Designation: E 1582 – 00

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°C. 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 be used.

NOTE 1—Since all electronic data treatments are not equivalent, the user shall verify equivalency prior to use.

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 2—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 SI units are standard.

1.8 This practice is related to ISO 11358 but provides information and methods not found in ISO 11358 .

1.9 *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 473 Terminology Relating to Thermal Analysis[2]

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

E 967 Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers[2]

E 1142 Terminology Relating to Thermophysical Properties[2]

2.2 *Other Standards:*

ISO 11358 Thermogravimetry (TG) of Polymers — General Principles[3]

3. Terminology

3.1 *Definitions*—Technical terms used in this document are defined in Terminology E 473 and E 1142.

[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.

[3] Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

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 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 3—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.

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 extrapolated endpoint temperature, (see 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.

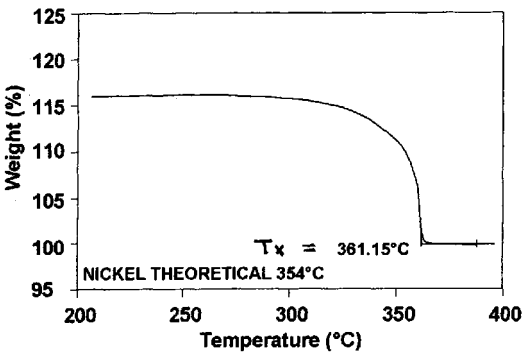


FIG. 1 Magnetic Reference Temperature

4.4 *Calibration of Analyzers That Have Simultaneous Thermogravimetry-Differential Scanning Calorimeter or Thermogravimetry-Differential Thermal Analysis Capability*—These instruments may be calibrated using melting temperature standards following Practice E 967.

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.

6.3 The atmosphere, purge gas type, purge gas flow rate, and heating will affect the calibration. These rates and conditions must be the same for both calibration and analysis. In addition, high heating rates should be avoided, if possible. Due to the differing heat exchange (emissivity and heat capacity) during the calibration and analysis, higher heating rates increase the eroor in the temperature measurement. The ICTAC Sixth International Test Program **(4)**[4] warns that heating rates above 6°C/min can produce errors in the temperature calibration.

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 *Thermobalance*, composed of:

7.1.1.1 *Furnace*, to provide uniform controlled heating of a specimen from 25°C to a constant temperature or at a constant rate within the applicable temperature range of this test method.

7.1.1.2 *Temperature Sensor,* to provide an indication of the specimen/furnace temperature to ± 0.1°C.

[4] The boldface numbers in parentheses refer to the list of references at the end of this practice.

7.1.1.3 A continuously recording *Balance,* to measure the specimen weight with a minimum capacity of 50 mg and a sensitivity of ± 5 µg.

7.1.1.4 A means of maintaining the specimen/container under *Atmospheric Control,* of nitrogen or other inert gas of 99.9+% purity at a purge rate of 50 to 100 mL/min constant to within ± 5 mL/min.

7.1.2 A *Temperature Controller,* capable of executing a specific temperature program by operating the furnace between selected temperature limits at a specified heating rate between 0.5 to 20°C/min constant to within ± 0.1°C/min or to an isothermal temperature that is maintained constant to ± 0.5°C for a minimum of 10 min.

7.1.3 A *Recording Device,* either analog or digital, capable of recording and displaying any fraction of the specimen weight (TGA thermal curve) including the signal noise, on the *Y*- axis versus any fraction of temperature, including signal noise, on the *X*- axis.

7.1.4 *Containers (pans, crucibles, and the like),* that are inert to the specimen and will remain dimensionally stable within the temperature limits of this test method.

8. Calibration and Standardization

8.1 *Calibration of Apparatus*—If necessary, calibrate the temperature sensor of the instrument at room temperature using the procedure described in the instrument manual.

8.2 *Calibration Materials*:

8.2.1 *Melting Point Standards*—For the temperature range covered by many applications, the melting transition of the 99.9+ % pure materials listed in Table 1 may be used for calibration.

NOTE 4—It is recommended that the size of the wire used be 0.25 mm in diameter. For sources of very pure fine metal wire, contact the ASTM Information Center.

NOTE 5—The melting temperatures of the first seven materials given in this table are taken from Mangum and Furukawa **(1)** and have been selected as primary fixed points for the International Temperature Scale of 1990. The remaining melting temperatures give in this table are taken from Bedford, Bonnier, Mass, and Pavese **(2)**.

