BSR/ASHRAE Standard 86-2013R

Public Review Draft

Methods of Testing the Floc Point of Refrigeration Grade Oils

Second Public Review (April 2026)
(Draft Shows Complete Proposed Standard)

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FOREWORD

This standard describes a standard test method for measuring the floc point (waxing tendency) of refrigeration-grade oils. Petroleum-derived oils are mixtures of large numbers of chemically distinct hydrocarbon molecules. At the low temperatures encountered in the low-pressure side of refrigeration units, some of the larger molecules separate from the bulk of the oil in the form of wax-like deposits. Wax deposition of these molecules in refrigeration systems is undesirable, as they are known to clog capillary tubes and to cause expansion valves to stick. The floc point procedure requires, in addition to the oil, a fluid in which the oil is completely miscible to a temperature below that at which floc will form. Refrigerant-12 (R-12) was selected because it fits this requirement. Refrigerant-22 (R-22), for example, cannot be used because phase separation would, generally, result before the floc point was reached. The ensuing hazing, clouding, and separation into two different layers precludes obtaining a floc point. The industry has a great deal of experience in successfully extrapolating floc point data from the test tube to field application.

Development of a floc point using some other fluid besides R-12 is feasible. However, it would require a research project and extrapolation of the data from another fluid without the field correlation, which has been available for R-12 floc point data, could result in major field problems.

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1. PURPOSE

This standard provides a method for measuring the waxing tendency of refrigeration-grade oils.

2. SCOPE

The floc point measurement indicates the waxing tendency of refrigeration-grade oils at low temperatures. The floc point is defined as the highest temperature at which wax or other solid substances precipitate when a mixture 10% by volume of oil and 90% by volume of R-12 is cooled under specified conditions. The results can be used to compare the waxing tendency of several different oils.

3. DEFINITIONS

floc point: the highest temperature at which solid substances precipitate when a mixture 10% by volume of oil and 90% by volume of R-12 is cooled under specified conditions.

flocculent: containing, or occurring in the form of, loosely aggregated particles or soft flakes.

refrigeration-grade oil: a naphthenic/paraffinic type oil that is stable with system components, will form a friction-reducing film between rubbing surfaces and seal critical clearances, and has low-temperature transport properties suitable for the application for which it is used.

wax: higher molecular weight materials that are not soluble in R-12/mineral oil mixtures at a particular temperature.

4. APPARATUS

4.1 Cooling Bath. A cooling bath is required for obtaining the necessary temperature. It is prepared by using dry ice or a refrigeration system as the cooling source. The cooling bath shall be large enough for testing three sample tubes at one time. A large, wide-mouthed, clear Dewar flask is one cooling bath option. Dry ice in a liquid such as ethanol or acetone is required. The use of liquids such as ethanol is necessary because this liquid has the advantage of reduced frosting when the sample tube is raised from the bath for observation. A mechanical stirrer shall be provided for stirring the bath. Proper stirring is important to eliminate temperature

differences. A small piece of aluminum foil in the liquid will indicate the degree of stirring obtained. If an electric motor is used, it is required to be of a type safe for use above the flammable cooling medium. A wire mesh basket with small openings shall be provided in the bath for the introduction of the dry ice so small pieces of dry ice cannot come into contact with the sample tube and cause local cold spots. The wire mesh basket can be raised or lowered to control the bath temperature**.**

4.2 Temperature-Measuring Devices. The temperature is measured with a precision electrical digital thermometer, a mercury thermometer, or an alcohol thermometer. ASTM standard thermometers 6F or 6C shall be used for liquid-in-glass thermometers and calibrated as prescribed by ASTM E1 [1]. Other digital thermometers or temperature-measuring devices shall have measurement uncertainty of ±1°F (±0.5°C) or less and shall be calibrated in accordance with ANSI/ASHRAE Standard 41.1 [2].

4.3 Sample Tube. The sample tube consists of a thick-walled borosilicate glass tube with a flared end and round bottom, as shown in Figure 1. Tube dimensions shall be 0.375 in. (9.5 mm) ID × 0.600 in. (15 mm) OD × 9 in. (228 mm) long. The tube shall be permanently graduated at the 0.34 oz (10.0 mL) volume (calibrated at room temperature).

4.4 Metal Connector Fittings. The metal connector fittings shall be shown as in Figure 1.

4.5 Graph Paper. In certain instances, graph paper is used with a glass plate to judge the cloudiness of the test sample. The graph shall have 20 lines per 1.0 in. (25.4 mm).

4.6 Mechanical Vacuum Pump. A mechanical vacuum pump that provides an absolute pressure of 13.3 Pa (0.1 mm Hg) shall be used.

