Designation: ~~D4539 – 17~~ D4539 – 22

Standard Test Method for Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT)[1]

This standard is issued under the fixed designation D4539; 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 estimating the filterability of diesel fuels in some automotive equipment at low temperatures.

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 ~~EPA and~~ many ~~state~~regulatory agencies as a hazardous ~~material~~substance that can cause ~~central nervous system, kidney, and liver damage.~~ serious medical issues. Mercury, or its vapor, ~~may~~ has been demonstrated to be hazardous to health and corrosive to materials. ~~Caution should be taken~~ Use caution when handling mercury and mercury-containing products. See the applicable product ~~Material~~ Safety Data Sheet ~~(MSDS) for details and EPA's website (http://www.epa.gov/mercury/faq.htm) for additional information. Users should be aware~~ (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, ~~in your state may be prohibited by state law.~~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 warning statements, see 1.3, 9.1, 9.2.1, 9.3, 9.5, and Annex A1.

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
D975 Specification for Diesel Fuel
D1655 Specification for Aviation Turbine Fuels
D2500 Test Method for Cloud Point of Petroleum Products and Liquid Fuels
D3117 Test Method for Wax Appearance Point of Distillate Fuels (Withdrawn 2010)[3]
D3699 Specification for Kerosine
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

[1] This test method is under the jurisdiction of 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.

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

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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D7962 Practice for Determination of Minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift
E1 Specification for ASTM Liquid-in-Glass Thermometers
E644 Test Methods for Testing Industrial Resistance Thermometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
E2877 Guide for Digital Contact Thermometers
2.2 *Coordinating Research Council, Inc.*
CRC Report No. 528 Diesel Fuel Low-Temperature Operability Field Test[4]
2.3 *Canadian General Standards Board:*
CAN/CGSB-3.0, No. ~~14.01-M86,~~140.1-M86, Low Temperature Flow Test (LTFT) for Diesel Fuels[5]

NOTE 1—CAN/CGSB-3.0, No. 140.1-M86 is essentially equivalent to Test Method D4539, but the differences in apparatus and procedures may or may not yield different results.

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 a "digital thermometer."

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 The temperature of a series of test specimens of fuel is lowered at a prescribed cooling rate. Commencing at a desired test temperature and at each 1 °C interval thereafter, a separate specimen from the series is filtered through a 17 µm screen until a minimum LTFT pass temperature is obtained. The minimum LTFT pass temperature is the lowest temperature, expressed as a multiple of 1 °C, at which a test specimen can be filtered in 60 s or less.

4.2 Alternatively, a single specimen may be cooled as described under 4.1 and tested at a specified temperature to determine whether it passes or fails at that temperature.

5. Significance and Use

5.1 The Low Temperature Flow Test results are indicative of the low temperature flow performance of the test fuel in some diesel vehicles (according to CRC Report No. 528). The test method is especially useful for the evaluation of fuels containing flow improver additives.

5.2 The test method can be used to supplement other measurements of diesel fuel low temperature behavior (in accordance with Test Methods D97, D2500, and D3117).

6. Apparatus

6.1 *Glass Specimen Vessels, (Borosilicate heat-resistant glass or equivalent)* several 300 mL, clear, heat resistant, wide-mouthed glass bottles having markings indicating 200 mL ± 10 mL and 50 mm to 60 mm ID or clear, heat resistant, tall form beakers with no pour spouts and equivalent dimensions.

[4] Available from Coordinating Research Council, Inc., 219 Perimeter Center Parkway, Atlanta, GA 30346.
[5] Available from CGSB Sales Centre, Ottawa, Canada K1A 1G6.