8.2.2 *Magnetic Transition Standards*.

NOTE 6—Materials with known magnetic transitions determined with high precision are required. For sources of materials of known or certified Curie transition temperatures, contact the ASTM Information Center. The values for Curie transition temperatures may chnage from lot to lot of the material. Curie point temperatures given in the table were obtained from Refs. **(4, 3, 5)**.

TABLE 1 Recommended Melting Temperature Standards

Calibration Material	Melting Temperature, °C (K)
Indium[A]	156.5985 (429.7485)
Tin[A]	231.928 (505.078)
Zinc[A]	419.527 (692.677)
Aluminum[A]	660.323 (933.473)
Silver[A]	961.78 (1234.93)
Gold[A]	1064.18 (1337.33)
Copper[A]	1084.62 (1357.77)
Nickel[B]	1455 (1728)
Palladium[B]	1554.8 (1828.0)
Platinum[B]	1768.2 (2041.3)

[A] Primary fixed points, ITS-90 **(1)**
[B] Secondary reference points, ITS-90 **(2)**

9. Procedure A—Melting Point Standard Test for Horizontal Balance Types

9.1 *Positioning of the Temperature Sensor*—If the system employs a temperature sensor that is movable, it shall be located as close to the specimen as possible without touching it or the balance pan. In addition, it must be located in exactly the same position during calibrations as used during analytical determinations.

NOTE 7—This position may be inside or outside the balance pan.

9.2 *Action-Reaction Procedure*:

9.2.1 Flatten one end of a fine platinum wire (approximately 0.34-mm diameter and 2 cm in length), and spot weld it to the outside of a specimen container as shown in Fig. 2. Carefully bend the wire into a U shape so that the cantilevered end is located in the center of the specimen container.

9.2.2 Suspend this specimen container from the balance mechanism so that it hangs freely, and locate the temperature sensor as outlined in 9.1.

9.2.3 Bend a 5-mm length (0.25-mm diameter) of the wire temperature standard into a sigmoid shape, and suspend it from the end of the platinum wire in the middle of the specimen container.

NOTE 8—The selection of wire standard depends on the part of the temperature axis that is to be calibrated. Two or more standards may be run consecutively to enable one to obtain a calibration curve.

9.2.4 Close the balance assembly, and purge the system with the desired atmosphere at the selected rate. Select the appropriate heating rate.

9.2.5 Zero the balance and the recorder.

9.2.6 Open the system and carefully suspend a platinum mass of approximately 50 mg from the end of the wire standard.

NOTE 9—This mass can be prepared by tightly winding approximately 50 mm of 0.25-mm diameter platinum wire and distending one loop of the wire to provide a convenient connecting loop.

9.2.6.1 Close the system.

9.2.7 For analog systems using a chart recorder, adjust the *y*-axis pen recorder control sensitivity so that the pen is located about the middle of the chart paper.

9.2.8 Rapidly heat the system to 50°C below the theoretical melting temperature, and allow the system to equilibrate for 5 min. Then heat at the selected programmed rate up through the melting temperature of the standard. When the standard melts, the platinum mass falls into the specimen boat or pan. This produces an action-reaction blip on the recorded thermal curve without any mass loss. A typical thermal curve is shown in Fig. 3.

NOTE 10—The recorder action is fast and must ordinarily be observed at high sensitivity and data acquisition rates. The measurement of the transition temperature for the action-reaction blip is usually determined by manual means, since data analysis programs typically treat events of this nature as noise.

NOTE 11—Alloying of silver and gold with a platinum support and specimen boat or pan occurs over time, ultimately making the holder unusable.

9.2.9 Measure the temperature of the peak of the blip.

9.3 *Drop Procedure*:

