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Methods of testing petroleum and associated products - Low temperature flow test (LTFT) for diesel fuels

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Preface

This National Standard of Canada CAN/CGSB-3.0 No. 140.1-2022 supersedes the 2017 edition.

Changes since the previous edition

- Added reference to ASTM E1 for liquid-in-glass thermometers and ASTM D8164 for digital contact thermometers (5.4).
- Added description of additional procedures for cleaning filter screens (6.2.2).
- Relocated note from 6.11 to 6.6, added reference to microcrystalline wax, and removed final sentence.
- Clarified instructions for minimum low temperature flow test (LTFT) pass temperature vs. LTFT pass-fail (6.11, 6.12).
- Allowed digital contact thermometers in verification procedure (A.4.1, A.4.2).
- Clarified washing of filter assembly in verification procedure (A.5.2).
- Calculated new temperature correction factors for Table A.1 and revised footnote describing calculations.
- Removed bibliography.

The following definitions apply in understanding how to implement this National Standard of Canada:

- "shall" indicates a **requirement**;
- "should" indicates a **recommendation**;
- "may" is used to indicate that something is **permitted**;
- "can" is used to indicate that something is **possible**, for example, that an organization is able to do something.

Notes accompanying clauses do not include requirements or alternative requirements. The purpose of a note accompanying a clause is to separate explanatory or informative material from the text. Annexes are designated normative (mandatory) or informative (non-mandatory) to define their application.

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Methods of testing petroleum and associated products - Low temperature flow test (LTFT) for diesel fuels

1 Scope

1.1 This method describes a procedure for determining the filter plugging tendency of diesel fuels at low temperature, usually due to the formation of wax crystals (see note in 6.1).

1.2 The method applies to all distillate diesel fuels. Fuels are expected to provide satisfactory fuel flow in automotive equipment at temperatures equal to or higher than the minimum pass temperature as described in 3.1.1.

1.3 This method is referenced in ASTM D4539-17 (Note 1).

1.4 The testing and evaluation of a product against this method may require the use of materials and/or equipment that could be hazardous. This document does not purport to address all the safety aspects associated with its use. Anyone using this method has the responsibility to consult the appropriate authorities and to establish appropriate health and safety practices in conjunction with any applicable regulatory requirements prior to its use.

1.5 Units of measurement – Quantities and dimensions used in this standard are provided in units from the International System of Units (SI units). This standard expresses the industry standard nominal measurements in North America of “% by mass” and “% by volume”. The SI equivalent expressions for these units are % (m/m) and % (V/V) respectively.

2 Normative references

The following normative documents contain provisions that, through reference in this text, constitute provisions of this method. The referenced documents may be obtained from the sources noted below.

Note: The contact information provided below was valid at the date of publication of this method.

An undated reference is to the latest edition or revision of the reference or document in question, unless otherwise specified by the authority applying this method. A dated reference is to the specified revision or edition of the reference or document in question.

2.1 ASTM International

ASTM D2500 — *Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels*

ASTM D4539-17 — *Standard Test Method for Filterability of Diesel Fuels by Low-Temperature Flow Test (LTFT)*

ASTM D5771 — *Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Optical Detection Stepped Cooling Method)*

ASTM D5772 — *Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Linear Cooling Rate Method)*

ASTM D5773 — *Standard Test Method for Cloud Point of Petroleum Products and Liquid Fuels (Constant Cooling Rate Method)*

ASTM D8164 — *Standard Guide for Digital Contact Thermometers for Petroleum Products, Liquid Fuels, and Lubricant Testing*

ASTM E1 — *Standard Specification for ASTM Liquid-in-Glass Thermometers*

2.1.1 Contact information

The above may be obtained from ASTM International. Telephone: 610-832-9585. Fax: 610-832-9555. Web site: www.astm.org, or from IHS Markit. Telephone: 613-237-4250 or 1-800-267-8220. Fax: 613-237-4251. Web site: www.global.ihs.com.

