



Designation: E793 – 24

Standard Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry¹

This standard is issued under the fixed designation E793; 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 describes the determination of the enthalpy (heat) of fusion (melting) and crystallization by differential scanning calorimetry (DSC).

1.2 This test method is applicable to solid samples in granular form or in any fabricated shape from which an appropriate specimen can be cut, or to liquid samples that crystallize within the range of the instrument. Note, however, that the results may be affected by the form and mass of the specimen, as well as by other experimental conditions.

1.3 The normal operating temperature range is from -120°C to 600°C . The temperature range can be extended depending upon the instrumentation used.

1.4 This test method is applicable to thermally stable materials with well-defined endothermic or exothermic behavior.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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. Referenced Documents

2.1 ASTM Standards:²

E473 Terminology Relating to Thermal Analysis and Rheology

E794 Test Method for Melting And Crystallization Temperatures By Thermal Analysis

E968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters (Withdrawn 2023)³

E1142 Terminology Relating to Thermophysical Properties

E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers

E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 *Definitions*—Specialized terms used in this test method are defined in Terminologies E473 and E1142 including *calorimeter*, *Celsius*, *crystallization*, *derivative*, *differential scanning calorimetry*, *endotherm*, *enthalpy*, *extrapolated onset*, *Kelvin*, *peak*, *temperature*, and *thermal curve*.

4. Summary of Test Method

4.1 This test method involves heating (or cooling) a test specimen at a controlled rate in a controlled environment through the temperature region of fusion or crystallization. The heat flow associated with fusion, an endothermic process (and crystallization, an exothermic process), is recorded and integrated over time. Absolute values for the enthalpy of fusion (and enthalpy of crystallization) or relative values for comparative purposes can thus be obtained.

NOTE 1—Melting (or crystallization) temperatures are sometimes determined in conjunction with measurements of the enthalpy of fusion or

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The last approved version of this historical standard is referenced on www.astm.org.

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

crystallization. These temperature values may be obtained by Test Method [E794](#).

5. Significance and Use

5.1 Differential scanning calorimetry provides a rapid method for the determination of enthalpic changes accompanying first-order transitions of materials.

5.2 This test method is useful for quality control, specification acceptance, and research.

6. Apparatus

6.1 *Differential Scanning Calorimeter (DSC)*, the essential instrumentation required to provide the minimum differential scanning calorimetric capability for this method includes:

6.1.1 Test Chamber composed of:

6.1.1.1 *A Furnace(s)*, to provide uniform controlled heating and cooling of a specimen and reference to a constant temperature or at a constant rate from -120°C to 600°C .

6.1.1.2 *A Temperature Sensor*, to provide an indication of the specimen temperature to $\pm 0.1^{\circ}\text{C}$.

6.1.1.3 *Differential Sensors*, to detect a heat flow difference between specimen and reference with a range of at least $\pm 100\text{ mW}$ and a sensitivity of $\pm 5\text{ mW}$.

6.1.1.4 A means of sustaining a test chamber environment of an inert purge gas as at rate of 10 mL/min to $50\text{ mL/min} \pm 5\text{ mL/min}$.

NOTE 2—Typically, 99.99+ % pure nitrogen, argon or helium is used when oxidation in air is a concern. Unless effects of moisture are to be studied, use of dry purge gas is recommended and is essential for operation at subambient temperatures.

6.1.2 *A Temperature Controller*, capable of executing a specific temperature program by operating the furnaces(s) between selected temperature limits at a rate of temperature change of up to at least 20°C/min constant to $\pm 0.1^{\circ}\text{C/min}$. or at an isothermal temperature constant to $\pm 0.1^{\circ}\text{C}$.

6.1.3 *A Recording Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required are heat flow, temperature, and time.

6.2 *Specimen Containers*, (pans, crucibles, vials, lids, closures, seals, etc.) that are inert to the specimen and reference materials and that are of suitable structural shape and integrity to contain the specimen and reference.

6.3 *Nitrogen*, or other inert gas supply for purging purposes.

6.4 *Balance*, with capacity greater than 100 mg , capable of weighing to the nearest 0.01 mg , or better.

NOTE 3—Balances readable to 0.01 mg are suitable for use with test specimens on the order of 10 mg in mass. A balance readable to 0.001 mg is required for test specimens on the order of 1 mg in mass. to achieve the precision described in this standard.

6.5 Auxiliary instrumentation considered useful or necessary for conducting this method includes:

6.5.1 *Data Analysis* capability of integrating the heat flow signal as a function of time to produce enthalpy information in units of mJ to a precision of $\pm 1\%$.

6.5.2 A means, tool or device to close, encapsulate, or seal the container of choice.

6.5.3 A cooling capability to hasten cool down from elevated temperatures, to provide constant cooling rates, or to sustain an isothermal subambient temperature.

7. Hazards and Interferences

7.1 Since milligram quantities of specimens are used, it is essential that samples are homogeneous.

7.2 Toxic or corrosive effluents, or both, may be released when heating the material and could be harmful to the personnel or the apparatus.

7.3 Samples that release volatiles upon heating will change mass and invalidate the test.

7.4 In the use of commercial instrumentation, the operator should read the manufacturer's operations manual to be aware of potential hazards of operation, such as burn hazards from hot surfaces.

8. Sampling

8.1 Powdered or granular materials should be mixed thoroughly prior to sampling and should be sampled by removing portions from various parts of the container. These portions, in turn, should be combined and mixed well to ensure a representative specimen for the determination. Liquid samples may be sampled directly after mixing.

8.2 In the absence of other information, samples are assumed to be analyzed as received. If some heat or mechanical treatment is applied to the sample prior to analysis, this treatment, and any mass loss resulting from this treatment, should be noted in the report.

9. Calibration

9.1 Using the same heating rate, purge gas, and flow rate to be used for specimens, calibrate the heat flow axis of the instrument, using the procedure in Practice [E968](#).

NOTE 4—Committee E37 recommends calibration of all signals at least annually.

10. Procedure

10.1 Weigh 1 mg to 15 mg of specimen to an accuracy of $\pm 0.1\%$ into a clean, dry specimen capsule.

NOTE 5—The specimen mass to be used depends on the magnitude of the transition enthalpy and the volume of the capsule. For comparing multiple results, use similar mass ($\pm 5\%$) and encapsulation. Weighing to less accuracy than one part per thousand may limit the accuracy of the enthalpy determination.

10.2 Seal or crimp the specimen capsule with a lid under ambient conditions. Minimize the free space between the specimen and the lid. For specimens sensitive to oxidation, hermetic sealing under an inert atmosphere may be desirable.

10.3 Load the specimen into the instrument chamber. Purge the chamber with dry nitrogen (or other inert gas) at a flow rate of 10 mL/min to 50 mL/min throughout the experiment.

10.4 The specimen may be heated rapidly to 50°C below the expected melting temperature and allowed to equilibrate.

NOTE 6—For some materials, it may be necessary to start the scan substantially lower in temperature, for example below the glass transition,