Designation: E2040 – 08 (Reapproved 2014)

Standard Test Method for Mass Scale Calibration of Thermogravimetric Analyzers[1]

This standard is issued under the fixed designation E2040; 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 mass (or weight) scale of thermogravimetric analyzers and is applicable to commercial and custom-built apparatus.

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 There is no ISO standard equivalent to this test method.

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:*[2]

E473 Terminology Relating to Thermal Analysis and Rheology

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

E1142 Terminology Relating to Thermophysical Properties

3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in Terminologies E473 and E1142.

4. Summary of Test Method

4.1 The mass signal generated by a thermogravimetric analyzer is compared to the mass of a reference material traceable to a national reference laboratory. A linear correlation using two calibration points is used to relate the mass (or weight) signal generated by the thermogravimetric analyzer and that of the reference material.

5. Significance and Use

5.1 This test method calibrates or demonstrates conformity of thermogravimetric apparatus at ambient conditions. Most thermogravimetry analysis experiments are carried out under temperature ramp conditions or at isothermal temperatures distant from ambient conditions. This test method does not address the temperature effects on mass calibration.

5.2 In most thermogravimetry experiments, the mass change is reported as weight percent in which the observed mass at any time during the course of the experiment is divided by the original mass of the test specimen. This method of reporting results assumes that the mass scale of the apparatus is linear with increasing mass. In such cases, it may be necessary only to confirm the performance of the instrument by comparison to a suitable reference.

5.3 When the actual mass of the test specimen is recorded, the use of a calibration factor to correct the calibration of the apparatus may be required, on rare occasions.

6. Apparatus

6.1 The essential equipment required to provide the minimum thermogravimetric analytical capability for this test method includes the following:

6.1.1 *Thermobalance,* composed of a *furnace*; a *temperature sensor*; a *balance* to measure the specimen mass with a minimum capacity within the range to be calibrated and a sensitivity of ±1 µg; and a means of maintaining the specimen/container under atmospheric control of the gas to be used at a purge rate between 10 to 100 ± 5 mL/min.

NOTE 1—Excessive purge rates should be avoided as this may introduce noise due to buoyancy effects and temperature gradients.

6.1.2 *Temperature Controller,* capable of maintaining ambient temperature to ±1K.

6.1.3 *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 thermogravimetric analysis are mass, temperature, and time.

6.1.4 *Containers (pans, crucibles, etc.),* which are inert to the specimen and which will remain gravimetrically stable.

[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 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.

7. Reagents and Materials

7.1 A reference material of known mass, which is traceable to a national standards laboratory, such as the National Institute of Standards and Technology (NIST). Such mass reference materials are available from most general laboratory equipment suppliers.

7.2 The mass of the reference material should correspond to the working range of the analysis. For most work, the mass maximum should be 25 to 50 % greater than the material being examined.

8. Calibration and Standardization

8.1 Perform any mass signal calibration procedures recommended by the manufacturer of the thermogravimetric analyzer as described in the operator's manual.

9. Procedure

9.1 Prepare the thermogravimetric analyzer for operation under the test conditions to be used for the characterization of test specimens, including loading an empty specimen container and initiating a purge gas. The temperature to be used is ambient.

9.2 Tare the apparatus by setting the mass of the empty specimen container to 0.00 mg.

9.3 Open the apparatus, place the reference material into the specimen container, and reassemble the apparatus under the test conditions to be used for the characterization of the test specimens.

9.4 Record the mass observed by the apparatus as m_o.

9.5 Record the mass of the reference material from its certificate as m_s, retaining all available decimal places in the measured value.

9.6 Calculate and report the value for the slope (S) and conformity (C) using Eq 2 and Eq 3.

10. Calculation

10.1 For the purpose of this test method, it is assumed that the relationship between observed mass (m_o) and the reference mass (m_s) is linear and governed by the slope (S) of Eq 1:

$$m_s = (m_o \times S) \quad (1)$$

10.2 By using the mass values taken from 9.4 and 9.5, calculate S using Eq 2:

$$S = m_s/m_o \quad (2)$$

NOTE 2—When performing this calculation, retain all available decimal places in the calculated value.

10.3 Using the value of S from 10.2, the percent conformity of the instrument mass scale, C, may be calculated using Eq 3:

$$C = (1.00000 - S) \times 100\,\% \quad (3)$$

NOTE 3—The percent conformity usually is 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, its expression as a percent is used in this procedure.

10.3.1 Conformity may be estimated to one significant figure using the following table of criteria:

10.3.1.1 If S is between 0.9999 and 1.0001, then conformity is better than 0.01 %.

10.3.1.2 If S is between 0.9990 and 0.9999 or between 1.0001 and 1.0010, then conformity is better than 0.1 %.

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

10.3.1.4 If S is between 0.9000 and 0.9900 or between 1.0100 and 1.1000, then conformity is better than 10 %.

10.4 Report the value of S and the conformity, C.

10.5 Using the determined value of S from Eq 2, Eq 1 may be used to calculate the true corrected mass (m) from an observed mass (m_o).

11. Report

11.1 The report shall include the following information:

11.1.1 Details and description, including the manufacturer and instrumental model number, where applicable, of the thermogravimetric analyzer.

11.1.2 The value of S as determined in 10.2, reported to at least four places to the right of the decimal point.

11.1.3 The conformity, C, as determined in 10.3.

11.1.4 The specific dated version of this test method used.

12. Precision and Bias

12.1 An interlaboratory study was conducted in 1998 that included participation by seven laboratories using instruments from a single manufacturer (TA Instruments). The results were treated by Practice E691.

12.2 *Precision:*

12.2.1 The mean value for the calibration constant was S = 0.99818.

12.2.2 The repeatability (within laboratory) standard deviation for S was 0.00047.

12.2.3 Two values, each the mean of duplicated determinations within a single laboratory, should be considered suspect if they differ by more than 95 % repeatability limit r = 0.0013.

12.2.4 The reproducibility (between laboratory) standard deviation for S was 0.0030.

12.2.5 Two values, each the mean of duplicated determinations in differing laboratories, should be considered suspect if they differ by more than 95 % reproducibility limit R = 0.0084.

12.3 *Bias:*

12.3.1 The measurement of conformity in this test method is a comparison of the calibration constant S with the theoretical value of 1.0000000 and provides an indication of bias.

12.3.2 The mean value for conformity was C = 0.18 %.

12.3.3 Conformity was found to vary widely among instrument models but in no case exceeded C = 0.66 %. This value is far better than the nominal conformity of 1 % required for most thermal analysis experiments.

13. Keywords

13.1 calibration; conformity; mass; thermogravimetry; thermogravimetric analyzer