



Standard Method for Enthalpy Measurement Validation of Differential Scanning Calorimeters¹

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1. Scope

1.1 This standard provides procedures for validating enthalpic measurements of differential scanning calorimeters (DSC) and analytical methods based upon the measurement of enthalpy or heat by DSC. Performance parameters determined include calorimetric repeatability (precision), detection limit, quantitation limit, linearity and bias. This method is applicable to both exothermic and endothermic events.

1.2 Validation of apparatus performance and analytical methods is requested or required for quality initiatives or where results may be used for legal purposes.

1.3 SI units are the standard.

1.4 *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 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods²

E 473 Terminology Relating to Thermal Analysis²

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

E 1142 Terminology Relating to Thermophysical Properties²

E 1860 Test Method for Elapsed Time Calibration of Thermal Analyzers²

E 1970 Practice for Statistical Treatment of Thermoanalytical Data²

E 2161 Terminology Relating to Performance Validation in Thermal Analysis²

2.2 Other Standard:

United States Food and Drug Administration, Q2B Validation of Analytical Procedures: Methodology, 62 FR 27464, May 19, 1997³

3. Terminology

3.1 Technical terms used in this standard are defined in Practice E 177 and in Terminologies E 473, E 1142, and E 2161.

4. Summary of Test Method

4.1 Temperature and time are the primary independent parameters and heat flow is the primary dependent experimental parameter provided by DSC. Integration of heat flow, as a function of time, yields enthalpy (heat).

4.1.1 Time, measured by the DSC apparatus, shall conform to better than 0.1 % verified by Test Method E 1860 and reported.

4.1.2 Heat flow, a measured value, is validated by its integration over time to obtain the desired calorimetric (enthalpic) information of interest. Determination and verification of enthalpy is the primary scope of this test method.

4.2 Calorimetric validation of a differential scanning calorimetric apparatus at a single temperature is performed using the indium metal melt as an analyte.

4.3 Validation of a DSC method based upon enthalpic measurement may be performed using the test specimen as the analyte.

4.4 The enthalpy of three (or more) specimens (nominally representing the maximum, midpoint and minimum of the range of the test method) are measured in triplicate (or more). A fourth blank specimen, containing no analyte, is also measured in triplicate.

NOTE 1—Repeatability is determined by performing a sufficient number of determinations to calculate statistically valid estimates of the standard deviation or relative standard deviation of the measurement.

4.4.1 Calorimetric linearity and bias are determined from the best-fit straight-line correlation of the results from measurements of the three (or more) specimens.

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 Thermal Analysis Methods.

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² Annual Book of ASTM Standards, Vol. 14.02.

³ Available from FDA, 5600 Fishers Lane, Rockville, MD 20857.