Designation: D2532 – 22

Standard Test Method for Viscosity and Viscosity Change After Standing at Low Temperature of Aircraft Turbine Lubricants[1]

This standard is issued under the fixed designation D2532; 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 U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of the kinematic viscosity of aircraft turbine lubricants at low temperature, and the percent change of viscosity after a 3 h and a 72 h standing period at low temperature.

1.1.1 The range of kinematic viscosities covered by this test method is from 7700 mm^2/s to 14 000 mm^2/s at –40 °C and from 7000 mm^2/s to 17 500 mm^2/s at –51 °C. The precision has only been determined for those materials, kinematic viscosity ranges, and temperatures as shown in the precision section. Kinematic viscosities and percent change of viscosity may be measured and reported at other temperatures and other thermal soak period intervals as agreed by the contracting parties.

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

1.2.1 The SI unit used in this test method for Kinematic Viscosity is mm^2/s. For user reference, 1 $mm^2/s = 10^{-6}$ m^2/s = 1 cSt.

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.* For specific hazard statements, see Section 7.

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]

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

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants

E1 Specification for ASTM Liquid-in-Glass Thermometers

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E644 Test Methods for Testing Industrial Resistance Thermometers

E1137 Specification for Industrial Platinum Resistance Thermometers

E1750 Guide for Use of Water Triple Point Cells

[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 D02.07 on Flow Properties.

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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.

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

E2593 Guide for Accuracy Verification of Industrial Platinum Resistance Thermometers
E2877 Guide for Digital Contact Thermometers

2.2 *Other Documents:*
MIL-PRF-7808 Lubricating Oil, Aircraft Turbine Engine, Synthetic Base[3]
MIL-PRF-23699 Lubricating Oil, Aircraft Turbine Engine, Synthetic Base, NATO Code Number O-156[3]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D4175.

3.1.2 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.1.2.1 *Discussion*—This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device. These devices are sometimes referred to as "digital thermometers."

3.1.2.2 *Discussion*—PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

4. Summary of Test Method

4.1 Kinematic viscosity and percent change is determined at low temperature using apparatus defined in Test Method D445 and Specifications D446 at time intervals of 3 h and 72 h following an initial measurement at 35 min.

5. Significance and Use

5.1 Aircraft turbine lubricants, upon standing at low temperatures for prolonged periods of time, may show an increase in kinematic viscosity. This increase may cause lubrication problems in aircraft engines. Thus, this test method is used to ensure that the kinematic viscosity does not exceed the maximum kinematic viscosity in certain specifications for aircraft turbine lubricants.

6. Apparatus

6.1 Viscometers, drying tubes, low-temperature bath, thermometer, timer, secondary viscosity standard, filter, and cleaning supplies are described in detail in Test Method D445.

6.2 *Viscometer*—The viscometer shall meet the requirements of Test Method D445 and Specifications D446 and be of the type in which the sample can be rerun without cleaning the viscometer. Suitable holders should be used. For convenience it is recommended that the viscometer size be chosen to keep the efflux time between 200 s and 1000 s.

NOTE 1—Consult Specifications D446 regarding calibration constant correction for the actual test temperature when using Specifications D446 viscometers that are not suspended level types.

6.3 *Drying Tubes*—Fit the viscometer openings with drying tubes filled with indicating silica gel, using cotton at top and bottom to hold the loosely packed desiccant in place. Provide a cross-connection on the viscometer side of the drying tubes (which can be closed by a pinch clamp or stopcock while liquid is being drawn into the efflux bulb) so that the restriction to air flow will not cause error. Replace the silica gel when a lavender color is noticeable.

6.4 *Viscosity Temperature Bath*—The constant-temperature bath must be capable of holding several viscometers at once. It must have adequate stirring of the liquid medium (Note 2) and balance between heat losses such that the bath temperature can be maintained at the required temperature ±0.03 °C.

NOTE 2—Isopropanol or other clear, low-freezing point liquid may be used as a bath liquid.

6.5 *Temperature Measuring Device*—Use either a digital contact thermometer as described in 6.5.2 with equal or better accuracy or a calibrated ASTM Kinematic Viscosity Test Thermometer such as 73C (–40 °C) or 74C or 43C (–51 °C) conforming to the requirements as prescribed in Specification E1 which have an accuracy after correction of ±0.03 °C or better. The ASTM 74C thermometer has a specification between –55.4 °C and –52.6 °C but is available from some suppliers with the scale expanded to cover –51 °C. The 43C thermometer is only graduated in 0.1 °C increments so interpretation at midpoints between the lines may be required.

6.5.1 When using liquid-in-glass thermometers, use a magnifying device to read the thermometer to the nearest ⅕ division (for example, 0.02 °C on thermometers graduated in 0.1 °C increments) to ensure that the required test temperature and temperature control capabilities are met.

6.5.1.1 Check the thermometer's ice point at least annually using an ice bath or a constant temperature bath against a reference thermometer. If the corrected temperature reading error is greater than the temperature tolerance, then the thermometer must be recalibrated.

