



Designation: E2160 – 23

Standard Test Method for Heat of Reaction of Thermally Reactive Materials by Differential Scanning Calorimetry¹

This standard is issued under the fixed designation E2160; 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 determines the exothermic heat of reaction of thermally reactive chemicals or chemical mixtures, using milligram specimen sizes, by differential scanning calorimetry. Such reactive materials may include thermally unstable or thermoset materials.

1.2 This test method also determines the extrapolated onset temperature and peak heat flow temperature for the exothermic reaction.

1.3 This test method may be performed on solids, liquids or slurries.

1.4 The applicable temperature range of this test method is 25 °C to 600 °C.

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 is related to Test Method E537, but provides additional information.

1.7 *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.*

1.8 *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

E537 Test Method for Thermal Stability of Chemicals by Differential Scanning Calorimetry

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

E968 Practice for Heat Flow Calibration of Differential Scanning Calorimeters (Withdrawn 2023)³

E1142 Terminology Relating to Thermophysical Properties

E1231 Practice for Calculation of Hazard Potential Figures of Merit for Thermally Unstable Materials

E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers

E3142 Test Method for Thermal Lag of Thermal Analysis Apparatus

3. Terminology

3.1 Specific technical terms used in this standard are defined in Terminologies E473 and E1142.

4. Summary of Test Method

4.1 A small (milligram) quantity of the reactive material is heated at 10 °C/min through a temperature region where a chemical reaction takes place. The exothermic heat flow produced by the reaction is recorded as a function of temperature and time by a differential scanning calorimeter. Integration of the exothermic heat flow over time yields the heat of reaction. If the heat flow is endothermic, then this test method is not to be used.

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.

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

4.2 The test method can be used to determine the fraction of a reaction that has occurred in a partially reacted sample. The heat of reaction is determined for a specimen that is known to be 100 % unreacted and is compared to the heat of reaction determined for the partially reacted sample. Appropriate calculation yields the fraction of the latter sample that was unreacted.

4.3 Subtracting the reaction fraction remaining from unity (1) yields the fraction reacted. The fraction reacted may be expressed as percent. If the sample tested is a thermoset resin, the percent reacted is often called the percent of cure.

4.4 The extrapolated onset temperature and peak heat flow temperature are determined for the exothermic heat flow thermal curve from 4.1.

5. Significance and Use

5.1 This test method is useful in determining the extrapolated onset temperature, the peak heat flow temperature and the heat of reaction of a material. Any onset temperature determined by this test method is not valid for use as the sole information used for determination of storage or processing conditions.

5.2 This test method is useful in determining the fraction of a reaction that has been completed in a sample prior to testing. This fraction of reaction that has been completed can be a measure of the degree of cure of a thermally reactive polymer or can be a measure of decomposition of a thermally reactive material upon aging.

5.3 The heat of reaction values may be used in Practice E1231 to determine hazard potential figures-of-merit Explosion Potential and Shock Sensitivity.

5.4 This test method may be used in research, process control, quality assurance, and specification acceptance.

6. Apparatus

6.1 *Differential Scanning Calorimeter* (DSC), capable of measuring and recording heat flow as a function of temperature and time. Such a DSC is composed of:

6.1.1 *Test Chamber*, composed of:

6.1.1.1 *Furnace(s)*, to provide uniform controlled heating of a specimen and reference to a constant temperature or at a constant rate within the temperature range of 25 °C to 600 °C.

6.1.1.2 *Temperature Sensor*, to provide an indication of the specimen or furnace temperature readable to within ± 0.5 °C.

6.1.1.3 *Differential Sensor*, to detect a heat flow difference between the specimen and reference equivalent to 0.2 mW.

6.1.1.4 *Means of Sustaining a Test Chamber Environment*, of inert (for example, nitrogen, helium or argon) or reactive (for example, air) gas at a purge rate of 50 mL/min \pm 5 mL/min.

