Designation: D6922 – 13 (Reapproved 2018)

Standard Test Method for Determination of Homogeneity and Miscibility in Automotive Engine Oils[1]

This standard is issued under the fixed designation D6922; 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 if an automotive engine oil is homogeneous and will remain so, and if it is miscible with certain standard reference oils after being submitted to a prescribed cycle of temperature changes. This test method is very similar to the homogeneity and miscibility test described in FED–STD–791/3470.1.

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 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D97 Test Method for Pour Point of Petroleum Products

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID) (Withdrawn 2003)[3]

D6557 Test Method for Evaluation of Rust Preventive Characteristics of Automotive Engine Oils

E1 Specification for ASTM Liquid-in-Glass Thermometers

E344 Terminology Relating to Thermometry and Hydrometry

2.2 *Federal Test Method Standard:*[4]

FED–STD–791/3470.1 Homogeneity and Miscibility of Oils

3. Terminology

3.1 *Definitions:*

3.1.1 *calibrate, v*—to determine the indication or output of a measuring device with the respect of that of a standard. **E344**

3.1.2 *homogeneity, n*—the ability of a test oil itself to remain the same in appearance throughout (uniform) after submission to a series of temperature changes.

3.1.3 *miscibility, n*—the ability of a reference oil and test oil to form a uniform mixture after blending and not separate into two phases after submission to a series of temperature changes.

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

[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 on Automotive 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

3.1.4.1 *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. D5844

3.1.5 *specimen, n*—a piece or portion of test oil used to make a test.

3.1.6 *test oil, n*—any oil subjected to evaluation in an established procedure. D6557

4. Summary of Test Method

4.1 Visual color determinations and observations are made on an undiluted test oil specimen, along with six blends of the same test oil that have been combined with specific reference oils. The pour point is then determined for the undiluted test oil specimen and the six blends. The undiluted test oil specimen and six blends are then allowed to warm to room temperature. Color determinations and observations are again made on the undiluted test oil specimen and six blends. The undiluted test oil specimen and six blends are heated to 232 °C, then allowed to cool to room temperature, and then stored at their pour point temperatures for 18 h to 24 h. The undiluted test oil specimen and six blends are then allowed to thaw and a series of color determinations and observations are made as they reach room temperature. All data are recorded on a report form.

5. Significance and Use

5.1 It is important that engine oils from different manufacturers be homogeneous and miscible with each other, because operators of automotive engines often do not have prior knowledge of the manufacturer of the oil that is currently used in their application, and engine failure can occur if oils are combined that do not stay homogeneous and function properly.

6. Apparatus

6.1 *Test Jar*—cylindrical, of clear glass, flat bottom, 33.2 mm to 34.8 mm outside diameter, and 115 mm to 125 mm in height. The inside diameter of the jar can range from 30.0 mm to 32.4 mm, within the constraint that the wall thickness be no greater than 1.6 mm. The jar shall have a line to indicate a sample height 5 mm ± 3 mm above the inside bottom.

6.2 *Thermometers*—ASTM 6C, calibrated with a range of −80 °C to +20 °C, conforming to Specification E1. In addition, temperature measuring devices such as liquid-in-glass thermometers, thermocouples, or platinum resistance thermometers may be used, if they provide equivalent or better accuracy and precision, and cover the required temperature range.

6.3 *Stoppers*—clean stoppers, some unbored and others centrally bored for test thermometers.

6.4 *Jacket*—a glass or metal, water tight, cylindrical form with a flat bottom and approximately 120 mm in depth. The inside diameter of the jacket shall be 9.0 mm to 12.0 mm greater than the outside diameter of the test jar.

6.5 *Disk*—cork or felt, about 6 mm in thickness and of the same diameter as the inside of the jacket.

6.6 *Gasket*—a ring gasket, about 5.0 mm in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

6.7 *Bath*—a liquid cooling bath suitable to obtain the required temperatures.

6.8 *Oven*—a constant temperature oven suitable to obtain the required temperatures.

