Designation: E220 – 13

Standard Test Method for Calibration of Thermocouples By Comparison Techniques[1]

This standard is issued under the fixed designation E220; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method describes the principles, apparatus, and procedure for calibrating thermocouples by comparison with a reference thermometer. Calibrations are covered over temperature ranges appropriate to the individual types of thermocouples within an overall range from approximately −195 to 1700 °C (−320 to 3100 °F).

1.2 In general, this test method is applicable to unused thermocouples. This test method does not apply to used thermocouples due to their potential material inhomogeneity—the effects of which cannot be identified or quantified by standard calibration techniques. Thermocouples with large-diameter thermoelements and sheathed thermocouples may require special care to control thermal conduction losses.

1.3 In this test method, all values of temperature are based on the International Temperature Scale of 1990. See Guide E1594.

1.4 *This standard may involve hazardous materials, operations and equipment. 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 requirements prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers
E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples
E344 Terminology Relating to Thermometry and Hydrometry
E452 Test Method for Calibration of Refractory Metal Thermocouples Using a Radiation Thermometer
E563 Practice for Preparation and Use of an Ice-Point Bath as a Reference Temperature
E644 Test Methods for Testing Industrial Resistance Thermometers
E1129/E1129M Specification for Thermocouple Connectors
E1594 Guide for Expression of Temperature
E1684 Specification for Miniature Thermocouple Connectors
E1751 Guide for Temperature Electromotive Force (EMF) Tables for Non-Letter Designated Thermocouple Combinations (Withdrawn 2009)[3]
E2846 Guide for Thermocouple Verification

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 *check standard, n*—a measurement instrument or standard whose repeated results of measurement are used to determine the repeatability of a calibration process and to verify that the results of a calibration processes are statistically consistent with past results.

3.2.2 *isothermal block, n*—a piece of solid material of high thermal conductivity used to promote thermal equilibrium between two or more thermometers.

3.2.3 *reference junction compensation, n*—the adjustment of the indication of a thermocouple such that the adjusted indication is equivalent to the emf or temperature that the thermocouple would indicate if the reference junctions were maintained at 0°C.

3.2.3.1 *Discussion*—In most cases, the thermocouple indication is adjusted by measuring the temperature of a terminal block where the thermocouple is connected, and then adding to the thermocouple emf an additional emf equal to the emf of the

[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 Nov. 1, 2013. Published December 2013. Originally approved in 1963. Last previous edition approved in 2007 as E220 – 07A. DOI: 10.1520/E0220-13.

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

thermocouple reference function evaluated at the temperature of the terminal block. Because the emf-temperature relationship of any actual thermocouple differs slightly from that of the reference function, reference junction compensation typically introduces higher uncertainties compared to the use of a well-prepared ice bath.

3.2.4 *reference junction compensator, n*—a device that implements reference junction compensation.

3.2.5 *reference thermometer, n*—thermometer that establishes the value of temperature in a given system containing additional temperature sensors.

3.2.5.1 *Discussion*—In a calibration system the reference thermometer is a calibrated thermometer capable of indicating values of temperature with known uncertainty. The reference thermometer provides the standard temperature for the system at the time of test.

3.2.6 *thermocouple type, n*—a standardized thermoelectric class of thermoelement materials that, used as a pair, have a normal relationship between relative Seebeck emf and temperature.

3.2.6.1 *Discussion*—For common, commercially available thermocouples, a thermocouple type is identified by a letter designation (types B, C, E, J, K, N, R, S, and T). The letter designation scheme is given in Guide E2846. The tables in E1751 and E1751 give temperature-EMF relationships for a number of additional thermocouple compositions that are not identified by a letter designation.

4. Summary of Test Method

4.1 Comparison calibration consists of measuring the emf of the thermocouple being calibrated in an isothermal medium while simultaneously measuring the temperature of the medium with a reference thermometer. The reference thermometer may be any thermometer with sufficient accuracy at the temperature of calibration.

5. Significance and Use

5.1 For users or manufacturers of thermocouples, this test method provides a means of verifying the emf-temperature characteristics of the material prior to use.

5.2 This test method can be used to calibrate a thermocouple for use as a reference, or it can be used to calibrate thermocouples representing a batch of purchased, assembled thermocouples.

5.3 This test method can be used for the verification of the conformance of thermocouple materials to temperature tolerances for specifications such as the tables in Specification E230 or other special specifications as required for commercial, military, or research applications.

6. Interferences

6.1 Since the success of this test method depends largely upon the ability to maintain the measuring junction of the thermocouple being calibrated and the reference thermometer at the same temperature, considerable care must be taken in choosing the media and conditions under which the comparisons are made. Stirred liquid baths, uniformly heated metal blocks, tube furnaces, and dry fluidized baths, properly used, are acceptable temperature comparison environments. In the case of large diameter thermoelements and sheathed thermocouples, special attention must be given to effects of thermal conduction.

6.2 Voltage measurement instruments with sufficiently high input impedance must be used for measuring thermocouple emf to eliminate instrument loading as a significant source of error. The ratio of input impedance to thermocouple loop resistance should be significantly (at least 10^4) greater than the ratio of the measured emf to the desired emf uncertainty.

6.3 The test method relies on the assumption that test thermoelements are homogeneous. If so, their output voltage at a given measuring junction temperature is independent of temperature variations along the length of the thermocouple. Departures from this ideal contribute to uncertainty in the use of test results. The effects typically are negligibly small for new, unused thermocouple material, but not for used thermocouples, especially those of base-metal composition. The effects of inhomogeneity can be identified, but not accurately quantified, by the techniques described in Appendix X4 in this test method and in section 8.2 of Guide E2846. Descriptions of the testing of used thermocouples may be found Guide E2846 and Manual MNL 12 (**1**).[4]

6.4 This test method presumes that the tested thermocouples are suitable for use in air throughout the range of calibration temperatures. To avoid oxidation of the thermoelements, refractory-metal thermocouples that have not been hermetically sealed in a sheath suitable for use in air should be tested in an inert gas environment at temperatures above approximately 500 °C. In this case, use of this test method is recommended in combination with the furnaces and related procedures described in Test Method E452.

7. Apparatus

7.1 The choice of apparatus used for the comparison test will depend primarily on the temperature range to be covered and on the desired calibration uncertainty. The apparatus required for the application of this test method will depend in detail upon the temperature range being covered but in all cases shall be selected from the equipment described as follows:

7.2 *Comparison Baths and Furnaces*—A controlled temperature comparison medium (bath or furnace) shall be used in which the measuring junction of the thermocouple to be calibrated is brought to the same temperature as a reference thermometer. The spatial uniformity of temperature within the nominally isothermal calibration zone shall be established. Acceptable methods include measurements of the calibration zone at the time of testing or the use of control charts that display the periodic calibration of check standards or the periodic characterization of the calibration zone. The frequency of such testing will depend on the inherent stability of the bath or furnace. The uniformity of the calibration zone shall be remeasured sufficiently often such that any deviations in

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

uniformity may be corrected prior to significant adverse affect on the readings. All thermocouples being calibrated and the reference thermometer must be immersed into this zone to an extent sufficient to ensure that the measuring junction temperature is not significantly affected by heat conduction along the thermocouple and reference thermometer assemblies. To avoid contaminating the thermoelements and insulation of unsheathed thermocouples, direct contact with calibration bath fluids should be avoided.

7.2.1 *Liquid Baths*—In the range from −150 to 630 °C (−240 to 1170 °F) the comparator bath shall usually consist of a well stirred liquid bath provided with controls for maintaining a constant and uniform temperature. Suitable types are described in the appendix to Test Method E77. At the liquid nitrogen boiling point, −196 °C (−321 °F), an isothermal block of copper suspended in an open dewar of liquid nitrogen can provide a very effective single-point liquid bath. In the range between −196 °C (−321 °F) and −150 °C (−240 °F), the bath construction is relatively complex, and commercial systems that rely on liquid nitrogen for cooling are recommended. A properly constructed liquid bath will have temperature gradients that are small relative to either fluidized powder baths or tube furnaces. A disadvantage of liquid baths is the relatively small operating range of any one bath fluid. The temperature gradients in a liquid bath will be repeatable provided that the bath liquid does not thermally decompose at high temperatures and that the conditions of bath heating and cooling are comparable to those that existed when the bath gradients were characterized. Periodic evaluation of bath gradients is necessary when using oil baths, since oil viscosity can increase significantly after use at high temperatures. Baths with multiple heaters require a monitoring system that enables the user to readily determine that all heaters are operational.