NOTE 1—Typically, at least 99 % pure nitrogen, helium or argon is employed when oxidation in air is a concern. Unless effects of moisture are to be studied, use of dry purge gas is recommended.

6.1.1.5 *Temperature Controller*, capable of executing a specific temperature program by operating the furnace(s) between

selected temperature limits (ambient temperature to 600 °C) at a heating rate between 2 °C/min and 20 °C/min constant to within ± 0.1 °C/min.

6.1.1.6 *A Data Collection 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 *Containers*, (pans, crucibles, vials, etc. and lids) that are inert to the specimen and reference materials and that are of suitable structural shape and integrity to contain the specimen and reference in accordance with the specific requirements of this test method.

6.3 *Balance*, with a capacity of 100 mg or greater to weigh specimens and containers, or both, readable to ± 1 μ g.

7. Safety Precautions

7.1 The use of this test method for materials of unknown potential hazards requires that precautions be taken during the sample preparation and testing.

7.2 Where particle size reduction by grinding is necessary, the user of this test method shall presume that the material is hazardous.

7.3 Toxic or corrosive effluents, or both, may be released when heating the test specimen and could be harmful to personnel or the apparatus. Use of an exhaust system to remove such effluents is recommended.

8. Calibration

8.1 Perform any calibration procedures recommended by the apparatus manufacturer as described in the Operations Manual.

8.2 Calibrate the temperature signal to within ± 2 °C using Test Method E967.

8.3 Calibrate the heat flow signal to within ± 0.5 % using Practice E968.

8.4 Calibrate the elapsed time signal, or ascertain its accuracy, to within ± 0.5 % using Test Method E1860.

NOTE 2—Calibration or calibration verification of all signals is recommended at least annually.

9. Procedure

9.1 Into a tared sample container, weigh to within ± 1 μ g, 1 mg to 2 mg of the test specimen. Record this mass as *M* in mg. Close the sample. Weigh the sealed container to within ± 1 μ g and recorded this mass as *N* in mg.

NOTE 3—Because of the reactive nature of the materials examined by this test method, small specimen sizes shall be used unless the approximate reactivity of the test specimen is known. Other specimen sizes may be used but shall be reported. Make sure that the specimen is homogenous and represents the sample.

NOTE 4—Some substances may have non-reactive components mixed with the thermally reactive material. An example would be reinforcing fibers mixed with a thermally-curing polymer. A specification of the fraction of inert material in the mixture may accompany these materials. The user should be aware that such specifications involve tolerances so that the actual fraction of inert material may vary within these tolerances from lot to lot. In such cases, the actual fraction of inert material must be taken into account.

NOTE 5—For highly reactive materials, the selection of sample containers can be particularly important. The material from which the container is constructed may catalyze the reaction or react with the sample material. Sealed containers may cause an autocatalytic effect or possibly a pressure effect. In open containers loss of material, and thereby loss of heat, could be an issue. Excessive pressurization of a sample container can be avoided by using vented containers; however, vented or unsealed containers may cause the measured heat of reaction to be much smaller than the true value. See 12.4 for an example of such an effect.

9.2 Heat the test specimen at a controlled rate of 10 °C/min ± 0.1 °C/min from ambient until the thermal curve returns to baseline following the exothermic event. If the upper limit of temperature for this test method, 600 °C, is reached before the thermal curve returns to baseline, then this test method is not applicable.

NOTE 6—Other heating rates may be used but shall be reported (see Appendix X1).

9.3 Cool the test specimen to ambient temperature upon completion of the experiment.

9.4 Reweigh the sample container. Compare this mass of the sealed sample container weight with *N* determined in 9.1. Report any specimen weight loss observed.

9.5 Construct a line connecting the baseline before the exothermic reaction to that after the reaction (see Fig. 1).

NOTE 7—For highly energetic reactions, a significant change may occur in the baseline prior to and following the exothermic reaction, due to a significant change in the heat capacity of the reacted material in the sample container. Such an instance might be handled by the construction of a baseline that is not a straight line. If a nonlinear baseline (for example, a sigmoidal baseline) is used it should be stated in the report and an example of the constructed baseline and the thermal curve should be included also.

