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**Designation: E 220 – 86 (Reapproved 1996)<sup>e1</sup>**



Designation: E 220 – 02

## Standard Test Method for Calibration of Thermocouples By Comparison Techniques<sup>1</sup>

This standard is issued under the fixed designation E 220; 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.

~~ε<sup>1</sup> Note—Keywords were added editorially in November 1996.~~

<sup>1</sup> This 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 March 27, 1986. Published May 1986. 10, 2002. Published September 2002. Originally published as E 220 – 63 T. Discontinued January 1995 and reinstated as E 220 – 86 (1996)<sup>ε1</sup>.

### 1. Scope

1.1 ~~This test method covers~~ describes the techniques of thermocouple calibration based upon comparisons of thermocouple indications principles, apparatus, and procedure for calibrating thermocouples by comparison with those of a reference thermometer, different from methods involving the use of fixed points. The precise evaluation of the electromotive force (emf)-temperature relation of a thermocouple is accomplished by determining its emf output at each of a series of measured temperatures: thermometer. Calibrations are covered over temperature ranges appropriate to the individual types of thermocouples within an over-all range from ~~about –180~~ approximately –195 to 1700 °C (–3290 to 2660°F)–3100 °F).

1.2 ~~In general, the test~~ this method is applicable to bare wire unused thermocouples. This 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. The latter thermocouples may require special care to control thermal conduction losses.

1.3 In this method, all values of temperature are based on the International Temperature Scale of 1990. See Guide E 1594.

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:

E 1 Specification for ASTM Thermometers<sup>2</sup>

E 77 Test Method for Inspection and Verification of Thermometers<sup>2</sup>

E 230 Specification for Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples<sup>2</sup>

E 344 Terminology Relating to Thermometry and Hydrometry<sup>2</sup>

~~E 563 Practice 452~~ Test Method for Calibration of Refractory Metal Thermocouples Using a Radiation Thermometer<sup>2</sup>

~~E 563 Practice for Preparation and Use of Freezing Point Reference Baths<sup>2</sup>~~

#### 2.2 ANSI Standard:

~~C 100.2 Direct-Current Ratio Devices: High Precision Laboratory Potentiometers<sup>3</sup>~~

#### 2.3 NIST Publications:

~~Circular 590—Methods of~~

~~E 644 Test Methods for Testing Industrial Resistance Thermometers<sup>2</sup>~~

~~E 988 Standard Temperature-Electromotive Force (EMF) Tables for Tungsten-Rhenium Thermocouples and<sup>2</sup>~~

~~E 1129 Specification for Thermocouple Materials<sup>4</sup>~~

~~Monograph 126—Platinum Resistance Thermometry<sup>4</sup>~~

~~Monograph 150—Liquid-in-Glass Thermometry<sup>4</sup> Connectors<sup>2</sup>~~

~~E 1594 Guide for Expression of Temperature<sup>2</sup>~~

<sup>2</sup> Annual Book of ASTM Standards, Vol 14.03.

<sup>3</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

<sup>3</sup> The boldface numbers in parenthesis refer to the list of references at the end of this standard.

<sup>4</sup> Available from National Institute

<sup>4</sup> For information on welding of Standards and Technology, U.S. Department of Commerce, Washington, DC 20234, measuring junctions, see Manual MNL-12.

E 1684 Specification for Miniature Thermocouple Connectors<sup>2</sup>

E 1751 Guide for Temperature Electromotive Force (EMF) Tables for Non-Letter Designated Thermocouple Combinations<sup>2</sup>

### **3. Terminology**

3.1 *Definitions*—The definitions given in Terminology E 344 shall be considered as applying apply to the terms used in 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 thermocouple—the type measurement are used to determine the repeatability of a thermocouple is represented by calibration process and to verify that the results of a letter designation as defined in accordance calibration processes are statistically consistent with Specification E 230: 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 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-w, 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-w 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-e 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, E, J, K, N, R, S, and T). The letter designation scheme is given in Specification E 230. The tables in E 988 and E 1751 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 By this test method a thermocouple is calibrated by comparing its indications with those

4.1 Comparison calibration consists of a reference thermometer at measuring the same temperature. The reference thermometer may be another thermocouple, a liquid-in-glass thermometer, or a platinum resistance thermometer, depending upon the temperature, the degree emf of accuracy required, or other considerations.

4.2 Since the success of the test method depends largely upon the ability to bring the thermocouple being calibrated in an isothermal medium while simultaneously measuring the standardized reference thermometer to the same temperature within the required limits of accuracy, 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, used medium with proper techniques, are specified for use in their respective temperature ranges.

4.3 Potentiometric instruments, or high-impedance electronic instruments, must be used for the measurement of emf eliminating instrument loading as a significant source of error. reference thermometer. The details of the test method, therefore, aim to provide assurance that the emf measured is actually the emf output of the thermocouple reference thermometer may be any thermometer with sufficient accuracy at the temperature of test and is not influenced by emf's arising from other sources. calibration.

### **5. Significance and Use**

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

5.2 This 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 state. Typically wire producers provide calibration thermocouples.

5.3 This method can be used for the verification of the individual conformance of thermocouple legs.

5.2 The test method provides for certifications materials to temperature tolerances for specifications such as the tables in Specification E 230 or other special specifications as required for commercial, military, or research applications.

5.3 The test method assumes that the materials are homogeneous.

### **6. Interferences**

6.1 Since the success of this 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 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. Comments on the testing of used thermocouples may be found in Manual MNL 12 (1).<sup>5</sup>

6.4 This 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 Method is recommended in combination with the furnaces and related procedures described in Method E 452.

## 7. Apparatus

67.1 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 below.

6.2 *Comparator 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 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.

67.2.1 *Liquid Baths*—In the range from ~~–160~~ from ~~–150~~ to 630 °C (~~–2540~~ to 1170 °F) the comparator bath shall usually consist of a well-stirred, insulated stirred liquid bath provided with controls for maintaining the temperature constant, a constant and uniform temperature. Suitable types are described in the appendix to Test Method E 77. ~~Laboratory type tube furnaces may be used above ambient~~ 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 but gradients that are not recommended for small relative to either fluidized powder baths or tube furnaces. A disadvantage of liquid baths is the most accurate work in this 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.

67.2.2 *Fluidized Powder Baths*—In the range from –70 to 980 °C (–100 to 1800 °F) the comparator bath may consist of an gas-fluidized bath of aluminum oxide or similar powder. ~~S~~ 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 ~~should be monitored to ensure consistency~~ 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.

67.2.3 *Tube Furnaces*—At temperatures above approximately 620 °C (1150 °F) an electrically heated tube furnace with a suitable nominally isothermal zone will usually constitute be used. Laboratory type tube furnaces may be used at any temperature provided that the comparator bath. increased uncertainty due to their spatial temperature variance is accounted for. Any one of a wide variety of designs may be suitable, but the furnace chosen should have the following capabilities:

6.2.3.1 Means should it shall be provided to control demonstrated that the furnace chosen can maintain a constant temperature

for short lengths stability of time (approximately  $\pm 1$  °C over a period of 10 min) at any temperature in the range over which the furnace is to be used:

~~6.2.3.2 There should~~ 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-z monitoring system that enables the user to visually determine that all heaters are operational and will require periodic remeasurement of-uniform the axial temperature into which profile. Single-zone furnaces may vary in temperature profile slowly as the-t heater element ages and will require only infrequent remapping of the temperature profile.