7.2.2 *Fluidized Powder Baths*—In the range from −70 to 980 °C (−100 to 1800 °F) the comparator bath may consist of a gas-fluidized bath of aluminum oxide or similar powder. Temperature equalizing blocks are almost always necessary within fluidized baths to minimize spatial and temporal temperature variations. The repeatability of thermal gradients within such a block depends on maintaining a constant fill level of powder in the bath and maintaining a uniform gas flow through the powder. The thermal gradients of a fluidized powder bath shall be verified by including either a second reference thermometer or a check-standard thermocouple in each comparison test.

7.2.3 *Tube Furnaces*—At temperatures above approximately 620 °C (1150 °F) an electrically heated tube furnace with a suitable nominally isothermal zone will usually be used. Laboratory type tube furnaces may be used at any temperature provided that the increased uncertainty due to their spatial temperature variance is accounted for. Any one of a wide variety of designs may be suitable, but it shall be demonstrated that the furnace chosen can maintain a temperature stability of ±1 °C over a period of 10 min at any temperature in the range over which the furnace is to be used. The axial temperature profile of a tube furnace shall be mapped to determine the location of the region with the best temperature uniformity. Furnaces with multiple heaters require a monitoring system that enables the user to visually determine that all heaters are operational and will require periodic remeasurement of the axial temperature profile. Single-zone furnaces may vary in temperature profile slowly as the heater element ages and will require only infrequent remapping of the temperature profile.

NOTE 1—Further discussions of suitable tube furnaces are given in Appendix X1.

7.2.4 *Other Baths*—The one essential design feature of any bath to be used with this test method is that it brings the measuring junction of the thermocouple being calibrated to the same temperature as the reference thermometer. Copper blocks immersed in liquid nitrogen have been used successfully at low temperatures. The blocks are provided with wells for the test thermocouples and the reference thermometer. Similarly, uniformly heated blocks have been used at high temperatures. Such baths are not excluded under this test method, but careful explorations of existing temperature gradients must be made before confidence may be placed in such an apparatus.

7.2.5 *Isothermal Blocks*—The use of an isothermal block can substantially reduce the temperature differences between the reference thermometer and the test thermocouples. Such a block should be manufactured from a material of high thermal conductivity that will not contaminate the thermocouples under test. High thermal conductivity reduces the spatial temperature variations in the block, resulting in better thermal equilibrium between the reference thermometer and the test thermocouples. An isothermal block may also be used to reduce temporal fluctuations of the thermometers. The fluctuations will decrease as either the heat capacity of the block is increased or the heat transfer to the surrounding furnace or bath is decreased. A consequence of this decrease in fluctuations is an increase in the time for the isothermal block to reach a steady-state temperature, so care must be exercised that the block is neither too large nor too well insulated. The temperature differences between the test thermocouples and the reference thermometer should be evaluated over the full temperature range of the apparatus by performing calibrations of check-standard thermocouples at a variety of immersions in the block and with the various thermometers inserted into different bores of the block. Similar temperature differences should also be measured as a function of time, following an adjustment of the furnace or bath temperature, to determine the length of time needed to reach thermal steady-state following a temperature change. Welding the measuring junctions of the test thermocouples and of a thermocouple used as a reference thermometer is a special case of an isothermal block formed by the common measuring junction.

7.3 *Reference Junction Temperatures*— A controlled temperature medium in which the temperature of the thermocouple reference junctions is maintained constant during a measurement cycle at a known or measured value shall be provided. A commonly used reference temperature is 0 °C (32 °F), usually realized through use of the ice point, but other temperatures may be used if desired. Reports of data taken with reference temperatures other than the ice point should be corrected to reflect the results that would have been obtained if the reference junction had been at the ice point, and the report shall state both the reference junction temperature and whether the correction is based on the reference function for that type of thermocouple or on the emf-versus-temperature response of the particular thermocouple under test. As an alternative, calibration data taken with a reference junction temperature other than the ice point may be reported without correction, but in such cases the calibration report must clearly state the actual reference junction temperature. With the exception of thermocouples that have very small Seebeck coefficients at the reference junction temperature (such as Type B thermocouples), a large uncertainty in the reference junction temperature will introduce a corresponding large uncertainty in the thermocouple calibration. Whatever reference junction technique is used, its uncertainty must be accounted for in the uncertainty of the thermocouple calibration being performed. An uncertainty in the reference junction temperature, $u(t_{\mathrm{ref}})$, will introduce an uncertainty in the measured temperature, $u_{rj}(t)$:

$$u_{rj}(t) = (S(t_{ref})/S(t))\, u(t_{ref})$$

where: $S(t_{ref})$ and $S(t)$ are the Seebeck coefficients of the thermocouple at temperatures t_{ref} and t, respectively.

7.3.1 *Method to Form a Reference Junction*—Reference junctions are formed by electrically connecting the ends of the thermoelements opposite the measuring junction to copper wires that lead to the emf measuring device. Any method that gives a reliable electrical connection may be used. The connection may be formed by welding, brazing, or soldering the wires, by a screw clamp, by crimping the wires together, or by a spring-loaded connector. Completely clean finished junctions of any harmful contaminants, especially if any soldering or brazing fluxes have been used.

7.3.2 *Use of an Ice-Point Bath to Maintain Reference Junction Temperature*—An acceptable method for utilizing the ice point as a reference junction is given in Practice E563. Each joined thermoelement/copper-wire pair leading to a reference junction shall be immersed in the reference junction bath to a sufficient depth such that the reference junction is in thermal equilibrium with the ice-point bath. The wires shall not be immersed directly into the ice-point bath, but shall be kept dry. For this purpose, a glass, plastic, or metal protection tube is usually used to contain the thermoelement/copper pair. If a metal protection tube is used, the wires shall be fully electrically insulated from the metal tube. In all cases, the thermoelements and the copper wires above the junction itself shall be insulated with a water-resistant insulation. If more than one reference junction is placed into a single protection tube, the junctions shall be electrically insulated from each other. A test to determine an appropriate depth of immersion into the ice-point bath is as follows: Prepare two ice-point baths, one containing the measuring junction of a thermocouple in an appropriate protection tube, and the other containing the reference junctions. Record the emf of the thermocouple at varying immersions of the reference junctions, allowing 10 min for thermal equilibration after each change of depth. A plot of emf-versus-immersion depth will show the minimum immersion depth necessary such that further increases in depth give negligible changes in emf. To promote good thermal equilibrium between the ice bath and the reference junctions, it is desirable to use copper leads of 0.5 mm (24 AWG) diameter or less, and to use protection tubes with the smallest inner diameter that still permits ready insertion of the reference junctions.

7.3.3 *Isothermal and Electronic Reference Junction Compensation*—For the rapid calibration of large numbers of thermocouples, the reference junctions can be made at an isothermal multiterminal strip. This avoids the thermal loading of the ice bath resulting from the large number of thermocouple and copper connecting wires. The temperature and isothermal condition of the strip shall be established and monitored by the use of a separate, reference temperature sensor. The spatial temperature variation across the terminations on the isothermal unit shall be mapped and accounted for. If desired, the thermocouple emf values obtained with use of an isothermal terminal strip may be compensated such that the compensated emf is equivalent to the thermocouple emf created by a thermocouple with reference junctions at 0°C (32°F). An electronic reference junction compensator accomplishes this task by accurately monitoring the temperature of the reference junctions and adding to the thermocouple emf an additional emf such that the sum is equivalent to the thermocouple emf produced with reference junctions at 0°C. The addition of emf to the thermocouple emf may be accomplished through software methods, as well as through addition of an actual emf. To minimize the uncertainty of an electronic reference junction emf as a source of error, the temperature equivalent of the emf produced by the electronic reference junction shall be known and measured with uncertainty less than that expected from the thermocouple calibration.

7.3.4 *Extension of Thermoelements to Reference Temperature* —Whenever possible, the thermoelements under test shall be continuous, extending from the measuring point through the temperature gradient, to the reference junction without any intermediate connections. In cases where this is not possible, several options exist:

7.3.4.1 *Matched Thermoelements*—Additional lengths of thermoelement materials from the same wire lots as those being calibrated may be used to extend the device under test to the reference bath. In such circumstances, no additional corrections are required.

7.3.4.2 *Thermoelements of the Same Type with Known Thermoelectric Response, but from a Different Lot*—Thermoelements being calibrated may be extended using thermoelement materials of the same type as the thermocouple under test. Such materials may be of thermocouple or extension grade, but shall have a known emf versus temperature relationship over the temperature interval to which they will be subjected, and corrections for the deviations of the extension material relative to the material under test over that interval shall be made. In general, it will be necessary to calibrate the test wire in the temperature range spanned by the extension wire and to measure the temperature of the junctions between the different materials in order to make this correction. It is acceptable to not apply a correction if the uncertainty budget for the calibration includes an appropriate allowance for temperature variations of the junction between the thermocouple and the extension material, and the calibration report specifies the range of transition junction temperatures for which the calibration is valid. This allowance may be experimentally determined by maintaining the measuring junction of the thermocouple at a fixed temperature, such as 0°C, and varying the temperature of the transition junction over a specified range.