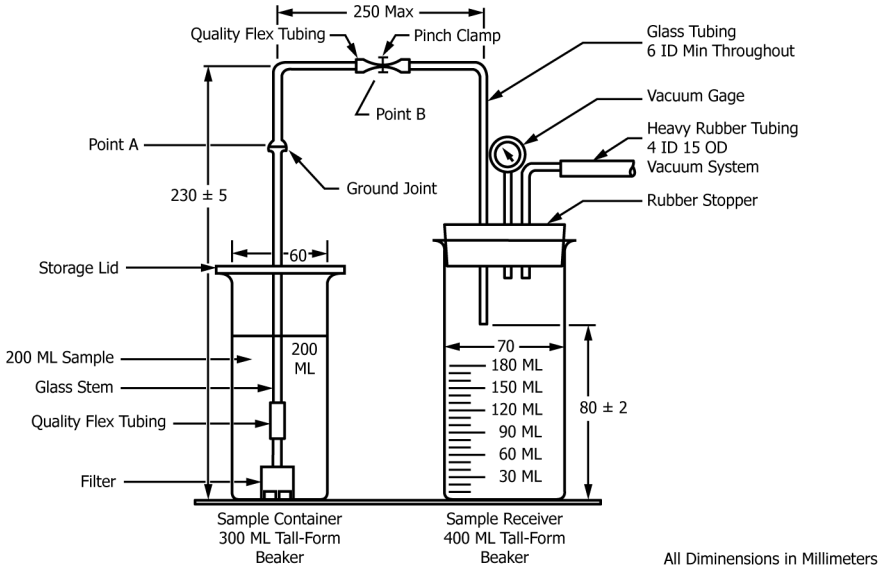


FIG. 1 LTFT Sample Filtration Assembly

6.2 *Glass Receiver Vessels,* clear, heat resistant, glass containers graduated through 180 mL in 10 mL ± 2 mL increments.

6.3 *Filtering Assembly* (see Fig. 1),including a storage lid or some other form of cover, glass tubing, flexible fuel resistant tubing, pinch clamp or valve, and rubber stopper, or other means to provide a vacuum seal.

6.4 *Filter Assembly*[6], as shown in detail in Fig. 2, for each sample container (300 mL beaker). 304SS sintered screen[7] is a twill Dutch weave mesh with a nominal filtration rating of 17 µm. The mesh is 65 wires/cm by 303/315 wires/cm. The wire strands have diameters of 0.0071 cm and 0.0046 cm, respectively. The nominal filtration rating indicates a 98 % removal by mass weight of all particles equal to or greater than 17 µm.

6.5 *Programmable Cooling System,* capable of cooling multiple specimens to the desired temperature at a mean rate of 1.0 °C per hour between +10 °C and −30 °C. Absolute deviation of any single temperature point along the prescribed ramp function must not exceed 0.5 °C in any specimen. The system's size and shape are optional. Either liquid or air baths are acceptable.