7.2.4 Other Baths—The one essential design feature of any bath to be used with this 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 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 red-uce the temperature differences between the reference thermometer and the-length test thermocouples. Such a block should be manufactured from a material of high thermal conductivity that will not contaminate the-f 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-adequate used to-permit a-depth reduce temporal fluctuations of-i the thermometers. The fluctuationsuff 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-measuring junction temperature 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-gradients-along 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-w used as a reference thermometer is a special case of an isothermal block formed by the common measuring junction.

NOTE 1—Further discussions of suitable tube furnaces are given in-X1.1 Appendix X1 and-X1.2.

~~6.2.4 Other Baths~~—The one essential design feature of any bath to be used with this method is that it brings the thermocouple being calibrated to the same temperature as the reference thermometer. Copper blocks immersed in liquid oxygen or some other refrigerant have been used successfully. 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 apparatus.

~~6.3~~ Appendix X2.

7.3 Reference Junction Temperatures—A controlled temperature-bath must medium shall be provided in which the temperature of the thermocouple reference junctions is maintained constant during a measurement cycle at a-chosen known or measured value. 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. The An acceptable method for utilizing the ice point as a reference junction is given in Practice E 563. Reports of data taken with reference temperatures other than the ice point should be-controlled corrected to reflect the results that would have been obtained if the reference junction had been at the ice point. As an alternative, calibration data taken with a-better accuracy 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. The reference junction temperature shall be known or measured with uncertainty less than that expected from the thermocouple calibration, to minimize this temperature variation as a source of-error. An acceptable method for utilizing error.

7.3.1 Isothermal and Electronic Reference Junction Compensation—For the ice point as a reference junction temperature is given in Practice E 563:

~~6.3.1~~ For the rapid calibration of large numbers of thermocouples, the reference junctions can be made at an isothermal multiterminal strip, whose temperature is determined by a reference thermocouple whose reference junction is in an ice-point bath. strip. This system avoids the thermal loading of the ice bath-by-a resulting from the large number of thermocouple-wires and copper connecting-wires.

~~6.3.2~~ Minimum error can wires. The temperature and isothermal condition of the strip shall be achieved-only established and monitored by-running 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 s;trip 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. Whatever reference junction technique is used, its uncertainty must be accounted for in the uncertainty of the thermocouple calibration being performed.

**7.3.2 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:

**7.3.2.1 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-j bath. In such circumstances, no additional corrections are required. A

**7.3.2.2 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 circuit due temperature range spanned by the extension wire and to measure the mismatch temperature of nominally similar alloys. The magnitude the junctions between the different materials in order to make this correction. No correction is necessary if both ends of the extension material are at temperatures within 1 °C (1.8 °F). It is also 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.

**7.3.2.3 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 E 1684 and E 1129 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-g differences.

**7.3.2.4 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  $\pm 2$  °C), thermocouple or extension grade wires of matching thermocouple type may be used in calibration circuits without correction.

**6.7.4 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 instrument can be chosen from one of three groups of commercially available, laboratory, high-precision types with thermocouple emf ranges suitable for use with thermometers. The first two groups are manually balanced potentiometers that are not self-contained and that require will be measured using a more-or-less permanent bench setup with a number of accessories, including a storage battery, high-sensitivity galvanometer or null detector, and a laboratory-type standard cell. All instruments require periodic calibration by digital voltmeter. For the National Institute of Standards and Technology or some other laboratory similarly qualified.

**6.4.1 Group A Potentiometers** shall be used where the highest accuracy is required. Potentiometers level of this group accuracy, voltmeters shall have a maximum uncertainty no-slide wires, all settings being made by means of dial switches. All design features will be consistent with greater than  $10^{-4}$  times the attainment of the highest accuracy. Such instruments emf reading and shall have input impedances larger than the thermocouple loop resistance by at least a limit factor of error of 0.2  $\mu$ V at 1000  $\mu$ V and 5  $\mu$ V or better at 50–000  $\mu$ V in accordance with ANSI C100.2.

**6.4.2 Group B Potentiometers** will normally be sufficiently accurate  $10^4$ . Reference junction compensation is required for thermocouple measurement with voltmeters. Such p In order to avoid forming unintended reference junctions at voltmeter terminals whose temperature may contain a slide wire, but all design features shall be directed toward high accuracy. Instruments poorly controlled, thermocouples must not be connected directly to the input terminals of this class shall have limits voltmeters without the use of error of 1  $\mu$ V at 1000  $\mu$ V and 12  $\mu$ V at 50–000  $\mu$ V.

**6.4.3 Group C Instruments** include appropriate electronic digital voltmeters reference junction compensation and analog-to-digital converters connection of potentiometric or other high-impedance design. Instruments of this class have limits of error similar the voltmeter to those in 6.4.1 and 6.4.2. These instruments permit fast readings of a large number of thermocouples. Such fast readings demand less temperature stability of the bath compensator with untimmed copper wires.

**6.7.5 Connecting Wire Assembly**—Connecting wires from the reference junction to the potentiometer are of voltmeter shall be insulated copper and should 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-conduit electrical shield or braided-cable if they are subject to electrical pickup.

**6.5.1 Selector switches** 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 and the standard thermocouple, calibrated.~~ Such switches ~~should~~ 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 ~~entirely disconnected from~~ electrically isolated. All of the ~~potentiometer.~~ The scanner switches ~~should~~ shall be constructed ~~with copper contacts, connections,~~ of the same material and terminals and ~~must~~ shall be located in the copper portion free of the circuit to preserve the all-copper circuit from the reference junction to the potentiometer, extraneous emf production (see Appendix X3). Precautions should be taken to protect the switches from temperature fluctuations due to ~~air currents~~ convection, conduction, or radiation from hot sources.

~~6.5.2 Terminal blocks may~~ 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 the connecting circuit, if convenient, calibration circuits, but should if terminal blocks are used for convenience, they shall be provided with copper binding posts and should be protected against the development of temperature gradients in across the blocks.~~

~~6.7.6 Thermocouple Insulation and Protection Tubes—Two-hole ceramic—~~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 conductors of a thermocouple. ~~Only suitable ceramic should be used, chosen of a thermoelements.~~ Use only insulation material ~~which that~~ will not contaminate the thermocouple (for example, clean, high-purity insulators such as 99.8 % aluminum oxide) and ~~which 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 and ~~should have bore~~ walled. Bore diameters that ~~should~~ provide a loose fit for the thermocouple wires ~~without binding.~~ wires. During the test, the thermocouples may be inserted in a protection tube ~~which should be~~ that is resistant to thermal shock, and noncontaminating to the thermocouple materials, and gastight.

