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National Standard of Canada

Methods of testing petroleum and associated products

Low temperature flow test (LTFT) for diesel fuels

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Introduction

This 2017 edition of CAN/CGSB-3.0 No.140.1 includes a note to address cases whereby wax can form at temperatures significantly above cloud point, as well as clarifying information on testing equipment and sample handling.

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No. 140.1-2005

Low temperature flow test (LTFT) for diesel fuels

1 Scope

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).

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.

This method is referenced and compared in ASTM D4539.

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.

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 addresses provided below were 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

D341 — *Standard Practice for Viscosity-Temperature Charts for Liquid Petroleum Products*

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

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

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

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

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

2.1.1 Source

The above may be obtained from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, U.S.A., telephone 610-832-9585, fax 610-832-9555, Web site www.astm.org, or from IHS Markit, 200-1331 MacLeod Trail SE, Calgary, Alberta T2G 0K3, telephone 613-237-4250 or 1-800-267-8220, fax 613-237-4251, Website www.global.ihs.com.

2.2 Coordinating Research Council (CRC)

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

2.2.1 Source

The above may be obtained from the Coordinating Research Council at jantucker@crcao.org. You may also contact the CRC as follows: 5755 North Point Parkway Suite 265; Alpharetta, GA 30022; 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 Source

The above may be obtained from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, U.S.A. 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 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 single 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 temperature (refer to CRC Report No. 528). The test method is especially useful for evaluating fuels containing 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 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: Thermometer (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 thermocouple or RTD thermister.

5.5 Cooling 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 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 is suitable for this purpose.

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 may remove contaminants that affect the low temperature flow properties of the fuel in actual service. Users of this test method may omit the pre-filtration step in order to evaluate potential service problems, but shall identify that they modified the test procedure and that the precision of this test method will 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: first 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.

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

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[1] Bulk filter screen material not verified according to Annex A is available in sheets from Pall Canada Limited, 7205 Millcreek Drive, Mississauga, Ontario L5N 3R3, 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 the Innotech Alberta , Fuels & Lubricants Group, 250 Karl Clark Road, Edmonton, Alberta, Canada T6N 1E4, telephone (780) 450-5104, fax (780) 988-9053, Web site http://innotechalberta.ca/OurTeams/FuelsandLubricants.aspx

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 thermometer, thermocouple or thermister 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 temperatures down to -40°C. The bulb of the thermometer or the tip of the thermocouple or thermister 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. During multiple samples testing, a sufficient number of temperature monitoring vessels (see 6.5) shall be distributed throughout the cooling bath to insure all test sample temperatures conform to precision requirements. Allow the samples to reach temperature equilibrium.

6.7 Start the temperature programmer 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 r/s) 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 sucking 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 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.

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 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 (e.g., a passing

LTFT) following an overnight cold soak. If this condition is not met at 5°C above the cloud point, then cold soaks at successively higher temperatures can be conducted until a suitable starting temperature is determined.

6.12 Alternatively, cool a single sample to a desired temperature and determine whether a passing result (see 6.10.1) or a failing result (see 6.10.2) is obtained.

7 Report

7.1 Report the temperature to the nearest whole degree recorded in 6.10.1 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.

