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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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# Standard Practice for Calibration of Temperature Scale for Thermogravimetry[1]

This standard is issued under the fixed designation E 1582; 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 practice covers the temperature calibration of thermogravimetric analyzers over the temperature range from 25 to 1500°C and is applicable to commercial and custom-built apparatus. This calibration may be accomplished by the use of either melting point standards or magnetic transition standards.

1.2 The mass change curve in thermogravimetry results from a number of influences, some of which are characteristic of the specimen holder assembly and atmosphere rather than the specimen. The variations from instrument to instrument occur in the point of measurement of the temperature, the nature of the material, its size and packing, the geometry and composition of the specimen container, the geometry and design of the furnace, and the accuracy and sensitivity of the temperature sensor and displaying scales. These all contribute to differences in measured temperatures, which may exceed 20 K. In addition, some sample holder assemblies will show variations of measured temperature with sample size or heating/cooling rate, or both. Since it is neither practical nor advisable to standardize sample holders or thermobalance geometries, instruments may be calibrated by measurement of the deviation of a melting or magnetic (Curie Point) transition temperature from the standard reference temperature. This deviation can be applied as a correction term to subsequent measurements.

1.3 This practice assumes that the indicated temperature of the instrument is linear over the range defined by a two-point calibration and that this linearity has been verified. These two calibration temperatures should be as close to the experimental measurements to be made as possible.

1.4 This practice describes three procedures for temperature calibration of thermogravimetric analyzers using any type balance. Procedures A and B use melting point standards with vertical balances. Procedure C uses magnetic transition standards for calibration. Procedure A is designed specifically for use with horizontal-type balances using external furnaces. Procedure B is designed specifically for use with vertical hang-down balances using either internal or external furnaces. No procedure is restricted to the use of the furnace type described in that procedure.

1.5 Computer or electronic-based instruments, techniques, or data treatment equivalent to this procedure may also be used. Users of this practice are expressly advised that all such instruments or techniques may not be equivalent. It is the responsibility of the user of this practice to determine the necessary equivalency prior to use. In the case of dispute, only the manual procedures, described in this practice, are to be considered valid.

1.6 The data generated by these procedures can be used to correct the temperature scale of the instrument by either a positive or negative amount using either a two-point temperature calibration procedure or a multi-point temperature calibration with best line fit for the generated data.

NOTE 1—A single-point calibration may be used where this is the only procedure possible or practical. The use of a single-point procedure is not recommended.

1.6.1 Many of the newer computer-controlled instruments have features for using calibration data of the latter type.

1.7 The values stated in SI units are to be regarded as the standard.

1.8 *This standard does not purport to address all of the safety problems, 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 472 Practice for Reporting Thermoanalytical Data[2]

E 473 Definitions of Terms Relating to Thermal Analysis[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

## 3. Terminology

3.1 *Definitions*—The term thermogravimetry is defined in Definitions E 473. Thermal Curve is defined in Terminology E 1142.

3.1.1 *magnetic reference temperature*—the observed temperature at which a change in the magnetic properties of a material in a magnetic field produces an apparent mass change. This temperature is read from the dynamic TG curve as the point of intersection of the extrapolated higher temperature

[1] This practice 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.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

portion of the base line with a tangent drawn to the point of greatest slope of apparent mass-change curve. This temperature most closely represents the Curie Point, that point on the mass change curve where the magnetic effect of the standard material has disappeared completely (see Fig. 1).

NOTE 2—The position of the magnet and the design of the instrument will affect the direction of the mass change.

## 4. Summary of Practice

4.1 This practice provides a set of different procedures since thermogravimetric apparatus is often of significantly differing design.

4.2 *Calibration of Analyzers Using Melting Point Standards*—The calibration material is heated at a controlled rate in a controlled atmosphere through its melting region. The temperature of the standard is monitored and recorded continuously. In this practice, a small platinum mass is suspended within a thermogravimetric analyzer specimen boat or pan from a fusible link of the standard calibration material. As the standard specimen is heated through the melting region, the platinum mass is released. The mass is either caught in the specimen boat or pan, producing an "action/reaction" blip on the thermal curve, or is allowed to drop through a hole in the bottom of the specimen boat or pan, producing a sharp, discontinuous mass loss. These events may be used to calibrate the thermogravimetric analyzer for the experimental conditions used.