~~6.6.1 Sheathed 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. The sheathed wire should extend, without splicing, to the reference junction for minimum error (see 6.3.2).~~

~~7.~~ typically by immersing the thermocouple into the bath until no further indication in temperature change is noted.

## **8. Reference Thermometers**

~~78.1 The reference thermometer to be used for the comparison calibration of thermocouples will depend upon the temperature range covered, whether a laboratory furnace or stirred liquid bath is used, the accuracy desired type of calibration apparatus, the calibration, accuracy desired, or in cases where more than one type of thermometer will suffice, the convenience or 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.~~

~~78.2 Platinum Resistance Thermometers—~~Platinum Resistance thermometers are an excellent choice as a reference in cases where the highest accuracy is required. Standard platinum resistance thermometer is (SPRTs) are the most accurate reference thermometers for use in stirred liquid baths at temperatures from approximately ~~-180.96 to 630°C (-300.962 °C (-310 to 1176.4 °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 cases where accuracy approaching 0.1°C (0.2°F) is required at temperatures below about ~~-60°C (-70°F) or above 200°C (400°F)~~, addition, there are few alternatives to the use 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 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 X2.23.

~~78.3 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—This type of thermometer—~~Liquid-in-glass thermometers may be used ~~from -180°C (-300°F), 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. The uncertainties~~ Further discussion of different types of liquid-in-glass thermometers ~~are~~ is given in X2.34. Specifications for ASTM thermometers are given in Specification E 1.

~~7.4~~

~~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 24-gage (0.51-mm) 0.5-mm (24-gage) diameter wire is recommended as the reference thermometer for temperatures from 9630 °C (11760 °F) to 1200 °C (2190 °F). Their use may also be extended down to room temperature. Accuracies Uncertainties attainable with careful use are given in Tables 1 and 2. Group A and B potentiometers (6.4.1 and 6.4.2) and Group C instruments (6.4.3) can be used with these thermocouples.

**7.5 Type B Thermocouples (Platinum-Rhodium/Rhodium-Platinum)**—The platinum-30 % rhodium/platinum-6 % rhodium (Type B) thermocouple, formed from 24-gage (0.51-mm) or larger size wire, is recommended as the reference thermometer for temperatures above 1200°C (2190°F). The uncertainties of temperature measurements with this type of thermometer are given in Table 1. Group A and B potentiometers (6.4.1 and 6.4.2) and Group C instruments (6.4.3) are suitable for use with this type of thermocouple.

**7.6 Type T Thermocouples (Copper-Constantan)**—This type of thermocouple may serve as a useful reference thermometer in the range of –180 to 370°C (–300 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 (400°F), and by the accuracy of the emf measurements and the inhomogeneity of the wire below 200°C. Twenty-four gage (0.51-mm) wire is a useful compromise between the lesser stability of smaller wire and the greater heat leakage of large wire. The uncertainties of temperature measurements with this thermocouple are given in Table 1. If measurements approaching an uncertainty of 0.1°C are to be made, a Group A potentiometer (6.4.1) must be used.

**7.7 Similar Thermocouples**—When Procedure C (9.4) is used, the reference thermometer shall be a previously calibrated thermocouple having the same composition as the test thermocouples.

## 8. Sampling

8.1 Sampling is normally specified in the ASTM material specification that calls for the calibration. As a guideline for compliance testing, a minimum of three samples are often taken for calibration compliance of a lot of wire or of thermocouples. In the case of wire, the samples should preferably be widely separated within the lot, for example, both ends and the middle of a coil. Users should be aware that in some instances compliance testing will cause changes to occur in the thermoelectric properties of the thermocouple wire.

## 9. General Procedures

9.1 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 a standard thermocouple or other thermometer standard. 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. One of the following three general methods may be used in the calibration procedure. 2.

**8.6 Procedure A** is applicable when a standard thermocouple and two potentiometers can be employed. The method is particularly adapted to the calibration Type B Thermocouples (Platinum-Rhodium versus Rhodium-Platinum)—The platinum-30 % rhodium versus platinum-6 % rhodium (Type B) thermocouple, of the thermocouples at any number of selected points when the same furnace 0.5-mm (24-gage) or larger diameter wire, is to be used over a wide temperature range, its temperature being changed for each point. Through recommended as the use of two potentiometers it is possible to make simultaneous readings of a standard thermocouple and a thermocouple being calibrated without waiting reference thermometer for the furnace or bath to stabilize at each temperature. temperatures above 1200 °C (2190 °F). The standard thermocouple is connected to one potentiometer and the thermocouple being calibrated to the other, as shown in Fig. 1. Each potentiometer is provided uncertainties of temperature measurements with a reflecting galvanometer. The spots this type of light are reflected from the two galvanometers onto a single scale, the galvanometers being adjusted so that the spots coincide at the zero of the scale when the circuits are open and, therefore, also when the potentiometers are set to balance the emf of each thermocouple. When more than one thermocouple is to be calibrated, a selector switch is introduced between the reference junctions of the thermocouples being calibrated and the associated potentiometer. The above procedure for taking data is then repeated for each thermocouple are given in turn. Tables 1 and 2.

**8.7 Procedure B** is applicable when only one potentiometer is used. The arrangement when several thermocouples are being calibrated using a Type T Thermocouples (Copper versus Constantan)—The type T thermocouple-thermometer may serve as a useful reference is shown thermometer in Fig. 2. This method requires that the bath or furnace including the thermocouple be stabilized at the desired temperature before readings are taken. Each thermocouple is connected range of –195 to the potentiometer 370 °C (–320 to 700 °F) in someq instances, although its accuracy is, in geneeral, limited by means the stability of the selector switch, as shown. The reference thermocouple should be read just before wire at temperatures above approximately 200 °C (390 °F), and just after by the reading accuracy of the emf of each thermocouple. After measuring measurements and the emf inhomogeneity of each thermocouple once, the whole sequence should be repeated at least once at the same temperature to give check readings of emf. When a large volume of work wire below 200 °C. One-half millimeter diameter (24 gauge) wire is involved, or when for some other reason it is inconvenient to use only one furnace or bath for all of the calibration temperatures, a series of furnaces or baths may be used, each being maintained at useful compromise between the different temperatures corresponding to the desired calibration temperatures. After insertion in each furnace or bath, time must be allowed for steady state conditions to be reached before readings are taken. Variations lesser stability of this method would include smaller wire and the use greater heat conduction of a platinum resistance thermometer or liquid-in-glass thermometer as reference in a stirred liquid bath. large wire.

