



# Standard Test Method for Mass Scale Calibration of Thermogravimetric Analyzers<sup>1</sup>

This standard is issued under the fixed designation E 2040; 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 ( $\epsilon$ ) 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 mass (or weight) scale of thermogravimetric analyzers and is applicable to commercial and custom-built apparatus.

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 There is no ISO standard equivalent to this test method.

1.5 *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:*

E 473 Terminology Relating to Thermal Analysis<sup>2</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>2</sup>

E 1142 Terminology Relating to Thermophysical Properties<sup>2</sup>

## 3. Terminology

3.1 *Definitions*—Specific technical terms used in this test method are defined in accordance with Terminologies E 473 and E 1142.

## 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  $\pm 1 \mu\text{g}$ ; and a means of maintaining the specimen/container under atmospheric control of the gas to be used at a purge rate between 10 to  $100 \pm 5 \text{ mL/min}$ .

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  $\pm 1\text{K}$ .

6.1.3 *Recording Device*, either analog or digital, capable of recording and displaying any fraction of the specimen mass signal including the signal noise.

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

## 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

<sup>1</sup> 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 and Recommended Practices.

Current edition approved Sept. 10, 1999. Published October 1999.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.