Designation: D4539 – 16

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 agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken 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 that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

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 and health 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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D97 Test Method for Pour Point of Petroleum Products

D975 Specification for Diesel Fuel Oils

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D3117 Test Method for Wax Appearance Point of Distillate Fuels (Withdrawn 2010)[3]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

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

E1137 Specification for Industrial Platinum 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, 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 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

3.1.1.1 *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

[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.

[4] Available from Coordinating Research Council, Inc., 219 Perimeter Center Parkway, Atlanta, GA 30346.

[5] Available from CGSB Sales Centre, Ottawa, Canada K1A 1G6.

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

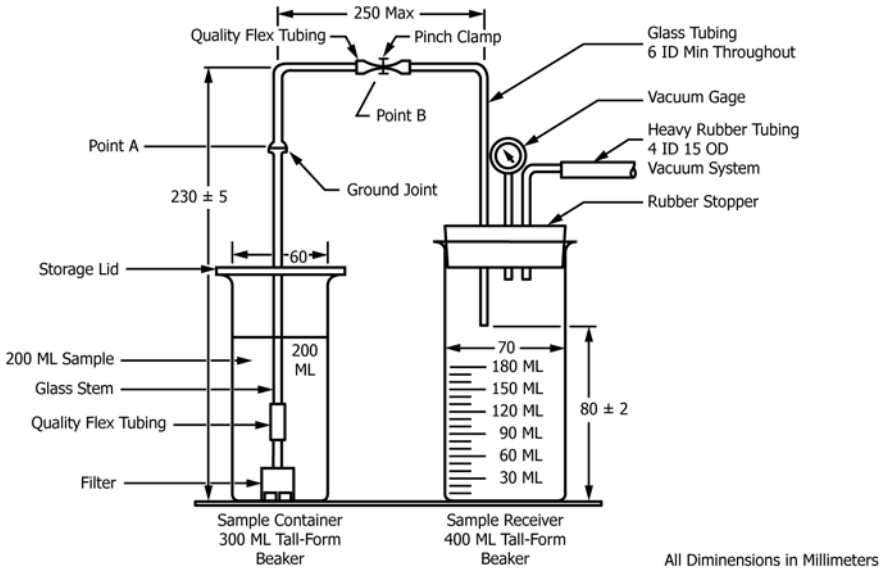


FIG. 1 LTFT Sample Filtration Assembly

display and/or recording device that may be internal or external to the device. These devices are sometimes referred to as a "digital thermometer."

3.1.1.2 *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.

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

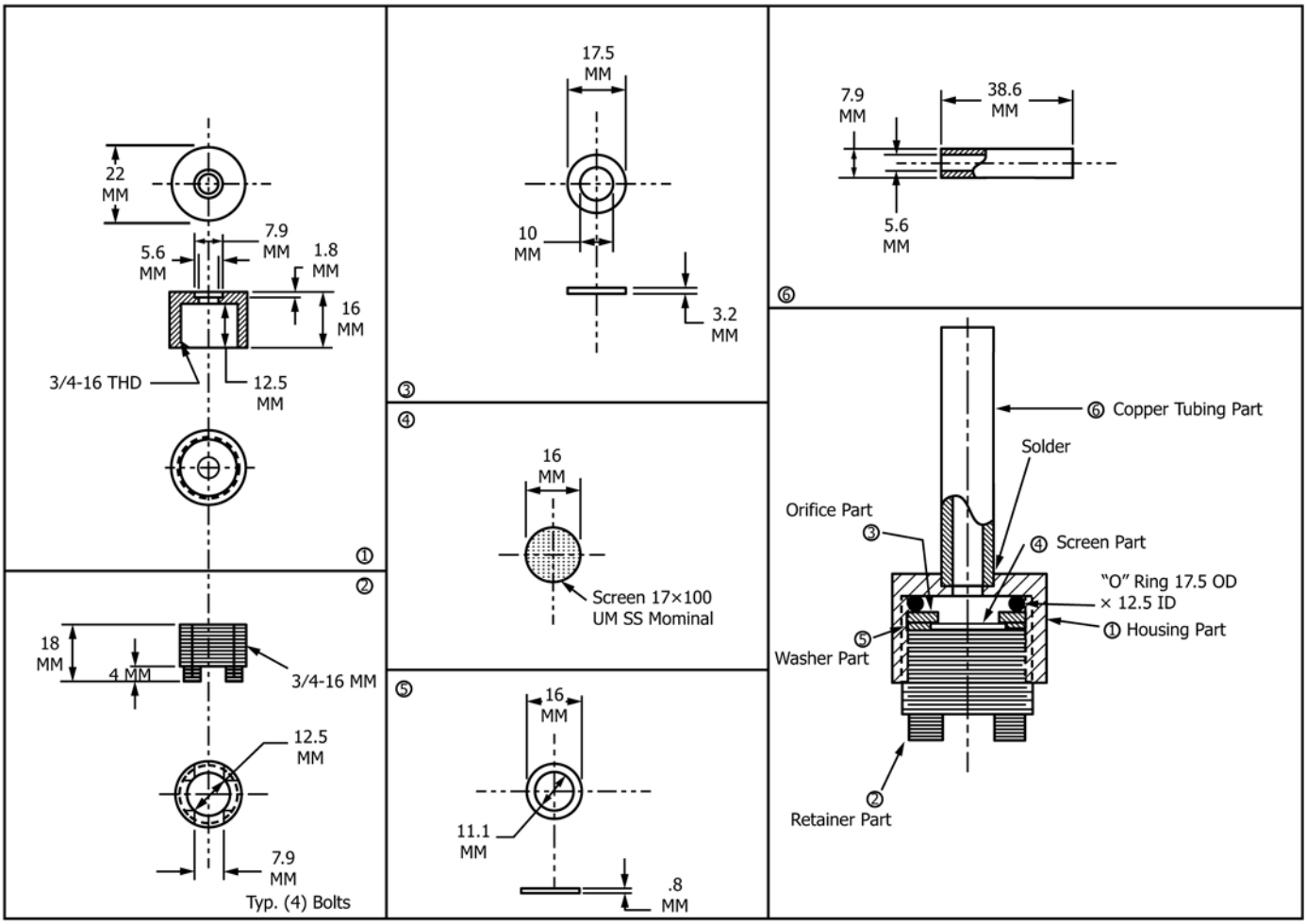
6.3 *Filtering Assembly* (see Fig. 1), including a storage lid or some other form of cover, glass tubing, flexible fuel resistant tubing, pinch clamp or valve, and rubber stopper, or other means to provide a vacuum seal.

