



Designation: E3142 – 18a (Reapproved 2023)

Standard Test Method for Thermal Lag of Thermal Analysis Apparatus¹

This standard is issued under the fixed designation E3142; 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.

INTRODUCTION

In thermal analysis, the temperature of a test specimen is changed while a physical property is measured. The measured physical property is the dependent variable and temperature (also measured) is the independent variable. In the majority of thermal analysis apparatus, temperature sensors cannot be attached directly to the specimen but can only touch the surface or be placed adjacent or close to the specimen such that the indicated temperature will be different to that of the specimen itself. In consequence the specimen temperature will lag the indicated temperature upon heating and cooling due to thermal resistance between sensor and specimen. The larger the test specimen, the greater the thermal lag is likely to be. To obtain the correct specimen temperature, thermal analysis apparatus is temperature calibrated so that the recorded temperature correctly indicates the specimen temperature. Such temperature calibration compensates for the temperature offset (τ) between the specimen temperature and that of the temperature sensor. This temperature offset changes linearly with temperature rate-of-change (β) (heating or cooling). The slope of this linear relationship is known as “thermal lag” ($\Delta T/\Delta \beta$). Knowing the thermal lag for an apparatus permits temperature calibration determined at one temperature rate-of-change to be adjusted to that at other rates. It is the purpose of this standard to aid the user to determine the thermal lag for an apparatus and to apply that thermal lag to measurements made at temperature rates-of-change different from that at which the temperature calibration is performed.

1. Scope

1.1 This test method addresses the dependence of temperature calibration on the temperature rate-of-change. This test method describes the determination of the thermal lag of thermal analysis apparatus and its application to the modification of the temperature calibration for that apparatus obtained at alternative linear temperature rates-of-change.

1.2 This test method is applicable, but not limited to, the temperature calibration of differential thermal analyzers (DTAs), differential scanning calorimeters (DSCs), thermogravimetric analyzers (TGAs), thermomechanical analyzers (TMAs), and dynamic mechanical analyzers (DMAs).

1.3 This test method is applicable only to apparatus demonstrating a linear relationship between indicated temperature and temperature rate-of-change.

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

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

1.6 *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

E473 Terminology Relating to Thermal Analysis and Rheology

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.

Current edition approved June 1, 2023. Published July 2023. Originally approved in 2018. Last previous edition approved in 2018 as E3142 – 18a. DOI: 10.1520/E3142-18AR23.

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.

- E698** Test Method for Kinetic Parameters for Thermally Unstable Materials Using Differential Scanning Calorimetry and the Flynn/Wall/Ozawa Method
- E967** Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers
- E1142** Terminology Relating to Thermophysical Properties
- E1363** Test Method for Temperature Calibration of Thermo-mechanical Analyzers
- E1582** Test Method for Temperature Calibration of Thermogravimetric Analyzers
- E1867** Test Methods for Temperature Calibration of Dynamic Mechanical Analyzers
- E1970** Practice for Statistical Treatment of Thermoanalytical Data
- E2069** Test Method for Temperature Calibration on Cooling of Differential Scanning Calorimeters

3. Terminology

3.1 Definitions:

3.1.1 Terms applicable to this test method and can be found in Terminologies **E473** and **E1142** and include the terms *calorimeter*, *differential*, *differential scanning calorimeter*, *differential thermal analysis*, *dynamic mechanical analysis*, *temperature*, *thermal analysis*, *thermogravimetric analysis*, and *thermomechanical analysis*.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *temperature offset*, n —the difference between the actual specimen temperature and that reported by a temperature sensor.

3.2.2 *thermal lag*, n —the change in indicated temperature offset with temperature rate-of-change.

4. Summary of Test Method

4.1 Following apparatus temperature calibration with a reference material at an identified temperature rate-of-change, the temperature offset of the apparatus is measured with that sample reference material as a function of additional temperature rates-of-change. A display of temperature (T) versus temperature rate-of-change (β) produces a linear relationship from which the slope ($\Delta T/\Delta \beta$) may be obtained. This slope value is known as thermal lag.

4.1.1 The thermal lag may be used to modify the temperature calibration of an apparatus at temperature rates-of-change other than at which it is calibrated.

4.1.2 The thermal lag of an apparatus may change with experimental conditions such as temperature, purge gas pressure, purge gas thermal conductivity, or other experimental conditions or instrument properties. Thermal lag shall be determined under the same experimental conditions as that of the test specimen.

4.1.3 Thermal lag calculations shall not be used for temperature rate-of-change values greater than 10 K/min.

4.1.4 Thermal lag is known to vary with temperature. If a multipoint temperature calibration is used, then thermal lag shall be determined at each calibration temperature.

5. Significance and Use

5.1 Differing temperature rates-of-change may be required for different measurements (for example, Test Method **E698**). Temperature calibration changes as a function of temperature rate-of-change. The use of the known thermal lag of an apparatus may be used to adjust the temperature calibration of the apparatus obtained at one temperature rate-of-change with that at another required for a given applications. This adjustment procedure for temperature calibration is described in **8.1**.

5.2 This test method may be used in research, quality assurance, and specification acceptance.

6. Interferences

6.1 Thermal lag on heating may differ from that on cooling.

6.2 Where thermal lag is examined upon cooling, the reference material shall be selected to show no supercooling. Liquid crystal materials and adamantane are examples of materials without supercooling (see Test Method **E2069**).

7. Procedure

7.1 Select the appropriate ASTM standard for temperature calibration of the apparatus (Test Method **E967** for differential scanning calorimeters or differential thermal analyzers, Test Method **E1582** for thermogravimetric analyzers, Test Method **E1363** for thermomechanical analyzers, or Test Methods **E1867** for dynamic mechanical analyzers).

7.2 Perform the procedure described in the calibration standard and record the determined temperature as T_1 and the observed temperature rate-of-change as β_1 .

NOTE 1—The temperature rate-of-change shall be measured. Use of the programmed temperature rate-of-change is not satisfactory.

7.3 Perform the procedure described in the calibration standard at two additional heating rates other than β_1 representing the low, medium, and high heating rate capability of the apparatus. Record these additional data pair values as T_2 and β_2 , and T_3 and β_3 .

NOTE 2—The reliability of the results of this determination increases as temperature rate-of-change increases.

7.4 Prepare a display of the values for **7.2** and **7.3**, with T -values on the Y-axis and the corresponding β -values on the X-axis.

NOTE 3—The display shall approximate a straight line. If not, then this test method is not applicable.

7.5 Using linear regression (see Practice **E1970**), determine and report the slope ($\Delta T/\Delta \beta$) and its standard deviation ($\sigma \Delta T/\Delta \beta$) of the best-fit straight line for these three points.

NOTE 4—The appropriate SI units for this value is seconds. However, it is easier to leave this value in the units of minutes for the purpose of its application. Alternatively, one may use the conversion factor of 1 min = 60 s in the calculations.

NOTE 5—The thermal lag is known to vary with temperature. If a multipoint temperature calibration is used, then thermal lag shall be determined at each calibration temperature.

NOTE 6—For the purpose of this test method, a goodness-of-fit R^2 value greater than 0.95 is considered acceptable.