6.9 *Automated Pour Point Tester (Alternative)*—There are automated pour point testers available and in use that may be advantageous. They save test time, permit the use of smaller samples, and have other factors which may merit their use. If automated testers are used, the user shall ensure that all of the manufacturer's instructions for calibration, adjustment, and operation of the instrument are followed. Report that the pour point was determined by an automatic instrument. In some cases, precision of automatic pour point testers may not have been determined. In any case of dispute, consider the pour point as determined by the manual method described herein the referee test.

7. Reagents

7.1 *Reference Oils, HMA through HMF*—These are available from the Test Monitoring Center.[5]

8. Sampling

8.1 Take samples to be tested in accordance with the instructions in Practice D4057.

9. Test Preparation

9.1 Place the seven clean test jars in a holder.

9.2 Measure and mark each test jar as follows:

9.2.1 Measuring inside the test jar, mark each jar at 23 mm and 54 mm from the bottom.

9.2.2 Mark the first test jar for use with the undiluted test oil specimen.

9.2.3 Mark six test jars with the letters A through F to designate which reference oil will be mixed with the test oil.

9.3 Vigorously shake the test oil.

9.4 Using test oil, fill the six jars (that were marked for use with reference oil) to the 23 mm mark.

9.5 Fill the test jar that was marked for the undiluted test oil, to the 54 mm mark.

9.6 Vigorously shake the six reference oils.

9.7 Fill the test jars marked A through F with the corresponding reference oil to the 54 mm mark.

9.8 Firmly place the unbored stoppers in each of the test jars and shake the test jars vigorously.

9.9 Remove the stoppers from each test jar.

[5] ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206-4489. This test method is supplemented by Information Letters and Memoranda issued by the ASTM Test Monitoring Center; users of this test method can contact the ASTM Test Monitoring Center to obtain the most recent of these.

10. Procedure

10.1 Heat the test specimens in the test jars to 46 °C ± 2 °C in a constant temperature oven or liquid bath.

10.2 Remove the test jars from the oven and allow to cool to room temperature.

10.3 For each test specimen record evidence of separation, its location, color, and particle size, and the color of the test specimen on the record form under *Before Treatment* (see Fig. A1.1).

10.4 *Determination of Pour Point:*

NOTE 1—This pour point procedure is based on the method described in Test Method D97 but with the exception indicated in Note 2.

10.4.1 Place a ring gasket around each test jar just below the oil level.

10.4.2 Place a calibrated thermometer with bored stopper in each of the test jars. Ensure that the thermometer bulb is immersed such that the beginning of the capillary is 3 mm below the surface of the oil.

10.4.3 Place a calibrated thermometer in the cooling bath that is maintained at a temperature of −18 °C to −15°C.

10.4.4 Place the test jars into the jacket in the cooling bath.

10.4.5 Pour points are expressed in integers that are positive or negative multiples of 3 °C. Begin to examine the appearance of each test specimen when its temperature is 12 °C above the expected pour point (estimated as a multiple of 3 °C). Remove the test jar from the jacket carefully and tilt it just enough to ascertain whether there is movement of the test specimen in the jar.

10.4.6 If there is movement of the test specimen in the test jar, return it immediately to the jacket. (The complete operation of removal and replacement shall require no more than 3 s.)

10.4.7 If the test specimen in the test jar does not flow when the jar is tilted:

10.4.7.1 Hold the test jar in a horizontal position for 5 s as noted by a timing device.

10.4.7.2 If the test specimen shows any movement under these conditions, return the jar to the jacket.

10.4.7.3 If the test specimen shows no movement, record the test temperature for this test jar on the record form, in degrees Celsius, under *Cooled to Pour Point* (see Fig. A1.1). Place the test jar in a holder to thaw.

NOTE 2—Test Method D97 requires that 3 °C be added to the temperature and reported. This is not the case here. The actual observed temperature is reported.

10.4.8 If the test specimen has not ceased to flow when it reaches −6 °C, place the test jar and jacket in a second bath maintained at a temperature of −35 °C to −32 °C.

