



# Standard Guide for Measuring Electromotive Force (EMF) Stability of Base-Metal Thermoelement Materials With Time in Air<sup>1</sup>

This standard is issued under the fixed designation E601; 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 guide provides a method for measuring the emf stability of base-metal thermoelement materials in air referenced to platinum at specified constant elevated temperatures using dual, simultaneous, emf indicators, or using a single emf indicator, with the test and reference emf measured alternately. This test is conducted over a period of weeks.

1.2 A calibrated platinum-rhodium/platinum thermocouple is used as a reference standard to establish the test temperature.

1.3 The useful life of a thermocouple depends on the stability of the emf generated at given temperatures for a required time interval. This method provides a quantitative measure of the stability of individual thermoelements. By combining the results of the positive (P) and negative (N) thermoelements, the stability of a thermocouple comprised of both P and N thermoelements may be obtained. The emf of an individual thermoelement is measured against platinum, which may be the platinum leg of the platinum-rhodium/platinum reference thermocouple, or an additional platinum reference.

NOTE 1—Some thermoelements may show insignificant emf drift while undergoing relatively rapid oxidation. In these cases, failure of the thermoelement may be indicated only by a large rise in the electrical resistance between joined thermoelements, as measured at the reference junctions.

NOTE 2—See ASTM MNL12 for recommended upper temperature limits in air.<sup>2</sup>

NOTE 3—This guide is only applicable for initially new unused thermoelements. Base-metal thermoelements exposed to temperatures above 200 °C become thermoelectrically inhomogeneous, and stability testing of inhomogeneous thermoelements will give ambiguous results.

1.4 *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.*

1.5 *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:*<sup>3</sup>

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E230/E230M Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples

E344 Terminology Relating to Thermometry and Hydrometry

E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature

E1159 Specification for Thermocouple Materials, Platinum-Rhodium Alloys, and Platinum

2.2 *NIST Document:*<sup>4</sup>

Monograph 175-1118696454ee/astm-e601-20

## 3. Terminology

3.1 *Definitions:*

3.1.1 The definitions given in Terminology E344 shall apply to this guide.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *emf indicator, n*—an instrument that measures the emf and displays the value, for example, a digital voltmeter (DVM).

3.2.2 *emf stability, n*—change in emf (or in equivalent temperature) with time, with the thermocouple junctions held at fixed temperatures and with the thermal profile along the thermoelements held constant; this is also referred to in this standard as drift.

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<sup>2</sup> Park RM, et al., *Manual on the Use of Thermocouples in Temperature Measurement: Fourth Edition, ASM MNL12*, ASTM International, 1993. Available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428, www.astm.org.

<sup>3</sup> 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.

<sup>4</sup> Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

3.2.3 *gradient zone, n*—the section of a thermocouple that is exposed during a measurement to temperatures in the range from  $t_{\text{amb}} + 0.1(t_m - t_{\text{amb}})$  to  $t_{\text{amb}} + 0.9(t_m - t_{\text{amb}})$ , where  $t_{\text{amb}}$  is ambient temperature and  $t_m$  is the temperature of the measuring junction.

3.2.4 *half-maximum heated length, n*—the distance between the tip of the temperature sensor and the position along the length of the sensor leads or sheath where the temperature equals the average of the calibration-point and ambient temperatures.

3.2.5 *normalize, v*—to mathematically adjust experimental emf data acquired at a set of temperatures to values corresponding to a common reference temperature.

3.2.6 *reference thermocouple, n*—calibrated Type S or Type R thermocouple.

3.2.7 *test thermocouple, n*—thermocouple composed of the thermoelement being tested and the platinum reference thermoelement.

