Designation: D 3117 – 03

An American National Standard

Standard Test Method for Wax Appearance Point of Distillate Fuels[1]

This standard is issued under the fixed designation D 3117; 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 detection of wax in burner fuels, diesel fuels, and turbine engine fuels in the range from −26 to +2°C. It is applicable to a dark-colored oil if the stirrer can be seen under illumination.

1.2 *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.* For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids[2]

D 2386 Test Method for Freezing Point of Aviation Fuels[2]

D 2500 Test Method for Cloud Point of Petroleum Products[2]

E 1 Specification for ASTM Thermometers[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *wax appearance point*—the temperature at which wax first begins to separate from the liquid when it is cooled under prescribed conditions.

4. Summary of Test Method

4.1 A specimen of distillate fuel is cooled under prescribed conditions while being stirred. The temperature at which wax first appears is recorded as the wax appearance point.

5. Significance and Use

5.1 Wax appearance point is the temperature at which wax crystals begin to precipitate out of a fuel under specified cooling conditions. The presence of wax crystals in the fuel may restrict flow or plug the fuel filter. In critical fuel systems, wax appearance point may define the lower limit of acceptable operability.

6. Apparatus

6.1 *Specimen Tube*—A double-walled (Dewar-type) jacketed tube with dimensions shown in Fig. 1.

6.2 *Temperature Measuring Device*—Liquid-in-glass thermometer conforming to specifications for ASTM Thermometer 62C in accordance with Specification E 1, or any other temperature measuring device with equal or better accuracy and equal temperature response.

6.3 *Stirrer Assembly*—A stainless steel wire configured in the manner described in Fig. 2 and manipulated by a motor or other suitable device in a vertical direction. The frequency of movement shall be 55 ± 5 cycles/min with an amplitude of 50 ± 5 mm. The stirrer shall be concentric with the temperature measuring device and shall be fitted with the moisture proof collar specified in Test Method D 2386. A No. 3, two-hole neoprene rubber stopper shall be used to seal the top of the specimen tube.

6.4 *Cooling Bath*—Use an unsilvered vacuum flask having minimum dimensions of 200-mm depth and 65-mm internal diameter. The bath temperature, below −45°C, may be maintained by refrigeration or suitable freezing mixtures (7.1). Bath temperature is monitored with an appropriate temperature measuring device such as ASTM Thermometer 6C.

NOTE 1—Solid carbon dioxide chips (dry ice) and isopropanol is a recommended mixture for coolant. An excess of dry ice should be avoided to prevent obscuring the sample tube in a continuous stream of bubbles. Isopropanol should be replaced daily or when low temperature viscosity is noticeably higher than a fresh bath. Liquid nitrogen may also be used as coolant instead of liquids cooled with solid carbon dioxide.

6.5 *Illumination*—A 150 to 230-mm long, 5 to 8-W fluorescent tube shall be mounted behind the specimen to illuminate it with transmitted light. Observations shall be made with the sample tube between the observer's eye and the lamp.

6.6 *Clock*—Use a clock or other timing device readable to 10 s to monitor the cooling rate.

[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 on Flow Properties.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 14.03.

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

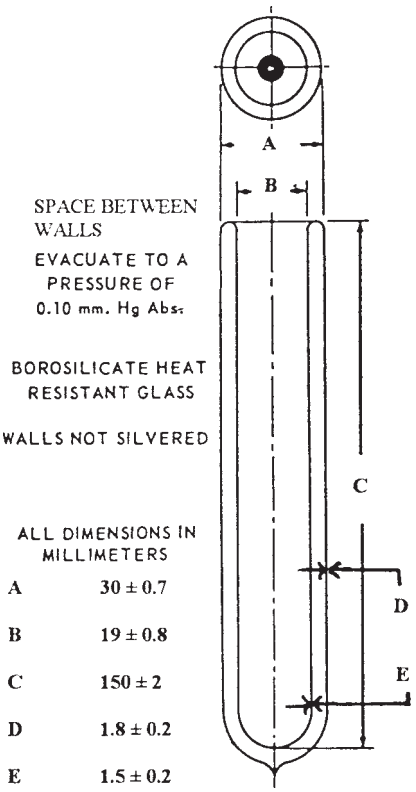


FIG. 1 Specimen Tube

7. Materials

7.1 *Carbon Dioxide (Solid) or Dry Ice*—(**Warning—** Extremely cold, −78°C.) A commercial grade of dry ice is suitable for use in the cooling bath (Note 2).