2.2 Coordinating Research Council

CRC Report 528 (9/83) — *Diesel Fuel Low-Temperature Operability Field Test*

2.2.1 Contact information

The above may be obtained from the Coordinating Research Council at jantucker@crcao.org. Telephone: 678-795-0506. Fax: 678-795-0509.

2.3 SAE International

SAE Technical Paper 982576 — *The Use of Flow Improved Diesel Fuel at Extremely Low Temperatures*

2.3.1 Contact information

The above may be obtained from SAE International. Telephone: 1-877-606-7323 (Canada and U.S. only) or 724-776-4970 (outside Canada and U.S.). Fax: 724-776-0790. Web site: www.sae.org.

3 Summary of method

3.1 The temperature of a series of 200 mL samples of test fuel is gradually lowered to the desired testing temperature at a controlled cooling rate of 1 °C/h. At 1 °C intervals, a sample from the series is filtered through a 17 µm screen at 20 kPa gauge vacuum (20 kPa below atmospheric pressure).

3.1.1 This procedure is repeated until a sample from the series does not pass the test. The minimum LTFT pass temperature is the lowest temperature, expressed as a multiple of 1 °C, at which a minimum 180 mL of sample, when cooled under the prescribed conditions, can be filtered in 60 s or less.

3.2 Alternatively, a sample may be cooled as described in 3.1 and tested at a specified temperature to determine if it passes or fails at that temperature.

4 Significance and use

4.1 The low temperature flow test helps evaluate the filterability of diesel fuels at low temperatures where wax plugging of diesel fuel systems can restrict the operation of automotive equipment. Fuels passing this test usually provide satisfactory flow performance at temperatures equal to or higher than the minimum LTFT pass temperature (refer to CRC Report No. 528). The test method is especially useful for evaluating fuels containing cold flow improver additives.

4.2 This test can be used as a measure of the flow performance of diesel fuels at low temperatures by petroleum refiners, marketers, distributors, consumers, and others concerned with tailoring and handling diesel fuels for automotive use.

5 Apparatus

5.1 Glass bottles

Several clear, heat-resistant, wide-mouthed bottles or tall form beakers having 240 to 300 mL capacity and 50 to 60 mm internal diameter (I.D.).

5.2 **Filtration assembly** (see figure 1)

5.3 **Filter** (see figure 2)

The wire filter screen[1] (fine wire mesh cloth, 304 SS sintered screen) is a twill Dutch weave mesh with a nominal filtration rating of 17 µm. The mesh is 65 wires/cm (165 wires/in.) by 303 to 315 wires/cm (770 to 800 wires/in.). The wire strands have diameters of 0.0071 cm (0.0028 in.) and 0.0046 cm (0.0018 in.), respectively. The nominal filtration rating indicates a 98 % removal by mass of all particles equal to or greater than 17 µm.

5.4 Temperature measuring device

Liquid-in-glass thermometer (ASTM E1 type 114C for air baths or type 5C for liquid baths) calibrated under total immersion conditions, spanning the temperature range of the test and calibrated in at least 0.5 °C increments, or a digital contact thermometer calibrated to provide equivalent accuracy to the liquid-in-glass thermometer over the temperature range of use (see ASTM D8164 for guidance on digital contact thermometers).

5.5 Cooling bath

Bath capable of cooling multiple samples to the required temperature, in a controlled manner of 1.0 ± 0.1 °C/h with a maximum deviation of ± 0.5 °C from the ramp, using either refrigerated air or liquid. The size and shape of the bath are optional. In an air bath, the air should circulate around all sides of the test bottle or beaker, and the bottle or beaker should sit on an insulating disk or by other means be kept from direct contact with a directly cooled surface (example: a rack in an air cooled chamber). When immersed in a liquid coolant, the liquid level in the test bottle or beaker should be at or below the liquid coolant level. A lid or cover should be placed over the bottles or beakers during the cooling to minimize condensation.