## 4. Summary of Guide

4.1 In this guide, the emf of a test thermocouple, comprised of a base-metal thermoelement relative to a platinum reference thermoelement, is determined as a function of time for a specified test temperature and thermal profile. If care is taken to maintain the chemical purity and annealed metallurgical state of the platinum thermoelement, the platinum will be thermoelectrically stable. In that case, variation in this emf value is attributed to instability of the base-metal thermoelement. The emf of the reference thermocouple ( $E_{\text{ref}}$ ) is used to measure the test temperature, and the emf of the test thermocouple ( $E_{\text{test}}$ ) is measured either simultaneously or alternately with  $E_{\text{ref}}$ . The test consists of the measurement of  $E_{\text{test}}$  at specified time intervals and at a specified constant value of  $E_{\text{ref}}$  which corresponds to a specified, constant temperature, until the required time of the test is exceeded or until an open circuit in the base-metal thermoelement results.

4.2 This guide is based on Method A of Test Method E220, where the reference thermocouple of Test Method E220 becomes the reference thermocouple used to measure the test temperature and one specified constant temperature replaces the series of measured temperatures.

## 5. Significance and Use

5.1 This test is important because the accuracy of a temperature measurement by a thermocouple is directly related to the emf stability of the thermoelements.

5.2 This test is used to verify that the tested thermoelements meet the intended requirements.

5.3 This test is useful in comparing the emf stability of two base metal thermoelements under the same conditions. The test and reference emf may be measured either simultaneously or alternately.

5.4 The relative stabilities of base metal thermoelements determined by this test are valid only under the specified test conditions. Results will be affected by changes in any of the following conditions: (1) temperature profile or gradient along

the length of the thermoelements, (2) abundance, velocity and composition of the air surrounding the test pieces, (3) thermoelectric inhomogeneity of the test thermoelements, and (4) stability of the platinum thermoelement.

5.5 The test does not address the determination of base metal thermoelement stabilities over a series of temperature changes.

5.6 The reliability of this test depends on the emf stability of the reference platinum thermoelement. For testing the relative emf stability of base-metal thermoelements, a reference element of platinum that has sufficient thermoelectric stability to determine any significant change in emf of base-metal thermoelements shall be used. To ascertain that the experimental method protects the platinum sufficiently from degradation, the method shall be validated by performing the procedure described in Appendix XI prior to the actual test.

5.7 The test result does not apply to applications in which the temperature distribution, for a given measuring junction temperature, changes with time.

## 6. Apparatus

6.1 *Thermocouple Used to Measure the Test Temperature*—A reference Type S or Type R thermocouple with 0.50 mm diameter (24 AWG) thermoelements or larger shall be used to measure the test temperature. The reference thermocouple shall consist of either standard tolerance or special tolerance wire in accordance with Table 1 in Specification E230/E230M. The choice of tolerance will not affect the determination of thermoelement drift. This thermocouple shall be of sufficient length to minimize the effect of heat conduction along the lengths of the wires upon the measuring junction temperature. (Platinum is a better heat conductor than most base metal thermocouple wires.) The length shall be sufficient to enable the reference thermocouple's measuring junction to be located within the test furnace's zone of nearly uniform temperature (refer to 6.5.2).

6.2 *Platinum Reference Thermoelement*—The emf of the test thermoelements shall be measured relative to a 0.50 mm diameter (24 AWG) platinum wire. This wire may be the platinum wire of the Type S or R reference thermocouple or a second 0.50 mm diameter (24 AWG) platinum wire. The length of this wire shall exceed that of the test specimen to minimize the transfer of heat from the measuring junction to the reference junction during testing (see 6.3). For more information concerning a platinum reference thermoelement see Specification E1159.

6.3 *Test Specimens*—The test specimens shall be lengths of wires, rods, ribbons, or strips of the coils or spools of the base-metal thermoelements to be evaluated. Their lengths shall be adequate to minimize the transfer of heat from the measuring junctions to the reference junctions during the period of testing. The lengths shall be at least 0.8 m (31 in.) depending on the length of the testing medium and the transverse sizes of the thermoelements. The specimens shall be free of kinks or other defects due to mechanical deformation, and shall be continuous without splices between the measuring and reference junctions.

