



Standard Test Method for Mass Loss and Residue Measurement Validation of Thermogravimetric Analyzers¹

This standard is issued under the fixed designation E2402; 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 method provides procedures for validating mass loss and residue measurements by thermogravimetric analyzers (TGA) and analytical methods based upon the measurement of mass loss or residue content. Performance parameters determined include mass loss and residue repeatability (precision), detection limit, quantitation limit, linearity and bias.

1.2 Validation of apparatus performance and analytical methods is requested or required for quality initiatives or where results may be used for legal purposes.

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

1.4 There is no ISO standard equivalent to this 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:²

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E473 Terminology Relating to Thermal Analysis and Rheology

E1142 Terminology Relating to Thermophysical Properties

E1582 Practice for Calibration of Temperature Scale for Thermogravimetry

E1970 Practice for Statistical Treatment of Thermoanalytical Data

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.

E2040 Test Method for Mass Scale Calibration of Thermogravimetric Analyzers

E2161 Terminology Relating to Performance Validation in Thermal Analysis

2.2 Other Standard:

United States Food and Drug Administration, Q2B Validation of Analytical Procedures: Methodology, 62 FR 27464, May 19, 1997³

3. Terminology

3.1 Technical terms used in this standard are defined in Practice E177 and in Terminologies E473, E1142, and E2161.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *highly volatile matter*—materials (such as moisture, plasticizer, residual solvent, etc.) that boil at temperatures below 200 °C.

3.2.2 *medium volatile matter*—materials (such as oil and polymer degradation products) that boil in the temperature range between 200 and 400 °C.

3.2.3 *residue*—material remaining (such as metal components, filler content or inert reinforcing materials) after more volatile components are vaporized.

3.2.4 *mass loss plateau*—a region of a thermogravimetric curve with a relatively constant mass (that is, accompanied by a minima in the first derivative of mass with respect to time).

4. Summary of Test Method

4.1 Mass is the primary dependent parameter and temperature is the primary independent parameter measured by TGA.

4.2 Mass loss and residue measurements are validated by their direct measurement using thermogravimetric apparatus over a specified temperature range using reference materials of known volatiles content as an analyte.

4.3 Alternatively, validation of a TGA method based upon mass loss and residue measurements may be performed using a specific test specimen as the analyte.

³ Available from Food and Drug Administration (FDA), 10903 New Hampshire Ave., Silver Spring, MD 20993-0002, <http://www.fda.gov>.