9.6 Integrate the area, as a function of time, bounded by the thermal curve and the baseline constructed in 9.5. Record this area as the heat of the reaction (*A*) in mJ.

NOTE 8—The area bounded by the thermal curve and the constructed

baseline gives the heat of the reaction. Instrument software is most often used to integrate this area. Although such software packages display thermal curves as in Fig. 1, they calculate the bound area on a basis of time. If older instruments without these software packages are used, or if manual checks are performed on newer instruments, then the manual integration must be performed with the abscissa presented as a time (seconds) coordinate.

NOTE 9—The amount of material should be chosen such that the maximum heat flow is less than 8 mW. This requirement reduces the potential of obtaining adiabatic heating of the sample. Adiabatic heating of the sample results in “leaning” peaks, an example of which is shown in Fig. 2 (adapted from Figure 11 of Jones (1996)).⁴ For highly energetic materials, it might be impossible to satisfy simultaneously the direction of 9.1 (using 1 mg to 2 mg of the test specimen) and the condition of this note (maximum heat flow less than 8 mW). If heat flow is larger than 8 mW and the peak is not “leaning”, it should not be necessary to reduce sample mass. Or, in other words, when both directions cannot be met simultaneously, sample mass need be reduced only if the observe peak leans.

9.7 Construct a tangent to the leading edge of the exothermic peak at the point of maximum rate of change and extrapolate that tangent to the baseline constructed in 9.5. Record the intersection of the tangent with the baseline as the onset temperature (*T_o*).

NOTE 10—In some cases, reactions may have induction periods or other effects that are manifested as exothermic deviations from the established baseline well before the onset temperature obtained by 9.7. Because of the importance of these effects for highly reactive materials, an additional onset temperature, the temperature of first deviation (*T_f*), is to be reported also. The temperature of first deviation is the temperature for which the thermal curve first deviates from the established baseline. The temperature of first deviation is to be noted in the report.

NOTE 11—Peak temperatures from two different determinations are comparable only if the same conditions were used for both measurements, for example, sample mass and vent diameter.

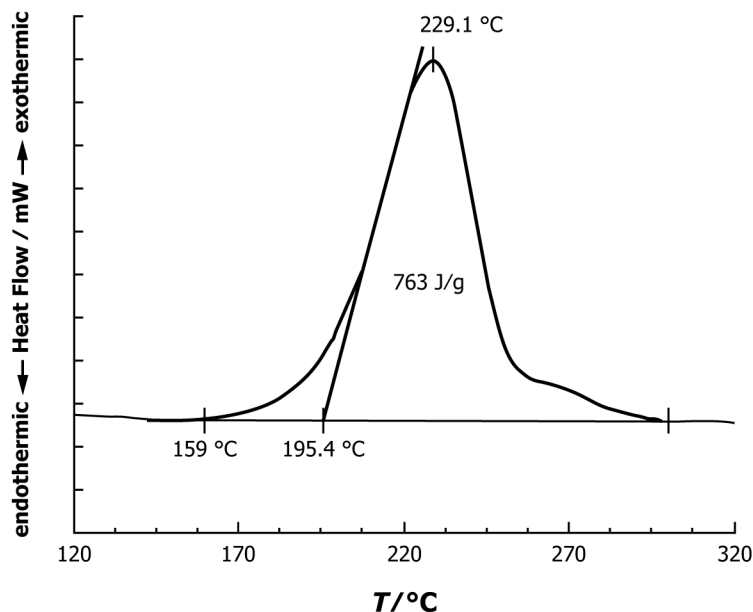


FIG. 1 Thermal Curve, Determination of Reported Values

⁴ Jones, D.E.G., and Augsten R.A., “Evaluation of Systems for Use in DSC Measurements on Energetic Materials,” *Thermochemica Acta*, Vol 286, 1996, pp. 355–373.