Designation: ~~E487 – 14~~ E487 – 20

Standard Test ~~Method~~Methods 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~~These test ~~method describes~~methods describe the assessment of constant-temperature stability (CTS) 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~~these test ~~method,~~methods, 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~~These test ~~method is~~methods are limited to ambient temperatures and above.

1.3.2 ~~This~~These test ~~method determines~~methods determine 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~~these test ~~method is~~methods are used to order the relative thermal stability of materials, the tests must be run under the same confinement condition (see 8.3).

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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~~ concerns, if any, associated with its use. It is the responsibility of ~~whoever uses~~ the user of this standard to ~~consult and~~ establish appropriate ~~safety~~ safety, health, and ~~health~~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.*

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

[1] ~~This~~These test ~~method is~~methods are under the jurisdiction of ASTM Committee E27 on 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~~standard's Document Summary page on the ASTM website.

3. Terminology

~~3.1 *Definitions:*~~

~~3.1.1 *constant-temperature stability (CTS) value*—the maximum temperature at which a chemical compound or mixture may be held for a 120-min period under the conditions imposed in this test without exhibiting a measurable exothermic reaction.~~

3.1 ~~The specialized terms in this standard are described in Terminologies E473 and E1445 including differential scanning calorimetry, differential thermal analysis, exotherm, and first-deviation-from-baseline.~~*Definitions:*

3.1.1 The specialized terms in this standard are described in Terminologies E473 and E1445, including *differential scanning calorimetry, differential thermal analysis, exotherm*, and *first-deviation-from-baseline*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *constant-temperature stability (CTS) value, n*—the maximum temperature at which a chemical compound or mixture may be held for a 120-min period under the conditions imposed in this test without exhibiting a measurable exothermic reaction.

4. Summary of Test ~~Method~~Methods

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 120-min period or until an exothermic reaction is recorded. Test temperatures are decreased in ~~10°C~~ 10 °C intervals until no exothermic reaction is observed in the 120-min test period. The ~~Constant Temperature Stability~~ constant-temperature stability is determined and reported using either Test Method A or Test Method B.

NOTE 2—Test periods other than two 120-min periods may be used but shall be reported.

NOTE 3—The processing times in many industrial scale unit operations (for example, drying, distillations, and the like) normally significantly exceed the 120-min 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~~ These test methods are 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~~These test ~~method described~~methods describe 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.~~ ±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~~ 400 °C at a rate of ~~1°C/min to 50°C/min~~ 1 °C/min to 50 °C/min and maintaining an isothermal temperature constant within that range to ~~±1°C~~ ±1 °C for 120 min.

6.2.3 A *Data Collection Device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for differential scanning calorimetry are heat flow, temperature and time.

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~~or containers (pans, crucibles, vials, and the ~~like)~~ like), or both, readable to ±0.1 mg (see Note 6).