7.3.4.3 *Thermocouple Connectors*—In all cases where there are junctions between the thermocouple under test and thermocouple lead wires, the temperature variations across the junctions shall be minimized. Thermocouple connectors as described in Specifications E1684 and E1129/E1129M will introduce no more than 1.1°C (2°F) error for a 40°C (70°F) temperature difference across the connector. This error will be proportionately reduced for smaller temperature differences.

7.3.4.4 *Circumstances with Small Temperature Differences*—In special cases where the temperature differences from end-to-end along the length of the wires used to extend a thermocouple for calibration purposes are very small (less than ±2°C), thermocouple or extension grade wires of matching thermocouple type may be used in calibration circuits without correction.

7.4 *Emf-Measuring Instruments*—The choice of a specific instrument to use for measuring the thermocouple emf will depend on the accuracy required of the calibration being performed. Generally the thermocouple emf will be measured using a digital voltmeter. For the highest level of accuracy, voltmeters shall have a maximum uncertainty no greater than 10^{-4} times the emf reading and shall have input impedances larger than the thermocouple loop resistance by at least a factor of 10^4. Reference junction compensation is required for thermocouple measurement with voltmeters. In order to avoid forming unintended reference junctions at voltmeter terminals whose temperature may be poorly controlled, thermocouples must not be connected directly to the input terminals of voltmeters without the use of appropriate electronic reference junction compensation and connection of the voltmeter to the compensator with untinned copper wires.

7.5 *Connecting Wire Assembly*—Connecting wires from the reference junction to the voltmeter shall be insulated copper and shall be configured as twisted pairs for wire lengths greater than 0.3 m (1 ft.), to reduce electromagnetic noise pickup. If the environment contains substantial electromagnetic noise, it may also be useful to run the wires in a grounded electrical shield or braided cable. Copper connections should be clean and free from oxides.

7.5.1 Scanner systems may be used to switch between the reference thermometer and the different thermocouples being calibrated. Such switches shall be of rugged construction and designed so that both connecting wires are switched when switching from one thermocouple to the next, leaving thermocouples not in use electrically isolated. All of the scanner switches shall be constructed of the same material and shall be free of extraneous emf production (see Appendix X3). Precautions should be taken to protect the switches from temperature fluctuations due to convection, conduction, or radiation. Scanning performance shall be evaluated to ensure adequate settling time before measurement.

7.5.2 It is preferable to use wire-to-wire connections in calibration circuits, but if terminal blocks are used for convenience, they shall be protected against the development of temperature gradients across the blocks.

7.6 *Thermocouple Insulation and Protection Tubes* —In the case where bare wire thermocouples are tested, two-hole insulation tubing may be used to support and electrically insulate the immersed portion of the two bare thermoelements. Use only insulation material that will not contaminate the thermocouple (for example, clean, high-purity insulators such as 99.8 % aluminum oxide) and that will provide the necessary electrical insulation at the highest temperature of the calibration. To prevent contamination of thermocouples by residues left by previously tested thermocouples, each insulator shall only be used with thermocouples of one type and the positive and negative thermoelements shall always be inserted in the same bore. The only exceptions allowed are: type R and type S thermocouples may be calibrated in the same insulators, and the thermoelements of type B thermocouples may be mounted in either bore. To avoid unnecessary mass and to minimize axial heat conduction in the region of the measuring junction, the tubing should be relatively thin walled. Bore diameters should provide a loose fit for the thermocouple wires. During the test, the thermocouples may be inserted in a protection tube that is resistant to thermal shock, and noncontaminating to the thermocouple materials.

7.6.1 Sheathed thermocouples may be tested without further protection or support in liquid or dry fluidized baths, provided that the bath medium is compatible with the sheath material. Thermocouples insulated with fibrous insulation must not be immersed directly into any bath liquid. 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 indication in temperature change is noted.

8. Reference Thermometers

8.1 The reference thermometer to be used for the comparison calibration of thermocouples will depend upon the temperature range covered, the type of calibration apparatus, the accuracy desired, or in cases where more than one type of thermometer will suffice, the preference of the calibrating laboratory. All reference thermometers shall be calibrated to indicate values of temperature corresponding to the International Temperature Scale of 1990. The condition of the reference thermometer shall be verified both before and after a calibration or a documented number of calibrations by checking its indication at a thermometric fixed point or by using a comparison measurement of total uncertainty less than the allowed uncertainty of the reference thermometer. Specific methods of verification for each type of reference thermometer are described in 8.10.

8.2 *Platinum Resistance Thermometers*— Platinum Resistance thermometers are an excellent choice as a reference in cases where the highest accuracy is required. Standard platinum resistance thermometers (SPRTs) are the most accurate reference thermometers for use at temperatures from approximately −196 to 962 °C (−310 to 1764 °F), with calibration uncertainties as low as 0.001 °C (0.002 ° F). SPRTs must meet a set of criteria specified by the ITS-90. In addition, there are a variety of platinum resistance thermometers that do not meet the criteria for SPRTs that have sufficient accuracy for use as a reference thermometer with this test method. Standard platinum resistance thermometers are described in X2.1, other platinum resistance thermometers are described in X2.2, and measurement instruments are described in X2.3.

8.3 *Thermistors*—For temperatures in the approximate range −40 °C (−40 °F) to 150 °C (300 ° F), a thermistor may serve as a reference thermometer with uncertainty of 0.001 to 0.01 °C. Section X2.5 provides additional information.

8.4 *Liquid-in-Glass Thermometers*—Liquid-in-glass thermometers may be used from –80 °C (–110 °F), or lower, to 400 °C (750 °F), or even higher with special types. Generally, the accuracy of these thermometers is less below −60 °C, where organic thermometric fluids are used, and above 400 °C where dimensional changes in the bulb glass may be relatively rapid, requiring frequent calibration. Further discussion of liquid-in-glass thermometers is given in X2.4. Specifications for ASTM thermometers are given in Specification E1.

8.5 *Types R and S Thermocouples (Platinum-Rhodium versus Platinum)*—The platinum-10 % rhodium versus platinum (Type S), or the platinum-13 % rhodium versus platinum thermocouple (Type R) of 0.5-mm (24-gauge) diameter wire is recommended as the reference thermometer for temperatures from 960 °C (1760 °F) to 1200 °C (2190 °F). Their use may also be extended down to room temperature. Uncertainties attainable with careful use are given in Tables 1 and 2.

8.6 *Type B Thermocouples (Platinum-Rhodium versus Rhodium-Platinum)*—The platinum-30 % rhodium versus platinum-6 % rhodium (Type B) thermocouple, of 0.5-mm (24-gauge) or larger diameter wire, is recommended as the reference thermometer for temperatures above 1200 °C (2190 °F). The uncertainties of temperature measurements with this type of thermocouple are given in Tables 1 and 2.

TABLE 1 Calibration Uncertainties in Calibrating Thermocouples by the Comparison Method—Temperatures in Degrees Celsius[A] (see Refs. 2 and 3)

	Temperature [°C]	Expanded Uncertainty (k=2) [°C]
Base metal thermocouples[A] (in a tube furnace by comparison with a calibrated Type S thermocouple	200	0.2
	400	0.4
	600	0.6
	800	0.7
	1000	0.9
	1200	1.0
Base metal thermocouples[A] (in stirred liquid baths, by comparison with an SPRT)	−196 (type E)	0.2
	−100 (type E)	0.1
	0	0.02
	200	0.2
	400	0.4
	500	0.5
Type R and S thermocouples (in a tube furnace, by comparison with a calibrated Type S thermocouple)	200	0.2
	400	0.2
	600	0.3
	800	0.3
	1000	0.3
	1100	0.2
Type B thermocouples (in a tube furnace, by comparison with calibrated Type S or Type B thermocouples)	200	0.8
	400	0.6
	600	0.5
	800	0.4
	1100	0.3
	1450	1.6

[A] Uncertainties for calibration of base metal thermocouples include an allowance for the inhomogeneity of the unused thermoelements.