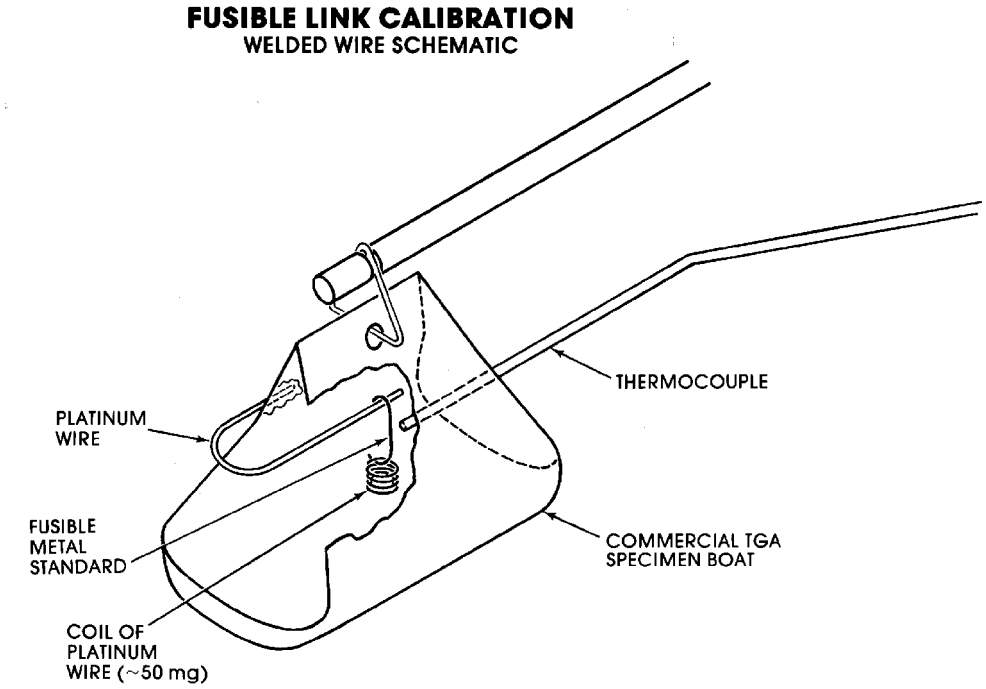
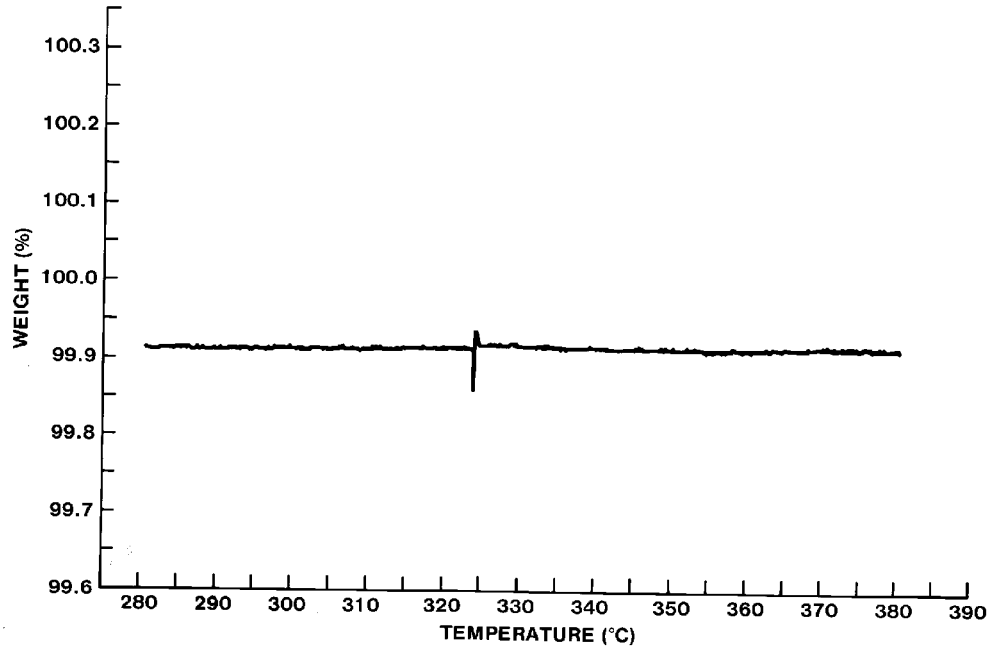


FIG. 2 Fusible Link Calibration; Welded Wire Schematic



NOTE—Sample: TGA STD LEAD CAL; Size: 44.55 mg; Rate: °C/min; N_2 at 50mL/min

FIG. 3 Typical Thermal Curve

9.3.1 Pierce a hole on both sides of the specimen container far up on the sides using a needle. Thread a short length of small diameter quartz rod (1 mm) or platinum wire through these holes so that the rod or wire is horizontal when the container is suspended. Alternatively, the container prepared in 9.2.1 may be used.

9.3.2 Cut a hole in the bottom of the specimen container, from the inside out, by placing it onto a soft surface and using a razor blade or sharp knife. See Fig. 4.

9.3.3 Suspend this specimen container from the balance mechanism so that it hangs freely, and locate the specimen temperature sensor as outlined in 9.1. See Note 7.

