



Designation: D 1016 – 99

An American National Standard

Standard Test Method for Purity of Hydrocarbons from Freezing Points¹

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1. Scope

1.1 This test method covers the sampling and determination of purity of essentially pure compounds for which the freezing points for zero impurity and cryoscopic constants are given.² The compounds to which the test method is applicable are: (**Warning:** Extremely flammable liquids and liquefied gases.)

<i>n</i> -butane	1,3-butadiene
isobutane	isoprene(2-methyl-1,3-butadiene)
<i>n</i> -pentane	benzene
isopentane	toluene (methylbenzene)
<i>n</i> -hexane	ethylbenzene
<i>n</i> -heptane	<i>o</i> -xylene (1,2-dimethylbenzene)
<i>n</i> -octane	<i>m</i> -xylene (1,3-dimethylbenzene)
2,2,4-trimethylpentane	<i>p</i> -xylene (1,4-dimethylbenzene)
methylcyclohexane	styrene (ethenylbenzene)
isobutene	

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 Sections 1, 6, 8, and 10-26.

NOTE 1—This test method covers systems in which the impurities form with the major component a substantially ideal or sufficiently dilute solution, and also systems which deviate from the ideal laws, provided that, in the latter case, the lowering of the freezing point as a function of the concentration is known for each most probable impurity in the given substance.

2. Referenced Documents

2.1 ASTM Standards:

D 1015 Test Method for Freezing Points of High-Purity Hydrocarbons³

¹ This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0D on Physical Methods.

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² Numerical constants in this test method were taken from the most recently published data appearing in "Tables of Physical and Thermodynamic Properties of Hydrocarbons and Related Compounds," or *ASTM DS 4A, Physical Constants of Hydrocarbons C₁ to C₁₀*, or both, prepared by the American Petroleum Institute, Research Project 44.

³ *Annual Book of ASTM Standards*, Vol 05.01.

3. Summary of Test Method

3.1 After measurement of the freezing point of the actual sample, purity can be calculated from the value of the determined freezing point and the values given for the freezing point for zero impurity and for the applicable cryoscopic constant or constants.⁴

3.2 For the equilibrium between an infinitesimal amount of the crystalline phase of the major component and a liquid phase of the major component and one or more other components, the thermodynamic relation between the temperature of equilibrium and the composition of the liquid phase is expressed by the equation:⁵

$$-\ln N_1 = -\ln(1 - N_2) = A(t_f - t_p)[1 + B(t_f - t_p) + \dots] \quad (1)$$

where:

- N_1 = mole fraction of the major component,
- N_2 = $(1 - N_1)$ = sum of the mole fractions of all the other components,
- t_f = freezing point, in degrees Celsius, of the given substance (in which the mole fraction of the major component is N_1), defined as the temperature at which an infinitesimal amount of crystals of the major component is in thermodynamic equilibrium with the liquid phase (see Note 3 of Test Method D 1015),
- t_{f0} = freezing point for zero impurity, in degrees Celsius, for the major component when pure, that is, when $N_1 = 1$ or $N_2 = 0$,
- A = first or main cryoscopic constant, in mole fraction per degree, and
- B = secondary cryoscopic constant, in mole fraction per degree.

Neglecting the higher terms not written in the brackets, Eq 1 can be transformed to the equation:

$$\log_{10} P = 2.00000 - (A / 2.3026)(t_{f0} - t_p)[1 + B(t_{f0} - t_p)] \quad (2)$$

⁴ For a more complete discussion of this test method, see Glasgow, Jr., A. R., Streiff, A. J., and Rossini, F. D., "Determination of the Purity of Hydrocarbons by Measurement of Freezing Points," *Journal of Research*, JRNBA, National Institute of Standards and Technology, Vol 35, No. 6, 1945, p. 355.

⁵ For details, see Taylor, W. J., and Rossini, F. D., "Theoretical Analysis of Time-Temperature Freezing and Melting Curves as Applied to Hydrocarbons," *Journal of Research*, JRNBA, Nat. Bureau Standards, Vol 32, No. 5, 1944, p. 197; also Lewis, G. N., and Randall, M., "Thermodynamics and the Free Energy of Chemical Substances," 1923, pp. 237, 238, McGraw-Hill Book Co., New York, NY.