**TABLE 1 Calibration Uncertainties in Calibrating Thermocouples by the Comparison Method—Temperatures in Degrees Celsius<sup>A</sup> (see Refs. 2 and 3)**

Thermocouple Temperature [°C]		Expanded Uncertainty $\overline{U}_y(k=2)$ [°C]	
Base metal thermocouple <sup>B</sup>	Temperature Range Base metal thermocouples <sup>A</sup>	Calibration Points <sup>C</sup> Temperature (range) At Observed Points	Uncertainty <sup>D</sup> In a tube furnace Points <sup>C</sup> Of Interpolated Values <sup>E</sup>
At comparison with calibrants		Of Interpolated Type S Values <sup>E</sup>	
<u>E</u>	200	0 to 870 <sup>F</sup>	every 100
—0.5	—1	—0.6	—0.2
400	0.4	—0.6	—0.2
600	0 to 870 <sup>F</sup>	—0.6	—0.2
300, 600, and 870	—0.5	2	—0.2
800	0.5	27	—0.2
1000	0 to 350 <sup>G</sup>	every 100	0.4
	0 to 350 <sup>G</sup>	every 100	0.4
	—160 to 0 <sup>G</sup>	every 50	—0.1-0
0.5	—J	1200	1.0
	—J	0 to 760 <sup>F</sup>	400, 300, 500, and 750
		0 to 350 <sup>G</sup>	every 100
		0 Base metal thermocouples <sup>A</sup>	400, 300, 500, and 750 (in stirred liquid baths, by—196 (type S) comparison with an SPRT)
—K	0 to 125	0 to 350 <sup>G</sup>	—0.1-0
—0F	0.1		—1.0
0	0.02		—1.0
every 100	—0.5	—1	—1.0
	200	—0.2	—1.0
	0 to 1250 <sup>F</sup>	—300, 600, 900, and 1200	—0.5
	0 to 350 <sup>G</sup>	400	—0.5
		every 100	0.4
		500	0.4
		every 50	—0.1
	—160 to 0 <sup>G</sup>		—0.1
		Type R and S thermocouples	200
		—160 to 0	—0.1
		comparison with a calibrated Type S thermocouple)	—0.1
		—R and —S	—0.1
		400	—0.1
		every 100	—0.3
600	—0.5 to 1100 and 2 at 1450	0.3	—0.3
800	0.3		—0.3
	0 to 1450 <sup>F</sup>	—0.3	—0.3
1000	0 to 1450 <sup>F</sup>	—0.3	—0.3
600 and 1200	—0.3	—1 to 1100 and 3 at 1450	—0.3
	1100	00 and 3 at 1450	—0.3
—2	—0 to 1700 <sup>F</sup>	every 100	—0.8
—	Type B thermocouples	200	0.8
B	comparison with calibrated Type S or Type B thermocouples)	—0.3	—0.5 to 1100 and 3 at 1700
		400	0.6
600	—0.3	—0.5	—0.5
—600 and —1200	—0.3	—0.5	—0.5
800	0.3	1 to 1100 and 5 at 1700	—0.5
—F	0 to 370 <sup>G</sup>	1 to 1100 and 5 at 1700 <sup>4</sup>	—0.5
	0 to 100 <sup>G</sup>		—0.5
	—160 to 0 <sup>G</sup>		—0.5
1100	0.370 <sup>G</sup>		—0.5
	0 to 100 <sup>G</sup>		—0.5
	—160 to 0 <sup>G</sup>		—0.5
every 100	—0.1-0.050-1	0.2	—0.5
50 and 100		0.1-0.2	—0.5
every 60	1450	1.6	—0.5

<sup>A</sup> Values given in this table are extracted from National Bureau of Standards Circular 590.

<sup>B</sup> See 3.2.

<sup>C</sup> Approximate calibration points.

<sup>D</sup> With homogeneous thermocouples and reasonable experimental care.

**TABLE 2 Calibration Uncertainties in Calibrating Thermocouples by the Comparison Method—Temperatures in Degrees Fahrenheit<sup>A</sup> (see Refs. 2 and 3)**

Thermocouple Temperature [°F]		Expanded Uncertainty $\bar{y}(k=2)$ [°F]	
Base metal thermocouple <sup>B</sup>	Temperature Range Base metal thermocouples <sup>A</sup>	Calibration Points <sup>C</sup> Temperature (range) At Observed Points	Uncertainty <sup>D</sup> In tube furnace Points <sup>C</sup> Of Interpolated Values <sup>E</sup>
	At comparison with a calibration <sup>E</sup>	Of Interpolated Type S Values <sup>E</sup>	
	<u>E</u>	32 to 1600 <sup>F</sup>	every 200 .4
1	400		
800	0.8		
1200	32 to 1600 <sup>F</sup>	.1	
600, 1100, and 1600	1600 <sup>F</sup>	.1	
1600	1	4	
2000	1	4.5	
32 to 650 <sup>G</sup> every 200	1.9	4	
	Base metal thermocouples <sup>A</sup> every 200	0.2	4 -256 to +32 <sup>G</sup> every 200
		-256 to +32	0.2
	comparison with an SPRT <sup>J</sup>		
	32 to 1400 <sup>F</sup>	300, 600, 1000, and 1400	1 2
	32 to 650 <sup>G</sup>	300, 600, 1000, and 1400 every 200	1 0.2 4
32	32 to 2300 <sup>F</sup>	every 200	0.2 4.04
K	32 to 2300 <sup>F</sup>	every 200	1 2
K	32 to 2300 <sup>F</sup>	400	1 20.4
	32 to 2300 <sup>F</sup>	600, 1200, 1800, and 2300	1 4
	32 to 650 <sup>G</sup>	800	1 40.7
		every 200	0.2 9
		1000	0.2 9
4		-256 to +32 <sup>G</sup> Type R and S -256 to +32 furnace, by comparison with a calibrated Type S thermocouple)	every 100 400 0.2 0.2
	R and S	32 to 2700 <sup>F</sup>	.4 .4
every 200	800		
1200	0.5		
1600	0.5		
2000	1 to 2000 and 4 at 2700		
1400 and 2200	0.5		
	32 to 2700 <sup>F</sup>	.4	
	600 <sup>F</sup>	.4	
	0.5	2 to 2000 and 5 at 2700	
	Type B thermocouples (in a tube furnace, by comparison with calibrated Type S or Type B thermocouples)	400	1.3
1200	0.7	B	0 to 3100 <sup>F</sup>
1600	0.7	800	1.00 <sup>F</sup>
1400 and 2200	0.5	every 200	0.5 1 to 2000 and 5 at 3100 <sup>F</sup>
2000	1.5	0.5	1 to 2000 and 5 at 3100 <sup>F</sup>
	32 to 700 <sup>G</sup>	2 to 2000 and 9 at 3100	
	32 to 200 <sup>G</sup>	2 to 2000 and 9 at 3100 every 200	0.20.4 0.4
	-256 to +32 <sup>G</sup>	110 and 200	0.2 0.4
		every 100	0.4 0.2
		2500	2.4 0.2 0.49

<sup>A</sup>This table is based upon the values in Table 1, but Fahrenheit temperatures are given in round numbers rather than exact equivalents of the Celsius temperature.

<sup>B</sup>See 3.2.

<sup>C</sup>Approximate calibration points.

<sup>D</sup>With homogeneous thermocouples and reasonable experimental care.

<sup>E</sup>Using difference curve from reference table.