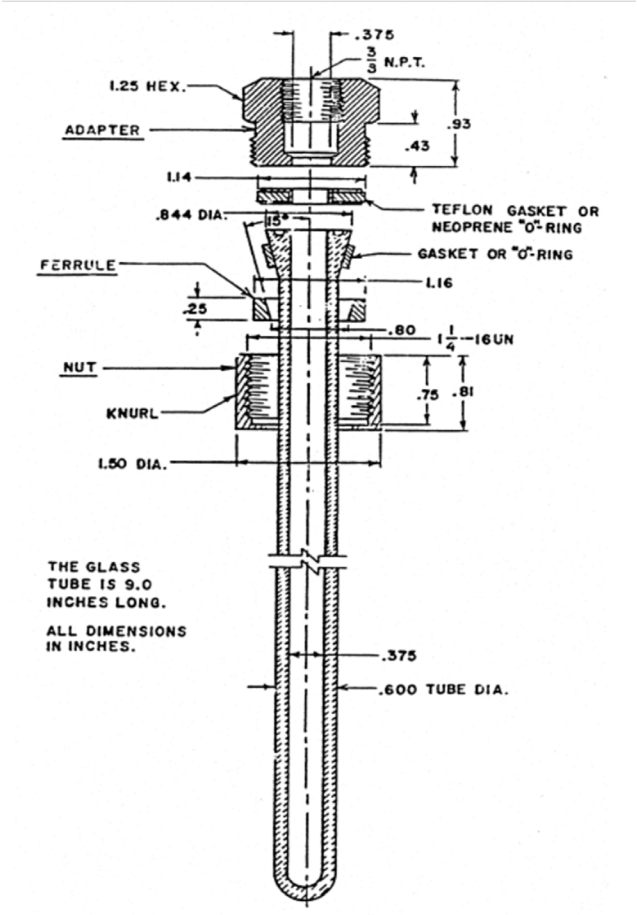

Figure 1 Floc tube with metal connector.

5. TEST PROCEDURE

5.1 Use proper personal protective equipment (PPE), such as a face mask, protective shield, and heavy gloves when handling the charged pressure tube outside the cooling bath or when evacuating the tube. The glass tube is under pressure and a hazard exists whenever the temperature is above –21°F (–30°C). When using ethanol or acetone, take appropriate precautions to prevent fire. These solvents and their vapors are highly flammable.

Do not use a glass tube at a pressure exceeding 80 psig (552 kPa) or temperatures exceeding 200°F (93°C). The tube shall not be subjected to any bending strain. Inspect the glass components for wear on a regular basis. Erosion, stress, cracks, nicks, or deep scratches provide early warning of the need for glass replacement. The glass may erode evenly so wear is not visibly noticeable. If wear is detected, replace the component to eliminate the potential cause of breakage.

5.2 The sample tube shall be cleaned, dried, and stored in a desiccator prior to use. Appropriate pre-cautions shall be taken to eliminate the introduction of water into the sample tube while charging with oil or refrigerant. Prepare a manifold or other system for evacuating and charging the sample tube. The refrigerant tubing shall be dried before use.

5.3 Ensure that stirring of the liquid is adequate for obtaining uniform temperature within the cooling bath. Install the thermometer in a tube with the same tube dimensions as the floc tube. Immerse the tube in the bath and fill it with the same fluid used in the cooling bath.

5.4 Transfer 0.03 oz (1.0 mL) of the sample oil into the sample glass tube using a 0.07 oz (2.0 mL) syringe for precise oil measurement (at room temperature). Attach a small brass valve to the sample tube using the metal connector fittings. Evacuate the sample tube to 13.3 Pa (0.1 mm Hg) absolute pressure to de-gas the oil.

5.5 Introduce a quantity of liquid R-12 so that the total volume of oil and R-12 is 0.34 oz (10.0 mL). The method of introduction shall preclude the introduction of water into the tube. This addition of R-12 and the measurement of the volume shall be done in the cooling bath at a temperature of –25°F (–32°C) to –30°F (–34°C).

5.6 The tube shall be disconnected from the charging assembly and allowed to warm up to the point where the refrigerant/oil mixture may be made homogeneous by shaking. Proper PPE shall be used. Any excess R-12 can then be bled off at a temperature above –20°F (–29°C). In this way, the volume of the refrigerant/oil mixture can be adjusted to 0.34 oz (10.0 mL) when measured at –25°F (–32°C) to –30°F (–34°C).

5.7 The sample tube containing the refrigerant/oil mixture shall then be immersed in the cooling baht, which has previously been adjusted to a temperature of approximately 0°F (–18°C). The temperature of the cooling bath shall then be lowered at a rate of 1°F (0.5°C) to 2°F (1°C) per minute by careful addition of dry ice or use of a controlled chilling device. Temperature readings with the thermometer or thermocouple in the test tube in the bath give values comparable to the temperature in the test sample tube.