7.2 *Isopropanol or Isopropyl Alcohol*—(**Warning—** Extremely flammable.) A commercial or technical grade of isopropanol is suitable for the cooling bath.

7.3 *Liquid Nitrogen*—(**Warning—**Extremely cold, −196°C.) A commercial or technical grade of liquid nitrogen is suitable for the cooling bath. See Note 2. (**Warning—**Carbon dioxide (solid) and liquid nitrogen liberate gases that can cause suffocation. Contact with skin causes burns, freezing, or both.)

8. Procedure

8.1 Ensure that the temperature measuring device is in calibration. For liquid-in-glass temperature measuring devices, see the Annex on Calibration in Test Method D 445.

NOTE 2—Because ambient temperatures are well above the range of ASTM Thermometer 62C, the liquid-in-glass thread will extend upward into the expansion chamber. During cooling, the liquid-in-glass temperature measuring device must be examined to make certain that no separation is observed in the liquid.

8.2 Sample temperature shall not be lower than 10°C when starting the measurement. Dry the specimen by filtration through a lintless filter paper.

8.3 Assemble the unit as shown in Fig. 3.

8.4 With the oil at 10°C or above, introduce a 25 ± 1-mL specimen into the tube. Add three drops of anhydrous isopropanol.

8.5 Adjust the stopper, temperature measuring device, and stirrer so that the bottom of the temperature measuring device is 25 mm above the bottom of the specimen tube and the stirrer does not break the surface at the upper end of its stroke.

8.6 When the stopper has been firmly seated, start the stirrer, raise the cooling bath around the specimen tube, and cool rapidly to 2°C.

8.7 Once the temperature reading is below 2°C, adjust the bath height to keep the cooling rate between 1 and 2°C/min. The preferred cooling rate is 1.5°C/min.

8.8 Continue cooling until wax distinctly appears when the specimen tube is examined by transmitted light. This is manifested by the appearance of very small wax crystals which make the stirring pattern quite obvious. An example of the change observed is shown in Fig. 4 and Fig. 5. Observe the temperature reading immediately to the nearest 0.2°C.

NOTE 3—The wax appearance point should not be misinterpreted to be the appearance of the very first crystals. The temperature at which a distinct swirl of wax particles around the stirrer occurs is the correct interpretation.

9. Report

9.1 The temperature at which the wax crystals are obviously present is the wax appearance point and is reported to the nearest 0.2°C.

10. Precision and Bias

10.1 *Precision*—The precision of this test method obtained by the statistical examination of interlaboratory test results is as follows:

10.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on the same fuel sample would, in the long run, in the normal and correct operation of the test method, exceed 0.8°C only in one case in twenty.

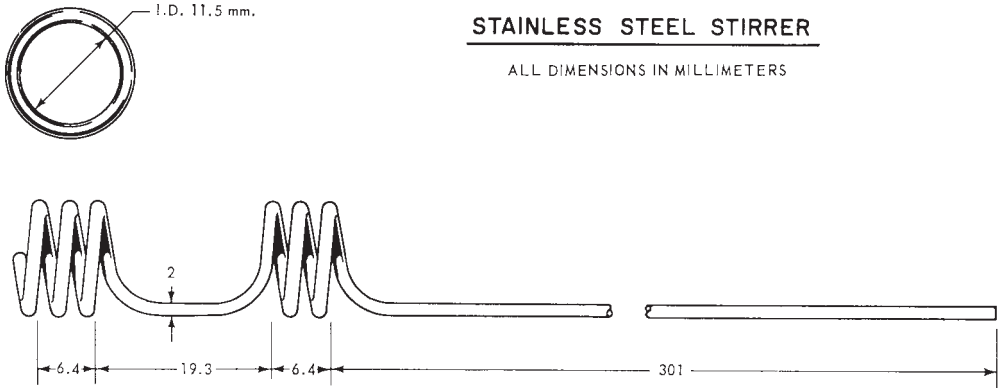
10.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on the same fuel sample would, in the long run, in the normal and correct operation of the test method, exceed 2.2°C only in one case in twenty.