TABLE 2 Calibration Uncertainties in Calibrating Thermocouples by the Comparison Method—Temperatures in Degrees Farenheit[A] (see Refs. 2 and 3)

	Temperature [°F]	Expanded Uncertainty (k=2) [°F]
Base metal thermocouples[A] (in a tube furnace by comparison with a calibrated Type S thermocouple	400	0.4
	800	0.8
	1200	1.1
	1600	1.5
	2000	1.9
Base metal thermocouples[A] (in stirred liquid baths, by comparison with an SPRT)	−321 (type E)	0.4
	−150 (type E)	0.2
	32	0.04
	400	0.4
	800	0.7
	1000	0.9
Type R and S thermocouples (in a tube furnace, by comparison with a calibrated Type S thermocouple)	400	0.3
	800	0.4
	1200	0.5
	1600	0.5
	2000	0.4
Type B thermocouples (in a tube furnace, by comparison with calibrated Type S or Type B thermocouples)	400	1.3
	800	1.0
	1200	0.8
	1600	0.7
	2000	1.1
	2500	2.9

[A] Uncertainties for calibration of base metal thermocouples include an allowance for the inhomogeneity of the unused thermoelements.

8.7 *Type T Thermocouples (Copper versus Constantan)*—The type T thermocouple may serve as a useful reference thermometer in the range of −195 to 370 °C (−320 to 700 °F) in some instances, although its accuracy is, in general, limited by the stability of the wire at temperatures above approximately 200 °C (390 °F), and by the accuracy of the emf measurements and the inhomogeneity of the wire below 200 °C. One-half millimeter diameter (24 gauge) wire is a useful compromise between the lesser stability of smaller wire and the greater heat conduction of large wire.

8.8 *Gold versus Platinum Thermocouples*— The gold versus platinum thermocouple is useful as a reference thermometer over the range 0 to 1000 °C (32 to 1830 °F). With proper construction and annealing, a gold versus platinum thermocouple will have uncertainties of approximately 0.01 to 0.02 °C (0.02 to 0.04 °F). To attain this performance, care in the emf measurements and protection of the thermoelements from contamination is necessary.

8.9 *Single-use Base-metal Thermocouples*—For tests to elevated temperature, a base metal thermocouple taken from a calibrated lot of wire of verified homogeneity may be used as a reference thermometer. Lot homogeneity may be determined by calibrating thermocouples fabricated from a statistical sample of the wire lot, and determining the standard deviation of emf values of the set of thermocouples, at each calibration temperature. In this application, the base-metal reference thermocouple would be used only at increasing test temperatures. Single-use base metal thermocouples are not amenable to recalibration, and an additional uncertainty must be included to account for drift of the reference during the test. In particular, type E and type K thermocouples that have not been specially heat-treated are known to exhibit shifts of up to the equivalent of 4 °C (7 °F) in thermoelectric response after relatively short exposures to temperatures in the range 250 ° C (480 °F) to 550 °C (1020 °F).

NOTE 2—In general, any thermometer may be employed as a reference thermometer provided that it has a known amount of measurement uncertainty.

8.10 *Verification of Reference Thermometer Performance*—When platinum resistance thermometers or thermistors are used as reference thermometers, the reference thermometer shall be verified by checking its indication at a thermometric fixed point. The ice point or the triple point of water is commonly used. Liquid-in-glass thermometers shall be measured at the ice point after each thermal cycle to temperatures exceeding 100 °C (212 °F). Measurements may be corrected for a change in ice-point reading following the procedures in Test Method E77. Thermocouples, other than Au/Pt thermocouples, are not amenable to recalibration in an apparatus different than the one used for the actual test. Therefore, thermocouple reference thermometers shall be verified by a comparison test against a second reference thermometer of equal or lesser uncertainty in the same apparatus as used for the test. Au/Pt thermocouples shall be verified at a thermometric fixed-point or by a comparison measurement. For thermocouples of all types, verification points at temperatures close to the temperature of the reference junctions are not a sensitive test of possible changes in the thermocouple. Verification points should be chosen to have a temperature as far from the reference junction temperature as practical

9. Sampling

9.1 Sampling is normally specified in the ASTM material specification that calls for the calibration. As a guideline for compliance testing, a minimum of two samples are often calibrated to ensure that a lot of wire or assembled thermocouples conforms to standardized emf-temperature relations within specified tolerances. In the case of wire, the samples should preferably be widely separated within the lot, for example, opposite ends of a coil. Users should be aware that in some instances compliance testing will cause changes to occur in the thermoelectric properties of the samples of thermocouple wire tested.

10. General Procedures

10.1 The calibration procedure consists of measuring the emf of the thermocouple being calibrated at selected calibration points, the temperature of each point being measured with the selected reference thermometer. The number and choice of test points will depend upon the type of thermocouple, the temperature range to be covered, and the accuracy required. Table 1 or Table 2 will serve as a guide to the selection. Both the nominally isothermal temperature calibration environment and the thermocouples must be stabilized at the calibration temperature before readings are taken. For any particular apparatus, the necessary stability may be determined by performing comparison measurements at a variety of drift rates of the reference thermometer temperature. A plot of the test thermocouple reading, normalized as described in 12.1.1, versus drift rate will indicate what magnitude of temperature drift can be tolerated without unacceptable variation in the calibration results. A minimum of three consecutive readings that yield the same emf value within measurement uncertainty is required. Instead of employing a single nominally isothermal temperature calibration environment whose temperature is changed to each calibration temperature, a series of nominally isothermal environments (for example, isothermal metal blocks, stirred fluid baths, or tube furnaces), each maintained at a calibration temperature, may be used provided the rates of immersion and extraction are not so large as to damage either the thermocouples being calibrated or the reference thermometer. After insertion of the thermocouple into each temperature calibration environment, time shall be allowed for steady state conditions to be reached before readings are taken. The depth of immersion ideally shall be the same throughout the test. Otherwise, the depth shall not be less than any previous immersion. Techniques similar to that described in Appendix X4, but covering only the range of immersion depth encountered in the calibration process, can be useful in determining if changes in immersion during the course of a calibration significantly affect the results. One of the following two general methods may be used in the calibration procedure.

10.2 *Method A, Automated Method*—Fig. 2 illustrates the schematic of a typical data acquisition system—assuming a thermocouple is used as the reference thermometer. This method permits the rapid testing of any number of thermocouples. The reference thermometer may be of any type meeting the uncertainty requirements for the calibration, provided that the data acquisition system can accommodate the corresponding type of signal. The thermocouples to be tested and the reference thermocouple are terminated at the reference junction temperature unit. This unit must meet the requirements of 7.3. A scanning unit sequentially connects each thermocouple to the input of a voltmeter. The voltmeter must meet the requirements of 7.4. In the time interval between a reading of the reference thermometer and of a test thermocouple, the temperature of the calibration zone will vary. Measurements shall be made rapidly enough so that this variation in temperature is small in comparison to the uncertainty of the temperature measurement. The emf-measuring system is comprised of a voltmeter, a display, and a data storage system. It may be desirable to provide a calibrated emf source and a zero (or shorted) input to the data acquisition system as references to improve the accuracy of the measurement. For a set of n test thermocouples, the recommended order of readings is: reference thermometer, test thermocouple 1, test thermocouple 2, … test thermocouple n, reference thermometer, test thermocouple n, … test thermocouple 1, reference thermometer. If the variations in temperature indicated by the reference thermometer are greater than desired with the above method, an alternative order of reading may be made, provided that the reference thermometer is read at least once for every reading of the test thermocouple and that the order of readings is symmetric in both forward and reverse directions.

NOTE 3—The reference temperature unit, the scanner, and the emf-measuring system are typically combined into a single system. They are described separately so that their characteristics may be understood and specified to meet the required uncertainty criteria.