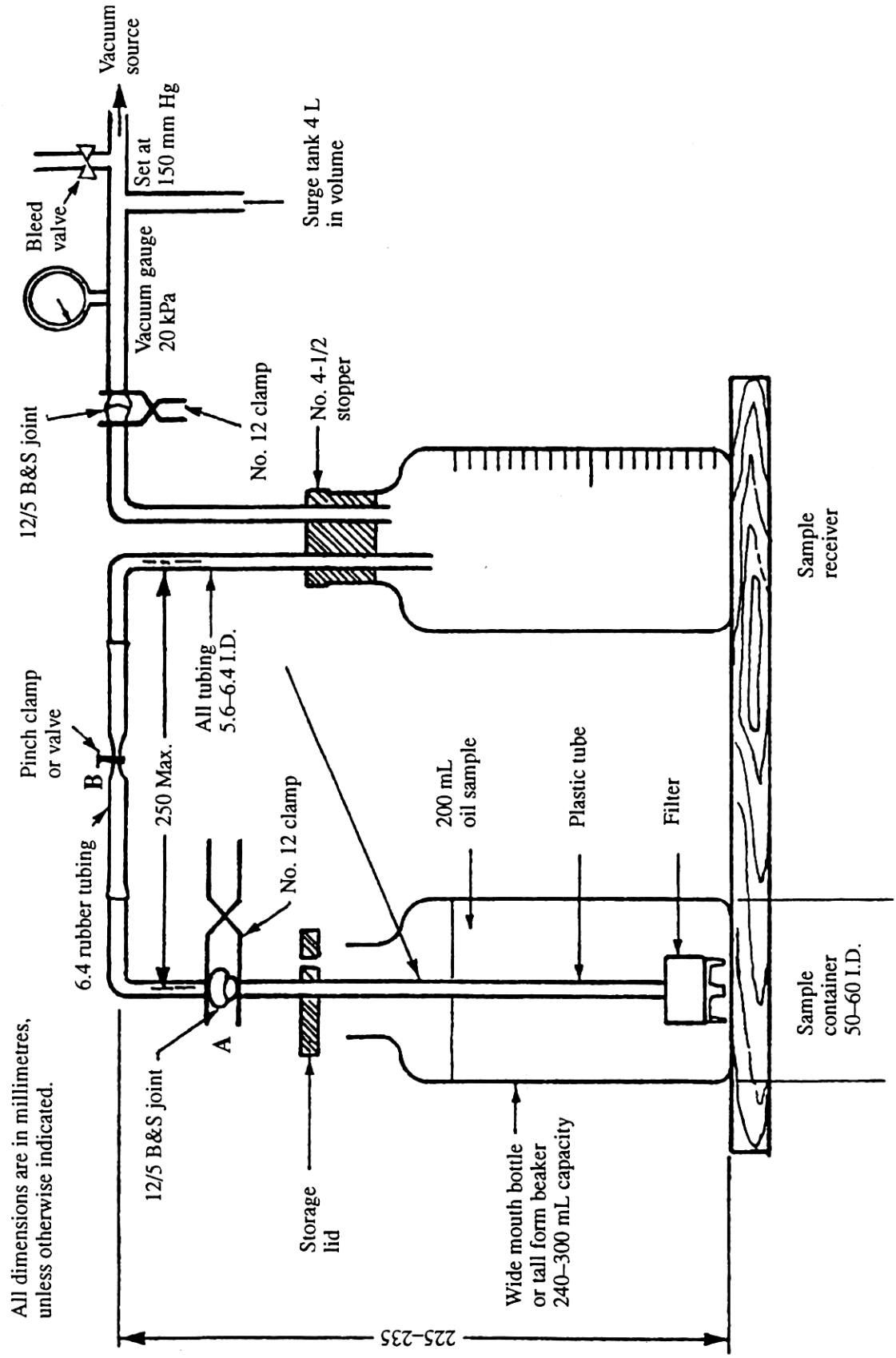

Figure 1 – Sample Filtration Assembly

All dimensions are in millimetres,
unless indicated otherwise.

