Designation: E487 – 04

Standard Test Method for Constant-Temperature Stability Of Chemical Materials[1]

This standard is issued under the fixed designation E487; 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 assessment of constant-temperature stability of chemical materials that undergo exothermic reactions. The techniques and apparatus described may be used on solids, liquids, or slurries of chemical substances.

1.2 When a series of materials is tested by this method, the results permit ordering the materials relative to each other with respect to their thermal stability.

1.3 *Limitations of Test*:

1.3.1 This test method is limited to ambient temperatures and above.

1.3.2 This test method determines neither a safe storage temperature nor a safe processing temperature.

NOTE 1—A safe storage or processing temperature requires that any heat produced by a reaction be removed as fast as generated and that proper consideration be given to hazards associated with reaction products.

1.3.3 When this test method is used to order the relative thermal stability of materials, the tests must be run under the same confinement condition (see 8.3).

1.4 SI units are the standard.

1.5 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

E537 Test Method for The 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

E1445 Terminology Relating to Hazard Potential of Chemicals

E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers

3. Terminology

3.1 *Definitions:*

3.2 *constant-temperature stability (CTS) value*—the maximum temperature at which a chemical compound or mixture may be held for a 2–h period under the conditions imposed in this test without exhibiting a measurable exothermic reaction.

3.3 The specialized terms in this standard are described in Terminologies E473 and E1445E1445.

4. Summary of Test Method

4.1 A sample of the chemical compound or mixture is placed in a glass or metal tube that is heated to a test temperature of interest. The sample temperature and heat flow or the difference between the sample temperature and the temperature of an inert reference material, are monitored over a 2-h period or until an exothermic reaction is recorded. Test temperatures are decreased in 10 °C intervals until no exothermic reaction is observed in the 2-h test period. The Constant Temperature Stability is determined and reported using either Method A or Method B.

NOTE 2—Test periods other than two 2 h may be used but shall be reported

[1] This test method is under the jurisdiction of ASTM Committee E27on Hazard Potential of Chemicals and is the direct responsibility of E27.02 on Thermal Stability and Condensed Phases.

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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.

NOTE 3—The processing times in many industrial scale unit operations (for example, drying, distillations, and the like) normally significantly exceed the 2 h time period in this CTS test procedure. Therefore, for the effective application of the CTS data for industrial scale operations, the CTS time must be extended to be greater than the processing time in the actual operation.

5. Significance and Use

5.1 This test method is a useful adjunct to dynamic thermal tests that are performed under conditions in which the sample temperature is increased continuously at a programmed rate. Results obtained under dynamic test conditions present difficulties in determining the temperature at which an exotherm initiates because onset temperature is dependent on heating rate. The test method described in the present standard attempts to determine the onset temperature under isothermal conditions where the heating rate is zero.

6. Apparatus

6.1 The design and complexity of the apparatus required for this method depends upon the size of the sample to be used. In general, observance of an exothermic reaction in small samples (less than 50 mg) is best done using differential thermal analysis or differential scanning calorimetry equipment and techniques. Larger samples (up to 2 g) may be tested using a Kuhner Micro CTS apparatus.

6.2 The following items are required to obtain the appropriate experimental data:

6.2.1 A test chamber composed of:

6.2.1.1 *Furnace(s)*, to provide uniform controlled heating of a specimen and reference to a constant temperature.

6.2.1.2 *Temperature Sensor*, to provide an indication of the specimen/furnace temperature to ± 0.1 °C.

6.2.1.3 *Differential Sensor*, to detect a difference in heat flow or temperature between specimen and reference specimen equivalent to 1 mW or 40 mK.

NOTE 4—Sample temperature may be measured either absolutely or differentially. When differential temperature measurements are made, and a reference material is used, the reference material should match the physical state and heat capacity of the sample as closely as practical. Typical reference materials are calcined aluminum oxide, glass beads, silicone oils, and a combination of these.

NOTE 5—Commercially available differential thermal analysis or differential scanning calorimetry apparatus capable of operating in an isothermal mode may be used. Alternatively, the apparatus may be assembled or fabricated from commercially available components (see 12.1).

6.2.2 A temperature *Controller* capable of heating from ambient to 400 °C at a rate of up to 50 °C/min and maintaining an isothermal temperature constant within that range to ± 1 °C for 120 min.

6.2.3 A *Recording Device*, to record and display differential heat flow or differential temperature, test specimen temperature and time to the sensitivities described above.

6.2.4 *Containers* (pans, crucibles, vials, test tubes, etc.) which are inert to the specimen and reference material and which are of suitable structure, shape, and integrity to contain the specimen and reference in accordance with the temperature and specimen mass requirements described in this section.

6.3 A *Balance* with a capacity of 100 mg or more to weigh specimens and/or containers (pans, crucibles, vials, and the like) to ± 0.1 mg (see Note 6).

7. Hazards

7.1 Dynamic thermal tests are normally carried out on small samples before the present test is undertaken. Therefore, the experimenter should have some knowledge of the magnitude of hazard associated with the material. Larger samples should be used only after due consideration is given to the potential for hazardous reaction. Thermodynamic calculations also can be used to determine the potential hazard.

7.2 Special precautions should be taken to protect personnel and equipment when the apparatus in use requires the insertion of samples into a heated block or furnace. These should include adequate shielding and ventilation of equipment, and face and hand protection.

8. Sampling

8.1 Specimens should be representative of the material being studied and should be prepared to achieve good thermal contact between the sample and container.

8.2 Specimen size depends upon the sensitivity of the available apparatus (see 12.1).

NOTE 6—Specimen size of 4–7 mg is typically used in thermal analysis apparatus. The Kuhner Micro CTS uses up to 2 g of sample. For test specimen size greater than 1 g, record mass to ± 0.1 g.

8.3 Specimens may be run in an unconfined or in a sealed specimen container, depending upon which condition has the more relevance for the end use of the data.

8.4 In selecting the material of construction of the specimen container, consideration should be given to possible interaction with the specimen.

9. Calibration

9.1 Apparatus temperature calibration shall be performed according to Practice E967 at a heating rate of 1 °C/min.

9.2 Apparatus heat flow calibration shall be performed according to PracticeE968 E968 for differential scanning calorimeters. Differential thermal and Kuhner Micro CTS apparatus shall be calibrated according to the manufacturers' instructions.

9.3 Apparatus elapsed time shall be calibrated according to Test Method E1860.

10. Procedure

10.1 Bring the sample holder of the apparatus to a temperature 10°C below that approximated as the onset temperature in a previous differential thermal analysis measurement. Maintain control at the set temperature at no more than ±1 °C.

NOTE 7—The onset temperature may be determined using Practice E537

10.2 Place the samples and containers in the heated sample holder at the control temperature. Note the starting time as the time of sample insertion and begin a temperature record versus time immediately.

NOTE 8—If the test apparatus allows the sample to be brought to the test