5.6 **Stop watch** (or electric timer)

Capable of measuring tenths of a second.

5.7 Vacuum system

Capable of maintaining a constant vacuum of 20 ± 0.2 kPa gauge (150 ± 1.5 mm Hg) and containing a surge tank with a minimum volume of 4 L.

6 Procedure

6.1 Filter a fresh sample of test fuel (CAUTION: combustible liquid) through dry, lintless filter paper, at 15 °C or higher, to remove any foreign material, sediment and/or water. Whatman No. 1 filter paper or equivalent product is suitable for this purpose.

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[1] Bulk filter screen material not verified according to Annex A is available in sheets from Pall Canada Limited, 3450 Ridgeway Drive, Unit 6, Mississauga, Ontario L5L 0A2, telephone: 905-542-0330. The catalogue description is: Rigimesh Sintered Woven Wire Mesh, Grade M, 304 SS.

Suitable wire filter screens (17 µm, 16 mm O.D.) verified according to Annex A are available from InnoTech Alberta, Fuels & Lubricants Group, 250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4, telephone: 780-450-5538, fax: 780-988-9053, Web site: http://innotechalberta.ca/OurTeams/FuelsandLubricants.aspx.

Note: 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 can remove contaminants that affect the low temperature flow properties of the fuel in actual service. If the pre-filtration step is omitted, report that the test procedure has been modified and that the precision of this test method does not apply.

6.2 Clean and inspect the filter screen and assembly before each test. Verify filters before initial use and after every 20 tests as specified in Annex A.

6.2.1 Clean the assembled filter with two solvents, using vacuum to draw the solvents through the screen. Use three successive washes of at least 50 mL of heptane (CAUTION: flammable) followed by three successive washes of at least 50 mL of acetone (CAUTION: extremely flammable).

Note: Acetone will degrade neoprene O-rings over time, so regular replacement of these parts of the filter assembly is advised. Air-dry the filters after washing.

6.2.2 Visually inspect each filter assembly for screen damage or the presence of particulates. Discard any damaged filter screens. Re-clean and verify or replace any filter screens containing particulates. Reversing the filter screen orientation to backwash the screen during the washing step and ultrasonic cleaning of the screen in a solvent may be used to help dislodge particulates.

6.3 Pour 200 mL of clean, dry sample at room temperature into a wide-mouthed bottle or tall form beaker.

6.4 Insert a clean filter assembly (filter, housing and tubing to point A, see figure 1) into each sample container and tightly cover the joint (point A of figure 1) with aluminum foil or other suitable cover to minimize introduction of condensation into the fuel samples.

6.5 Insert either a liquid-in-glass thermometer or a digital contact thermometer into one or more separate bottles or beakers containing 200 mL of light middle distillate fuel such as JET A or JET A-1 (CAUTION: combustible liquid) that will not phase separate at the lowest bath temperature used. The bulb of the liquid-in-glass thermometer or the tip of the digital contact thermometer should be as close as possible to the centre of the fuel sample.

6.6 Place the bottles or beakers (see 6.3 and 6.5) into the cooling bath at a temperature at least 5 °C above the cloud point (as measured in accordance with ASTM D2500, ASTM D5771, ASTM D5772 or ASTM D5773) of the fuel under test.

Note: In some cases, wax can form at temperatures significantly above the measured cloud point due to slow crystallization of paraffinic wax (see SAE 982576) or poor detection of microcrystalline (isoparaffinic) wax. In such cases, starting cooling at just 5 °C above the cloud point could yield results that do not reflect low-temperature operability, as conditions for wax formation would not represent those encountered in the field. More representative results can be obtained by starting cooling at a higher temperature, above the range where wax can form. A suitable starting temperature can be demonstrated by the absence of wax (for example, a passing LTFT) following an overnight cold soak.

During testing of multiple samples, sufficient temperature monitoring vessels (see 6.5) shall be distributed throughout the cooling bath to ensure all test sample temperatures conform to precision requirements. Allow the samples to reach temperature equilibrium.