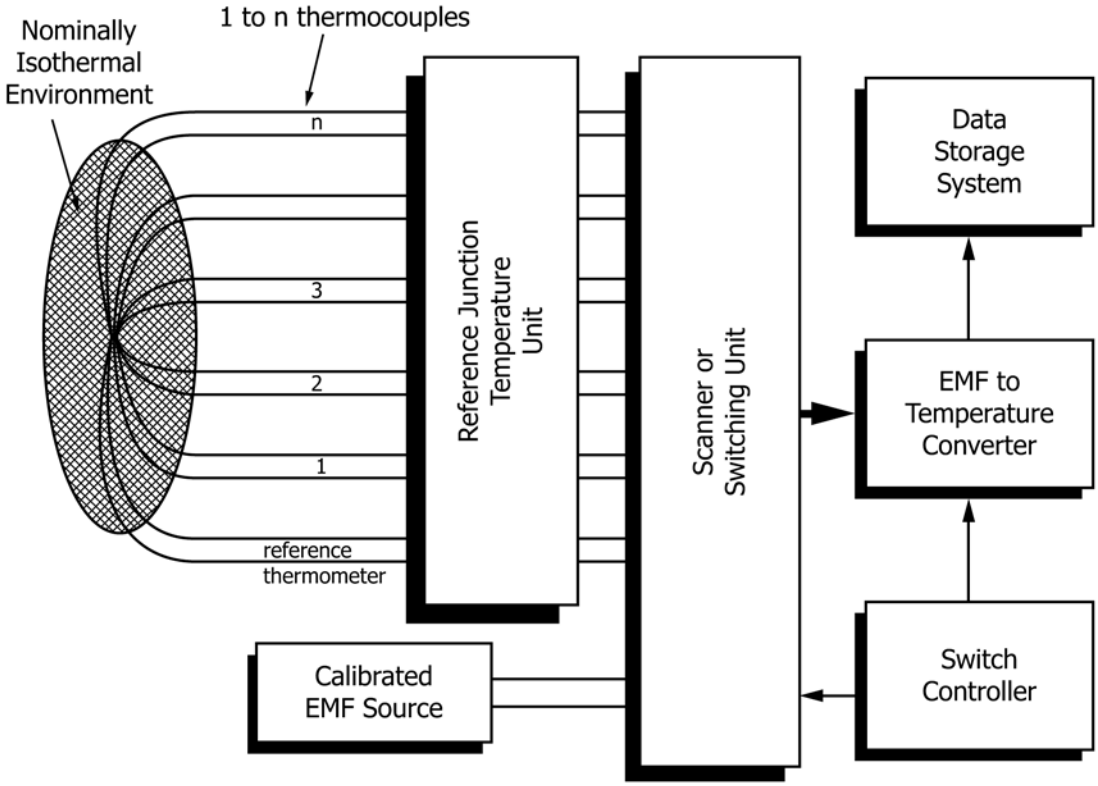

FIG. 1 Automated Thermocouple Data Acquisition System Layout—With Thermocouple Reference

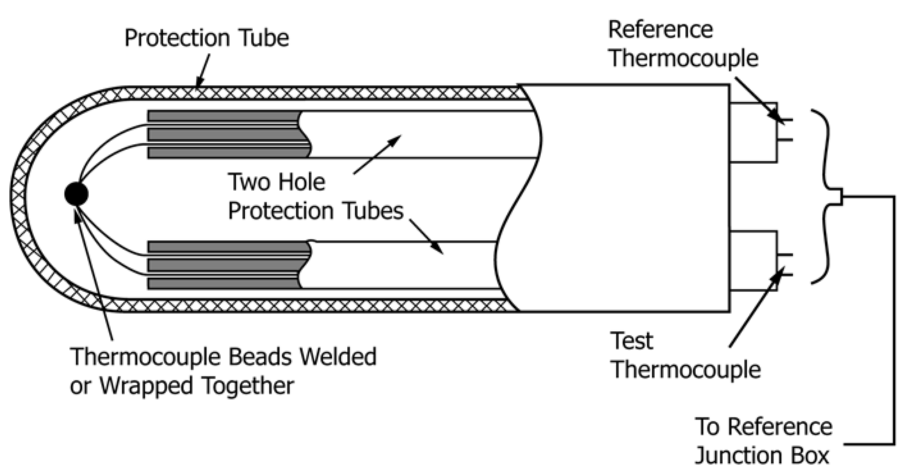

FIG. 2 Thermocouple Assembly in Protection Tube (Multilayered Cutaway View)

NOTE 4—Any program that performs manipulations on the measured data shall be checked for accuracy by evaluating the output response to known inputs.

10.3 *Method B, Manual Method*—This method may be used when one or more thermocouples are to be calibrated with manually operated switches. Each thermocouple is connected to the voltmeter in sequence. The reference thermometer should be read just before and just after the reading of each thermocouple under calibration. After measuring the emf of each test thermocouple, the entire sequence should be repeated at the same temperature.

10.4 *Method C, Differential Method*— When the thermocouple being calibrated is of the same type as the reference thermometer a differential emf measurement method may be employed. In this method, the emf of the reference thermocouple is measured and the differential emf between the thermocouple being calibrated and the reference thermocouple is directly measured by connecting the thermocouples in series, with the polarity of one of the thermocouples reversed. Note that in order to make a serial connection the thermocouple junctions must be electrically isolated. This technique can offer improved stability since the difference will remain relatively constant over small temperature intervals. For this technique a voltmeter with uncertainty up to 0.5 μV can be used.

11. Preparation of Thermocouples for Test

11.1 *Bare-Wire Thermocouples for Laboratory Furnaces or Fluidized-Bed Baths*—In preparation for test, a suitable thermocouple protection tube shall be chosen that is long enough to provide sufficient immersion and to extend out from the furnace or bath for 50 to 75 mm (2 to 3 in.). A two-hole ceramic insulating tube, somewhat longer than the protection tube, shall be selected for each thermocouple. The thermoelements of each thermocouple are threaded through the holes in its respective tube, and the group of thermocouple tubes loosely bundled together. 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 thermocouples.[5] If it is not convenient to weld the junctions together the junction of each thermocouple must be welded separately and the junctions brought into good thermal contact by wrapping them with a thermally-conductive wire or foil of an element such as platinum. Slip the insulating tubes down on the thermoelements as close to the measuring junctions as possible without stressing the wires. Insert the bundle of thermocouples to the bottom of the protection tube; then place the tube at the proper depth in the furnace or bath. Such an assembly is shown schematically in Fig. 2. As an alternative to the closed-end protection tube shown in Fig. 2, an open-ended protection tube may be used, provided that both ends of the tube are outside of the furnace. In this process, take care not to stress or cold work the wires. Special care must also be taken during handling of the thermoelements to avoid contamination (see 7.6).

11.2 *Ungrounded Junction Thermocouples*— For ungrounded junction thermocouples, the measuring junctions must be maintained in good thermal contact by keeping the measuring junctions well immersed within the nominally isothermal calibration zone (see 7.2).

11.3 *Procedures for Types S, R, and B Thermocouples (Platinum-Rhodium versus Platinum or Platinum-Rhodium) in Laboratory Furnaces or Fluidized-bed Baths*—This procedure is applicable for the calibration of platinum-10 % rhodium vs. platinum (Type S) or platinum-13 % rhodium vs. platinum (Type R) thermocouples in the temperature range from 0 °C to 1200 °C (32 °C to 2200 °F) and for platinum-30% rhodium vs. platinum-6% rhodium (Type B) thermocouples in the range from 0 °C to 1700 °C (32 °F to 3100 °F).

11.3.1 Prior to calibration, annealing of the bare thermoelements is recommended. First clean the thermoelements by wiping with a soft-fiber wipe soaked with ethanol. Suspend the thermocouple freely in air from two binding posts, which should be close together so that the tension in the wires and stretching while hot are kept to a minimum. Shield the thermocouple from drafts. Electrically anneal the thermocouple in air for a period of 45 min at approximately 1450 °C (2650 °F). Then cool it slowly (over a period of approximately 1 min) to 750 °C (1380 °F) and hold it at that temperature approximately 30 min. Next, allow the thermocouple to cool to room temperature within a few minutes. Alternating current from an adjustable transformer is a convenient source of controlled power for heating the thermocouple wires, about 12 A being required for 0.5 mm wire. The temperature is most readily determined by sighting on the platinum thermoelement with a disappearing filament or two-wavelength radiation thermometer. A radiation thermometer reading of 1300 °C (2380 °F) will correspond to a wire temperature of about 1450 °C. This correction is necessary to account for the emissivity of the wire. If the above annealing procedure is not performed, a subcomponent must be added to the uncertainty budget to account for annealing of the test thermocouple during use. This uncertainty may be determined from the differences of the calibration results for thermocouple material annealed as stated above and for thermocouple material in the "as-received" state.

11.3.2 After annealing, thread the thermocouple through its insulating tube and mount it, together with a reference thermometer, in a protection tube as described in 7.6. The reference temperature may be established using any calibrated thermometer capable of indicating values of temperature with known and acceptably small uncertainty; type B, R, or S thermocouples are commonly used. Special care must be exercised during handling of the annealed thermocouple wires to avoid contaminating or stressing them.

11.4 *Procedure for Base-Metal Thermocouples in Laboratory Furnaces or Fluidized-Bed Baths*—This procedure is applicable to the calibration of the bare wire base-metal thermocouples in the temperature range 0 to 1260 °C (32 to 2300 °F). Base metal thermocouples should be calibrated in the "as-received" condition, with no further annealing.

NOTE 5—This method is intended for use with new wire. Base-metal thermocouples undergo changes with use at high temperature that often render them unfit for recalibration. Therefore, these thermocouples shall not be recalibrated.