9.3.4 Bend a 5-mm length of the wire temperature standard into a sigmoid shape, and suspend it from the middle of the rod

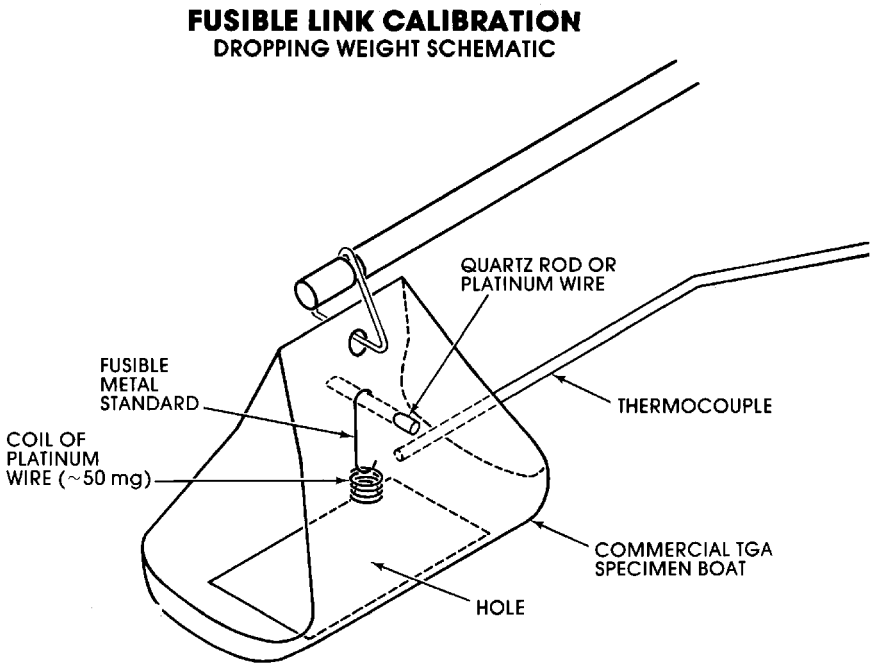


FIG. 4 Fusible Link Calibration; Dropping Weight Schematic

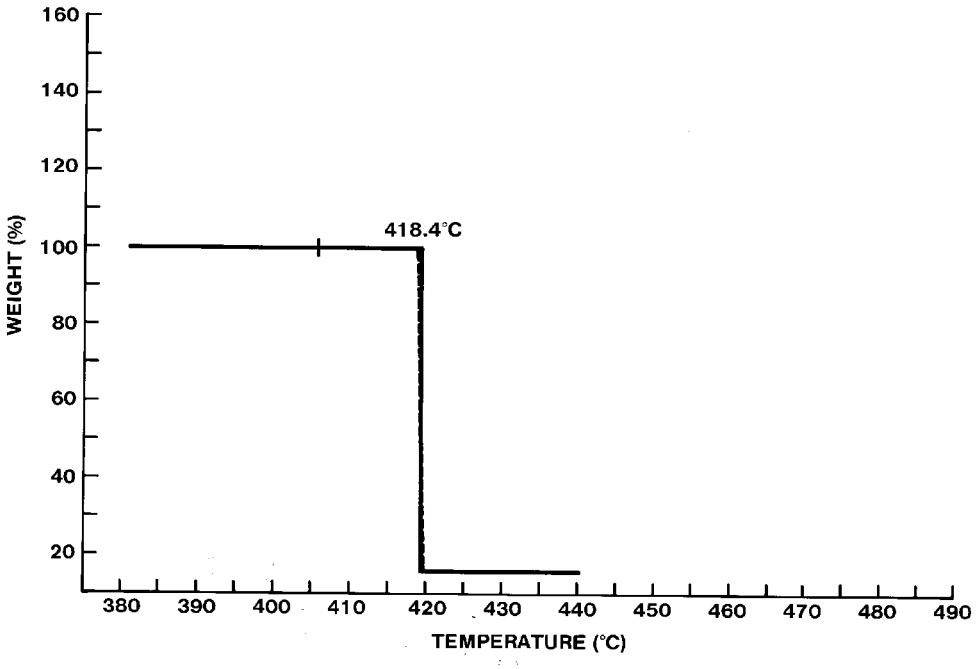
or wire. See Note 8 (9.2.3).