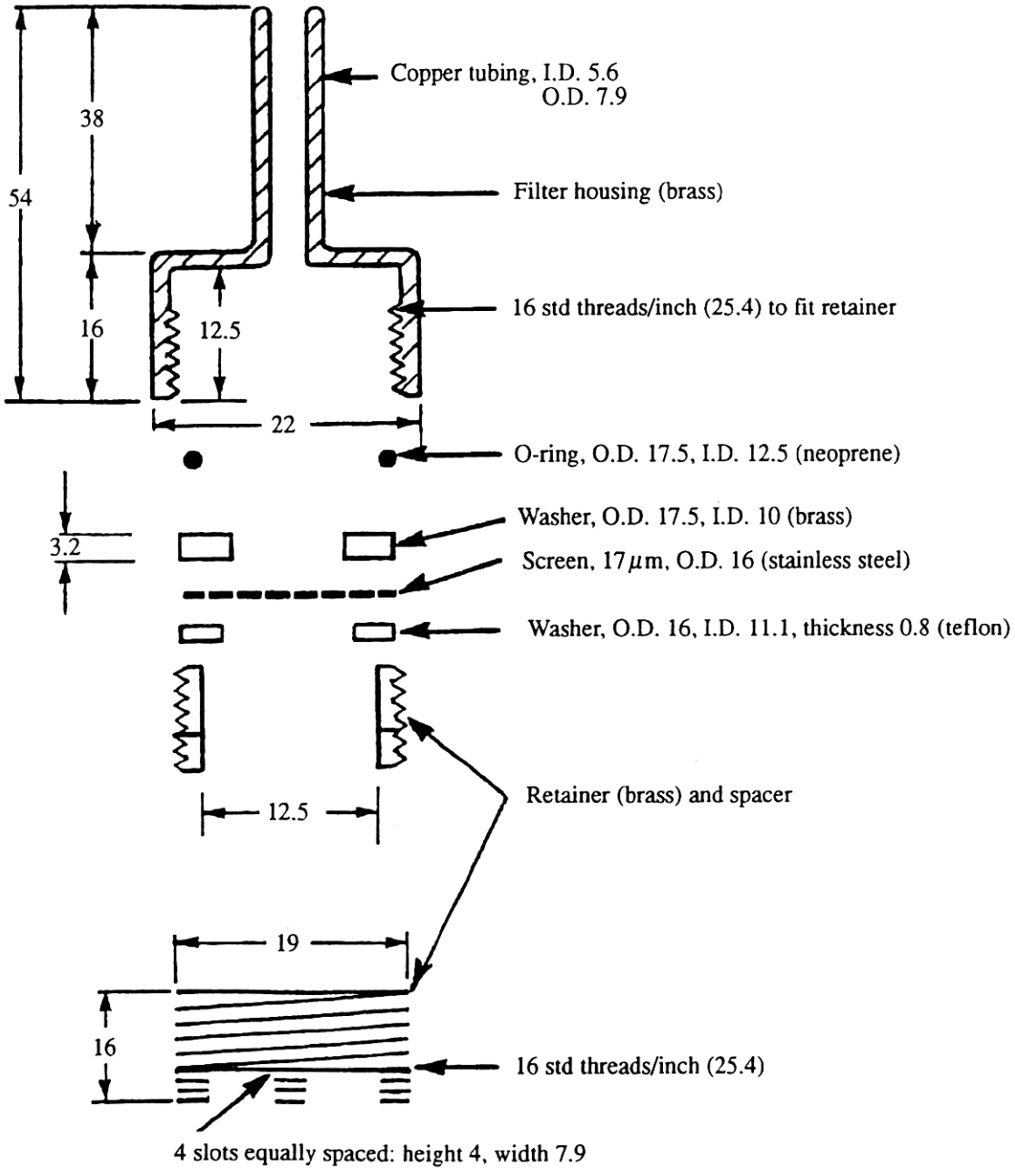

Figure 2 — Filter details

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 Filtering assembly and filter as depicted in Figures 1 and 2 of the LTFT procedure except that a total immersion thermometer shall be inserted in the sample container.

A.4.2 Total immersion thermometer having a range of -2 to 80°C to the nearest 0.5°C.

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 filter assembly in heptane and acetone using vacuum to draw solvent through the screen. 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 A5.1).

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.

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[2] Ditridecyl adipate reference oil is available from Imperial Oil Chemicals Division as Esterex™ A51.

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 A1.

A.5.9 Multiply the actual filtration time in seconds by the correction factor to obtain the corrected filtration time. (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 A1[3] — Temperature correction for the filter screen verification procedure

Filtration temperature	Correction factor	Filtration temperature	Correction factor	Filtration temperature	Correction factor
+10.0	0.567	+20.0	1.000	+30.0	1.627
10.5	0.585	20.5	1.026	30.5	1.664
11.0	0.603	21.0	1.053	31.0	1.701
11.5	0.621	21.5	1.081	31.5	1.739
12.0	0.640	22.0	1.109	32.0	1.778
12.5	0.659	22.5	1.137	32.5	1.817
13.0	0.679	23.0	1.166	33.0	1.857
13.5	0.699	23.5	1.196	33.5	1.898
14.0	0.719	24.0	1.226	34.0	1.939
14.5	0.740	24.5	1.256	34.5	1.980
15.0	0.761	25.0	1.287	35.0	2.022
15.5	0.783	25.5	1.319	35.5	2.065
16.0	0.805	26.0	1.351	36.0	2.109
16.5	0.828	26.5	1.383	36.5	2.152
17.0	0.851	27.0	1.416	37.0	2.197
17.5	0.875	27.5	1.450	37.5	2.242
18.0	0.899	28.0	1.484	38.0	2.288
18.5	0.924	28.5	1.519	38.5	2.334
19.0	0.949	29.0	1.554	39.0	2.381
19.5	0.974	29.5	1.590	39.5	2.429

[3] This table is based on the following viscosities for Vistone A-30 (previous name for ditridecyl adipate reference oil from Imperial Oil Chemicals Division): 27.04 cSt at 40°C and 5.38 cSt at 100°C. Viscosities at the temperatures above were calculated from equation 1 in Section X1.1 of ASTM D341, where Z = (v + 0.07). Alternately, the temperature correction factor may be calculated in accordance with ASTM D4539, Annex 1.

Bibliography

[1] ASTM International. ASTM D97 — *Standard Test Method for Pour Point of Petroleum*. See 2.1.1 for the source where to obtain a copy.

[2] ASTM International. ASTM D975 — *Standard Specification for Diesel Fuel Oils*. See 2.1.1 for the source where to obtain a copy.

[3] ASTM International. ASTM D1655 — *Standard Specification for Aviation Turbine Fuels*. See 2.1.1 for the source where to obtain a copy.

[4] ASTM International. ASTM E1 — *Standard Specification for ASTM Liquid-in-Glass Thermometers*. See 2.1.1 for the source where to obtain a copy.