Designation: E1860 − 23

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

This standard is issued under the fixed designation E1860; 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 calibration or performance confirmation of the elapsed-time signal from thermal analyzers.

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

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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
E473 Terminology Relating to Thermal Analysis and Rheology
E487 Test Methods for Constant-Temperature Stability of Chemical Materials
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1142 Terminology Relating to Thermophysical Properties
E1858 Test Methods for Determining Oxidation Induction Time of Hydrocarbons by Differential Scanning Calorimetry
E1868 Test Methods for Loss-On-Drying by Thermogravimetry
E2070 Test Methods for Kinetic Parameters by Differential Scanning Calorimetry Using Isothermal Methods
E2161 Terminology Relating to Performance Validation in Thermal Analysis and Rheology

3. Terminology

3.1 *Definitions:*

3.1.1 The technical terms used in this test method are defined in Terminologies E473, E1142, and E2161, including *calibration, conformance, relative standard deviation,* and *thermal analysis*.

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 metrology institute. 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 increasing temperature 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 (Test Methods E2070), Thermal Stability (Test Method E487), Oxidative Induction Time (OIT) (Test Methods D3895, D4565, D5483, E1858, and Specification D3350) and Loss-on-Drying (Test Methods E1868) are common examples of these kinds of experiments.

[1] This test method is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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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.

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 %.

6. Apparatus

6.1 *Timer or Stopwatch,* with timing capacity of at least 3 h (10 800 s), a resolution of 0.1 s or better and an accuracy of 1.5 s per day which performance has been verified using standards and procedures traceable to a national metrology institute (such as the National Institute of Standards and Technology (NIST)). Such timers are available from most laboratory equipment suppliers.

7. Calibration

7.1 Perform any elapsed time signal calibration procedures recommended by the manufacturer of the thermal analyzer as described in the operator's manual.

8. Procedure

8.1 Obtain the instrument reaction time (*I*).

8.1.1 Reset the timer and the elapsed time signal for the thermal analyzer to zero elapsed time.

8.1.2 Simultaneously start the timer and the elapsed time signal for the thermal analyzer. Allow them to run for 6 s to 10 s. Simultaneously stop the timer and the elapsed time signal for the thermal analyzer. Record the elapsed time from the timer as t_1. Record the elapsed time from the thermal analyzer as t_2.

NOTE 1—The elapsed time of the timer (t_1) is equal to the elapsed time of the thermal analyzer (t_2) plus the instrument reaction time (*I*). The instrument reaction time is that required for the thermal analyzer to initialize and terminate the thermal analysis experiment and may be up to several seconds. The instrument start up time does not affect the elapsed time of the thermal analysis experiment since the experiment is exclusive of this time.

NOTE 2—Data acquisition rate shall be set to the maximum available.

NOTE 3—Time measurements shall be recorded in seconds retaining all available digits.

8.1.3 Calculate the instrument reaction time *I* by Eq 2 (9.2).

8.2 Obtain the calibration constant (*S*).

8.2.1 Reset the timer and the elapsed time signal for the thermal analyzer to zero elapsed time.

8.2.2 Simultaneously start the timer and the elapsed time signal for the thermal analyzer. Allow them to run for a minimum of 10 000 s (= 167 min = 2.8 h = 2 h, 47 min). Simultaneously stop the timer and the elapsed time signal for the thermal analyzer (see Note 2, Note 3, and Note 4). Record the elapsed time from the timer as t_t. Record the elapsed time from the thermal analyzer as t_o.

8.2.3 Calculate the value for *S* using Eq 3 (see 9.3).

8.3 Using the values for *I* and *S* from 8.1.3 and 8.2.3, calculate the percent conformity (*C*) using Eq 4 or table of percent conformity values (see 9.4).

9. Calculation

9.1 For the purpose of these procedures, it is assumed that the relationship between observed elapsed time (t_o) and the actual elapsed time (t) is linear and is governed by Eq 1:

$$t = t_o S \tag{1}$$

where:

t = true experimental elapsed time (s),
t_o = thermal analyzer observed elapsed time (s), and
S = slope (nominal value = 1.00000).

9.2 Using the values for t_1 and t_2 from 8.1, the instrument reaction time (*I*) may be calculated by:

$$I = t_1 - t_2 \tag{2}$$

9.3 Using the values for t_t and t_o from 8.2, the calibration constant *S* may be calculated by:

$$S = (t_t - I)/t_o \tag{3}$$

where:

t_t = observed time of reference timer.

9.3.1 When performing this calculation, retain all available decimal places in the measured value and in the value of *S*.

9.4 Using the value for *S* from 9.3, the percent conformity of the instrument elapsed time indicator may be calculated as follows:

$$C = (1.00000 - S) \times 100\,\% \tag{4}$$

NOTE 4—The percent conformity is usually a very small number and expressing it as a percent value may be inconsistent with SI metric notation. Because of its effect on the experiment and because of common use, it is expressed as a percent is this procedure.

9.4.1 Conformity may be estimated to one significant figure using the following criteria:

9.4.1.1 If *S* lies:

Between 0.9999 and 1.0001, then conformity is better than 0.01 %,

Between 0.9990 and 0.9999 or between 1.0001 and 1.0010, then conformity is better than 0.1 %, and

Between 0.9900 and 0.9990 or between 1.0010 and 1.0100, then conformity is better than 1 %.

9.5 Using the determined value for *S*, Eq 1 may be used to calculate the true elapsed time (t) from an observed elapsed time (t_o).

10. Report

10.1 Report the following information:

10.1.1 Model number and description of the thermal analyzer used,

10.1.2 The value of *S* as determined in 8.2.3 reported to at least five places to the right of the decimal point, and

10.1.3 Conformity as determined in 9.4.