Designation: E 1860 – 97a

Standard Test Method for Elapsed Time Calibration of Thermal Analyzers[1]

This standard is issued under the fixed designation E 1860; 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 calibration or performance confirmation of the elapsed-time signal from thermal analyzers.

1.2 Electronic instrumentation or automated data analysis and reduction systems or treatments equivalent to this test method may be used.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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:*

D 3350 Specification for Polyethylene Plastics Pipe and Fittings Materials[2]

D 3895 Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry[2]

D 4565 Test Methods for Physical and Environmental Performance Properties of Insulations and Jackets for Telecommunications Wire and Cable[3]

D 5483 Test Method for Oxidation Induction Time of Lubricating Greases by Pressure Differential Scanning Calorimetry[4]

E 473 Terminology Relating to Thermal Analysis[5]

E 487 Test Method for Constant-Temperature Stability of Chemical Materials[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

E 1142 Terminology Relating to Thermophysical Properties[5]

E 1858 Test Method for Determining Oxidative Induction Time of Hydrocarbons by Differential Scanning Calorimetry[5]

E 1868 Test Method for Loss-on-Drying by Thermogravimetry[5]

3. Terminology

3.1 *Definitions:*

3.1.1 The technical terms used in this test method are defined in Terminologies E 473 and E 1142.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *relative standard deviation, n*—the ratio of the standard deviation of a series of measurements to their mean value, usually expressed as percent.

4. Summary of Test Method

4.1 The elapsed time signal generated by a thermal analyzer is compared to a clock (or timer) whose performance is known and traceable to a National Reference Laboratory. The thermal analyzer may be said to be in conformance, if the performance of the thermal analyzer is within established limits. Alternatively, the elapsed time signal may be calibrated using a two point calibration method.

5. Significance and Use

5.1 Most thermal analysis experiments are carried out under temperature ramp conditions where temperature is the independent parameter. Some experiments, however, are carried out under isothermal temperature conditions where the elapsed time to an event is measured as the independent parameter. Isothermal Kinetics, Thermal Stability (Test Method E 487), Oxidative Induction Time (OIT) (Test Methods D 3895, D 4565, D 5483, Specification D 3350 and Method E AAAA) and Loss-on-Drying (Method E BBBB) are common examples of these kinds of experiments.

5.2 Modern scientific instruments, including thermal analyzers, usually measure elapsed time with excellent precision and accuracy. In such cases, it may only be necessary to confirm the performance of the instrument by comparison to a suitable reference. Only rarely will it may be required to correct the calibration of an instrument's elapsed time signal through the use of a calibration factor.

5.3 It is necessary to obtain elapsed time signal conformity only to 0.1 times the repeatability relative standard deviation (standard deviation divided by the mean value) expressed as a percent for the test method in which the thermal analyzer is to be used. For those test methods listed in Section 2 this conformity is 0.1 %.

[1] This test method is under the jurisdiction of ASTM Committee E-37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.01 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 08.02.

[3] *Annual Book of ASTM Standards*, Vol 10.02.

[4] *Annual Book of ASTM Standards*, Vol 05.03.

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