Designation: D 1015 – 05

An American National Standard

Standard Test Method for Freezing Points of High-Purity Hydrocarbons[1]

This standard is issued under the fixed designation D 1015; 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 a procedure for the precise measurement of the freezing points of high-purity hydrocarbons.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *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 hazard statements, see 5.1, 6.1 and 6.2.

NOTE 1—For the calculation of the molal purity of essentially pure compounds from measured freezing points and for procedures to be used for the sampling and determination of purity of certain specific compounds, see Test Method D 1016.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1016 Test Method for Purity of Hydrocarbons from Freezing Points

D 1265 Practice for Sampling Liquefied Petroleum (LP) Gases (Manual Method)

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

3. Summary of Test Method

3.1 The precise experimental measurement of the freezing point is made from interpretation of time-temperature freezing or melting curves.[3]

4. Significance and Use

4.1 The freezing point measured by this test method, when used in conjunction with the physical constants for the hydrocarbons listed in Test Method D 1016, allows the determination of the purity of the material under test. A knowledge of the purity of these hydrocarbons is often needed to help control their manufacture and to determine their suitability for use as reagent chemicals or for conversion to other chemical intermediates or finished products.

5. Apparatus

5.1 *Freezing-Point Apparatus,*[4,5] as shown in Figs. 1-3 comprising a freezing tube, a metal sheath for the freezing tube, a Dewar flask for the cooling bath, a Dewar flask for the warming bath, a stirring mechanism, suitable clamps and holders for the parts, and the absorption tubes. The outer walls of all Dewar flasks can be covered with adhesive tape to minimize danger from glass in case of breakage. (**Warning**—When using liquid nitrogen as a refrigerant, provide a means to prevent condensation of oxygen in the space between the freezing tube and the metal sheath and subsequent sealing of the space by ice forming on the ceramic (or glass) fiber collar. Provide the metal sheath with suitable openings in the *sides* and *bottom*. Failure to do this may result in breakage of the freezing tube when the liquefied oxygen evaporates within the sealed space.)

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04 on Hydrocarbon Analysis.

Current edition approved Nov. 1, 2005. Published November 2005. Originally approved in 1949. Last previous edition approved in 2004 as D 1015–99(2004).

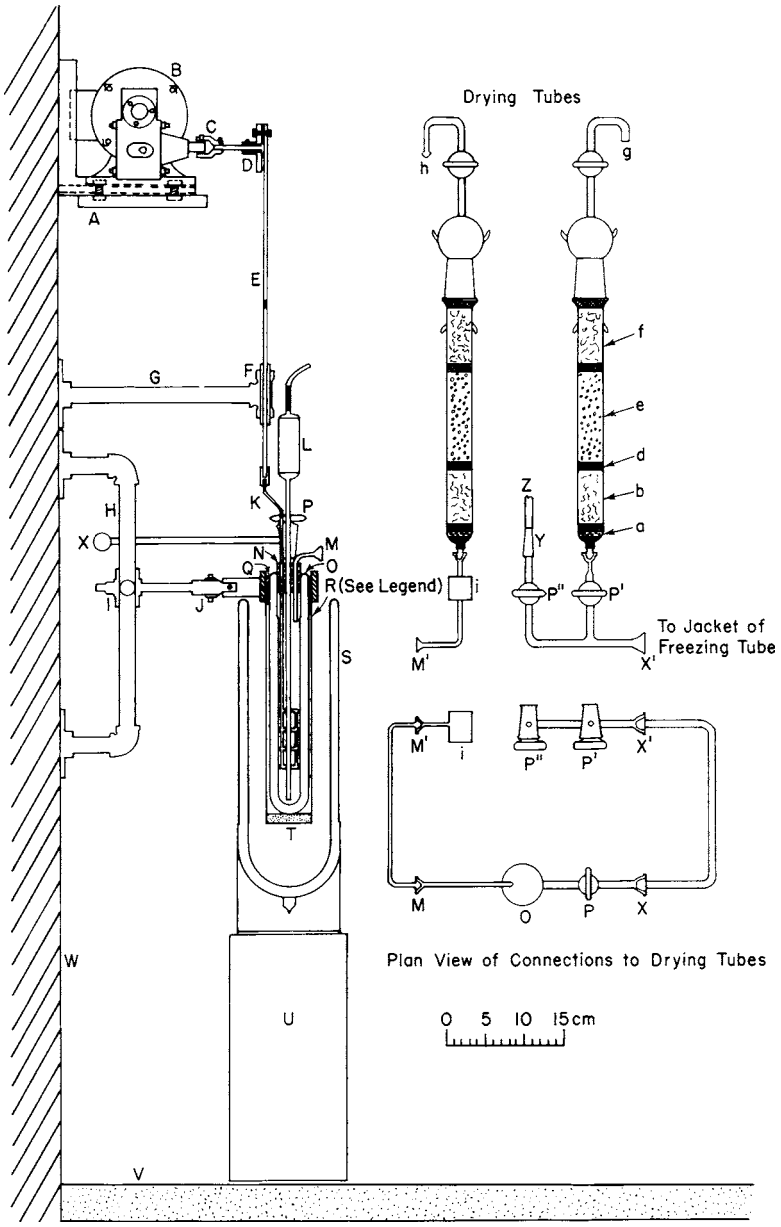
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] For details not given here, see Glasgow, A. R., Jr., Rossini, F. D., and Streiff, A. J., "Determination of the Purity of Hydrocarbons by Measurement of Freezing Points," *Journal of Research*, JNBAA, National Institute of Standards and Technology, Vol 35, No. 6, 1945, p. 355.

