Designation: ~~E2046 – 14~~ E2046 – 19

Standard Test Method for Reaction Induction Time by Thermal Analysis[1]

This standard is issued under the fixed designation E2046; 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 measurement of ~~Reaction Induction Time~~reaction induction time (RIT) of chemical materials that undergo exothermic reactions with an induction period. The techniques and apparatus described may be used for solids, liquids, or slurries of chemical substances. The temperature range covered by this test method is typically from ambient to ~~400°C.~~ 400 °C. This range may be extended depending upon the apparatus used.

1.2 The RIT is a relative index value, not an absolute thermodynamic property. As an index value, the RIT value may change depending upon experimental conditions. A comparison of RIT values may be made only for materials tested under similar conditions of apparatus, specimen size, and so forth. Furthermore, the RIT value may not predict behavior of large quantities of material.

1.3 The RIT shall not be used by itself to establish a safe operating temperature. It may be used in conjunction with other test methods (for example, Test Methods E487~~,~~ and E537, and Guide E1981) as part of a hazard analysis of a particular operation.

1.4 This test method may be used for RIT values greater than 15 min (as relative imprecision increases at shorter periods).

1.5 This test method is used to study catalytic, autocatalytic, and accelerating reactions. These reactions depend upon time as well as temperature. Such reactions are often studied by fixing one experimental parameter (that is, time or temperature) and then measuring the other parameter (that is, temperature or time). This test method measures time to reaction onset detection under isothermal conditions. It is related to Test Method E487 that measures detected reaction onset temperature under constant time conditions

1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this ~~standard.~~test method.

~~1.7 There is no ISO standard equivalent to this test method.~~

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 ~~consult and~~ establish appropriate ~~safety~~ safety, health, and ~~health~~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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3350 Specification for Polyethylene Plastics Pipe and Fittings Materials
D3895 Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry
D4565 Test Methods for Physical and Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable
D5483 Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry
D6186 Test Method for Oxidation Induction Time of Lubricating Oils by Pressure Differential Scanning Calorimetry (PDSC)
E473 Terminology Relating to Thermal Analysis and Rheology

[1] This test method is under the jurisdiction of ASTM Committee E27 on Hazard Potential of Chemicals and is the direct responsibility of Subcommittee 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.

E487 Test Method for Constant-Temperature Stability of Chemical Materials
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
E1858 Test Methods for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry
E1860 Test Method for Elapsed Time Calibration of Thermal Analyzers
E1981 Guide for Assessing Thermal Stability of Materials by Methods of Accelerating Rate Calorimetry
E2070 Test Methods for Kinetic Parameters by Differential Scanning Calorimetry Using Isothermal Methods

3. Terminology

3.1 The specialized terms used in this test method are described in Terminologies E473 and E1445, including *differential scanning calorimetry, differential thermal analysis, extrapolated onset value, first-deviation-from-baseline, onset value, isothermal*, and *reaction*.

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 minimum of two hours without exhibiting a measurable exothermic reaction. (See Test Method E487.)

3.2.2 *reaction induction time (RIT) value, n*—the time a chemical compound or mixture may be held under isothermal conditions until it exhibits a specified exothermic reaction.

4. Summary of Test Method

4.1 A specimen of the chemical compound or mixture is placed in an inert container that is then heated to an operator-selected test temperature of interest. The specimen temperature and the difference in heat flow or temperature between the test specimen and an inert reference are monitored until an exothermic reaction is recorded. The time from the attainment of the isothermal test temperature until the extrapolated onset to the exothermic reaction is taken as the ~~Reaction Induction Time.~~RIT.

4.2 Using fresh specimens measurements at more than one isothermal test temperature may be made.

4.3 The RIT is expressed as time at a specific test temperature. For example:

~~RIT~~

~~= 120 min at 100°C~~ RIT = 120 min at 100 °C

5. Significance and Use

5.1 This test method measures the time to extrapolated onset of an exothermic reaction under constant temperature (isothermal) conditions for reactions which have an induction period, for example, those which are catalytic, autocatalytic, or accelerating in nature or which contain reaction inhibitors.

5.2 The RIT determined by this test method is an index measurement that is useful for comparing one material to another at the test temperature of interest and in the same apparatus type only.

5.3 This test method is a useful adjunct to dynamic thermal tests, such as Test Method E537, which are performed under conditions in which the sample temperature is increased continuously at constant rate. Results obtained under dynamic test conditions may result in higher estimates of temperature at which an exothermic reaction initiates because the detected onset temperature is dependent upon the heating rate and because dynamic methods allow insufficient time for autocatalytic reactions to measurably affect the onset temperature.