9.3.5 Continue as directed in 9.2.4-9.2.7.

9.3.6 Continue as directed in 9.2.8. When the standard melts, the platinum mass falls through the bottom of the boat or pan, causing a large mass loss (100 %). A typical thermal curve obtained using this experimental arrangement is shown in Fig. 5. The transition temperature may be found by determining the extrapolated onset temperature of the curve. Record this as the melting temperature of the standard material.

10. Procedure B—Melting Point Standard Test for Vertical Hang-Down Balances

10.1 *Positioning of the Temperature Sensor (Thermocouple)*:



NOTE—Sample: TGA STD ZINC WIRE; Size: 72.68 mg; Rate: °C/min; N_2 at 50mL/min

FIG. 5 Typical Thermal Curve-Using Experimental Arrangement–See 9.3.2

10.1.1 For those systems using internal microfurnaces with an adjustable temperature sensor, it shall be adjusted so that it is just beneath (but not touching) the thermogravimetric analyzer specimen container (see Fig. 6).

10.1.2 For those systems using external furnaces with an adjustable temperature sensor, where the measuring temperature sensor, by design, is fixed in the horizontal plane but movable in the vertical plane (see Fig. 7), the vertical position of the thermocouple shall be adjusted until it is even with the bottom of the thermogravimetric analyzer specimen container.

10.1.3 The thermocouple location described in 10.1.1 and 10.1.2 shall also be the same as that used during the analytical determinations.

10.2 *Action-Reaction Procedure*:

10.2.1 Bend a fine platinum wire (approximately 0.34-mm diameter and 1 cm in length), and suspend it from the hang-down wire and above the specimen container, as shown in Fig. 8. Ensure that the lowest end of the platinum wire is bent into a U shape and is located above and near the center of the specimen container.

10.2.2 See 9.2.3. Suspend the sigmoid-shaped calibration wire (0.25-mm diameter) from the end of the platinum wire above the specimen pan.

10.2.3 Close the balance assembly. Purge the balance and furnace tube with the desired atmosphere and at the selected flow rate. Select the heating rate that will be used in subsequent analyses. See 6.3.

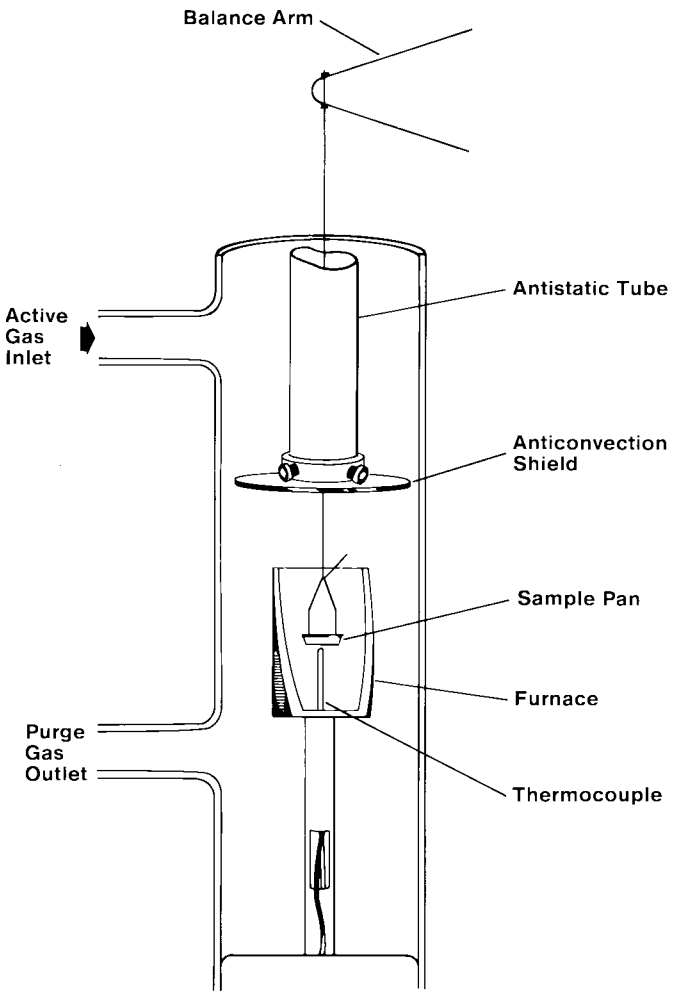


FIG. 6 Positioning of the Temperature Sensor (Thermocouple)–See 10.1.1.