[4] The sole source of supply of the apparatus known to the committee at this time is Reliance Glass Works, Inc., Bensenville, IL.

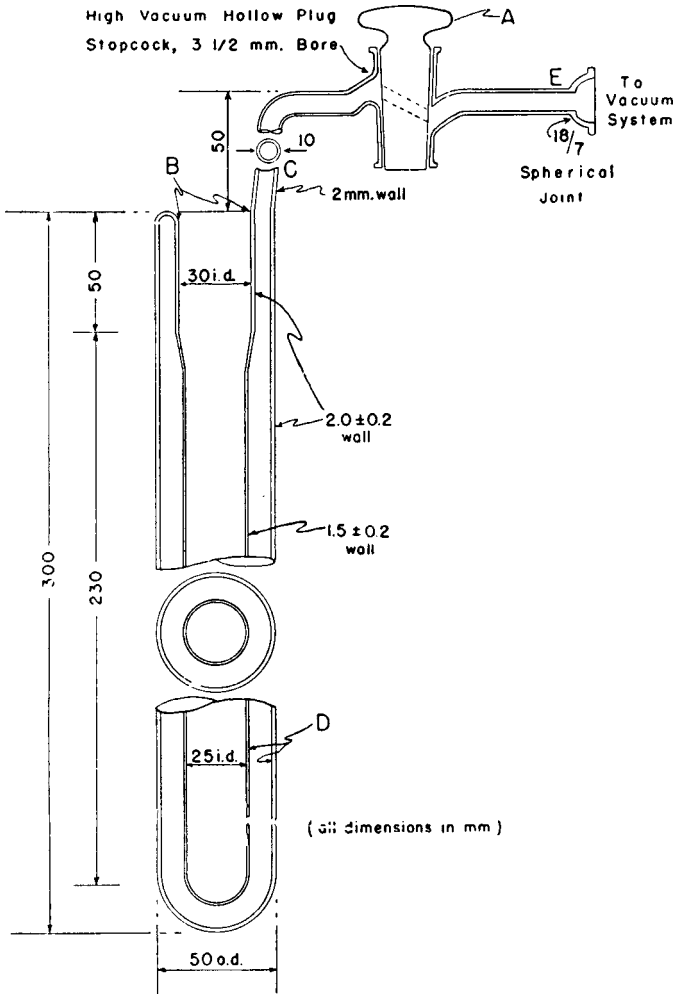
[5] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

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



A—Bracket for motor, with rubber pad.
B—Motor, with reduction gears, to give 120 r/min.
C—Coupling. (See Fig. 3).
D—Wheel. (See Fig. 3).
E—Steel rod. (See Fig. 3).
F—Bearing. (See Fig. 3).
G—Support for bearing. (See Fig. 3).
H—Support for freezing tube.
I—Adjustable clamp holder.
J—Clamp for freezing tube.
K—Stirrer. (See Fig. 3).
L—Thermometer.
M—Tube for inlet of dry air, with 12/5 spherical joint.
M′—12/5 spherical joint connection to rotameter.
N—Cork stopper, with holes as shown, plus a small hole for the "seed" wire.
O—Freezing tube, with silvered jacket. (See Fig. 2)
P—Stopcock on freezing tube.
P′—Stopcock (high vacuum) to drying tube.
P″—Stopcock (high vacuum) to vacuum line.
Q—Ceramic (or glass) fiber collar.
R—Brass cylinder, 317.5 mm (12½ in.) in length and 54 mm (2⅛ in.) in inside diameter, with bakelite collar; when liquid nitrogen is used, the metal shield must be provided with suitable openings in sides and bottom (see 5.1). If liquid air is used, the metal shield should be constructed so as to keep hydrocarbon from contact with liquid air (see 6.2).
S—Dewar flask, for cooling or warming bath; approximate inside diameter, 101 mm (4 in.); approximate inside depth, 330 mm (13 in.).
T—Ceramic (or glass) fiber pad at bottom of cylinder *R*.
U—Wood block support.
V—Table top.
W—Wall.
X,X′—Spherical joint, 18/7.
Y—Standard metal (copper or brass) to glass taper connections soldered.
Z—Connection to vacuum pump.
a—Anhydrous calcium sulfate, with indicator.
b—Anhydrous magnesium perchlorate, granular.
d—Separating layer of glass wool.
e—Ascarite.
f—Anhydrous calcium sulfate.
g—To air.
h—To source of compressed air.
i—Flow meter, for rates of 10 to 20 mL/min.