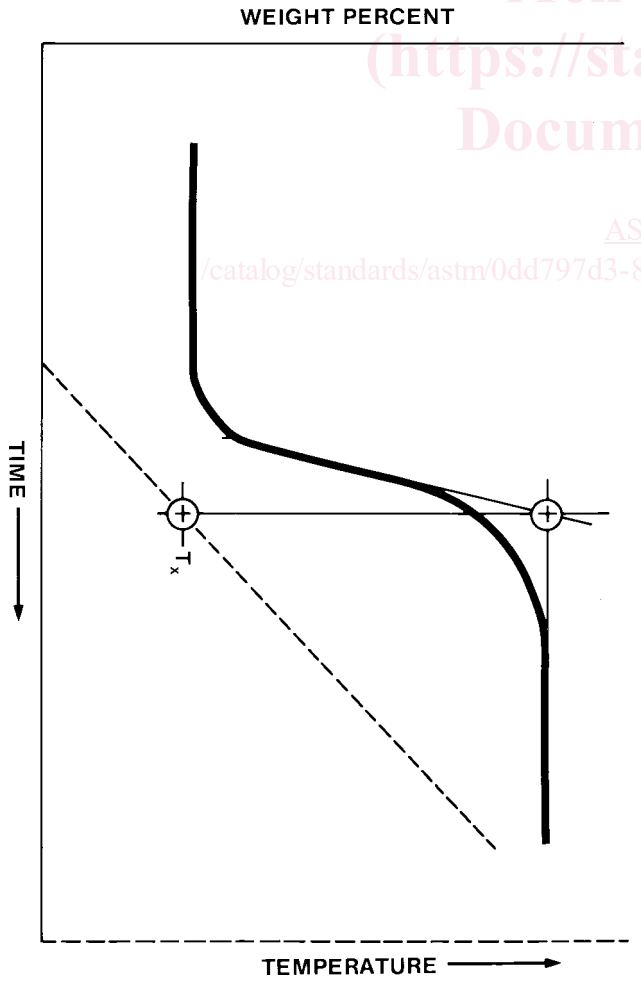


**FIG. 1 Magnetic Reference Temperature**

4.3 *Calibration of Analyzers Using Magnetic Transition Standards*:

4.3.1 In this procedure, the apparent mass change of one or more of the magnetic transition standards is obtained under the normal operating conditions of the instrument. The end temperature, $T_x$ (Fig. 1), is determined and compared with the established transition temperature for the material. The difference provides an adjustment or calibration that may be applied to the temperature scale of the instrument.

4.3.2 The apparent mass change of the magnetic transition materials is caused by the magnetic to nonmagnetic transition in the presence of a magnetic field.

## 5. Significance and Use

5.1 Thermogravimetric analyzers are used to characterize a broad range of materials. In most cases, one of the desired values to be assigned in thermogravimetric measurements is the temperature at which significant changes in specimen mass occur. Therefore, the temperature axis (abscissa) of all apparent-mass-change curves must be calibrated accurately, either by direct reading of a temperature sensor, or by adjusting the programmer temperature to match the actual temperature over the temperature range of interest. In the latter case, this is accomplished by the use of either melting point or magnetic transition standards.

5.2 This practice permits interlaboratory comparison and intralaboratory correlation of instrumental temperature scale data.

## 6. Interferences

6.1 The reference metals are sensitive to impurities and may oxidize at elevated temperatures. All runs shall be conducted in an oxygen-free inert purge gas of the same type to be used in the experimental procedures.

6.2 Care must be taken to stay below temperatures at which the magnetic transition standard will react with the specimen or its holder.

## 7. Apparatus

7.1 *Thermogravimetric Analyzer*—A system of related instruments that are capable of continuously measuring the mass of a specimen in a controlled atmosphere and in a controlled temperature environment ranging from ambient to at least 25°C above the temperature range of interest over a selected time period. This instrument shall consist of the following:

7.1.1 *Electrobalance*, sensitive to 5 µg.

7.1.2 *Furnace*, controllable from 25°C to the upper temperature limit of the instrument.

7.1.3 *Temperature Programmer*, capable of providing a linear rate of rise from 0.5 to 20°C/min (programmed rates in addition to those listed may be used).

7.1.4 *Temperature Measurement System*, consisting of a sensor, usually a thermocouple and a readout/recording device.

7.1.5 *Recording Device*, for example, *X-Y* or strip chart recorder, printer-plotter.

7.2 *Inert Atmosphere (Gas Purge)*—Purified grade $N_2$ or other inert gas.