11.4.1 Thread the thermocouple through its insulating sleeve and mount with a reference thermometer, in a protection tube as described in 7.6. The reference temperature may be established using any calibrated thermometer capable of indicating values of temperature with known and acceptably small uncertainty; type B, R, or S thermocouples are commonly used. When using a calibrated thermocouple as the reference thermometer, take appropriate precautions to prevent contamination of the reference thermocouple such as inserting the reference thermocouple into a two-hole ceramic insulating sleeve to within a few millimeters of the measuring junction, and sealing the end of the ceramic tube to the thermocouple with borosilicate glass or a small amount of kaolin and sodium silicate cement. This protective measure minimizes contamination of the reference thermocouple, with the exception of the small 2 or 3-mm length, which is necessarily in contact with the base-metal thermocouple. If the furnace is heated uniformly in this region, contamination of the exposed thermoelements will cause negligible error. If the thermoelements become brittle at the junction with use, cut off this part and draw enough thermoelement through the seal to form a new

[5] For information on welding of measuring junctions, see *Manual MNL-12*.

junction. Examine the seal after each use and remake it if it does not appear to be satisfactory.

11.4.2 If the thermoelements are large, one or more of the base-metal thermocouples may be welded together and a hole drilled in the common junction to accept the measuring junction of the reference thermocouple. It is recommended, however, to weld the measuring junction of the reference and test thermocouples into a common junction as shown in Fig. 2.

11.4.3 When thermoelements, insulators, and protection tubes of base-metal thermocouples are large, it is particularly important that tests be made (or have previously been made) to ensure that the depth of immersion is sufficient in the furnace or furnaces being used. Sheathed thermocouples can be calibrated in a tube furnace with no further protection provided that the furnace environment is compatible with the sheath.

11.5 *Procedure for Thermocouples in Stirred Liquid Baths*—This procedure is applicable to the calibration of thermocouples, either bare wire or sheathed, up to a temperature of approximately 630 °C (1170 °F). Bare-wire thermocouples will require initial mounting, and possibly annealing, as described in 7.6, 11.3, and 11.4. Usually no additional special preparation of the thermocouple will be required other than to insert it to the bottom of a protection tube for immersion in the liquid bath. Borosilicate glass tubing has been found convenient for use up to 540 °C (1000 °F). Above 540 °C, vitreous silica or ceramic tubing has been found satisfactory. It should be closed at the immersed end, and large enough to permit easy insertion of the thermocouple or thermocouples to be calibrated but no larger than necessary. Unfavorable heat transfer conditions in an unnecessarily large diameter tube will require a greater depth of immersion in the bath than would a close-fitting tube. If a bare-wire thermocouple is being calibrated, provide the thermoelements with electrical insulation over the length inserted in the protection tube. Slip a loose-fitting insulating sheath over one or both thermoelements of the thermocouple. Use any suitable commercially available material that will withstand the highest temperature to which it will be exposed without chemically contaminating the test thermocouple and that will maintain electrical insulation between typical thermoelements of at least 10^4 times the resistance of the thermoelements themselves. Immerse sheathed thermocouples directly in the bath liquid in cases where the sheath material will not be attacked by the liquid. Salt baths for use at high temperatures must have corrosion resistant (typically steel) thermowells into which the thermocouple protection tubes and standard thermometers are inserted for protection from the molten salt. Quartz sheath thermometers or protection tubes should not be used in direct contact with salt bath fluids. The reference thermometer may be a thermocouple inserted in the protection tube with the thermocouple being calibrated, or it may be any type of reference thermometer immersed in the bath liquid close to the thermocouple protection tube. The choice of a reference thermometer will be governed principally by the accuracy required (see and Appendix X2).

11.6 *Insulation of Bare-wire Thermocouples Exposed to Ambient Temperatures*—The bare thermoelements extending from the insulators used in the furnace or bath may be insulated with fiberglass or plastic sleeving to prevent electrical shorts between the thermoelements. A short (1 cm or 0.5 in.) section of heat-shrinkable tubing can be used to support the junction between the sleeving and the insulator used in the furnace or bath, thereby minimizing mechanical strain of the thermoelements. Large diameter thermoelements that are sufficiently rigid to be self-supporting will not require this section of heat-shrink tubing and may not require sleeving as well.

12. Calibration Procedures

12.1 The following methods for taking calibration data are applicable to platinum, refractory metal, and base-metal thermocouples.

12.1.1 *Thermocouples in Laboratory Furnaces*—Immerse the protection tube containing the thermocouples to the proper depth in a suitable electrically heated tube furnace (7.2.3 and Appendix X1), and assemble the reference junction, connecting wires, and switching as described in Section 7. For base-metal thermocouples, heat the furnace to the temperature of the lowest calibration point, and stabilize at this temperature for about 10 min, using the indications of the reference thermometer and all thermocouples to ensure temperature stability. Alternatively, a series of furnaces whose temperatures are maintained at fixed calibration points may be used provided sample immersion and extraction rates are not so large as to damage either the thermocouples being calibrated or the reference thermometer. Read the reference thermometer and test thermocouples, according to Method A, B, or C as described in Section 10. Inspection of the variation of the reference thermometer readings will indicate whether the furnace and sensor elements have been adequately stabilized. Under stable conditions, the emf reading of the test thermocouple will correspond to a temperature represented by the mean of the temperature readings of the reference thermometer. Take a second series of readings resulting in a second value of emf for the test thermocouple, at nearly the same temperature. Because of small variations in furnace temperature, it is unlikely that the two sets of emf readings will have been made at exactly the same temperature. Correct the second measurement of the emf of the test thermocouple by subtracting an emf term corresponding to the temperature difference between the second and first test readings. For example, if the temperature indicated by the reference thermometer is 650.0 °C at the time of the first measurement and the temperature indicated by the reference thermometer is 650.1°C at the time of the second measurement and a Type J thermocouple is being calibrated (Type J thermocouple emf is 36.071 mV at 650°C and 36.131 mV at 651°C), Corrected 2nd emf Reading = 2nd Reading emf − S(650°C) × (650.1°C − 650°C), where S (650°C) = Seebeck coefficient = (36.131 mV − 36.071 mV)/(651°C − 650°C) = 0.0603 mV/°C.

The two values of emf for the test thermocouple are then averaged and assigned to the first measurement temperature. Using the standard reference tables given in Specification E230 or Guide E1751, corrections can be applied for temperature differences of up to 5°C (9°F) without introducing an error greater than the equivalent of 0.1°C (0.2°F). Repeat the procedure at the next higher and succeeding test points. Noble metal thermocouples may be calibrated in either ascending or descending temperature steps. Normalization methods that are mathematically equivalent to the above method, to within the small interpolation errors of the above method, may be used for analyzing the data.

12.1.2 *Thermocouples in Stirred Liquid Baths*—The procedure for taking data in the calibration of thermocouples in stirred liquid baths is identical with that for laboratory furnaces described in 12.1.1, except that the reference thermometer is often a liquid-in-glass or resistance thermometer instead of a thermocouple. When measurements approaching an uncertainty of 0.1°C are to be made, voltmeters with a measurement uncertainty no greater than given in 7.4 must be used.

13. Calculations

13.1 *Emf Difference Function Calculation*—Having determined the emf of the thermocouple at a number of calibration points, complete the calibration by interpolating between the calibration points. Various interpolation methods may be used to accommodate special circumstances. The method involving the use of a difference curve from a reference table is often the simplest and most accurate to use. To use this method, choose the appropriate reference table in Specification E230 or Guide E1751 as a basis for establishing a difference curve. Calculate the emf difference $\Delta E = E_r - E$ for each calibration point, where E_r is the table value of emf and E is the emf of the test thermocouple at the temperature of the calibration point. Include the value of $\Delta E = 0$ at the reference junction temperature in the data set. Calculate a functional form to approximate the emf difference versus temperature by performing a least squares fit of the resulting data. It is often convenient to use a low-order polynomial to model the emf difference. As an approximate guide, the order of the polynomial should not be greater than the number of data points divided by two. Functional forms other than polynomials are generally acceptable provided that they result in a smoothly varying curve with no more than one point of inflection between the calibration points. The lowest fitting errors attainable when modeling a set of data over a large temperature range will be approximately 0.4 µV for type S and R thermocouples and from 1 µV to 5 µV for base metal thermocouples. The resulting function for ΔE as a function of temperature represents the difference curve between the table values of emf and the emf values of the measured thermocouple. From this relation, table values or an equation form of ΔE as a function of E of the test thermocouple are obtained. At any observed value of E add the corresponding value of ΔE from the curve and enter the table at this corrected value of emf to obtain the true temperature. The values ΔE take the sign of a correction to be added to the observed emf, producing a corrected emf with which one can enter the standard table to get the true temperature. Alternatively, the function $\Delta E(t)$ may be subtracted from the thermocouple reference function to create a single function giving the test thermocouple emf as a function of temperature. Emf functions shall not be used outside the range of calibration.

NOTE 6—These table values are based upon a reference junction temperature of 0 °C or 32 °F. If another reference junction temperature has been used, the reference table values must be adjusted by subtracting from each the table value of emf corresponding to the reference junction temperature used.