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.

[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

6.7 *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.

6.8.2 *Digital Contact Thermometer Requirements:*

Parameter	Requirement
DCT	Guide E2877 Class F or better
Temperature Range	–80 °C to 20 °C
Display Resolution	0.1 °C, minimum
Sensor Type	Platinum Resistance Thermometer (PRT)
Sensor	3 mm o.d. sheath with a sensing element less than 18 mm in length
Minimum Immersion	Less than 40 mm per Practice D7962
Accuracy	±500 mK (±0.5 °C) for combined probe and sensor
Response Time	Less than or equal to 25 s as defined in Specification E1137
Drift	Less than 500 mK (0.5 °C) per year
Calibration Error	Less than 500 mK (0.5 °C) over the range of intended use.
Calibration Range	–75 °C or lower to 0 °C or greater
Calibration Data	4 data points evenly distributed over calibration range with data 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.

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.

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 3—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 4—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 5—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.

$$\text{Repeatability} = 2\ ^\circ\text{C} \quad (1)$$

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.

$$\text{Reproducibility} = 4\ ^\circ\text{C} \quad (2)$$

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.)

A1.1.3 (**Warning—**Keep away from heat, sparks, and open flame.)

A1.1.4 (**Warning—**Keep container closed.)

A1.1.5 (**Warning—**Use with adequate ventilation.)

A1.1.6 (**Warning—**Avoid buildup of vapors, and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters.)

A1.1.7 (**Warning—**Avoid prolonged breathing of vapor or spray mist.)

A1.1.8 (**Warning—**Avoid contact with eyes or skin.)

A1.2 n-Heptane

A1.2.1 (**Warning—**Flammable. Harmful if inhaled.)

A1.2.2 (**Warning—**Keep away from heat, sparks, and open flame.)

A1.2.3 (**Warning—**Keep container closed.)

A1.2.4 (**Warning—**Use with adequate ventilation.)

A1.2.5 (**Warning—**Avoid prolonged breathing of vapor or spray mist.)

A1.2.6 (**Warning—**Avoid prolonged or repeated skin contact.)

A1.3 Combustible Liquid

A1.3.1 (**Warning—**Combustible. Vapor harmful.)

A1.3.2 (**Warning—**Keep away from heat, sparks, and open flame.)

A1.3.3 (**Warning—**Keep container closed.)

A1.3.4 (**Warning—**Use with adequate ventilation.)

A1.3.5 (**Warning—**Avoid prolonged breathing of vapor or spray mist.)

A1.3.6 (**Warning—**Avoid prolonged or repeated skin contact.)

A1.4 Mercury

A1.4.1 (**Warning—**Poison. May be harmful or fatal if inhaled or swallowed.)

A1.4.2 (**Warning—**Vapor harmful, emits toxic fumes when heated.)

A1.4.3 (**Warning—**Vapor pressure at normal room temperature exceeds threshold limit value for occupational exposure.)