FIG. 1 Assembly of the Freezing-Point Apparatus



A—High-vacuum stopcock, hollow plug, oblique 3½-mm bore.
B—Inside opening of freezing tube, which must have no bulge at this point.
C—Slanted connection to jacket of freezing tube.
D—Internal walls of jacket of freezing tube, silvered.
E—Spherical joint, 18/7.

FIG. 2 Details of the Freezing Tube

5.2 *Resistance Bridge*,[6] Mueller type, reading from 0.0001 to 50 Ω, in steps of 0.001 Ω.

5.3 *Platinum Resistance Thermometer*,[6] precision grade, with a resistance near 25.5 Ω at 0°C, calibrated by the National Institute of Standards and Technology for the range from −190 to 500°C.

5.4 *Null Point Indicator*, may be either a galvanometer or a microvolt ammeter.

5.4.1 *Galvanometer*,[6] having a sensitivity of 0.1 mV/m at 1 m for highest precision or a sensitivity of 0.5 mV/m at 1 m for routine precision.

5.4.2 *Microvolt Ammeter*.[5,7]

5.5 *Lamp and Scale*,[6] any suitable type.

5.6 *Stopwatch or Clock*, preferably having graduations in minutes and hundredths of minutes.

5.7 *High-Vacuum Oil Pump*,[5,8] capable of evacuating the jacket of the freezing tube to a pressure of 0.133 Pa in 10 min or less.

5.8 *Seeding Apparatus*, as shown in Fig. 4, for inducing crystallization.

5.9 *Silica Gel Funnel*, as shown in Fig. 5, for filtering compounds through silica gel to remove water. To be used only when specified in Test Method D 1016.

6. Materials

6.1 *Carbon Dioxide Refrigerant*—Solid carbon dioxide in a suitable liquid. (**Warning**—Extremely cold (−78.5°C). Liberates heavy gas which can cause suffocation. Contact with skin causes burns or freezing, or both. Vapors can react violently with hot magnesium or aluminum alloys.) Acetone is recommended. (**Warning**—Extremely flammable. Harmful if inhaled. High concentrations can cause unconsciousness or death. Contact can cause skin irritation and dermatitis. Use refrigerant bath only with adequate ventilation.)

6.2 *Liquid Nitrogen or Liquid Air*—(**Warning**—Extremely cold. Liberates gas which can cause suffocation. Contact with skin causes burns or freezing, or both. Vapors can react violently with hot magnesium or aluminum alloys.) For use as a refrigerant. If obtainable, liquid nitrogen is preferable because of its safety.

6.2.1 Use liquid nitrogen refrigerant only with adequate ventilation. If liquid air is used as a refrigerant, it is imperative that any glass vessel containing hydrocarbon or other combustible compound and immersed in liquid air be protected with a suitable metal shield. The mixing of a hydrocarbon or other combustible compound with liquid air due to the breaking of a glass container would almost certainly result in a violent explosion. If liquid nitrogen is used as a refrigerant, no hydrocarbon sample should ever be permitted to cool below the condensation temperature of oxygen (−183°C at 1 atm). This would not be likely to occur in normal operation, but might occur if the apparatus were left unattended for some time.

6.3 *Silica Gel*, for use in silica gel funnel.[5,9] If the gel has been exposed to the atmosphere because of punctured or loosely sealed containers, before use, dry the gel in a shallow vessel at 150 to 205°C for 3 h, then transfer while hot to an air-tight container.

[6] Apparatus described in 5.2, 5.3, 5.4, and 5.5 was manufactured by the Leeds and Northrup Co., Philadelphia, PA, under the following catalog numbers: resistance bridge, No. 8069 B; platinum resistance thermometer, No. 8163 B; galvanometer, highest precision, No. 2284 D; galvanometer, routine precision, No. 2430 A; lamp and scale, No. 2100. The galvanometer, routine precision, No. 2430-A, and the lamp and scale, No. 2100, are still available from Leeds and Northrup. The platinum resistance thermometer, No. 8163-B, is no longer available from Leeds and Northrup, but is available with the same part number from Yellows Springs Instrument Co., Yellow Springs, OH. The resistance bridge No. 8069-B, and the galvanometer, highest precision, No. 2284-D, are no longer available; however, they may be obtainable from instrument exchanges or used equipment suppliers. If other available instrumentation is substituted for the original, the precision statement of Section 13 will not apply.

[7] The sole source of supply of the apparatus known to the committee at this time is Keithley Instruments, Inc., 28775 Aurora Rd., Cleveland, OH.

[8] The sole source of supply of the apparatus known to the committee at this time is Boekel Industries, Inc. Philadelphia, PA.

[9] The sole source of supply of the apparatus known to the committee at this time is Davison Chemical Co., Baltimore, MD.