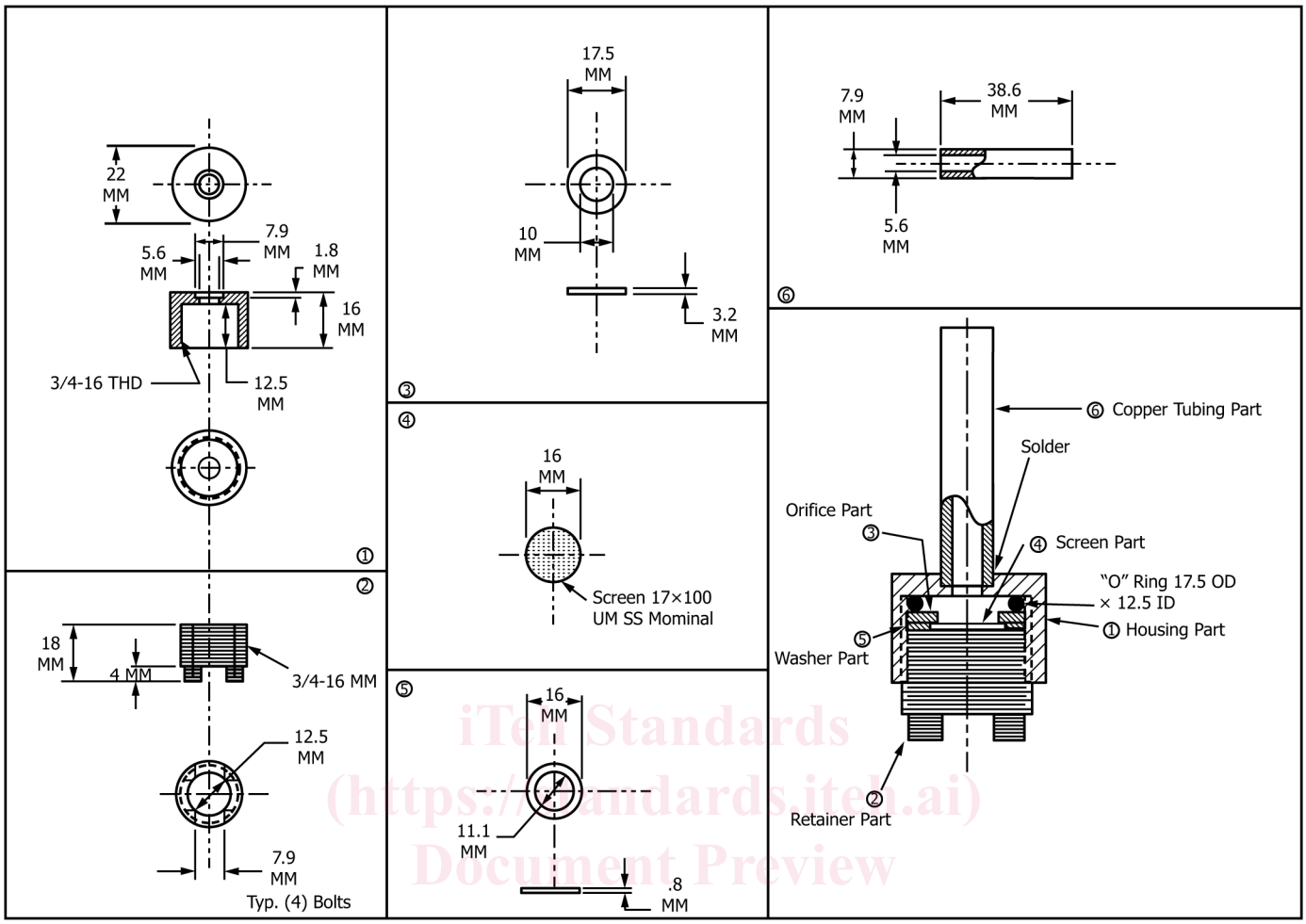
6.6 *Stop Watch or Electric Timer,* capable of measuring tenths of a second.

6.7 *Vacuum System,* capable of maintaining a constant vacuum of 20.0 kPa ± 0.2 kPa below atmospheric pressure at the receiver for the duration of each determination.

6.8 *Temperature Measuring Device*—Either a liquid-in-glass thermometer as described in 6.8.1 or a Digital Contact Thermometer (DCT) meeting the requirements described in 6.8.2.

6.8.1 *Liquid-in-glass Temperature Measuring Device*—Conforming to specifications for ASTM Thermometer 114C for air baths. For liquid baths use either ASTM Thermometer 5C in accordance with Specification E1, or ASTM Thermometer S5C in accordance with Specification E2251, or an alternative liquid-in-glass thermometer with equal or better accuracy and equal temperature response.

[7] The sole source of supply of suitable filter cloth known to the committee at this time is Pall Aerospace Co., Pall Aeropower Corp., 6301 49th St. N, Pinellas Park, FL 33781. 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.



Note: Material for ① ② ③ is brass; material for ⑤ is corrosion resisant polymer; for ⑥ is copper tubing.

FIG. 2 LTFT Filter Assembly

6.8.2 *Digital Contact Thermometer Requirements:*

Parameter	Requirement
DCT	Guide E2877 Class F or better
Nominal Temperature Range[A]	–38 °C to +50 °C for liquid bath –80 °C to 20 °C for air bath
Display Resolution	0.1 °C, minimum
Accuracy[B]	±500 mK (±0.5 °C)
Sensor Type	Platinum Resistance Thermometer (PRT)
Sensor Sheath[C]	4.2 mm OD maximum
Sensor Length[D]	Less than 18 mm
Immersion Depth[E]	Less than 40 mm per Practice D7962
Measurement Drift[E]	Less than 500 mK (0.5 °C) per year
Response Time[F]	Less than or equal to 4 s per Footnote F
Calibration Error	Less than 500 mK (0.5 °C) over the range of intended use.
Calibration Range	Consistent with temperature range of use

Calibration Data	Four data points evenly distributed over the calibration range that is consistent with the range of use. The calibration data is to be included in calibration report.
Calibration Report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body.