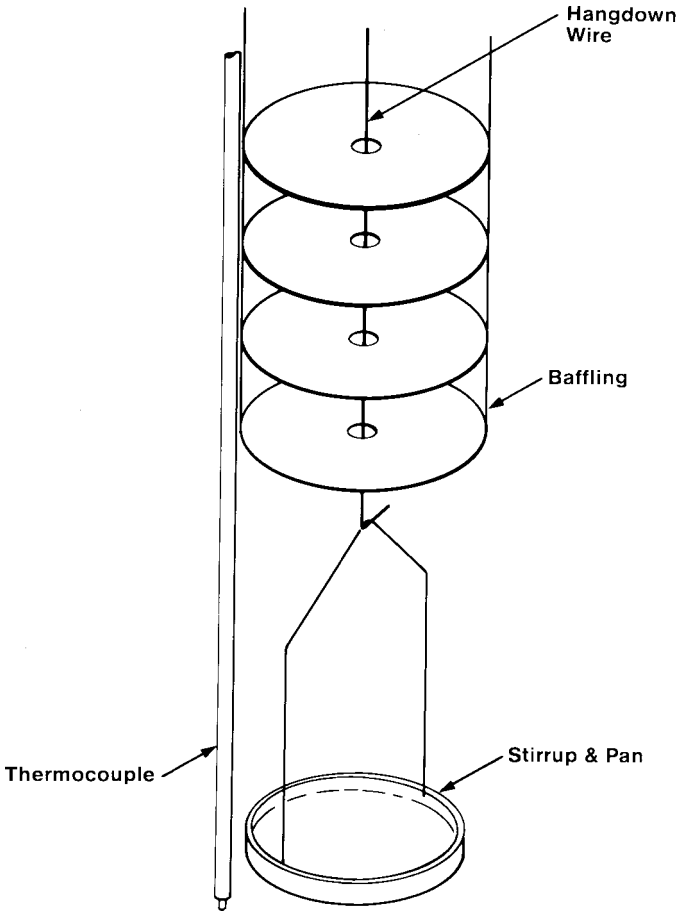


FIG. 7 Positioning of the Temperature Sensor (Thermocouple)–See 10.1.2.

10.2.4 Adjust the balance so that it now gives a zero mass signal.

10.2.5 Open the system and carefully;

10.2.6 Carefully suspend a platinum mass of approximately 50 mg from the freely hanging wire. See Note 8 (9.2.6). Close the system.

10.2.7 For analog systems using a chart recorder, adjust the *Y*-axis (mass or mass percent) pen position so that the mass of the suspended platinum weight that was added in 10.2.6 represents approximately 50 % of the ordinate scale.

10.2.8 Continue as directed in 9.2.8 and 9.2.9.

10.3 *Drop Procedure*:

10.3.1 Cut a large hole in the bottom of the specimen container. Use this specimen container as a replacement for the solid bottom container used in the action-reaction technique.

10.3.2 Using the specimen container with a hole in the bottom, follow the same procedure as that given for the action-reaction procedure in accordance with 10.2.

NOTE 12—In some cases, when using the external furnace, a larger pan/stirrup combination may be used. In this case, the dimensions of both the platinum wire and the metal wire temperature standard may be adjusted to meet the requirements of the larger pan/stirrup combination.

