Designation: E601 − 07a(Reapproved 2013)

Standard Test Method for Measuring Electromotive Force (emf) Stability of Base-Metal Thermoelement Materials with Time in Air[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method measures 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 MNL 12 for recommended upper temperature limits in air.[2]

NOTE 3—This test method is only applicable for initially new 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E220 Test Method for Calibration of Thermocouples By Comparison Techniques

E230 Specification and 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

3. Terminology

3.1 *Definitions*—The definitions given in Terminology E344 shall apply to this test method.

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.

3.2.3 *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.

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

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

[1] This test method is under the jurisdiction of ASTM Committee E20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.04 on Thermocouples.

Current edition approved May 1, 2013. Published May 2013. Originally approved in 1977. Last previous edition approved in 2007 as E601 – 07a. DOI: 10.1520/E0601-07AR13.

[2] *Manual on the Use of Thermocouples in Temperature Measurement: Fourth Edition*, Available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428, www.astm.org.

[3] 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.

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

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

4. Summary of Test Method

4.1 In this test method, 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_{ref}) is used to measure the test temperature, and the emf (E_{test}) of the test thermocouple is measured either simultaneously or alternately with E_{ref}. The test method consists of the measurement of E_{test} at specified time intervals and at a specified constant value of E_{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 test method is based on Method A of Test MethodE220, 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 of Test Method E220.

5. Significance and Use

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

5.3 This test method 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 method are valid only under the specified test conditions. Results would be affected by changes in 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; (4) stability of the platinum thermoelement.

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

5.6 The reliability of this test method 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 X1 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 as per Table 1 in Specification E230. 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. (Note: platinum is a better heat conductor than most base metal thermocouple wires.) 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, Specification E1159 may be consulted.

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 test. The lengths shall be at least 0.8 m (30 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 must be maintained at a known constant temperature during a measurement cycle. The uncertainty attributable to the reference junction temperature shall be less than ±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. Section 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 Section 7.2.3 of E220. The furnace employed shall have the following capabilities: The furnace tube shall be long enough to permit a depth of immersion of the thermocouple measuring junctions that is sufficient to assure that the temperature of the measuring junctions is not affected by heat conduction along the thermoelements.

6.5.1 Means shall be provided to control the temperature of the furnace to within ±10 °C (±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 a nearly isothermal work zone sufficiently large 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 $-(t_{fixed}(\text{time}) - t_{fixed}(\text{initial}))$, where t_{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 into the isothermal zone needed 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:

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

where k is the thermal conductivity of the thermoelement, and d is the diameter of the thermoelement. Calculate the distance L_{eq} for each tested thermoelement in units of W/(m · 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 BTU/(hr·ft·°F) is:

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

Calculate the distance L_{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_{mj} of the measuring junction assembly (see 7.1). Identify the maximum L_{max} of the set of all calculated L_{eq} values and $d_{mj.}$

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_{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 Section 6.5.3 between the measuring junction location and a distance L_{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.

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

6.6 *Electromotive Force Indicator* —The emf measuring instrumentation shall have a measurement uncertainty of not more than 1 µV at 1 000 µV and 12 µV at 50 000 µV for this test. The emf indicators may be potentiometers or digital voltmeters. Sections 6.2 and 7.4 of Test Method E220 may be consulted 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 is introduced into the copper part of the circuit between the reference junctions and the thermal-emf indicators. These switches shall comply with 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 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 (Al_2O_3) with total impurities of less than 0.5 % (mass), and the maximum limit for specific impurities shall be: 0.04 % (mass) for Fe_2O_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 (Al_2O_3) with total impurities of less than 0.5 % (mass), and the maximum limit for specific impurities shall be: 0.04 % (mass) for Fe_2O_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