



Standard Test Method for Temperature and Enthalpy Measurement Validation of Differential Scanning Calorimeters¹

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1. Scope*

1.1 This test method provides procedures for validating temperature and enthalpy measurements of differential scanning calorimeters (DSC) and analytical methods based upon the measurement of temperature or enthalpy (or heat), or both, by DSC. Performance parameters determined include temperature and calorimetric repeatability (precision), detection limit, quantitation limit, linearity, and bias. This test method is applicable to exothermic 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:²

[E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

[E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers](#)

[E968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters](#)

[E1142 Terminology Relating to Thermophysical Properties](#)

¹ This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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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.

[E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers](#)

[E1970 Practice for Statistical Treatment of Thermoanalytical Data](#)

[E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology](#)

2.2 *FDA Publications:*³

[Q2B Validation of Analytical Procedures Methodology](#), 62 FR 27464, May 19, 1997

3. Terminology

3.1 Technical terms used in this standard are defined in Practice [E177](#) and in Terminologies [E473](#), [E1142](#), and [E2161](#) including *analyte*, *detection limit*, *differential scanning calorimetry*, *enthalpy*, *extrapolated onset value (temperature)*, *linearity*, *mean*, *precision*, *quantitation limit*, *relative standard deviation*, *repeatability*, *standard deviation*, *thermal curve*, and *validation*.

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 [E1860](#) and reported.

4.1.2 Temperature is directly measured by a temperature sensor that is an integral part of the differential scanning calorimetry apparatus.

4.1.3 Heat flow, a measured value, is validated by its integration over time to obtain the desired calorimetric (enthalpic) information of interest.

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.

³ Available from Food and Drug Administration (FDA), 10903 New Hampshire Ave., Silver Spring, MD 20993-0002, <http://www.fda.gov>.

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