10.3.3 In this case, when the fusible link melts, the platinum

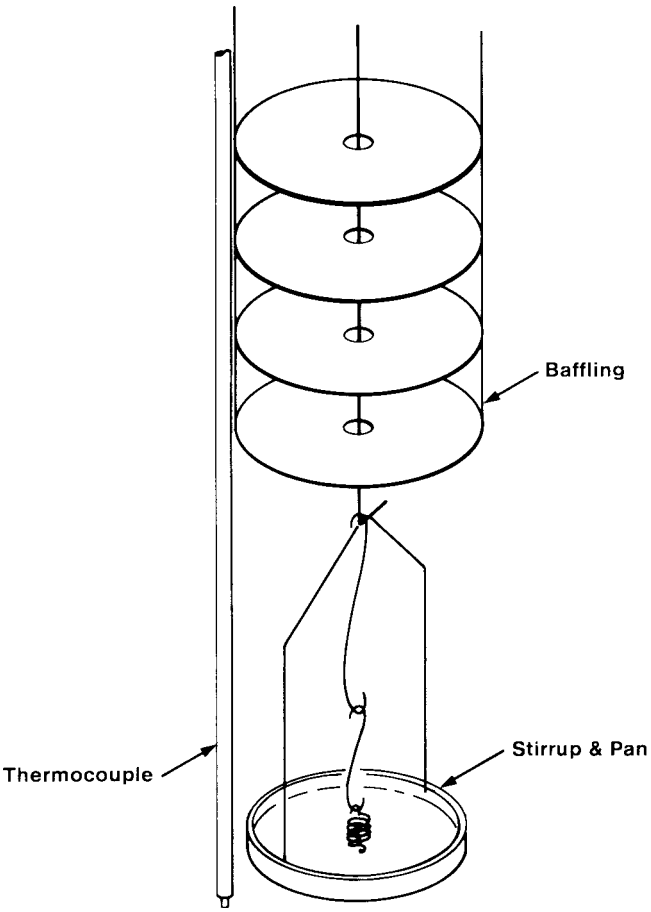


FIG. 8 Action-Reaction Procedure–See 10.2.1.

mass falls through the opening and a weight loss is observed. A typical thermal curve obtained using this experimental arrangement is shown in Fig. 5. Determine the extrapolated onset temperature of the curve. Record this as the melting temperature of the standard material.

11. Procedure C—Magnetic Transition Standard Calibration

11.1 Adjust the thermocouple position as described in 9.1 or 10.1.

11.2 Close the system, adjust the atmospheric flow rate to the selected rate, and zero the balance and chart recorder if used.

11.3 Open the system and place a specimen of the magnetic transition material in the specimen container in the same position as that in which one would place a specimen. Close the system.

11.4 Place a permanent magnet or electromagnet outside the furnace in a position as close to the furnace and specimen container as is practical in order to affect an apparent mass change at the reference material transition temperature. Remove (or turn off) the magnet to ascertain whether a conveniently measurable apparent mass change occurs.

NOTE 13—The choice of magnets or magnetic fields should be made so that at least a 2 % change in apparent mass is observed by turning the field on and off.

11.4.1 Adjust the specimen size or mass loss sensitivity, if necessary, and replace (or turn on) the magnet.

11.5 Rapidly heat the magnetic transition standard to a temperature at least 50°C below the transition temperature of the standard and hold that temperature for 5 min.

11.6 Heat the test specimen at the desired heating rate through the transition temperature region until an apparent mass change has been observed (T_x , Fig. 1).

NOTE 14—To allow full ramp equilibration prior to the transition, the scanning ramp should extend at least 5 min prior to the observed transition (hence, the 50°C mentioned would be appropriate for scanning rates below 10°C/min).

NOTE 15—More than one reference material can be used in one run. If the magnetic reference temperature of the reference material is sufficiently different from the mass change region of the test substance that the apparent and real mass changes do not interact, the reference material may be used during measurements on the test substance. Chemical interaction of any type with the sample may alter the temperature of the magnetic transition.

NOTE 16—The same specimen container and heating rates should be used in both the calibration procedures and the analytical determinations. See 6.3.

11.7 Determine the magnetic transition temperature by observing that point on the mass change curve that most closely agrees with that of T_x of Fig. 1. Computer or hand-made determination of this point can be made.

12. Calculation

12.1 For the purposes of these procedures, it is assumed that the relationship between observed temperature (*TO*) and actual specimen temperature (*T*) is a linear one governed by the following equation:

$$T = (TO \times S) + I \quad (1)$$

where:

S and I = slope and intercept, respectively.

(See 12.2 for the means to calculate *S* and *I*, used in (Eq 1)).

NOTE 17—For some instruments, the assumption of a linear relation between observed and actual specimen temperature may not hold. Under such conditions, calibration temperatures sufficiently close together shall be used so that the instrument calibration is achieved with a series of linear relations.

12.2 *Two-Point Calibration*:

12.2.1 Using the standard temperature values taken from Table 1 and Table 2 and the corresponding observed temperatures taken from the experimental sections above, calculate the slope and intercept using the following equations:

TABLE 2 Curie Temperature Standards

Metal	Curie Point (Magnetic) Transition, °C (K)
Alumel[A]	163 (436)
Permanorm 3[B]	266 (539)
Nickel[A]	354 (627)
MuMetal[B]	386 (659)
Permanorm 5[B]	459 (732)
Perkalloy[A]	596 (869)
Trafoperm[B]	754 (1027)
Iron[A]	780 (1053)
Hisat-50[A]	1000 (1273)
Cobalt[C]	1120 (1393)

[A]From Ref. **(3)**
[B]From Ref. **(4)**
[C]From Ref **(5)**.

$$S = (TS_1 - TS_2)/(TO_1 - TO_2) \quad (2)$$

$$I = [(TO_1 \times TS_2) - (TS_1 \times TO_2)]/(TO_1 - TO_2) \quad (3)$$

where:

S = slope (nominal value = 1.00),
I = intercept,
TS_1 = reference transition temperature for Standard 1 from Table,
TS_2 = reference transition temperature for Standard 2 from Table,
TO_1 = observed transition temperature for Standard 1 determined in either Section 9, 10, or 11,
TO_2 = observed transition temperature for Standard 2 determined in either Section 9, 10, or 11.