6.5.2 When using a digital contact thermometer (DCT), the following requirements shall apply:

Criteria	Minimum Requirements
DCT	E2877 Class A
Display resolution	0.01 °C, recommended 0.001 °C
Display accuracy	±30 mK (±0.03 °C) for combined probe and sensor
Sensor type	RTD, such as a PRT or thermistor
Drift	less than 10 mK (0.01 °C) per year
Response time	less than or equal to 6 s as defined in Specification E1137
Linearity	10 mK over range of intended use
Calibration Report	The DCT shall have a report of temperature calibration traceable to a national calibration or metrology standards body issued by a competent calibration laboratory with demonstrated competency in temperature calibration. An ISO 17025 accredited laboratory with temperature calibration in its accreditation scope would meet this requirement.
Calibration Data	The calibration report shall include at least 3 calibration temperatures at least 5 °C apart which are appropriate for its intended use.

[3] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://dodssp.daps.dla.mil.

6.5.2.1 For a constant temperature bath employed, the DCT probe shall be immersed by more than its minimum immersion depth in a constant temperature bath so that the center of the probe's sensing region is at the same level as the lower half of the working capillary provided the probe's minimum immersion depth is met and is no less than indicated on the calibration certificate. The end of the probe sheath shall not extend past the bottom of the viscometer. It is preferable for the center of the sensing element to be located at the same level as the lower half of the working capillary as long as the minimum immersion requirements are met.

NOTE 3—With respect to DCT probe immersion depth, a procedure is available in Test Methods E644, Section 7 (Minimum Immersion Length Test), for determining the minimum depth. With respect to an ice bath, Practice E563 provides guidance on the preparation of an ice bath, however variance from the specific steps is permitted provided preparation is consistent as it is being used to track change in calibration.

6.5.2.2 Verify the DCT calibration at least annually. The probe shall be recalibrated when the check value differs by more than 0.01 °C from the last probe calibration. Verification can be accomplished with the use of a water triple point cell, an ice bath, or other suitable constant temperature device which has a known temperature value of suitable precision. See Practice E563, Guide E1750, and Guide E2593 for more information regarding checking calibrations.

6.5.2.3 In the case of constant temperature baths used in instruments for automatic viscosity determinations, the user is to contact the instrument manufacturer for the correct DCT that has performance equivalence to that described here.

6.6 *Timing Device*—Use any timing device, spring-wound or digital, that is capable of taking readings with a discrimination of 0.1 s or better and has an accuracy within ±0.07 % (see Annex A3 of Test Method D445) of the reading when tested over the minimum and maximum intervals of expected flow times.

6.6.1 Timing devices powered by alternating electric current may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in kinematic viscosity flow time measurements.

6.7 *Secondary Viscosity Standards.*

7. Procedure for Cleaning Viscometers and Filter Screen

7.1 Apply air pressure or suction to the viscometer to remove any previous test specimen. Allow the viscometer to drain for 5 min.

7.1.1 Wash the viscometer four times, inside and out, with fresh toluene (**Warning—**Flammable) using suction as required. Allow the viscometer to drain.

7.1.2 Wash the viscometer four times, inside and out, with acetone, and allow to drain for 5 min. Then dry with vacuum suction.

7.2 If organic or other deposits are visible in the viscometer, clean the viscometer thoroughly by filling it completely with glass cleaning solution for several hours to remove residual traces of organic deposits. Allow to drain for 5 min. It is essential that strong alkaline cleaning solutions are not used as changes in the viscometer calibration can occur.

7.2.1 Rinse viscometer inside and out with distilled water until all traces of the cleaning solution are completely removed. Allow to drain for 5 min.

7.2.2 Dry with filtered dry air, a vacuum line, or in an oven at approximately 100 °C for approximately 30 min until all traces of water are removed.

7.3 Clean the filter screen by first disassembling the screen (if practicable).

7.3.1 Rinse thoroughly with fresh toluene (**Warning—** Flammable).

7.3.2 Rinse thoroughly with fresh acetone (**Warning—** Flammable).

7.3.3 Dry in oven at approximately 100 °C.

8. Procedure

8.1 For the duration of the test, maintain the bath temperature at the required temperature ±0.03 °C.

8.2 Charge the clean, dry viscometer as prescribed in Test Method D445 and Specifications D446.

8.2.1 Affix the drying tubes and carefully flush the moist room air from the viscometer by placing vacuum to the drying tubes. Draw the sample into the working capillary and timing bulb and then place rubber stoppers into the tubes to hold the sample in place so as to preclude the possibility of any traces of residue moisture condensing on the walls of the capillary and timing bulb while the sample cools to test temperature. Moisture must not be allowed to condense on the walls of the working capillary and efflux bulb.

8.2.2 Insert the viscometer into the constant-temperature bath, and vertically align the viscometer if a self-aligning holder has not been used. After insertion, allow 20 min to 30 min for the viscometer to reach bath temperature, and remove the stoppers. Where one bath is used to accommodate several viscometers, never add or withdraw, or clean a viscometer while any other viscometer is in use for measuring an efflux (flow) time.

8.2.3 Start the first determination of kinematic viscosity 35 min ± 1 min after the viscometer is placed in the bath. Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. Measure a single efflux time, in seconds to within 0.1 s or better, the time required for the advancing ring of contact (meniscus) to pass from the first timing mark to the second. If this flow time is less than the specified minimum (see Specifications D446), select a viscometer with a capillary of smaller diameter and repeat the operation. Calculate the kinematic viscosity as per subsection 9.1 and record the measurement. Be careful that the stopcock or pinch clamp joining each arm of the viscometer has been opened to ensure that there is no effect of the drying tubes on the efflux time.

8.3 At 3 h ± 5 min after the completion of the first kinematic viscosity determination (subsection 8.2.3), obtain