5.8 If an oil shows a cloud at 0°F (–18°C), the test shall be repeated to make certain that water has not been inadvertently introduced into the oil during the charging operation. If an oil is wet, it can be dried by heating the oil under vacuum or in an open container at 302°F (150°C) for 1 hour while bubbling nitrogen into the oil.

5.9 The tube shall be observed as the temperature is lowered, beginning at 0°F (–18°C). If a clear Dewar flask is used, the tube may be viewed through the Dewar flask. The tube may also be removed briefly (10 seconds) from the cooling bath for viewing. Avoid disturbing the contents by removing the tube carefully, without shaking. When distinct flocculent material is observed in the mixture, the test thermometer or thermocouple in the bath shall be read. This temperature shall be reported as the floc point. Some oils show a haze at temperatures of 5°F (3°C) to 10°F (6°C) above the floc point.

5.10 In some instances, the mixture becomes increasingly hazy or cloudy as the temperature is lowered but does not form distinct flocculent material. When this occurs, the tube shall be removed from the cooling bath and held against a glass plate with graph paper behind the glass plate (see Section 4.5). When the cloudiness in the tube prevents seeing the lines on the graph paper, then the temperature shall be read, and this condition shall be an endpoint to the floc point test. If desired, the tube may be warmed up and the test repeated using the same sample tube.

5.11 Repeat the test with a second oil sample, run at a different time. If the results of these two observations agree within 5°F (3°C), the average of the two shall be reported as the floc point of the oil.

6. REPRODUCIBILITY OF RESULTS

Duplicate determinations of floc point by the same operator in the same laboratory shall agree within 5°F (3°C).

Duplicate determinations between two different laboratories are suspect if they differ by more than 15°F (8°C). This reproducibility requirement is based on a round robin test conducted in 1982 by seven laboratories using three oil samples (see Informative Appendix A). The results were evaluated using ASTM E691-1979, *Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method.*

7. SIGNIFICANCE OF RESULTS

The floc point can be used to compare the waxing tendencies of different oils. In actual refrigeration systems, different refrigerant and oil concentrations are encountered, so test results cannot be used to directly predict actual system performance. The oil concentration in the expansion devices of most refrigeration and air-conditioning systems is lower than 10% by volume, resulting in a significantly lower temperature at which wax will separate from the refrigerant/oil mixture.

8. NORMATIVE REFERENCES

1. ASTM. 2014. ASTM E1-2014(R2025), *Standard Specification for ASTM Liquid-in-Glass Thermometers.* West Conshohocken, PA: ASTM International.
2. ASHRAE. 2024. ANSI/ASHRAE Standard 41.1, *Standard Methods for Temperature Measurement*. Peachtree Corners, GA: ASHRAE.

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INFORMATIVE APPENDIX A—ROUND ROBIN TEST RESULTS

Table A-1 is a summary of the data from seven independent laboratories that provided floc points on three oil types.

Table A-1 Round Robin Test Results–Test Conducted June 1982[a]

	Floc Point, °F			Floc Point, °C		
Lab	**Oil A**	**Oil B**	**Oil C**	**Oil A**	**Oil B**	**Oil` C**
1	–94, –93	–60, –60	–48, –49	–70, –70	–51, –51	–44, –45
2	–89, –92	–67, –66	–50, –58	–67, –69	–54, –55	–46, –50
3	—	–73, –72	–42, –38	—	–58, –58	–41, –39
4	–77, –82, –81	–48, –52, –52	–41, –38, –43	–61, –63, –63	–44, –47, –47	–41, –39, –42
5	–80, –79, –80	–57, –56, –56	–39, –39, –40	–62, –62, –62	–50, –49, –49	–39, –39, –40
6	–91, –87	–60, –55	–45, –44	–68, –66	–51, –48	–43, –42
7	–75, –75	—	–52, –48	–60, –60	—	–47, –44

a. Data developed by project committee members for the 1983 edition of this standard.

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INFORMATIVE APPENDIX B—BIBLIOGRAPHY

1. DIN. 2007. DIN 51351, Testing of lubricants – Determination of floc point of refrigerator oils by pressure tube method. Berlin, Germany: Deutsches Institut für Normung.
2. DIN. 1976. DIN 51590-2, Testing of lubricants – Determination of the content of material insoluble in R-12 refrigerator oils; method at –40 Deg C (233 K). Berlin, Germany: Deutsches Institut für Normung.
3. "Kinetic" Technical Bulletin 14, Handling 'Freon' Fluorinated Hydrocarbon Compounds in the Laborato-ry, Freon Products Div., E. I. DuPont de Nemours & Co.
4. ASTM. 1979. ASTM E691-1979, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method. West Conshohocken, PA: ASTM International.