6.7 Start the temperature programmer for the cooling bath to cool at a rate of 1 °C/h.

6.8 Before the sample reaches the desired test temperature, do the following:

a) clamp the tubing closed at point B (see figure 1);

b) place an empty sample receiver in position;

c) adjust the vacuum to 20 ± 0.2 kPa (150 ± 1.5 mm Hg);

d) reset the timer.

6.9 When the sample is cooled to the desired testing temperature, using the stem of the filter assembly, gently stir (15 revolutions at approximately 1 revolution per second) the sample to disperse any settled wax crystals. Connect the joint to the tubing of the filtration apparatus at point A (see figure 1) so that it rests on the bottom of the sample bottle.

Note: Early in development of this test procedure, it was noted that the stirring step improved precision.

6.10 Filter the sample by opening the valve or pinch clamp at point B (see figure 1) while starting the timer. If necessary, adjust the vacuum bleed to maintain a vacuum of 20 ± 0.2 kPa gauge.

6.10.1 If the sample can be filtered in less than 60 s, stop the timer the instant the filter assembly loses suction on the sample and begins drawing air. Stop the flow of fuel sample by disconnecting the glass joint at point A. This allows fuel contained in the glass tubing to flow into the glass receiver. Disconnect the vacuum source. Measure the volume of sample filtered (mL) after warming to room temperature (15 to 25 °C). Record the testing temperature (°C), the volume of sample filtered (mL), and the filtration time (s). If a volume of 180 mL or more has been filtered, this is considered a passing result.

6.10.2 If the sample cannot be filtered within 60 s, stop the timer at 60 s and disconnect the glass joint at point A (see figure 1). Measure the volume of sample filtered in millilitres after warming to room temperature. Record the testing temperature (°C) and the volume of sample filtered (mL). If less than 180 mL has been filtered, this is considered a failing result.

6.11 To determine the minimum LTFT pass temperature, repeat 6.8 through 6.10 at 1 °C intervals until at least one passing result (see 6.10.1) and one failing result (see 6.10.2) are obtained. Record the temperature (°C) of the last passing result that preceded the failing result.

6.12 Alternatively, the results from 6.10 may be used to report whether a sample passes (see 6.10.1) or fails (see 6.10.2) at a desired temperature.

7 Report

7.1 Report the temperature to the nearest whole degree recorded in 6.11 as: minimum LTFT pass temperature: _____ °C.

7.2 Alternatively, report the result and test temperature to the nearest whole degree recorded in 6.12 as *pass* or *fail* at _____ °C.

8 Precision

8.1 The precision of the method as determined by the statistical examination of interlaboratory test results falling in the range of -20.0 to -28.0 °C is as follows.

8.1.1 Repeatability

The difference between successive results obtained by the same operator with the same apparatus on identical test material would exceed 1.5 °C only in one case in twenty.

8.1.2 Reproducibility

The difference between two single results obtained by different operators in different laboratories on identical test material would exceed 3.7 °C only in one case in twenty.

