9.5 *Residue Observed*—Record that the test oil produced solid or liquid material in the compatibility test, identify the reference oils involved, and report as detailed in 9.2.2. If it is desired to quantify the amount of residue, proceed as directed in Appendix X1 and Appendix X2.

NOTE 2—Residues produced in the compatibility test might arise because of incompatibility between the test oil and reference oil, or from a storage stability problem associated with test oil itself, or both (see 8.2). Without further quantitative testing, as described in Appendix X1 and Appendix X2, it is not possible to identify the source of the residue in the compatibility test.

9. Report

9.1 *Storage Stability:*

9.1.1 If the storage stability test resulted in no visual liquid or solid residue, report zero separated residues were observed.

9.1.2 If the storage stability test resulted in visual liquid or solid residue, report separated residues were observed.

9.2 *Compatibility:*

9.2.1 If none of the test oil/reference oil mixes resulted in visual solid or liquid residues in the compatibility test, report zero separated residues were observed.

9.2.2 If one or more of the test oil/reference oil mixes resulted in visual solid or liquid residues in the compatibility test, report that the test oil resulted in separated residues in the compatibility test and identify the reference oil(s) involved.

10. Precision and Bias

10.1 No information is presented about either the precision or bias of Test Method D7603 for measuring the storage stability and compatibility of gear oils since the test result is nonquantitative.

11. Keywords

11.1 compatibility; gear oil; storage stability

APPENDIXES

(Nonmandatory Information)

X1. QUANTIFYING THE AMOUNT OF SOLID OR LIQUID RESIDUES

X1.1 The procedures described in this appendix allow the determination of the mass of solid residues or the volume of liquid residues in the event such residues are observed at the end of the storage stability and compatibility test storage period of 30 days.

X1.2 Observe the nature of any separated material found in 8.1.6.2 and 8.2.9.5.

X1.2.1 If the separated material observed in X1.2 is not sufficiently compacted by the centrifuging to permit decanting the supernatant oil, continue the centrifuging for intervals of 15 min. If after centrifuging for 15 min, the separated material is a solid, proceed as directed in X1.2.2. If after centrifuging for 30 min, the separated material is a liquid, or if it has not been compacted, proceed as directed in X1.2.1.1 and X1.2.1.2.

X1.2.1.1 Remove the tubes from the centrifuge. Read and record the volume of the separated liquid at the bottom of each tube as indicated in Table X1.1 and Fig. X1.1.

X1.2.1.2 Calculate and record the average volume of the separated liquid.

X1.2.2 If the separated material is solid, remove the stoppers and decant (and discard) the supernatant oil. Drain the centrifuge tubes in an inverted position at 25 °C ± 3 °C for a period of 2 h. Discard the drained fluid. Wash the solid residue with solvent three times to assure that it is free of oil. Centrifuge the solid residue between washes, as detailed in 8.1.5, in order to compact the residue before draining the wash solvent from the tube.

X1.2.2.1 To evaporate the solvent, place the centrifuge tubes in an oven controlled at 105 °C ± 3 °C for 120 min ± 5 min.

X1.2.2.2 Remove the centrifuge tubes from the oven and place them in a desiccator to cool to 25 °C ± 3 °C.

X1.2.2.3 Measure the mass of each tube and its contents to the nearest 1 mg. Determine and record the mass of the separated material in each tube by subtracting the mass of the empty centrifuge tube.

X1.2.2.4 Calculate and record, to the nearest 1 mg, the average mass of the separated material.

TABLE X1.1 Procedure for Reading Volume for the Cone-Shaped Centrifuge Tube

Volume of Separated Liquid, mL	Read to Nearest, mL
0.0 to 0.2	0.025
0.2 to 1.0	0.05
> 1.0	0.10