13.2 *Emf Function Calculation*—In a few special cases it may be desirable to model the functional relationship of emf with temperature ($E(t)$) without utilizing differences from a reference function. Various functional forms are acceptable provided that they result in a smoothly varying curve with no more than one point of inflection between any two adjacent calibration points. As an approximate guide, the number of coefficients of the functional form should not be much greater than the number of data points divided by two. In no case should the functional form have more parameters than the reference function in Specification E230 or other relevant specification. Modeling of data by direct calculation of the emf function often will require substantially more parameters and substantially more measurement data than the corresponding calculation at the same level of uncertainty by the method of emf differences. Emf functions shall not be used outside the range of calibration.

14. Report

14.1 Report the calibration results as required by the user. This may be the function $E(t)$, a table of values of E at a number of temperatures, a table of values of ΔE at selected values of E, or a table of values of the temperature equivalent, Δt, of ΔE, at selected values of temperature. The calibration report shall state the type of reference thermometer used, and the calibration uncertainty and corresponding level of confidence.

15. Precision and Bias

15.1 Due to the varying nature of the equipment used in this test method, no statement can be made about its precision and bias. Instead, an estimate of uncertainty, otherwise known as an uncertainty budget, is used. This uncertainty evaluation shall follow the method shown in the Uncertainty section.

16. Uncertainty

16.1 The single-operator repeatability and multilaboratory reproducibility of calibration conducted by this test method will depend on the optional techniques and equipment selected, the variability of the wires between samples, the bias between references used, and the skill of the operator. The uncertainties given in Tables 1 and 2 represent the capability of the test method as achieved by NIST (see Refs (**2,3**)), but actual results can vary significantly. The user is cautioned that the method is prone to significant errors if not done skillfully. A variety of effects, listed in Table 3, contribute to the uncertainty of calibrations performed according to this test method. An uncertainty budget including each of these terms should be derived. The accuracy obtained in comparison calibrations depends upon two principal factors, the accuracy realized at the calibration points and the accuracy with which interpolation is made.

16.2 *Accuracy of Calibration Points*— The accuracy attained at each calibration point will depend upon the degree to which the reference thermometer and the test thermocouple are maintained at the same temperature when measurements are

TABLE 3 Components of Calibration Uncertainties

Uncertainty Component	Evaluation Method
1. Test thermocouple inhomogeneity and drift	Literature, or Section 10.1 or Appendix
2. Reference thermometer calibration	Provider of thermometer calibration
3. Reference thermometer repeatability	Section 8.1
4. Reference thermometer measurement	Manufacturer's specifications
5. Bath or furnace temperature gradients	Section 7.1
6. Bath or furnace stability	Section 10.1
7. Extraneous emf from scanners, etc	Appendix X3
8. Emf measurement	Manufacturer's specifications
9. Interpolation between calibration points	Section 16.3

made, the accuracy of the reference thermometer and its related instruments, and the accuracy of the emf measurements, as listed in components 1 through 8 in Table 3. Uncertainty components covering reference thermometer repeatability, bath temperature stability, and thermocouple drift may be evaluated by statistical analysis of multiple measurements of check-standard thermometers. At temperatures in excess of approximately 200 °C (400 °F), base metal thermocouples may drift substantially during the calibration test, imposing the primary limitation on the uncertainty of the calibration. Metallurgical limitations of thermocouple materials result in compositional inhomogeneities along the length of the thermoelements, even when new. This effect limits the best accuracy attainable in a thermocouple calibration. As an approximate guide, base metal thermocouples may be calibrated to a fractional uncertainty of 10^{-3} of the temperature difference between the reference and measuring junctions, noble-metal alloy thermocouples may be calibrated to a fractional uncertainty of 10^{-4} of this temperature difference, and pure-element thermocouples may be calibrated to a fractional uncertainty of 10^{-5} of this temperature difference. The combined uncertainties that occur at the calibration points for the common types of thermocouples, as obtained at NIST, are given in Tables 1 and 2. Uncertainties for calibration of base metal thermocouples include an allowance for the inhomogeneity of the unused thermoelements.

16.3 *Uncertainty of Interpolated Values*— The uncertainty of interpolated values will depend upon the number of calibration points and the closeness with which the reference function used represents the behavior of the particular thermocouple being calibrated. The more accurately the values conform to the emf-temperature relationship of the reference function, the fewer the number of calibration points required for a given uncertainty. In general, the calibration points should bracket the temperature range over which the thermocouple is to be used, and no extrapolation should be attempted. Tables 4 and 5 give approximate uncertainties of interpolation for thermocouples that meet the standard tolerances of Specification E230, provided that the interpolation method is based on a smooth curve modeling the difference of the emf from the reference function. These uncertainties were extracted from Ref (**6**), which gives the uncertainty of a calibration both at the temperature values tested and of interpolated values at temperatures between calibration points. Data are not available for type N thermocouples, but the interpolation uncertainties are expected to be no worse than for type K thermocouples.

16.3.1 When a substantial number of calibration points is available for a single test thermocouple, the calibration data itself may be used to evaluate the interpolation error. The thermocouple calibration is determined by fitting a polynomial to the difference of the emf from the reference function, as described in 13.1. The number of data points shall equal or exceed twice the number of parameters fitted. The standard uncertainty of using the resulting polynomial to interpolate the emf between calibration temperature values is taken as the rms deviation of the polynomial from the data:

$$u = \sqrt{\frac{1}{N_{df}} \sum_i (E_i - E_{fit})^2}$$

TABLE 4 Additional Calibration Uncertainties Due to Interpolation—Temperatures in Degrees Celsius (6)

Thermocouple Type[A]	Temperature Range	Calibration Points	Expanded (k=2) Uncertainty of Interpolation, for Temperatures other than Calibration Points[B]
B	0 to 1700	every 100	0.2
	0 to 1700	600 and 1200	0.7 to 1100 and 5 at 1700
E	0 to 870	every 100	0.5
	0 to 870	300, 600, and 870	1.5
	−195 to 0	every 50	0.4
J	0 to 760	100, 300, 500, and 750	0.5
	0 to 350	every 100	0.4
K	0 to 1250	every 100	0.5
	0 to 1250	300, 600, 900, and 1200	1.5
	−195 to 0	every 50	0.4
R and S	0 to 1450	every 100	0.2
	0 to 1450	600 and 1200	0.7 to 1100 and 3 at 1450
T	0 to 370	every 100	0.1
	0 to 100	50 and 100	0.05
	−195 to 0	every 60	0.1

[A] See Specification E230.
[B] Using difference curve from reference table.

TABLE 5 Additional Calibration Uncertainties Due to Interpolation-Temperatures in Degrees Fahrenheit[A]

Thermocouple Type[B]	Temperature Range	Calibration Points	Expanded (k=2) Uncertainty of Interpolation, for Temperatures other than Calibration Points[C]
B	32 to 3100	every 200	0.4
	32 to 3100	1100 and 2200	1.3 to 2000 and 9 at 3100
E	32 to 1600	every 200	0.9
	32 to 1600	600, 1100, and 1600	3
	−320 to 32	every 100	0.7
J	32 to 1400	300, 600, 1000, and 1400	0.9
	32 to 650	every 200	0.7
K	32 to 2300	every 200	0.9
	32 to 2300	600, 1200, 1800, and 2300	3
	−320 to 32	every 100	0.7
R and S	32 to 2700	every 200	0.4
	32 to 2700	1100 and 2200	1.3 to 2000 and 5 at 2700
T	32 to 700	every 200	0.2
	32 to 200	110 and 200	0.1
	−320 to 32	every 100	0.2

[A] This table is based upon the values in Table 4, but Fahrenheit temperatures are given in round numbers rather than exact equivalents of the Celsius temperature.
[B] See Specification E230.
[C] Using difference curve from reference table.

where:

E_i = the emf value i of the test thermocouple, measured at temperature t_i,
E_{fit} = the emf of the fitted polynomial evaluated at t_i, and
N_{df} = the number of degrees of freedom in the fit = number of data points − number of fitted parameters.

To obtain the interpolation uncertainty for a coverage factor of two, this component (u) is multiplied by two. The interpolation uncertainty shall be added in quadrature to the uncertainties of the measured points to obtain the combined uncertainties of the interpolated points.