<sup>F</sup>In tube furnaces, by comparison with a calibrated Type S thermocouple.

<sup>G</sup>In stirred liquid baths, by comparison with a standard platinum resistance thermometer.

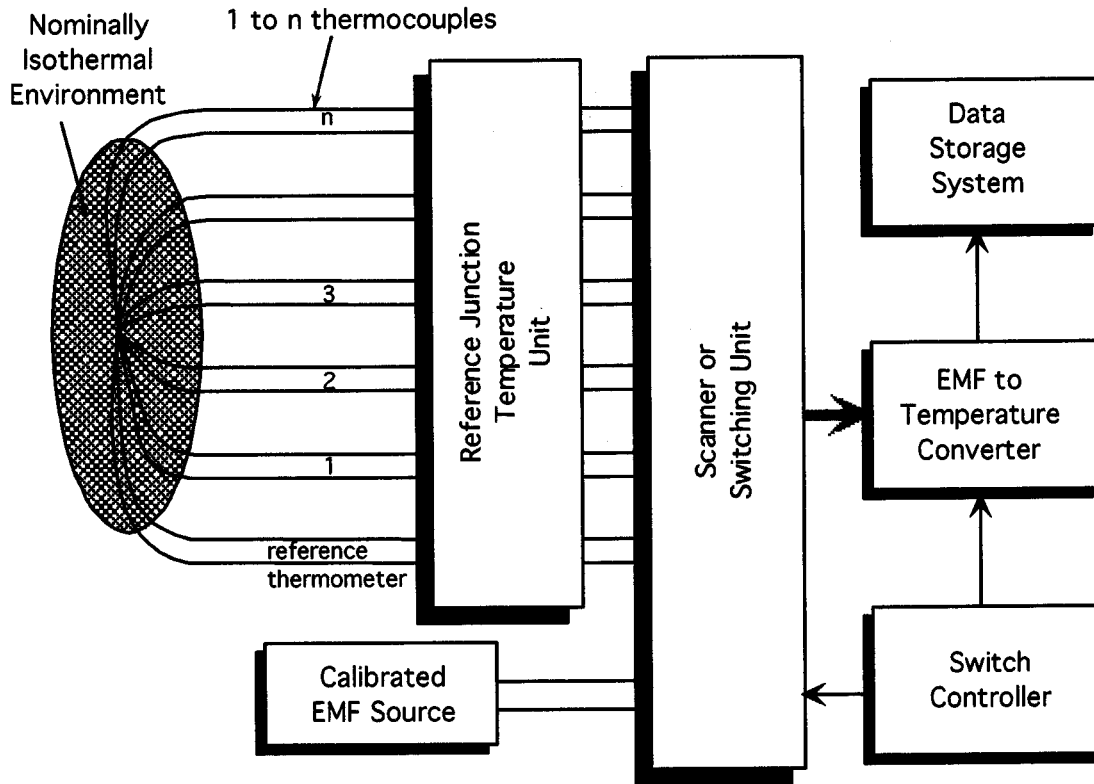


FIG. 1 Automated Thermocouple Data Acquisition System Layout—With Thermocouple Reference

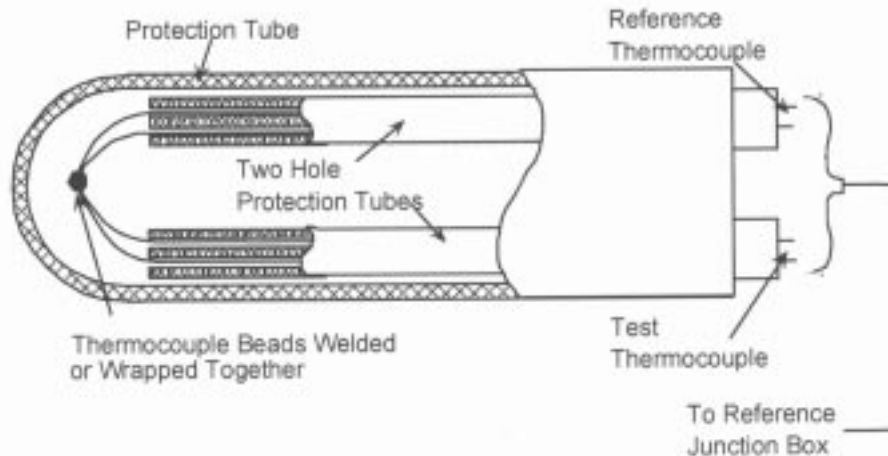


FIG. 2 Thermocouple Assembly in Protection Tube (Multilayered Cutaway View)

8.8 Procedure C

9.4.1 When the Gold versus Platinum Thermocouples—The gold versus platinum thermocouple being calibrated 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 same type as emf measurements and protection of the reference, as, for example, thermoelements from contamination is necessary.

8.9 Single-use Base-metal Thermocouples—For tests to elevated temperature, a Type S base metal thermocouple being taken from a calibrated against lot of wire of verified homogeneity may be used as a Type S reference thermometer. Lot homogeneity may be determined by calibrating thermocouple;s fabricated from a convenient variation statistical sample of the two potentiometer methods may be employed. With this technique, wire lot, and determining the standard deviation of emf values of the reference thermocouple is measured with one potentiometer, as in Procedure A, but in set of thermocouples, at each calibration temperature. In this ease application, the second potentiometer is used to measure simultaneously the relatively small emf difference between

the base-metal reference thermocouple would be used only at increasing test temperatures. Single-use base metal thermocouples are not amenable to recalibration, and the thermocouple being calibrated. At least two distinct advantages may an additional uncertainty must be realized through the use included to account for drift of this technique. First, since the emf differences are a small fraction of reference during the emf, they can be measured test. In particular, type E and type K thermocouples that have not been specially heat-treated are known to a higher degree exhibit shifts of absolute accuracy (microvolts). Secondly, if up to the emf differences vary equivalent of 4 °C (7 °F) in thermoelectric response after relatively slowly with change short exposures to temperatures in temperature, the actual temperature at the time of a measurement need not range 250 °C (480 °F) to 550 °C (1020 °F).

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

8.10 *Verification of furnace temperature can be Reference Thermometer Performance*—When platinum resistance thermometers or thermistors are used than that tolerated when using Procedure A. Because of these two circumstances as reference thermometers, the method may reference thermometer shall be readily automated.