NOTE 18—I has the same units (that is, °C or K) as TS_1, TS_2, TO_1, and TO_2, which are consistent with each other. The value for I will be different, depending on the units used. S is a dimensionless number whose value is independent of the units of I and T.

12.2.2 When performing these calculations, retain all available decimal places in the measured value and in intermediate values in the calculation such as the values for S and I. The final calculated or corrected temperature should be rounded to the decimal place equivalent to two significant places in the standard deviation. If, however, this temperature is to be used in a subsequent calculation, all available decimal places should be retained.

12.3 *One-Point Calibration*—If the slope value determined in 12.2.1 is between 0.999 and 1.001, only the intercept need be determined through a one-point calibration procedure.

$$I = TS_1 - TO_1 \quad (4)$$

12.4 Using the determined values for S and I, (Eq 1) may be used to calculate the actual specimen transition temperature (T) from an observed transition temperature (TO).

13. Report

13.1 Report the following information:

13.1.1 Complete identification and description of the standard reference materials used for calibration, including source and purity.

13.1.2 Model number and description of the instrument used for calibration, including the location of the thermocouple.

13.1.3 Details of the procedure used for calibration, including description of the type of specimen boat or pan used and any departures from the described procedure.

13.1.4 Identification of the specimen atmosphere by gas composition, flow rate, and purity.

13.1.5 Heating rate used.

13.1.6 Statement of the dimensions, geometry, and materials of the specimen holder; and the method of loading the specimen.

13.1.7 A copy of all original records that are presented.

13.1.8 If derivative thermogravimetry is used, the method of obtaining the derivative should be indicated and the units of the ordinate specified.

13.1.9 The specific dated version of this test method used.

14. Precision and Bias

14.1 The precision of magnetic transition and melting point tests were determined by interlaboratory testing with the statistical analysis conducted in accordance with Practice E 691. In each case, one material was used for the interlaboratory test. Six laboratories participated in the melting point procedure. Four laboratories participated in the magnetic transition procedure[5].

14.2 *Melting Point Test*:

14.2.1 *Repeatability*—Duplicate determinations on two specimens of the same sample by the same analyst should not differ by more than 5.3°C.

14.2.2 *Reproducibility*—Duplicate determinations on two specimens of the same sample analyzed in different laboratories should not differ by more than 11.9°C.

14.3 *Magnetic (Curie Point) Transition Test*:

14.3.1 *Repeatability*—Duplicate determinations on two specimens of the same sample by the same analyst should not differ by more than 5.4°C.

14.3.2 *Reproducibility*—Duplicate determinations on two specimens of the same sample analyzed in different laboratories should not differ by more than 10.8°C.

15. Keywords

15.1 Curie Point standards; magnetic transition standards; melting point standards; temperature calibration; thermogravimetry

[5] A Research Report is available from ASTM. Request RR:E37–1017.

REFERENCES

(**1**) Mangum, B.W. and Furukawa, G.I., , *Guidelines for Realizing the International Temperature Scale of 1990 (ITS-90)*, National Institute of Standards and Technology Technical Note 1265, 1990, p. 8.
(**2**) Bedford, R.E., Bonnier, G., Mass, H., and Pavese, F, Recommended Values of Temperature on the International Temperature Scale of 1990 for a Selected Set of SEcondary Reference Points*Metrologia*, Vol. 33, 1996, pp. 133–154.
(**3**) Norem, S. D., O'Neill, M. J., and Gray, A. P., "The Use of Magnetic Transitions in Temperature Calibration and Performance Evaluation of Thermogravimetric Systems," *Thermochim. Acta*, Vol 1, 1970, p. 29.
(**4**) *ICTA Certificate GM-761*, ICTA Certified Reference Materials for Thermogravimetry certified by the International Confederation for Thermal Analysis, National Institute for Science and Technology, Gaithersburg, MD.
(**5**) Moskalewicz, R., Proceedings of the IV-th ICTA Conference, Budapest, Hungary, 1974.