16.4 *Accuracy of Calibrated Thermocouples in Use*—In a strict sense, calibrations by the methods described here apply only for conditions of use similar to those under which the calibrations were made. Once a thermocouple, particularly one of base metals, has been heated to high temperature, changes may occur, even in relatively homogenous thermoelements, that will cause the emf output of the thermocouple to be dependent upon the particular temperature profile existing between the measuring and reference junctions. This is particularly true of base-metal thermocouples that have been calibrated at one depth of immersion and are used at a shorter depth of immersion. A general quantitative assessment of errors that can arise from this source is not feasible, but the possibilities of such errors should be recognized in analyzing uncertainties to temperature measurements made with calibrated base-metal thermocouples. For base metal thermocouples, it is preferred practice to calibrate a lot of thermocouple wire or a statistical sample of assembled thermocouple probes, and to then use a new thermocouple or probe for each thermal environment. Techniques for in situ validation of used thermocouples that eliminate errors resulting from the different thermal profiles of calibration apparatuses relative to the apparatus where the thermocouple is used are discussed in MNL-12 (**1**).

17. Keywords

17.1 calibration; comparison techniques; thermocouple; thermoelement

APPENDIXES

(Nonmandatory Information)

X1. APPARATUS

X1.1 Wire-Wound Electric Tube Furnaces

X1.1.1 Wire-wound electric tube furnaces suitable for thermocouple calibration may be obtained commercially. Such furnaces are typically equipped with a means of regulating the current or controlling the temperature and are available in models that will operate from various power mains. For temperatures up to about 1150 °C (2100 °F), a furnace with a heating element of nickel (80 %)-chromium (20 %) will suffice, whereas iron-chromium-aluminum wire alloys are frequently used in furnaces up to 1300 °C. Furnaces of wire-wound design with heating elements of platinum or platinum-rhodium are available for higher temperatures, alternatively ceramic element materials may be used (see X1.2). A convenient size of heating tube is 25 mm (1 in.) in diameter and 600 mm (24 in.) long. The heating tube may be mounted either horizontally or vertically, but a vertically mounted tube must be plugged at its lower end to minimize convection currents through the tube. A choice of tube dimensions and orientation may be influenced by such factors as the size and kinds of wires in the thermocouples to be calibrated, mounting convenience, or personal preference in a particular use. Before relying upon any furnace, however, a test should be made to ascertain that the depth of immersion is sufficient to eliminate cooling or heating of the junctions by heat flow along the thermocouple and the insulating and protecting tubes. This can be determined by observing the change in emf of the thermocouple as the depth of immersion is changed slightly. It is difficult to generalize upon what a sufficient depth of immersion may be, since in a particular instance this will depend upon the number and size of the thermocouple wires entering the furnaces as well as furnace characteristics, such as tube diameter and profile of thermal gradients along the tube.

X1.2 Ceramic Element Electric Tube Furnaces

X1.2.1 For temperatures above 1200 °C (2200 °F), laboratory tube furnaces with heating elements of materials such as silicon carbide (SiC) or molybdenum disilicide ($MoSi_2$) may be used as an alternative to platinum wire. Silicon carbide furnaces operate upto 1500 °C or 1600 °C (2700 °F or 2900 °F) and molybdenum disilicide to approximately 1700 °C (3100 °F). For thermocouple calibration purposes, these furnaces should always be fitted with a suitable ceramic tube.

X2. REFERENCE THERMOMETERS

X2.1 Standard Platinum Resistance Thermometers

X2.1.1 Standard platinum resistance thermometers (SPRTs) are the most accurate reference thermometers and are used in defining the ITS-90 from approximately −259 to 962 °C. The SPRT sensing element is made from pure platinum and supported essentially strain-free. Because of the delicate construction, the SPRT is easily damaged by mechanical shock and must be handled carefully to retain its calibration.

NOTE X2.1—No single SPRT covers the entire temperature range of −260 to 962 °C.

X2.2 Industrial Platinum Resistance Thermometers

X2.2.1 Certain industrial platinum resistance thermometers are specially manufactured and subjected to special heat treatment and calibration to establish their measurement uncertainty. These thermometers contain sensing element constructions that are not as easily affected by handling as SPRTs. However, they also typically have higher measurement uncertainties and narrower usage ranges than SPRTs. Testing of industrial platinum resistance thermometers is described in Test Methods E644.

X2.3 Measurement Instruments for Resistance Thermometers

X2.3.1 Several types of instruments can be used. They include analog and digital instruments and those that use resistance bridges, voltage comparison, or current and potential methods. AC and DC bridges and digital multimeters are becoming increasingly common due to their ease of use and compatibility with computerized data acquisition systems. These instruments typically provide the user the option of a digital display that can be set to provide readings in ohms, millivolts, or temperature. The operating current of these instruments must be low enough that any self-heating of the thermometer is minimized.

X2.4 Liquid-in-Glass Thermometers

X2.4.1 Liquid-in-glass thermometers can be used as relatively simple and accurate temperature references over a range of moderate temperatures when good usage techniques are followed. Discussions of the calibration and use of liquid-in-glass thermometers are given in Refs (**7**,**8**) and Test Method E77.

X2.5 Thermistors

X2.5.1 Thermistors are a type of resistance thermometer in which the sensing element is typically composed of electrically conductive oxides. Because thermistors have high sensitivity, instrumentation costs are often lower than for other types of resistance thermometers for a given level of accuracy. Most thermistors do not conform to a standard resistance-temperature relationship, and each thermistor must be individually calibrated. Glass-coated thermistors should be selected for the best stability.

X3. TEST FOR STRAY THERMALLY GENERATED EMFS IN COPPER CONNECTING WIRES

X3.1 A test for extraneous emfs in the copper connecting wires, switching, etc., between the reference junctions and the potentiometer may be made as follows: With the thermocouple assembly as shown in Fig. 1, remove one of the ends of the test thermocouple from the reference junction bath and connect a short piece of copper wire across the measurement terminals. This copper link will complete the circuit through the connecting wires to the voltmeter, which will now indicate any emf originating in the scanner system, binding posts, or other system components, as well as extraneous pickup from other electrical sources. Temperature gradients in the copper link will not induce an emf if a good grade of homogeneous copper wire is used. The zero offset of the voltmeter must be evaluated before performing this test.

X4. TEST FOR THERMOCOUPLE INHOMOGENEITY

X4.1 This procedure is intended to demonstrate that the calibration of a thermocouple will be insensitive to the temperature profile over its specified depth of immersion. To utilize this procedure, the test article should be attached to a calibrated resistance thermometer or a thermocouple of known homogeneity that will move with the test article, clamping or tying their measuring ends together. The reference thermometer must be able to cover the entire range of calibration temperatures desired. In the course of calibrating a thermocouple, at its highest calibration temperature, withdraw the test article from the furnace or bath in steps. The test article shall be held at that position until its temperature profile comes to steady state equilibrium. At least four steps should be held during the withdrawal process. The withdrawal temperature profile should provide a temperature gradient at least as sharp as that expected in the intended application of the test article. The sharpest gradients in the ambient-to-test temperatures are produced at the interface between a liquid metal bath and a blown air entrance region; the least sharp gradients are produced in an air-atmosphere entrance to a furnace without an equalizing block. At each step, compare the output of the test article with the temperature indicated by the reference thermometer. The withdrawal procedure should cover as much of the length of the thermocouple as is expected to encounter temperatures more than 100 °C above ambient. Alternatively, particularly for thermocouples much longer than the heated calibration environment, hold the thermocouple at each calibration temperature and heat the exposed length of the thermocouple. The user must determine if the deviations in indicated temperature between the unit under test and the reference thermometer are significant relative to the uncertainty of calibration or exceed tolerances set by the user. Conversely, the test may be used with thermocouples or thermoelements to determine suitable uncertainty subcomponents to account for variations in depth of immersion in use. This test merely indicates that inhomogeneity is tolerable for application for spatial variations in temperature less abrupt than those imposed in the test. More abrupt spatial temperature variations imposed on the thermocouple (as in transient or specialized thermometry) could result in much larger errors than suggested by the procedure. It is not intended that this procedures quantify the Seebeck coefficient of the thermocouple elements nor provide corrections for thermocouples that fail the immersion tests. It provides only a reasonable indication of the thermocouple's ability to meet the specified tolerances independent of the details of the temperature profile between the measurement and reference junctions of the test article. This procedure does not subject the test article to any temperatures beyond those required for its calibration, but the practitioner should be aware of changes in the properties of Type KP, EP, and JP thermocouple elements due to prolonged exposure to temperatures in the 200 °C (400 °F) to 600 °C (1100 °F) range. When conducted at temperatures above approximately 200 °C (400 °F) for base metal thermocouples, or above 450 °C (840 °F) for noble metal thermocouples, this test is primarily intended for testing of thermocouples that will be used in a manner that precludes use of the thermocouple at a single, fixed immersion into an apparatus where the temperature profiles imposed on the thermocouple will be reproducible. When the temperature of the measuring junction does not exceed the above limits, this test is useful for quantifying the uncertainties resulting from the interaction of thermocouple inhomogeneity and variations in thermocouple immersion.

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