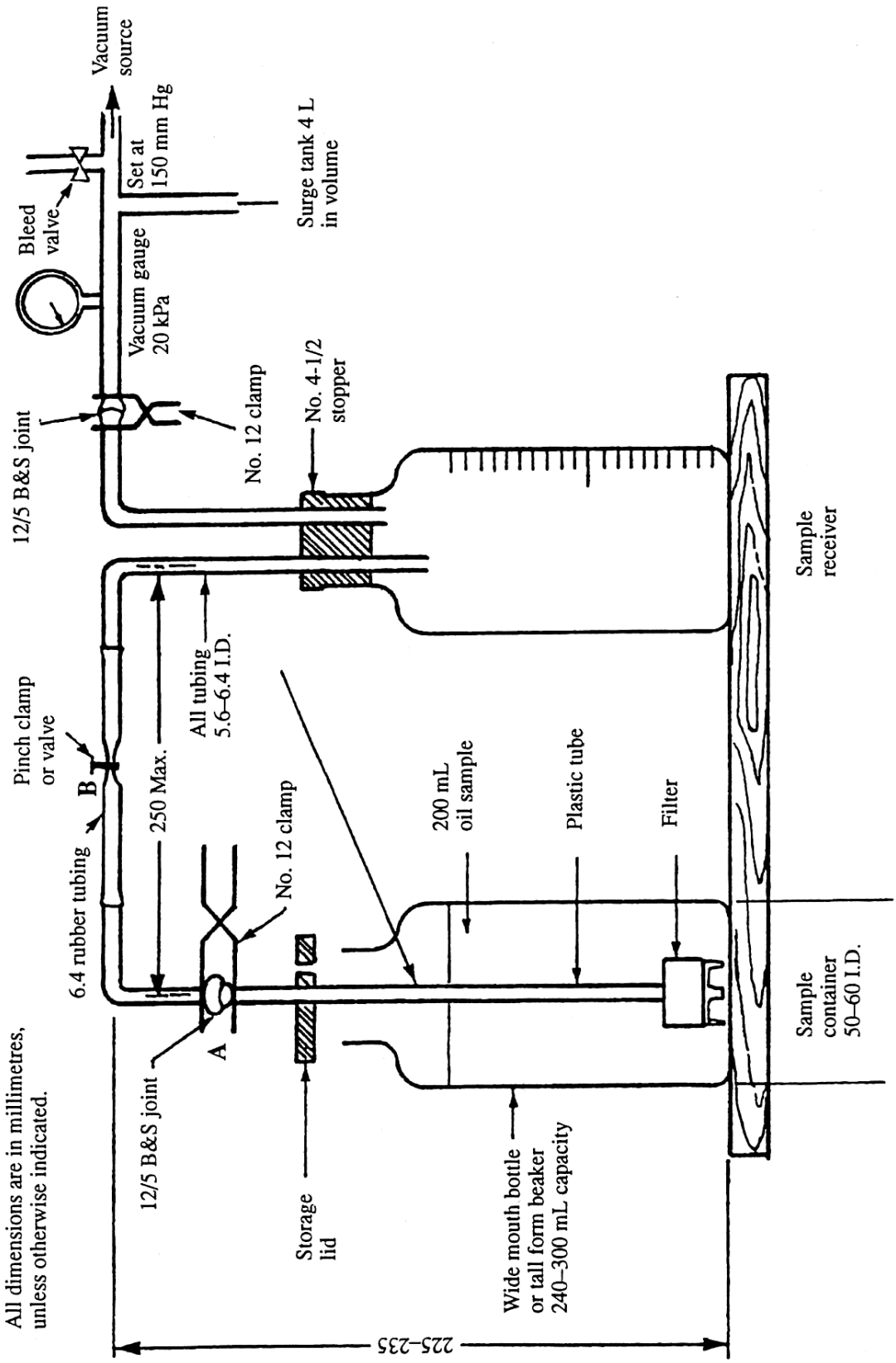

FIGURE 1

Sample Filtration Assembly

Figure 1 — Sample filtration assembly

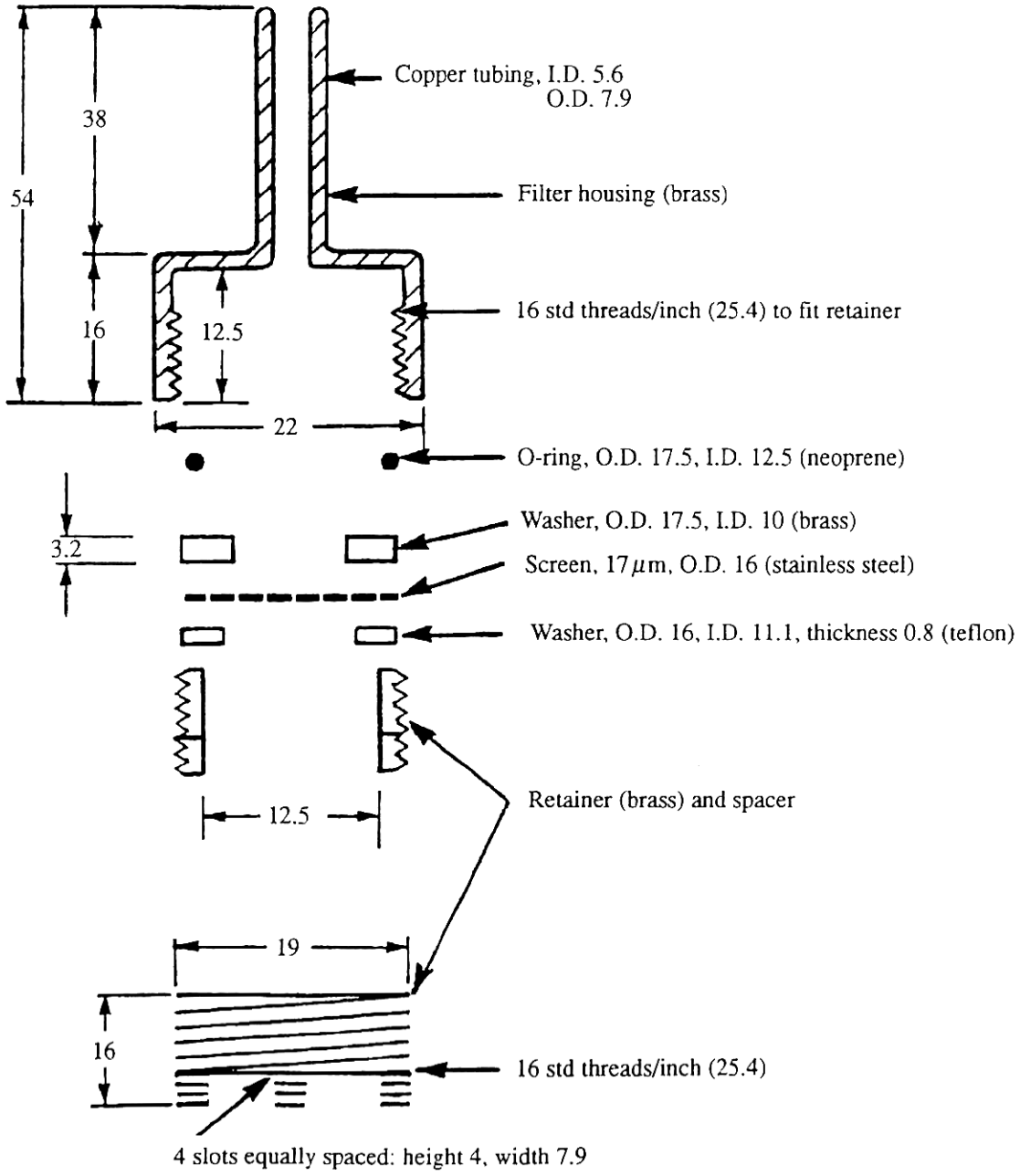

Figure 2 — Filter details

Annex A
(normative)

Verification procedure for LTFT wire filter screens

A.1 Scope

A.1.1 This procedure provides a rapid, accurate method for verifying the 17 µm wire filter screens used in the low temperature flow test for diesel fuels.

A.1.2 The procedure is aimed at guarding against the use of filter screens which would give spurious LTFT results due to improper mesh size, physical damage, blockage with foreign material, etc.

A.2 Summary of method

A.2.1 The method is similar to the filtration step of the LTFT except that a) a synthetic di-ester reference oil (ditridecyl adipate)[2] is used in place of a diesel fuel sample, b) the oil is filtered at room temperature, and c) filtration time is adjusted using temperature correction factors.