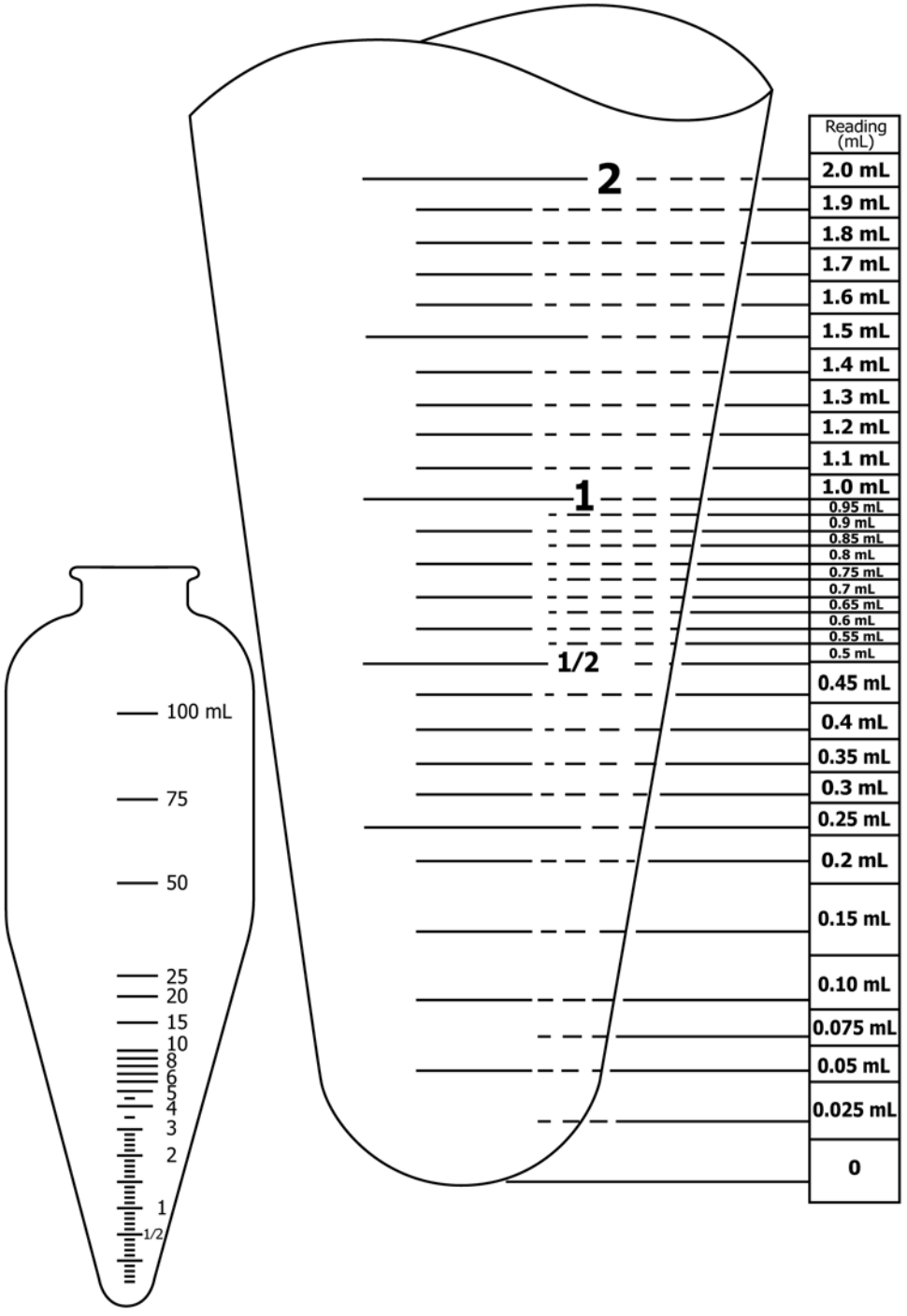

FIG. X1.1 Procedure for Reading Volume of Separated Liquid for the Centrifuge Tube

X2. CALCULATING THE PERCENT OF ADDITIVE MATERIAL THAT SEPARATES OUT

X2.1 This appendix allows the mass or volume of separated residue recorded in Appendix X1 to be converted to the percent of the additive material originally contained in the test oil.

X2.2 Storage Stability

X2.2.1 If the residue is a solid, calculate the mass percent additive precipitated in the test oil, using Eq X2.1:

$$\text{Mass percent additive precipitated} = 100\,M/(0.9\,m_t) \quad \text{(X2.1)}$$

where:

M = average mass (in grams) of separated residue from the two centrifuge tubes (see X1.2.2.4),
m_t = mass percent additive in the test oil,[7] and
0.9 = assumed specific gravity of the test oil.

X2.2.2 If the residue is a liquid (see X1.2.1.1 and X1.2.1.2), calculate the volume percent liquid additive deposited using Eq X2.2:

$$\text{Volume percent liquid additive deposited} = 100\,V/v_t \quad \text{(X2.2)}$$

where:

V = average volume (in millilitres) of separated liquid residue from the two centrifuge tubes (see X1.2.1.2),
v_t = volume percent additive in the test oil.[7]

NOTE X2.1—Obtain this information from the supplier of the gear oil.

X2.3 Compatibility

X2.3.1 If the residues from the test oil/reference oil mixes and the test oil alone are solids, calculate the mass percent incompatibility residue for each test oil/reference oil combination using Eq X2.3:

$$\text{Mass percent incompatibility residue} = \frac{100[M_X - (M_R + M_T)]}{0.9(50\,m_r + 50\,m_t)} \quad \text{(X2.3)}$$

where:

M_X = average mass (in grams) of separated material found in the test oil/reference oil compatibility test (see X1.2.2.4),
M_R = average mass of separated material in the reference oil alone, grams/50 mL of oil,
= 0 for the *SSCT Fluid (6-pack)* reference oils,
M_T = average mass of separated material in the test oil alone, grams/50 mL of oil,
= M/2 if the test oil has been independently subjected to the storage stability test,
m_r = mass percent of additive in the reference oil. This information is supplied by the TMC,[5]
m_t = mass percent additive in the test oil,[7]
0.9 = assumed specific gravity of both reference oil and test oil.

NOTE X2.2—Theoretical zero incompatibility corresponds to $M_X = M_R + M_T$, that is, the separated material arises only from storage instability of the test oil, or the reference oil alone, or both. Evidence of incompatibility is demonstrated when $M_X - (M_R + M_T) > 0$.

X2.3.2 If the residues from the test oil/reference oil mixes and the test oil alone are liquids, calculate the volume percent incompatibility residue for each test oil/reference oil combination using Eq X2.4:

$$\text{Volume percent incompatibility residue} = \frac{100[V_X - (V_R + V_T)]}{(50\,v_r + 50\,v_t)} \quad \text{(X2.4)}$$

where:

V_X = average volume (in millilitres) of separated material found in the test oil/reference oil compatibility test (see X1.2.1.2),
V_R = average volume of separated material in the reference oil alone, millilitres/50 mL of oil,
= 0 for the *SSCT Fluid (6-pack)* reference oils,
V_T = average volume of separated material in the test oil alone, millitres/50 mL of oil,
= V/2 if the test oil has been independently subjected to the storage stability test,
v_r = volume percent of additive in the reference oil. This information is supplied by the TMC,[5] and
v_t = volume percent additive in test oil.[7]

[7] Obtain this information from the supplier of the gear oil.

SUMMARY OF CHANGES

Subcommittee D02.B0.01 has identified the location of selected changes to this standard since the last issue (D7603 –10) that may impact the use of this standard.

(1) Only editorial changes were made, applying Form and Style (including SI 10) guidelines.