



Standard Test Method for Purity by Differential Scanning Calorimetry¹

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1. Scope

1.1 This test method describes the determination of purity of materials greater than 98.5 mole percent purity using differential scanning calorimetry and the van't Hoff equation.

1.2 This test method is applicable to thermally stable compounds with well-defined melting temperatures.

1.3 Determination of purity by this test method is only applicable when the impurity dissolves in the melt and is insoluble in the crystal.

1.4 There is no ISO method equivalent to this test method.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*²

E473 Terminology Relating to Thermal Analysis and Rheology

E793 Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry

E794 Test Method for Melting And Crystallization Temperatures By Thermal Analysis

E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

E968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters

¹ This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Calorimetry and Mass Loss.

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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.

E1970 Practice for Statistical Treatment of Thermoanalytical Data

3. Terminology

3.1 *Definitions*—The definitions relating to thermal analysis appearing in Terminology E473 shall be considered applicable to this test method.

4. Summary of Test Method

4.1 This test method is based upon the van't Hoff equation:³

$$T_s = T_o - (RT_o^2 \chi) / (H F) \quad (1)$$

where:

T_s = specimen temperature, K

T_o = melting temperature of 100 % pure material, K

R = gas constant (= 8.314 J mol⁻¹ K⁻¹),

χ = mole fraction of impurity,

H = heat of fusion, J mol⁻¹, and

F = fraction melted.

4.2 This test method consists of melting the test specimen that is subjected to a temperature-controlled program while recording the heat flow into the specimen as a function of temperature. The resulting melting endotherm area is measured to yield the enthalpy of fusion, H . The melting endotherm area is then partitioned into a series of fractional areas (about ten, comprising the first 10 to 50 % of the total area). The fractional area, divided by the total area, yields the fraction melted, F . Each fractional area is assigned a temperature, T_s .

4.3 Eq 1 has the form of $Y = mX + b$ where $Y = T_s$, $X = 1/F$, $m = -(R T_o^2 \chi) / H$, and $b = T_o$. A plot of Y versus X should produce a straight line with slope m and intercept b .

4.4 In practice, however, the resultant plot of T_s versus $1/F$ is seldom a straight line. To linearize the plot, an incremental amount of area is added to the total area and to each fractional area to produce a revised value for F . The process of incremental addition of area is continued until a straight line is obtained.

$$F = (A_{part} + c) / (A_{total} + c) \quad (2)$$

³ Brennan, W. P., DiVito, M. P., Fynas, R. L., Gray, A. P., "An Overview of the Calorimetric Purity Measurement", in *Purity Determinations by Thermal Methods*, R. L. Blaine and C. K. Schoff (Eds.), Special Technical Publication 838, American Society for Testing and Materials, West Conshohocken, PA, 1984, pp. 5–15.