A.2.2 A 150 mL sample of the reference oil is filtered through the wire filter screen at 20 ± 0.2 kPa (150 ± 1.5 mm Hg) gauge vacuum at room temperature. If the corrected filtration time is between 45 and 53 s inclusive, the screen is acceptable for use in the LTFT. If not, the screen should be rejected.

A.3 Precision

A.3.1 Repeatability

Exceeds 4 s once in twenty cases.

A.3.2 Reproducibility

Not known.

A.4 Apparatus

A.4.1 Filter assembly and filter, as depicted in figures 1 and 2 of the LTFT procedure except that a temperature measuring device shall be inserted in the sample container.

A.4.2 Total immersion liquid-in-glass thermometer having a range of -2 to 80 °C to the nearest 0.5 °C, or a digital contact thermometer.

A.4.3 Vacuum source capable of maintaining 20 ± 0.2 kPa (150 ± 1.5 mm Hg) gauge vacuum.

A.5 Procedure

A.5.1 Filter the reference oil through dry, lintless filter paper at room temperature. Whatman No. 1 or equivalent filter paper is suitable for this purpose.

A.5.2 Wash the filter assembly as described in 6.2. Air-dry the filters after washing.

A.5.3 Pour a 150 mL sample of the filtered reference oil into a wide-mouthed bottle or a tall form beaker (see A.5.1).

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[2] Ditridecyl adipate reference oil is available from ExxonMobil Chemical as Esterex™ A51. The reference oil was previously known as Vistone A-30.

A.5.4 Insert the filter assembly into the sample.

A.5.5 Filter the reference oil by applying 20 ± 0.2 kPa (150 ± 1.5 mm Hg) gauge vacuum while simultaneously starting the timer.

A.5.6 Stop the timer the instant the filter assembly loses suction on the oil and begins drawing in air.

A.5.7 Record the filtration time in seconds and the temperature of the oil filtered to the nearest 0.5 °C.

A.5.8 Select the correction factor corresponding to the filtration temperature from Table A.1.

A.5.9 Multiply the actual filtration time in seconds by the correction factor to obtain the corrected filtration time (for example, for an actual filtration time of 37 s at 24.5 °C, the corrected filtration time would be 37 x 1.256 = 46.5 s, and the screen would be reported as acceptable).

A.6 Report

A.6.1 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 reported as unacceptable.

Table A.1[3] — Temperature correction for the filter screen verification procedure

Filtration temperature	Correction factor	Filtration temperature	Correction factor	Filtration temperature	Correction factor
+10.0	0.582	+20.0	1.000	+30.0	1.595
10.5	0.599	20.5	1.025	30.5	1.630
11.0	0.617	21.0	1.051	31.0	1.666
11.5	0.635	21.5	1.077	31.5	1.702
12.0	0.653	22.0	1.104	32.0	1.738
12.5	0.671	22.5	1.131	32.5	1.775
13.0	0.691	23.0	1.159	33.0	1.813
13.5	0.710	23.5	1.187	33.5	1.851
14.0	0.730	24.0	1.215	34.0	1.889
14.5	0.750	24.5	1.244	34.5	1.928
15.0	0.771	25.0	1.274	35.0	1.968
15.5	0.792	25.5	1.304	35.5	2.008
16.0	0.813	26.0	1.334	36.0	2.049
16.5	0.835	26.5	1.365	36.5	2.090
17.0	0.857	27.0	1.397	37.0	2.132
17.5	0.880	27.5	1.428	37.5	2.174
18.0	0.903	28.0	1.461	38.0	2.217
18.5	0.927	28.5	1.494	38.5	2.261
19.0	0.951	29.0	1.527	39.0	2.305
19.5	0.975	29.5	1.561	39.5	2.349

[3] This table is based on viscosities for the reference oil (see A.2.1) of 27.04 cSt at 40 °C and 5.38 cSt at 100 °C. Temperature correction factors were calculated as described in ASTM D4539-17, Appendix X1. Alternately, temperature correction factors using different reference oil viscosities may be calculated in accordance with ASTM D4539-17, Appendix X1.