**6.4 Reference Junction Temperature**—The reference junction ends of the test specimens, of the platinum reference element, if used, and of the reference thermocouple shall be maintained at a known constant temperature during a measurement cycle. The uncertainty attributable to the reference junction temperature shall be less than  $\pm 0.1$  °C. Ice point reference junction baths provide a relatively simple and reliable means for maintaining the reference junction at 0 °C (32 °F) when proper precautions are exercised in their use. Practice E563 provides an acceptable method for utilizing the ice point as a reference junction bath. Subsection 7.3 of Test Method E220 may be consulted for alternative methods of providing a reference junction temperature.

**6.5 Tube Furnace**—The test shall be conducted in an electrically heated tube furnace such as described in Subsection 7.2.3 of Test Method E220. The furnace tube shall be sufficient in length to permit a depth of immersion of the thermocouple measuring junctions assuring the measuring junctions is not affected by heat conduction along length of the thermoelements.

**6.5.1** Means shall be provided to control the temperature of the furnace to within  $\pm 10$  °C ( $\pm 18$  °F) of a nominal temperature during the performance of the test.

**6.5.2** The test shall be conducted in a uniformly heated furnace providing an isothermal work zone of sufficient size and stability to maintain all junctions at the same temperature.

**6.5.3** To determine the uncertainty resulting from temperature non-uniformities in the work zone, measure the temperature profile along the thermocouple axis in the vicinity of the work zone, using a platinum-rhodium alloy thermocouple or a platinum resistance thermometer prior to commencement of the test. If the furnace temperature is not sufficiently stable to obtain a temperature profile with a single thermometer, it may be useful to place one thermometer at a fixed half-maximum heated length, and to move a second thermometer along the furnace-tube axis. Adjust the readings of the moveable thermometer by adding the correction  $-(t_{\text{fixed}}(\text{time}) - t_{\text{fixed}}(\text{initial}))$ , where  $t_{\text{fixed}}$  is the temperature indication of the thermometer at fixed half-maximum heated length.

**6.5.3.1** A thermoelement extending from ambient temperature into an isothermal zone of a furnace will come to equilibrium with the temperature of the isothermal zone through radiative, convective, and conductive heat transfer between the thermoelement and the surrounding furnace environment. The distance of immersion,  $L_{\text{eq}}$ , into the isothermal zone required to achieve thermal equilibrium depends significantly on both the thermoelement diameter and its thermal conductivity. The characteristic length for a wire to achieve thermal equilibrium with its surroundings is given by the approximate correlation:

$$L_{\text{eq}} = (2.5 \text{ cm}) \left( \frac{d}{1 \text{ mm}} \right)^{1/2} \left( \frac{k}{100 \text{ W/(m}\cdot\text{K)}} \right)^{1/2} \quad (1)$$

where:

$k$  = the thermal conductivity of the thermoelement, and  
 $d$  = the diameter of the thermoelement.

Calculate the distance  $L_{\text{eq}}$  for each tested thermoelement in units of  $\text{W}/(\text{m}\cdot\text{K})$ , and  $d$  is the diameter of the thermoelement in millimeters. The equivalent equation in English units, with  $d$  in units of inches and  $k$  in units of  $\text{BTU}/(\text{hr}\cdot\text{ft}\cdot\text{°F})$  is:

$$L_{\text{eq}} = (1 \text{ in}) \left( \frac{d}{0.04 \text{ in}} \right)^{1/2} \left( \frac{k}{58 \text{ BTU}/(\text{hr}\cdot\text{ft}\cdot\text{°F})} \right)^{1/2} \quad (2)$$

Calculate the distance  $L_{\text{eq}}$  for each tested thermoelement and the platinum reference thermoelement. The approximate thermal conductivities listed in Table 1 may be used for this purpose. Measure the diameter  $d_{\text{mj}}$  of the measuring junction assembly (see 7.1). Identify the maximum  $L_{\text{max}}$  of the set of all calculated  $L_{\text{eq}}$  values and  $d_{\text{mj}}$ .