A1.4.4 (**Warning—**Do not breathe vapor.)

A1.4.5 (**Warning—**Keep container closed.)

A1.4.6 (**Warning—**Use with adequate ventilation.)

A1.4.7 (**Warning—**Do not take internally.)

A1.4.8 (**Warning—**Cover exposed surfaces with water, if possible, to minimize evaporation.)

A1.4.9 (**Warning—**Do not heat.)

A1.4.10 (**Warning—**Keep recovered mercury in tightly sealed container prior to sale or purification. Do not throw in sink or in rubbish.)

APPENDIX

(Nonmandatory Information)

X1. LTFT WIRE FILTER SCREEN STANDARDIZATION PROCEDURE

X1.1 Procedure

X1.1.1 Dismantle and inspect the wire filter screen assembly. Discard any damaged screens.

X1.1.2 Reassemble and wash the filter assembly as specified in 9.2.

X1.1.3 Filter the Vistone[8] A-30 reference oil through dry, lintless filter paper, having a normal filtration rating of less than 17 µm at room temperature.

X1.1.4 Pour 150 mL of clean, dry Vistone A-30 into a 300 mL heat resistant tall-form beaker (Borosilicate heat-resistant glass or equivalent) with no pour spout.

X1.1.5 Insert the filter assembly into the sample.

X1.1.6 Insert a thermometer into the beaker and wait until the temperature reading stabilizes.

X1.1.7 Filter the Vistone A-30 by applying a vacuum of 20.0 kPa ± 0.2 kPa below atmospheric pressure while simultaneously starting the stopwatch.

X1.1.8 Stop the timer at the instant the filter assembly loses suction on the oil and begins drawing in air.

X1.1.9 Record the filtration time in seconds and the filtration temperature to the nearest 0.5 °C.

X1.1.10 Calculate the temperature correction factor corresponding to the filtration temperature using the following equations. The viscosity of the Vistone A-30 reference oil will be provided by the vendor.

$$\log\log(v_t + 0.7) = A - B\log T \quad \text{(X1.1)}$$

$$C_t = v_{20}/v_t \quad \text{(X1.2)}$$

where:

v_t = viscosity of reference fluid at specified temperature, mm^2

v_{20} = viscosity of reference fluid at 20 °C, mm^2,

A, B = constants to be solved,

C_t = temperature correction factor at specified temperature,

T = temperature in Kelvin at which v_t is determined,

T = 273.1 + °C.

Example: Determine temperature correction factor at 10 °C. If the viscosities for Vistone A-30 are:

27.04 mm^2/s (cSt) at 40 °C

5.38 mm^2/s (cSt) at 100 °C

X1.1.11 Enter the viscosity and corresponding temperature data in (Eq X1.1):

$$\log\log(v_t + 0.7) = A - B\log T \quad \text{(X1.3)}$$

$$\log\log(27.04 + 0.7) = A - B\log(273.1 + 40) \quad \text{(X1.4)}$$

$$\log\log(5.38 + 0.7) = A - B\log(273.1 + 100) \quad \text{(X1.5)}$$

X1.1.12 Solve for A and B:

$$A = 8.8500,\ B = 3.4823 \quad \text{(X1.6)}$$

X1.1.13 Determine the viscosity of Vistone A-30 at 20 °C and 10 °C using (Eq X1.1)

$$\log\log(v_{20} + 0.7) = 8.8500 - 3.4823\log(273.1 + 20) \quad \text{(X1.7)}$$

$$v_{20} = 64.75 \quad \text{(X1.8)}$$

$$\log\log(v_{10} + 0.7) = 8.8500 - 3.4823\log(273.1 + 10) \quad \text{(X1.9)}$$

$$v_{10} = 111.33 \quad \text{(X1.10)}$$

X1.1.14 Calculate the temperature correction factor at 10 °C using (Eq X1.2)

$$C_t = v_{20}/v_t \quad \text{(X1.11)}$$

$$C_{10} = 64.75/111.33 = 0.582 \quad \text{(X1.12)}$$

X1.1.15 Multiply the actual filtration time in seconds by the correction factor to obtain the corrected filtration time. (Example: for an actual filtration time of 79 s at 10 °C, the corrected filtration time would be 79 × 0.582 = 46 s (X1.1.11), and the screen would be reported as acceptable.)

[8] Vistone is a registered trademark of Infineum International Limited.

X1.2 Report

X1.2.1 If the corrected filtration time falls between 45 and 53 s, inclusive, the screen is reported as acceptable for use in the LTFT. If the corrected filtration time falls outside this range, the screen is unacceptable and should be discarded.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D4539 – 10) that may impact the use of this standard. (Approved Dec. 1, 2016.)

(1) Added requirements for DCT temperature measuring device.

(2) Reformatted units to conform to SI.