



Standard Test Method for Determination of Purity by Differential Scanning Calorimetry¹

This standard is issued under the fixed designation E 928; 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 the determination of purity by use of differential scanning calorimetry and the evaluation of the results using the van't Hoff equation.

1.2 This test method is generally 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 Computer- or electronic-based instruments, techniques, or data treatments equivalent to this test method may also be used. Users of this test method are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this test method to determine the necessary equivalency prior to use.

1.5 The values stated in SI units are to be regarded as the 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:

E 473 Terminology for Thermal Analysis²

E 793 Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry²

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers²

E 968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters²

3. Terminology

3.1 **Definitions**—The definitions relating to thermal analysis appearing in Terminology E 473 shall be considered applicable to this test method.

4. Summary of Test Method

4.1 This test method consists of melting the test specimen that is subjected to a temperature-controlled program while recording the heat flow (power) into the specimen as a function of time. The resulting endotherm area is often measured to yield the enthalpy of fusion. 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). Each fractional area is assigned a temperature. A plot of the reciprocal fractional areas and their corresponding temperatures is then produced. The resulting plot is seldom a straight line. To linearize the plot, an incremental amount is added to the enthalpy of fusion and the fractional areas recalculated and plotted. The purity level is then calculated from the slope of this straight line.

5. Significance and Use

5.1 The melting temperature range of a compound broadens as the impurity level rises. This phenomenon is described approximately by the van't Hoff equation for melting point depressions. Measuring and recording the instantaneous heat flow (power) into the specimen as a function of time or temperature during such a melting process is a practical way for the generation of data suitable for analysis by the van't Hoff equation.

5.2 The results obtained include: sample purity (expressed as mol percent); enthalpy of fusion (expressed as joules per mol); and the melting temperature (expressed in Kelvin) of the pure form of the major component.

5.3 Generally, the repeatability of this test method decreases as the purity level decreases. This test method is ordinarily considered unreliable when the purity level of the major component of the mixture is less than 98.5 mol % or when the incremental enthalpy correction (x) exceeds 20 % of the original detected enthalpy of fusion.

5.4 This test method is used for quality control, specification acceptance, and research.

6. Interferences

6.1 This test method is nonspecific. Many impurities may cause the melting temperature broadening. Thus, it is not useful in identifying the nature of the impurity or impurities but only the total mol percent of impurity present.

6.2 The van't Hoff theory assumes the following:

¹ This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods and Recommended Practices.

Current edition approved Dec. 10, 1996. Published April 1997. Originally published as E 928 – 83. Last previous edition E 928 – 85 (1989) ϵ^1 .

² Annual Book of ASTM Standards, Vol 14.02.