[A]The nominal temperature range may be different from the values shown provided the calibration and accuracy criteria are met.
[B]Accuracy is the combined accuracy of the DCT unit which is the display and sensor.
[C]Sensor sheath is the tube that holds the sensing element. The value is the outside diameter of the sheath segment containing the sensor element.
[D]The physical length of the temperature sensing element.
[E]As determined by Practice D7962 or an equivalent procedure.
[F]Response Time—The time for a DCT to respond to a step change in temperature. The response time is 63.2 % of the step change time as determined per Section 9 of Test Method E644. The step change evaluation begins at 20 °C ± 5 °C air to 77 °C ± 5 °C with water circulating at 0.9 m/s ± 0.09 m/s past the sensor.

NOTE 2—A DCT display mounted on the end to the probe's sheath is likely not suitable due to temperature exposure of the electronics. Consult manufacturer for temperature limitations.

NOTE 3—When making measurements below –40 °C with a PRT, it may be necessary to use a 1000 ohm sensor in order to obtain accurate measurements.

6.8.3 The DCT calibration drift shall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with 6.8.2. See Practice D7962.

NOTE 4—When a DCT's calibration drifts in one direction over several calibration checks, it may be an indication of deterioration of the DCT.

7. Reagents

7.1 *Jet A Aviation Turbine Fuel*—As specified in Specification D1655, kerosine, as specified in Specification D3699, Grade No. 1 (or Grade Low Sulfur No. 1), as specified in Specification D975, or equivalent liquid that will not separate at temperatures down to –30 °C.

7.2 *Heptane*—Reagent grade. (**Warning—**Flammable. See A1.2.)

7.3 *Acetone*—Reagent grade. (**Warning—**Flammable. See A1.1.)

8. Sampling

8.1 Obtain a sample in accordance with Practice D4057, or by Practice D4177.

8.2 Each specimen test requires a minimum of 200 mL. Ensure that sufficient sample is obtained to perform the subsequent series of test specimens according to the procedure followed (see Section 4).

9. Procedure

9.1 Filter a fresh specimen of test fuel at 15 °C or higher, through dry, lintless filter paper, having a nominal filtration rating of less than 17 µm. (**Warning—**Combustible liquid. See A1.3.)

NOTE 5—The purpose of this filtration step is to remove any contaminants that interfere with the effectiveness of low temperature flow improver additives. However, this pre-filtration step may remove contaminants that affect the low temperature flow properties of the fuel in actual service. Users of this test method may find it helpful to run the test with and without the pre-filtration step to compare results and in recognition that the precision of the test method will not apply if the pre-filtration step is not carried out.

9.2 Clean and inspect the filter assembly before each test. Filters obtained from the manufacturer are already standardized. Appendix X1 provides a procedure for checking the filter performance, if desired.

9.2.1 Clean the assembled filter with two solvents using a vacuum to draw the solvents through the screen. Begin with three successive washes of at least 50 mL of heptane (**Warning—**Flammable. See A1.2). Follow with three successive washes of at least 50 mL of acetone (**Warning—**Extremely flammable. See A1.1). Air dry the filters after washing.

9.2.2 Visually inspect each filter assembly for screen damage or the presence of particulates. Discard any damaged filter screens. Reclean any filter screens containing particulates. If the standardization of the filter is suspect, obtain a new filter. Alternately, return the filter to the manufacturer for verification; Appendix X1 provides a procedure for checking the filter performance.

9.3 Pour 200 mL of clean, dry fuel into each of the several 300 mL beakers. (**Warning—**Combustible liquid. See A1.3.)

9.4 Insert the clean filter assembly into each specimen container and tightly cover the joint (Point A in Fig. 1) and lid with aluminum foil to exclude condensation.

9.5 Insert a temperature measuring device into one or more separate, identical glass specimen bottles or beaker(s) containing 200 mL of Jet A aviation turbine fuel kerosine, or Grade No. 1 (or Grade Low Sulfur No. 1) or equivalent liquid that will not phase separate at temperatures down to −30 °C. (**Warning—**Combustible liquid. See A1.3.) Place the temperature measuring portion of the device at or near the center of the bottle or beaker approximately half way between the top and the bottom of the liquid.