10.4.9 Continue to perform steps 10.4.5 through 10.4.7 at 3 °C intervals for each of the test jars.

10.4.10 If the test specimen has not ceased to flow when the temperature of −24 °C has been reached, place the test jars in a third bath maintained at a temperature of −52 °C to −49 °C.

10.4.11 Continue to perform steps 10.4.5 through 10.4.7 at 3 °C intervals for each of the test jars.

10.4.11.1 For determinations of very low or very high pour points, additional cold baths should be maintained with temperature differentials of about 17 °C. In each case transfer the test jar when the temperature of the test specimen reaches a point of 28 °C above the temperature of the new bath.

10.4.12 Allow the test specimens to thaw.

10.4.13 When each of the test specimens become transparent and the cloudiness has disappeared observe for the evidence of separation, its location, color, and particle size, and the color of the test specimen. Record the information requested on the record form under *Warmed Just Above Cloud Point* (see Fig. A1.1).

10.5 *Heating of the Test Specimens:*

10.5.1 Wait until the test specimens have reached room temperature.

10.5.2 Remove the thermometers and stoppers from the test jars.

10.5.3 Remove the ring gaskets from the test jars.

10.5.4 Place all test jars in an appropriate oven or liquid bath where the temperature has been set to a point that will allow the test specimen temperatures to reach 232 °C ± 2 °C within 90 min.

NOTE 3—Heating rates of 25 min to 90 min have been found to be acceptable.

10.5.5 When the test specimen temperatures reach 232 °C ± 2 °C, immediately discontinue the heating process and allow the test jars to cool to room temperature.

10.6 *Storage of the Test Specimens at Pour Point:*

10.6.1 Store the undiluted test oil specimen and the six blends at the pour point of the undiluted test oil specimen for 18 h to 24 h in the appropriate cold box.

10.6.2 At the end of the storage period, remove the test specimens from the cold box and allow them to warm.

10.6.3 When the test specimens become transparent and cloudiness has disappeared, observe and record the information requested on the record form under *Heat to 232 °C – Stored at Sample Pour Point Temperature – Store 18 h – Warm Above Pour Point* (see Fig. A1.1).

10.6.4 Allow the test specimens to warm to room temperature.

10.6.5 Observe the test specimens and record the information requested on the record form under *Warm to Room Temperature* (see Fig. A1.1).

10.6.6 Discard the test specimens as required.

11. Report

11.1 Report the data on the homogeneity and miscibility test form located in Fig. A1.1.

12. Precision and Bias

12.1 No information is presented about either the precision or bias of Test Method D6922 for measuring homogeneity and miscibility since the test results are nonquantitative.

13. Keywords

13.1 automotive engine oil; homogeneity; miscibility

ANNEX

(Mandatory Information)

A1. REPORT FORM

Sample No.: _____ Date: _____

Reference Oil Added To Test Oil	None	HMA	HMB	HMC	HMD	HME	HMF
Thermometer Serial Number							
Before Treatment							
Evidence of Separation							
Location							
Color							
Particle Size							
Visual Appearance of the Test Specimen							
Cooled to Pour Point, °C							
Warmed Just Above Cloud Point							
Evidence of Separation							
Location							
Color							
Particle Size							
Visual Appearance of the Test Specimen							
Heat to 232 °C – Stored at Sample Pour Point Temperature – Store 18 h – Warm Above Pour Point							
Evidence of Separation							
Location							
Color							
Particle Size							
Visual Appearance of the Test Specimen							
Warm to Room Temperature							
Evidence of Separation							
Location							
Color							
Particle Size							
Visual Appearance of the Test Specimen							

Evidence of Separation
D – Definite
N – None

Color
w – White or very light
y – Yellow
b – Black

Visual Appearance of the Oil
d – Too dark to see through
m – Transparent but dark
l – Light

Location
T – Near top
B – Near bottom
F – Filament
U – Uniformly distributed

Particle Size
s – Small, as in cloud or haze
s_p – Specks or larger particles

Operator: _____

FIG. A1.1 Engine Oil Homogeneity and Miscibility Test