NOTE 4—Eq 1 was derived for a temperature of 200 °C, which is near the lower limit of observable thermoelement drift. For higher temperatures, the value of  $L_{\text{eq}}$  from Eq 1 will give an upper limit on the actual equilibration length.

**6.5.3.2** The standard uncertainty due to thermal non-uniformity is the maximum temperature variation in the profile from subsection 6.5.3 between the measuring junction location and a distance  $L_{\text{max}}$  away from the measuring junction.

**6.5.3.3** Alternative methods may be used to determine the standard uncertainty due to thermal non-uniformity, such as comparison of results in the test furnace with results obtained either in fixed-point cells or in a stirred liquid bath of high temperature uniformity; or numerical heat-transfer calculations.

**6.6 Electromotive Force Indicator**—The emf-measuring instrumentation shall have a measurement uncertainty of not more than  $1 \mu\text{V}$  at  $1000 \mu\text{V}$  and  $12 \mu\text{V}$  at  $50000 \mu\text{V}$  for this test. The emf indicators may be potentiometers or digital voltmeters. See subsections 6.2 and 7.4 of Test Method E220 for further discussions of thermal emf indicators and methods of emf measurement.

**6.7 Connecting Wires**—Connecting wires from the reference junctions to the emf indicator or indicators shall be electrically insulated copper. If the test is sensitive to electrostatic interference, the wires shall be electrically shielded. If electromagnetic interference is present, the conductors shall be twisted to minimize this effect.

**6.8 Selector Switches**—When more than one thermoelement is to be tested, a selector switch shall be introduced into the copper part of the circuit between the reference junctions and the thermal-emf indicators. These switches shall comply with subsection 7.5.1 of Test Method E220.

**6.9 Thermocouple Insulation**—For the segment of the thermoelements exposed to temperatures above ambient, ceramic tubing may be used to support and electrically insulate the test

**TABLE 1 Approximate Thermal Conductivities of Thermoelement Materials at 200 °C**

| Thermoelement Type | $k$ (W/(m · K)) |
|--------------------|-----------------|
| Pt                 | 72              |
| EP, KP             | 21              |
| EN, JN, TN         | 31              |
| JP                 | 62              |
| KN                 | 32              |
| NP                 | 19              |
| NN                 | 31              |
| TP                 | 380             |

thermoelement, the thermocouple used to measure the test temperature, and the platinum reference thermoelement, if used.

6.9.1 For the test thermoelements, the ceramic tubing shall be aluminum oxide ( $\text{Al}_2\text{O}_3$ ) with total impurities of less than 0.5 % (mass), and the maximum limit for specific impurities shall be 0.04 % (mass) for  $\text{Fe}_2\text{O}_3$ .

6.9.2 For the thermocouple used to measure the test temperature and for the platinum reference thermoelement, the ceramic tubing shall be aluminum oxide ( $\text{Al}_2\text{O}_3$ ) with total impurities of less than 0.5 % (mass), and the maximum limit for specific impurities shall be 0.04 % (mass) for  $\text{Fe}_2\text{O}_3$  and 0.08 % (mass) for Si.

6.9.3 To avoid unnecessary mass and to minimize axial heat conduction in the region of the measuring junction, the ceramic tubing should be relatively thin-walled and should have bore diameters of sufficient size to permit threading of the thermoelements without bending or straining them and to avoid binding. If possible the test thermocouple(s) and the reference thermocouple should be welded individually and then the measuring junctions welded all together to create a loose fitting bundle with a common measuring junction.

6.9.4 To minimize contamination, single lengths of ceramic insulation shall be used. Additionally, if using ceramics that were previously used, insert only thermocouples of the same type as previously used. Insert the positive and negative thermoelement into the bore previously used for that thermoelement to prevent cross contamination from previous testing.

