Designation: E2550 – 11

Standard Test Method for Thermal Stability by Thermogravimetry[1]

This standard is issued under the fixed designation E2550; 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 covers the assessment of material thermal stability through the determination of the temperature at which the materials start to decompose or react and the extent of the mass change using thermogravimetry. The test method uses minimum quantities of material and is applicable over the temperature range from ambient to 800°C.

1.2 The absence of reaction or decomposition is used as an indication of thermal stability in this test method under the experimental conditions used.

1.3 This test method may be performed on solids or liquids, which do not sublime or vaporize in the temperature range of interest.

1.4 This test method shall not be used by itself to establish a safe operating or storage temperature. It may be used in conjunction with other test methods (for example, E487, E537 and E1981) as part of a hazard analysis of a material.

1.5 This test method is normally applicable to reaction or decomposition occurring in the range from room temperature to 800 °C. The temperature range may be extended depending on the instrumentation used.

1.6 This test method may be performed in an inert, a reactive or self-generated atmosphere.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 There is no ISO standard equivalent to this test method.

1.9 *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. This standard may involve hazardous materials, operations, and equipment.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

E487 Test Method for Constant-Temperature Stability of Chemical Materials

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1142 Terminology Relating to Thermophysical Properties

E1445 Terminology Relating to Hazard Potential of Chemicals

E1582 Practice for Calibration of Temperature Scale for Thermogravimetry

E1981 Guide for Assessing Thermal Stability of Materials by Methods of Accelerating Rate Calorimetry

E2040 Test Method for Mass Scale Calibration of Thermogravimetric Analyzers

3. Terminology

3.1 Definition:

3.1.1 Specific technical terms used in this test method are defined in Terminologies E473, E1142 and E1445. These terms include thermogravimetry (TG), thermogravimetric analysis (TGA), thermal stability, onset temperature (T_o), derivative, and TG curve.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *DTG curve*, *n*—a plot of the first derivative of TG data with respect to temperature or time.

3.2.2 *mass change plateau*, *n*—a region of the TG curve with a relatively constant mass; it is accompanied by a minimum in the DTG curve for a mass loss, or a maximum for a mass gain.

[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] 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.

4. Summary of Test Method

4.1 A sample of the material to be examined is placed in an inert container and then heated at a controlled rate of 1 to 20°C min^{-1} under a controlled atmosphere. The sample mass is recorded continuously as a function of time and temperature.

4.2 When the sample undergoes a reaction or thermal decomposition involving a mass change, that change is indicated by a departure from the initially established baseline of the mass record (see Fig. 1).

4.3 The onset temperature and mass changes are determined and reported.

5. Significance and Use

5.1 TG provides a rapid method for determining the thermal decomposition and reaction mass change of a material.

5.2 This test method is useful in detecting potentially hazardous reactions and in estimating the temperatures at which these reactions occur. This test method is recommended as a screening test for detecting the thermal hazards of an uncharacterized material or mixture (see Section 8).

5.3 Energetic materials, pharmaceuticals and polymers are examples of materials for which this test might be useful. This test is especially useful for materials having melting points that overlap with the onset of reaction or decomposition.

NOTE 1—In Differential Scanning Calorimetry (DSC), the melting endotherm may interfere with the determination of the onset temperature for reaction or decomposition.

5.4 This test is not suitable for materials that sublime or vaporize in the temperature range of interest. A sample with volatile impurities needs to be purified prior to the TGA testing. Alternatively, the sample can be tested as is, however, special caution is required during the data analysis. The mass loss due to the loss of impurity should not interfere with the determination of reaction or decomposition temperature.

5.5 The four significant criteria of this test method are: the detection of a sample mass change; the extent of the mass change; the approximate temperature at which the event occurs; the observance of effects due to the atmosphere.

6. Limitations

6.1 Many environmental factors affect the existence, magnitude and onset temperature of a particular reaction or decomposition. Some of these, including heating rate, instrumental sensitivity, and atmosphere reactivity, will affect the detectability of a reaction or decomposition using this procedure. Therefore, it is imperative that the results obtained from the application of this test method be viewed only as an indication of the thermal stability of a material.

6.2 This test method can only be used to detect reaction or decomposition that involves a mass change, such as a production of gaseous species or a mass gain in reactive atmosphere. This test method is not suitable for materials that sublime or vaporize in the temperature of interest.

6.3 This test method may not be reliable for heterogeneous samples.

NOTE 2—For heterogeneous samples, it is recommended to perform replicate measurements to determine the variability of the results. If inconsistent results are obtained, the study should be carried out using larger-scale apparatus, such as accelerating rate calorimetry.

7. Apparatus

7.1 *Thermogravimetric Analyzer (TGA)*—The essential instrumentation required to provide the minimum thermogravimetric analytical capability for this practice includes:

7.1.1 A thermobalance composed of:

7.1.1.1 A furnace to provide uniform controlled heating of a specimen to a constant temperature or at a constant rate within the applicable temperature range of this test method.

7.1.1.2 A temperature sensor to provide an indication of the specimen/furnace temperature to ±0.1°C.

7.1.1.3 A continuously recording balance to measure the specimen mass with a minimum capacity of 10 mg and a sensitivity of ±10 µg.

NOTE 3—An apparatus with a larger capacity can also be used. The sensitivity must be at least ±0.1 mass %.

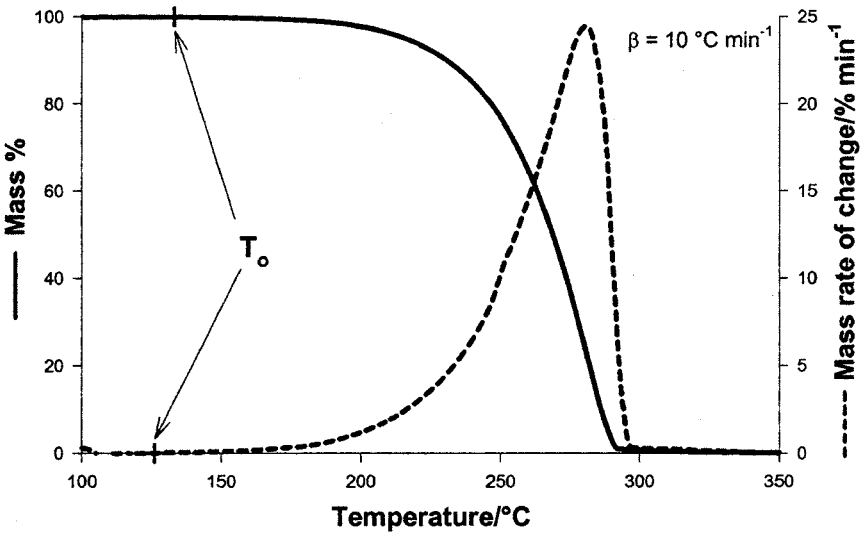

FIG. 1 Typical TG and DTG Curves