5.4 RIT values determined under a series of isothermal test conditions may be plotted as their logarithm versus the reciprocal of the absolute temperature to produce a plot, the slope of which is proportional to the activation energy of the reaction as described in Test ~~Method~~Methods E2070.

5.5 This test method may be used in research and development, manufacturing, process and quality control, and regulatory compliance.

5.6 This test method is similar to that for ~~Oxidation Induction Time~~oxidation induction time (OIT) (for example, Specification D3350 and Test Methods D3895, D4565, D5483, D6186, and E1858) where the time to the oxidation reaction under isothermal test conditions is measured. The OIT test method measures the presence of antioxidant packages and is a relative measurement of a ~~material's~~material's resistance to oxidation.

6. Apparatus

6.1 The design and complexity of the apparatus required for this test method depends upon the size of the specimen to be used. In general, observation of an exothermic reaction in small specimens (less than 50 mg) is performed using differential scanning calorimetry or differential thermal analysis equipment and techniques. Large samples (up to 2 g) may be tested using devices such as the Kuhner Micro CTS apparatus.

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

NOTE 1—Commercially available differential scanning calorimetry apparatus may be used. Alternatively, the apparatus may be assembled or fabricated from commercially available components.

6.2.1 *Test Chamber,* composed of the following:

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 specimen/furnace temperature readable to ±0.1 K.

6.2.1.3 *Differential Sensor,* to detect a difference in heat flow (or temperature) between the specimen and the reference specimen equivalent to 10 µW or 0.01 mK.

NOTE 2—A reference material is used when differential heat flow or differential temperature measurements are made. The reference material should match the physical state and heat capacity of the specimen as closely as practical. Typical reference materials include calcined aluminum oxide, glass beads, silicone oil, or combinations thereof.

6.2.1.4 *Means of Sustaining a Test Chamber Environment,* through the use of an air purge gas at a rate of 10 mL/min to 100 ± 5 mL/min.

NOTE 3—Typically, air or inert 99.9+ % pure nitrogen, argon, or helium is employed (when oxidation in air is a concern). Unless effects of moisture are to be studied, use of dry purge gas is recommended.

NOTE 4—For the Kuhner Micro CTS apparatus, the purge gas is provided by operation in a laboratory hood with the door(s) approximately 50 % closed.

6.2.2 *Temperature Controller,* capable of heating from ambient to ~~400°C~~ 400 °C at a rate of up to ~~20°C/min~~ 20 °C/min and maintaining an isothermal temperature constant within that range of ~~±0.4°C~~ ±0.4 °C for the duration of the test, or both.

6.2.3 *A Data Recording 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 or differential thermal analysis are heatflow (or differential temperature), temperature, and time.

6.2.4 *Containers* (pans, crucibles, vials, test tubes, and so forth, and lids), which are inert to the specimen and reference materials at the maximum temperature used and which are of suitable structure, shape, and integrity to contain the specimen and reference in accordance with the temperature and mass requirements as described in this section.

6.3 *Balance,* with a capacity of 100 mg or more to weigh specimens or containers to ±0.1 mg.

NOTE 5—A balance capacity of 10 g or more with a readability to ±0.1 g is required for use with the Kuhner Micro CTS device.

7. Hazards

7.1 Dynamic thermal tests, utilizing milligram quantities of materials, such as Test Method E537, are normally conducted on specimens before the present test is undertaken. The experimenter shall have sufficient knowledge of the magnitude of hazard associated with the material. Larger specimens shall be used only after due consideration is given to the potential for hazardous reaction. Thermodynamic calculations also may be used to determine the potential hazard.

7.2 Special precautions shall be taken to protect personnel and equipment when the apparatus in use requires the insertion of specimens into a heated block or furnace. These shall include adequate shielding and ventilation of equipment, and face and hand protection (see Note 8).

8. Sampling

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

8.2 Specimen size depends upon the sensitivity of the available apparatus.

NOTE 6—Specimen size of 1 mg to 7 mg is typically used in thermal analysis apparatus. Specimen size of 1 g to 2 g is typically used with the Kuhner Micro CTS apparatus.

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

9. Calibration

9.1 Apparatus temperature calibration shall be performed in accordance with ~~Practice~~ Test Method E967 at a heating rate of ~~1°C/min.~~ 1 °C/min.

9.2 Apparatus heat flow calibration may be performed in accordance with Practice E968.

9.3 Apparatus elapsed time shall be verified to be better than ±1 % by Test Method E1860.

10. Procedure

10.1 Weigh 1 mg to 7 mg with a ~~precision~~readability of ±1 mg of the test specimen into a clean specimen container. Seal the container, if desired.