## 7. Procedure

7.1 *Preparation of Thermocouples*—The thermoelement junction shall be prepared by welding, using a procedure proven by experience, or by testing, to produce junctions that are mechanically secure and electrically conductive at the test temperature for the life of the test.<sup>5</sup> The measuring junctions of all of the thermocouples may be welded together into a common bead to provide good thermal contact between the junctions of the different thermoelements. Weld a reference thermocouple to the test specimen to form a mechanically sound junction assembly. If a platinum wire other than the reference thermocouple platinum leg is to be used as the platinum reference then it shall be welded to the junction assembly. If it is not convenient to weld the junctions together, the junction of each thermocouple shall be welded separately and the junctions brought into good electrical and thermal contact by wrapping them with a thermally-conductive wire or platinum foil.

7.1.1 Electrically insulate the test specimens from each other, except at the junction, and insulate the reference thermocouple and the additional platinum leg from the test specimens with ceramic insulators (sufficiently loose to thread the thermoelements through the bore without bending or straining them). Slip the insulating tubes down on the thermoelements as close to the measuring junctions as possible without stressing the wires. Thermoelements may be inserted

into the insulating tubes before or after fabrication of the measuring junction. The number of test specimens joined to the platinum reference may be as many as the volume of the testing medium permits, provided that thermal conduction along the thermoelements does not impair isothermal conditions.

7.1.2 To prepare the reference junction, make the electrical connection between the individual legs of the test thermocouple and their respective copper leads using a screw or spring connector, or by soldering, welding, crimping, or any other suitable means. Then place these connections into individual clean glass, plastic, or metal tubes. If metal tubes are used, the thermoelements must be electrically insulated from the tubes, and the tubes shall be fabricated from stainless steel or another alloy that will not have excessive thermal conductance with the ambient environment. As stated in Test Method E220 care must be taken to keep thermal conduction losses within the limits of experimental error typically by immersing the thermocouple into the bath until no further change in indicated emf is noted. Completely clean finished junctions of any harmful contaminants, especially if any soldering or brazing fluxes have been used.

7.2 Heat the tube furnace to the test temperature. Insert the measuring junction end of the assembly into the furnace after the test temperature has been established, so that the junction is in the uniform temperature zone. Avoid bending or kinking of the wire in a zone of temperature gradient. Copper extension wires shall connect the emf-measuring instrument and reference junctions. Take care to ensure that the thermocouple assemblies do not move within the furnace with time in such a manner as to change the temperature distribution along the wires.

7.3 Make the initial emf readings as soon as the system has reached steady-state thermal conditions, especially if the recording of early drift is desired. Inspection of the variation of the reference thermometer readings will indicate whether the furnace and sensor elements have been adequately stabilized. The actual test temperature, as measured by the reference thermocouple, must be within  $\pm 10^\circ\text{C}$  of the nominal test temperature. If the reference thermocouple does not meet the  $\pm 10^\circ\text{C}$  of the nominal test temperature, the furnace control system will require adjustment and additional stabilization time to assure the system has once again reached the desired steady-state thermal condition. Measure and record the test temperature as indicated by the emf of the reference thermocouple. Measure and record the emf of the test specimens versus the platinum reference thermoelement while maintaining the test temperature. Measure the emf with the temperature changing at a rate not exceeding  $0.5^\circ\text{C}/\text{min}$  ( $1^\circ\text{F}/\text{min}$ ). A minimum of three consecutive readings that yield the same emf value within measurement uncertainty is required.

7.4 When using a single emf indicator to make measurements, either an automated method (Method A) or a manual method (Method B) may be used. Fig. 1 in Test Method E220 is a schematic of a typical data acquisition system using a thermocouple as the reference thermometer.

7.4.1 *Method A*—This method permits the rapid testing of any number of thermocouples. The thermocouples to be tested

<sup>5</sup> For more information on welding of measuring junctions, see *Manual on the Use of Thermocouples in Temperature Measurement: Fourth Edition, MNL12*.<sup>2</sup>