9.6 Place the specimen bottles or beaker (from 9.3 through 9.5) into the cooling bath at a temperature that is at least 5 °C above the wax appearance point (Test Method D3117) or cloud point (Test Method D2500) of the fuel under test. During multiple specimen testing, a sufficient number of temperature monitoring vessels (from 9.5) must be distributed throughout the cooling bath to insure all test specimen temperatures conform with precision requirements. The positioning of all bottles or beakers shall permit unimpeded circulation of the cooling medium across their bottoms and sides.

9.7 Close the cooling bath's door, if it has one.

9.8 Start the temperature programmer at a rate of −1.0 °C⁄h.

9.9 Before the sample reaches the desired test temperature, check the following:

9.9.1 Apply the pinch clamp or close the valve at Point B in Fig. 1.

9.9.2 Place an empty receiver vessel in position.

9.9.3 Adjust the vacuum to 20.0 kPa ± 0.2 kPa below atmospheric pressure.

9.9.4 Reset the timer.

9.10 When the specimen has cooled to the desired testing temperature, use the filter assembly stem to gently stir (15 revolutions at approximately 1 turn/s) the specimen to disperse any settled wax crystals. Remove the aluminum foil and connect the filtration apparatus joint at Point A in Fig. 1. If the specimen has to be removed from the cooling bath for filtration, these steps shall be completed within 1 min.

9.11 Filter the specimen by removing the pinch clamp or open the valve at Point B in Fig. 1 while simultaneously starting the timer. If necessary, adjust the vacuum system to maintain a vacuum of 20.0 kPa + 0.2 kPa below atmospheric pressure.

9.12 Reapply the pinch clamp or close the valve at Point B in Fig. 1 at precisely 60 s or when suction is lost, whichever occurs first. Record the volume of specimen filtered in millilitres and the testing temperature in degrees Celsius.

9.13 *Pass—Fail Criteria:*

9.13.1 *Passing Result*—The result is considered a pass if most of the specimen has been siphoned into the receiver vessel within 60 s, and suction is lost due to the low level of specimen remaining in the specimen vessel.

NOTE 6—Typically, a volume of approximately 180 mL will be collected in the receiver vessel in a passing result, but this volume may vary due to differences in specimen vessel dimensions and the temperature/volume characteristics of the fuel.

9.13.2 *Failing Result*—The result is considered a fail if suction is not lost within 60 s.

9.14 To determine the minimum LTFT pass temperature, repeat 9.9 through 9.12 on subsequent test specimens that have been cooled 1 °C lower than the previous test temperature, until at least one passing result and one failing result are obtained (see 9.13.1 and 9.13.2).

9.15 Alternatively, cool a single specimen to a desired temperature and determine whether a passing (9.13.1) or a failing (9.13.2) result is obtained.

10. Report

10.1 Report the temperature of the last passing result recorded in 9.14 as:
Minimum LTFT Pass Temperature =_____°C.

10.2 Alternatively, report the result recorded in Step 9.15 as: *Pass or Fail* at _____°C.

11. Precision and Bias

11.1 *Precision*—The precision data were obtained in a cooperative program in which fuels were investigated over the temperature range from −10 °C to −25 °C. This cooperative program used liquid-in-glass thermometers. The precision of this test method as determined by the statistical examination of interlaboratory test results is as follows:

11.1.1 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

$$\text{Repeatability} = 2\ ^{\circ}\text{C} \tag{1}$$

11.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

$$\text{Reproducibility} = 4\ ^{\circ}\text{C} \tag{2}$$

11.2 *Bias*—There being no criteria for measuring bias in these test product combinations, no statement of bias can be made.

12. Keywords

12.1 diesel fuel; filterability; flow; low temperature; LTFT

ANNEX

(Mandatory Information)

A1. WARNING STATEMENTS

A1.1 Acetone

A1.1.1 (**Warning—**Extremely flammable.)

A1.1.2 (**Warning—**Vapors may cause flash fire.)