9.4.2 When comparison measurements are being made verified by direct subtraction in this manner, there can be no electrical circuit between the various thermocouples except checking its indication at a thermometric fixed point. The ice point where or the difference voltage triple point of water is measured. In liquid and dry fluidized baths with common media, this requirement presents no problem, provided that commonly used. Liquid-in-glass thermometers shall be measured at the thermocouples do not touch ice point after each other. In tube furnaces it 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 E 77. Thermocouples, other than Au/Pt thermocouples, are not amenable to keep recalibration in an apparatus different than the various junctions at 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 temperature without electric contact. In this situation apparatus as used for the electrical isolation can test. Au/Pt thermocouples shall be achieved verified at the reference end a thermometric fixed-point or by use of an isolating potential comparator circuit.<sup>6</sup> The basic circuit is shown in Fig. 3. The isolating potential device is a double-pole, double-throw chopper (vibrating switch) and a capacitor which is first charged comparison measurement. For thermocouples of all types, verification points at temperatures close to the potential temperature of the reference thermocouple and then is moved into series opposition with the emf of the junctions are not a sensitive test thermocouple so that the difference potentiometer indicates the difference of possible changes in emf between the two. The total emf of thermocouple. Verification points should be chosen to have a temperature as far from the reference thermocouple junction temperature as practical

## 9. Sampling

9.1 Sampling is continuously monitored by normally specified in the other potentiometer. To obtain ASTM material specification that calls for the highest sensitivity, calibration. As a guideline for compliance testing, a minimum of two chopper and condenser units samples are used at 180-deg phase difference, as shown in Fig. 3. The potentiometers may be often calibrated to ensure that a lot of the manually balanced type, wire or if self-balancing potentiometers are used, assembled thermocouples conforms to standardized emf-temperature relations within specified tolerances. In the results may case of wire, the samples should preferably be presented on a recorder chart or other automatic data systems. With appropriate switching, automatic or manual operation, widely separated within the number lot, for example, opposite ends of thermocouples that may a coil. Users should be calibrated together aware that in some instances compliance testing will depend principally on cause changes to occur in the size thermoelectric properties of the furnace used. samples of thermocouple wire tested.

## 10. General Procedures

### 10.1 The calibration procedure consists of Thermocouples for Test

10.1 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 measuring the furnace or bath for 50 to 75 mm (2 to 3 in.). A two-hole ceramic insulating tube, somewhat longer than emf of the protection tube, shall be thermocouple being calibrated at selected for each thermocouple. Except when using Procedure C (9.4), calibration points, the wires temperature of each thermocouple are threaded through point being measured with the holes in its respective tube, selected reference thermometer. The number and choice of test points will depend upon the group type of thermocouple tubes loosely bundled together. The measuring junctions of all of thermocouple, the thermocouples may temperature range to be welded together into covered, and the accuracy required. Table 1 or Table 2 will serve as a common bead guide to provide good thermal contact between the junctions of selection. Both the different thermocouples.<sup>6</sup> If it is not convenient to weld nominally isothermal temperature calibration environment and the junctions together, the junction of each thermocouple thermocouples must be welded separately and stabilized at the junctions brought into good contact calibration temperature before readings are taken. For any particular apparatus, the necessary stability may be determined by wrapping them with platinum wire or foil. Slip performing comparison measurements at a variety of drift rates of the insulating

tubes down on reference thermometer temperature. A plot of the test thermocouple wires 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 measuring junctions rates of immersion and extraction are not so large as possible without stressing to damage either the wires. Insert thermocouples being calibrated or the bundle reference thermometer. After insertion of thermocouples to 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 protection tube; then place same throughout the tube at test. Otherwise, the proper depth shall not be less than any previous immersion. Techniques similar to that described in Appendix X4, but covering only the furnace or bath. Such an assembly is shown schematically range of immersion depth encountered in Fig. 4. In this process take care not to stress or cold work the wires. Special care must also calibration process, can be useful in determining if changes in immersion during handling the course of a calibration significantly affect the thermocouple wires to avoid contaminating them. results. One of the following two general methods may be used in the calibration procedure.

10.2 *Procedures for Types B, R, and S Thermocouples (Platinum-Rhodium/Platinum)*—This procedure for annealing is adapted to Method A, Automated Method—Fig. 2 illustrates the calibration schematic of bare wire platinum-10% rhodium/platinum (Type S) or platinum-13% rhodium/platinum (Type R) thermocouples in a typical data acquisition system—assuming a thermocouple is used as the temperature range from 0 to 1480°C (32 to 2700°F) and to reference thermometer. This method permits the calibration rapid testing of platinum-30% rhodium/platinum-6% rhodium (Type B) thermocouples in any number of thermocouples. The reference thermometer may be of any type meeting the range from 0 to 1700°C (32 to 3100°F). Suspend uncertainty requirements for the thermocouple freely in air from two binding posts, which should be close together so calibration, provided that the tension in data acquisition system can accommodate the wires and stretching while hot are kept corresponding type of signal. The thermocouples to a minimum. Shield be tested and the reference thermocouple from drafts. Electrically anneal are terminated at the reference junction temperature unit. This unit must meet the requirements of 7.3. A scanning unit sequentially connects each thermocouple in air for to the input of a period voltmeter. The voltmeter must meet the requirements of 4.5 min at approximately 1450°C (2650°F). Then cool it slowly (over 7.4. In the time interval between a period reading of approximately 1 min) to 750°C (1380°F) the reference thermometer and hold it at that temperature approximately 30 min. Following this anneal, allow of a test thermocouple, the thermocouple to cool to room temperature within a few minutes. Alternating current from a variable transformer is a convenient source of controlled power for heating the thermocouple wires, about 12 A being required for 24-gage (0.51-mm) wire. The calibration zone will vary. Measurements shall be made rapidly enough so that this variation in temperature is most readily determined by sighting on small in comparison to the platinum leg uncertainty of the thermocouple with an optical pyrometer. A pyrometer reading temperature measurement. The emf-measuring system is comprised of 1300°C (2380°F) will correspond a voltmeter, a display, and a data storage system. It may be desirable to provide a wire temperature of about 1450°C. This correction is necessary calibrated emf source and a zero (or shorted) input to account for the data acquisition system as references to improve the accuracy of the wire.

10.2.1 Following the anneal, thread the thermocouple through its insulating tube and mount, together with measurement. For a set of  $n$  test thermocouples, the recommended order of readings is: reference thermocouple, in a protection tube as described in 9.1. The thermometer, test thermocouple 1, test thermocouple 2, ... test thermocouple  $n$ , reference thermometer, test thermocouple shall be either a Type B, R, or S  $n$ , ... test thermocouple that has been calibrated at fixed points or 1, reference thermometer. If the variations in temperature indicated by comparison the reference thermometer are greater than desired with another thermocouple so calibrated. Special care must the above method, an alternative order of reading may be exercised during handling made, provided that the reference thermometer is read at least once for every reading of the annealed test thermocouple wires to avoid contaminating or stressing them.

10.3 *Procedure for Base-Metal Thermocouples in Laboratory Furnaces*—This procedure is applicable to and that the calibration order of the bare wire base-metal thermocouples readings is symmetric 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. both forward and reverse directions.

NOTE 2—This method is intended for use with new wire. Base-metal thermocouples undergo changes with use at high 3—The reference temperature which often render them unfit for recalibration. Therefore, these thermocouples should not be recalibrated.

