Designation: E 2160 – 01

Standard Test Method for Heat of Reaction of Thermally Reactive Materials by Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E 2160; 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 method is 25 to 600°C.

1.5 SI units are to be regarded as the standard.

1.6 There is no ISO method equivalent to this standard.

1.7 This standard is related to Test Method E 537 and to NAS 1613, but provides additional information.

1.8 *This standard may involve hazardous materials, operations, and equipment. 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 Relating to Thermal Analysis[2]

E 537 Test Method for Assessing the Thermal Stability of Chemicals by Methods of Thermal Analysis[2]

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[2]

E 968 Practice of Heat Flow Calibration of Differential Scanning Calorimeters[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

E 1231 Practice for Hazard Potential Figures-of-Merit for Thermally Unstable Materials[2]

E 1860 Test Method for Elapsed Time Calibration of Thermal Analyzers[2]

2.2 *Other Standard:*

NAS 1613 Seal Element, Packing, Preformed, Ethylene Propylene Rubber, National Aerospace Standard, Aerospace Industries Association of America, 1725 DeSales St., NM, Washington, DC 20036

3. Terminology

3.1 Specific technical terms used in this standard are defined in Terminologies E 473 and E 1142.

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.

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 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

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[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 Test Methods.

Current edition approved Sept. 10, 2001. Published December 2001.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

this 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 E 1231 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 to 600°C.

6.1.1.2 *Temperature Sensor*, to provide an indication of the specimen or furnace temperature 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 ± 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 and 20°C/min constant to within ± 0.1°C/min.

6.1.1.6 *Recording Device*, capable of recording and displaying any portion (including signal noise) of the differential heat flow on the ordinate as a function of temperature or time on the abscissa.

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 method.

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

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 Practice E 967.

8.3 Calibrate the heat flow signal to within ± 0.5 % using Practice E 968.

8.4 Calibrate the elapsed time signal, or ascertain its accuracy, to within ± 0.5 % using Test Method E 1860.

9. Procedure

9.1 Into a tared sample container, weigh to within ± 1µg, 1 to 2 mg of the test specimen. Record this mass as *M* in mg. Close the sample container but provide a vent to ensure that the internal pressure is in equilibrium with the ambient pressure. Weigh the sealed container to within ± 1 µg and recorded this mass as *N* in mg.

NOTE 2—Because of the reactive nature of the materials examined by this 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 3—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 4—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.

9.2 Heat the test specimen at a controlled rate of 10 ± 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 method, 600°C, is reached before the thermal curve returns to baseline, then this method is not applicable.

NOTE 5—Other heating rates may be used but shall be reported.

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 6—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 (e.g. a sigmoidal baseline) is used it should be stated in the report and an example