10.2 *Bias*—Because liquid hydrocarbon standards of known wax appearance point are not available, no statement of bias can be made.

NOTE 4—These precision data were obtained in a cooperative program among eight laboratories testing eight fuels for both wax appearance point and cloud point by Test Method D 2500. None of the fuels contained wax crystal modifying additives.

11. Keywords

11.1 distillate fuels; wax



NOTE—Tolerance on all dimensions are ±2 mm, except on wire thickness.

FIG. 2 Stirrer

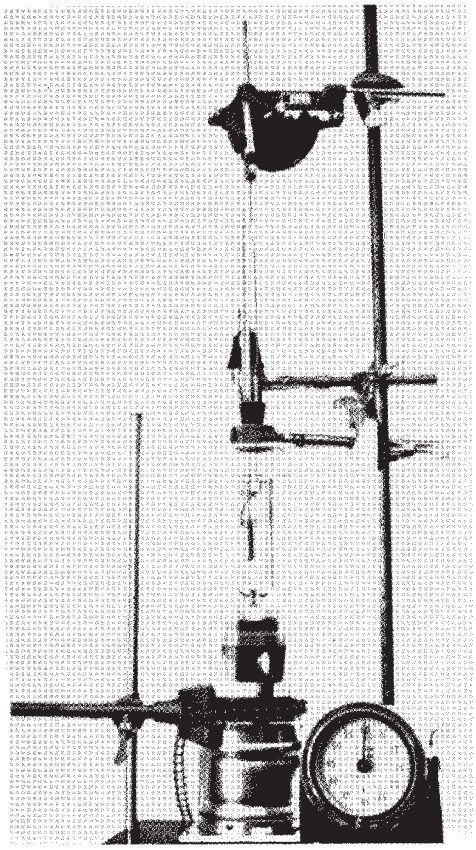


FIG. 3 Complete Assembly

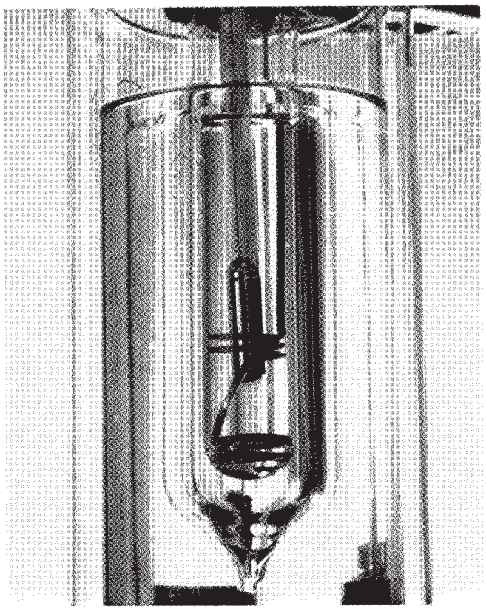


FIG. 4 Tube Before Wax Appearance

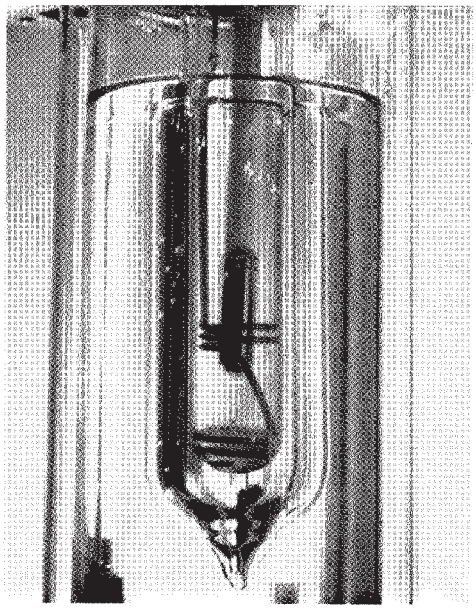


FIG. 5 Tube After Wax Appearance

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 3117–96$^{\epsilon 1}$) that may impact the use of this standard.

(1) Added Test Method D 445 to Section 2, Referenced Documents.

(2) Added "temperature measuring device" throughout.