10.3.1 Thread unit, the thermocouple through its insulating tube scanner, and mount with the emf-measuring system are typically combined into a reference thermocouple, in a protection tube as single system. They are described in 10.1. The reference thermocouple shall be either a Type B, S, or R thermocouple that has been calibrated at fixed points or by comparison with another thermocouple separately so calibrated. When calibrating base metal thermocouples against a Type B, S, or R thermocouple, Procedure C (9.4) cannot that their characteristics may be used. For Procedures A understood and B (9.2 and 9.3) protect the

reference thermocouple by a two-holed ceramic insulating tube specified to within a few millimetres of meet the measuring junction, and seal this end of required uncertainty criteria.

NOTE 4—Any program that performs manipulations on the ceramic tube to the thermocouple measured data shall be checked for accuracy by borosilicate glass or by a small amount of kaolin and sodium silicate cement. This protective measure minimizes contamination of evaluating 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 wires will not cause any error. If the wire becomes brittle at the junction with use, cut off this part of the wire and draw enough wire through the seal output response to form a new junction. Examine the seal after each use and remake it if it does not appear to known inputs.

**10.3 Method B, Manual Method**—This method may be good.

10.3.2 If the wires are large, used when one or more of the base-metal thermocouples may are to be welded together and a hole drilled in the common junction calibrated with manually operated switches. Each thermocouple is connected to accept the measuring junction of the voltmeter in sequence. The reference thermocouple. It is probably better practice, however, to weld thermometer should be read just before and just after the measuring junction reading of the reference each thermocouple i under calibration. After measuring the common junction as shown in Fig. 4.

10.3.3 When wires, insulators, and protection tubes emf of base-metal thermocouples are large, it is particularly important that tests be made (or have previously been made) to ensure that each test thermocouple, the depth of immersion is sufficient in the furnace or furnaces being used. Sheathed thermocouples can entire sequence should be calibrated in a tube furnace with no further protection provided that repeated at the furnace atmosphere is compatible with the sheath. The precaution discussed in 9.4 must be taken if differential comparison techniques are used. same temperature.

10.4 *Procedure for Base-Metal Thermocouples in Stirred Liquid Baths*—This procedure is applicable to Method C, Differential Method—When the thermocouple being calibrated ions of base-metal thermocouples, either bare wire or insulated, up to a temperature of approximately 980°C (1800°F). Usually no special preparation of the thermocouple will be required other than to insert it to same type as the bottom of reference thermometer a protection tube for immersion in the liquid bath. Borosilicate glass tubing has been found convenient for use up to 540°C (1000°F). From 540 to 620°C (1150°F), use vitreous silica or ceramic tubing. It should differential emf measurement method may be closed at employed. In this method, the immersed end, and large enough to permit easy insertion emf of the reference thermocouple  $\theta$  is measured and the differential emf between the thermocouples to being calibrated but no larger than necessary. Unfavorable heat transfer conditions in an unnecessarily large diameter tube will require a greater depth of immersion in and the bath than would a close-fitting tube. If a bare wire reference thermocouple is being calibrated, provide directly measured by connecting the w thermocouples in series, with electrical insulation over the length inserted in the protection tube. Slip a loose-fitting insulating sheath over polarity of one or both legs of the thermocouple. Use any suitable commercially available material, but select it to withstand the highest temperature to which it will be exposed. Immerse sheathed thermocouples directly reversed. Note that 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 steel thermowells into which the thermocouple protection tubes and standard thermometers are inserted for protection from the molten salt. The reference thermometer may be order to make a thermocouple inserted in serial connection the protection tube with the thermocouple being calibrated, or it may junctions must be a liquid-in-glass thermometer or resistance thermometer immersed in electrically isolated. This technique can offer improved stability since the bath liquid close to the thermocouple protection tube. The choice of a reference thermometer difference will remain relatively constant over small temperature intervals. For this technique a voltmeter with uncertainty up to 0.5  $\mu$ V can be governed principally by the accuracy required (see Section 7). 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.<sup>4</sup> 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 an 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 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 8 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 ~~both platinum~~ 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 (6.2.3), (7.2.3 and Appendix X1), and assemble the reference junction, connecting wires, and switching appropriate to Procedures A, B, or C as described in Section 9, whichever is selected to be used.

**11.1.1.1 Procedure A**—Measurements at 7. For base-metal thermocouples, heat the calibration points should start with the lowest temperature and be continued through succeeding higher temperatures. Set the potentiometer connected to the reference thermocouple to the emf corresponding to the desired temperature and start the furnace heating. Heating may be rapid until the calibration point is nearly reached, at which time the power is cut back so as to stop the temperature rise at a temperature just above that required. With power to the furnace reduced, or off, lower the furnace temperature through the calibration point at a rate not exceeding 0.5°C (1°F)/min. Occasional adjustment of the spots from the two galvanometers will be necessary to keep their null positions coincident on the common scale at all times. As the furnace cools, adjust the setting of the potentiometer connected to the test thermocouple continuously until its associated galvanometer spot crosses its null position at the same time as the galvanometer spot for the standard thermocouple crosses its null position. When this setting is reached, the emf of the test thermocouple corresponds to the temperature indicated by the reference thermocouple. Repeat the measurement with power to the furnace, and the temperature rising at nearly the same rate of temperature change as that which occurred in the first measurement with the temperature falling. The two emf measurements should not differ by more than 5 µV; take their average as the emf of the test thermocouple at the calibration point. Repeat the measurements at each successive calibration point.

**11.1.1.2 Procedure B**—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 thermocouple-fs to ensure temperature measurement. Read alternately the emf's 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 reference, thermocouples being calibrated or the test thermocouple, and reference thermometer. Read the reference again. A comparison thermometer and test thermocouples, according to Method A, B, or C as described in Section 10. Inspection of the two readings variation of the reference thermocouple thermometer readings will indicate whether the furnace has 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 two emf temperature readings of the reference thermocouple 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 multiplying by the ratio (Table subtracting an emf 1st measurement/2nd measurement) where term corresponding to the Table emf is that listed for temperature difference between the second and first test thermocouple 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 E 230, Standard E 988, or Guide E 1751, corrections can be applied for temperature differences of up to 10°C (18°F) 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.

**11.1.1.3 Procedure C**—The exact procedure to points. Noble metal thermocouples may be adopted will depend upon calibrated in either ascending or descending temperature steps. Normalization methods that are mathematically equivalent to the particular apparatus being used when some version of the isolating potential comparator circuit is being used. Therefore, no specific procedures can be outlined here, except above method, to point out that many within the small interpolation errors of the precautions and practices representing good techniques described above must be observed. The rate of change of furnace temperature method, may be significantly higher than is permissible with Procedure A, but it probably should not exceed 10°C (18°F)/min used for analyzing the most accurate work.

~~11.1.2~~ 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 ~~Procedure B~~ that for use with laboratory furnaces described in ~~11.1.1.2~~, ~~12.1.1~~, except that the reference thermometer may be is often a liquid-in-glass or resistance thermometer instead of a thermocouple. When measurements approaching an accuracy uncertainty of 0.1 °C are to be made. ~~Potentiometers made, voltmeters with a limit of error measurement uncertainty no greater than given in 6.4.1 7.4~~ must be used.

### 123. Calculations

~~123.1 Emf Difference Function Calculation~~—Having determined the emf of the thermocouple at a number of calibration points, complete the calibration is completed by interpolating between the calibration points. ~~Different Various interpolation methods may be used to accommodate special circumstances, but t.~~ The method using involving the use of a difference curve from an arbitrary a reference table is often the simplest and most accurate to use. To use this method, choose the appropriate reference table in Specification ~~E 230 to establish~~ E 230, Standard E 988, or Guide E 1751 as a basis for establishing a difference curve.

~~NOTE 3~~—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.

~~12.2 Calculate 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. ~~With values Include the value of  $\Delta E = 0$  as 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  $\mu\text{V}$  for type S and R thermocouples and from 1  $\mu\text{V}$  to 5  $\mu\text{V}$  for base metal thermocouples. The resulting function for  $\Delta E$  as abscissas, plot 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  $\Delta E$  for each calibration point and draw as a smooth curve through the points. The value function of  $\Delta E = 0$  at of the reference junction temperature is added as an additional point on the curve. Draw the straight line,  $\Delta$  test thermocouple are obtained. At any observed value of  $E = 0$ , which represents the difference curve for the table values of emf. At any observed value of  $E$  add the corresponding value of  $\Delta E$  from the curve and enter the table at this corrected value of emf to obtain the true temperature. A difference curve typical of platinum-10% rhodium/platinum is shown in Fig. 5. Note that the The values of  $\Delta E$  take the sign of a correction which is to be added to the observed emf to give emf, producing a corrected emf with which one can enter the standard table to get the true temperature. For example, in Fig. 5 at 7 mV. Alternatively, the correction is  $-2 \mu\text{V}$ . Therefore, with an observed emf of 7 mV enter function  $\Delta E(t)$  may be subtracted from the table at  $7.000 - 0.002 = 6.998 \text{ mV}$  thermocouple reference function to obtain create a single function giving the corresponding temperature.

~~12.3~~ When Procedure C is being used, the data may test thermocouple emf as a function of temperature. Emf functions shall not be recorded in any one used outside the range of calibration.

~~NOTE 6~~—These table values are based upon a reference junction temperature of ways. When manually balanced potentiometers are 0 °C or 32 °F. If another reference junction temperature has been used, the data may reference table values must be used to produce a difference curve adjusted by subtracting from a standard each the table as described above, or value of emf corresponding to the reference junction temperature used.

~~13.2 Emf Function Calculation~~—In a few special cases it may be used desirable to model the functional relationship of emf with temperature ( $E(t)$ ) without utilizing differences from a complete table by computer using standard numerical methods for curve fitting.<sup>6</sup> When self-balancing potentiometers 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 data may number of coefficients of the functional form should not be printed on much greater than the chart number of an X-Y recorder, where data points divided by two. In no case should the emf difference is plotted as a functional form have more parameters than the reference function in Specification E 230 or other relevant specification. Modeling of data by direct calculation of the total 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 reference thermocouple. Still further automation is possible using computerized systems.

~~13.~~ method of emf differences. Emf functions shall not be used outside the range of calibration.

### 14. Report

~~134.1~~ Report the calibration results in any convenient form, as required by the user. This may be the function  $E(t)$ , a table of values of  $E$  at a number of temperatures or it may be temperatures, a table of values of  $\Delta E$  at selected values of  $E$ .

**14. Precision, or a table of values of the temperature equivalent,  $\Delta t$ , of  $\Delta E$ , 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. Uncertainty**

15.1 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 skills of the operators in unspecified techniques. The uncertainties given in Tables 1 and 2 represent the capability of the committee members and are not supported method as achieved by NIST (see Ref (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 Method. An uncertainty budget including each of these terms should be derived. 14.2 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.

14.2.1

15.2 *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 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. These uncertainties may result when homogeneous uncertainties for calibration of base metal thermocouples are used and when reasonable care is exercised in include an allowance for the work.

14.2.2 *Accuracy inhomogeneity of the unused thermoelements.*

15.3 *Uncertainty of Interpolated Values*—The accuracy—The uncertainty of interpolated values will depend upon the number of calibration points and the closeness with which the reference table function used represents the behavior of the particular thermocouple being calibrated. The more accurately the values conform to the emf-temperature relationship of the actual thermocouple in the reference table, function, the fewer the number of calibration points required for a given accuracy 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. The Tables 4 and 5 give approximate uncertainties of interpolated values using interpolation for thermocouples that meet the method standard tolerances of Specification E 230, provided that the interpolation method is based on a smooth curve modeling the difference curves of the emf from the reference function. These uncertainties were extracted from Ref (4), which gives the uncertainty of a calibration both at the temperature values are given in Tables 1 tested and 2. These of interpolated values at temperatures between calibration points. Data is not available for type N thermocouples, but the listed interpolation uncertainties are expected to be no worse than for type K thermocouples.

15.3.1 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:

**TABLE 3 Components of Calibration Uncertainties**

Uncertainty Component	Evaluation Method
1. Test thermocouple inhomogeneity and drift	Literature, or Section 10.1 or Appendix X4.1
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 15.3

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

Thermocouple Type <sup>A</sup>	Temperature Range	Calibration Points	Expanded ( $k=2$ ) Uncertainty of Interpolation, for Temperatures other than Calibration Points <sup>B</sup>
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
J	-195 to 0	every 50	0.4
	0 to 760	100, 300, 500, and 750	0.5
K	0 to 350	every 100	0.4
	0 to 1250	every 100	0.5
R and S	0 to 1250	300, 600, 900, and 1200	1.5
	-195 to 0	every 50	0.4
T	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

<sup>A</sup> See Specification E 230.

<sup>B</sup> Using difference curve from reference table.

**TABLE 5 Additional Calibration Uncertainties Due to Interpolation—Temperatures in Degrees Fahrenheit<sup>A</sup>**

Thermocouple Type <sup>B</sup>	Temperature Range	Calibration Points	Expanded ( $k=2$ ) Uncertainty of Interpolation, for Temperatures other than Calibration Points <sup>C</sup>
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
J	-320 to 32	every 100	0.7
	32 to 1400	300, 600, 1000, and 1400	0.9
K	32 to 650	every 200	0.7
	32 to 2300	every 200	0.9
R and S	32 to 2300	600, 1200, 1800, and 2300	3
	-320 to 32	every 100	0.7
T	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

<sup>A</sup> 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.

<sup>B</sup> See Specification E 230.

<sup>C</sup> Using difference curve from reference table.

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

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.

14.2.3—

**15.4 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 elements, which thermoelements, that will cause the emf output of the thermocouple to be dependent upon the particular temperature gradient 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 which that can arise from this source is not feasible, but the possibilities of such errors should be recognized in assigning accuracies 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).

## 156. Keywords

15.1 comparator baths;

16.1 calibration; comparison calibration; thermocouple 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, designed to operate on either 110 or 220 V, and commercially. Such furnaces are typically equipped with an adjustable rheostat or other a means for 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. Specific information is known, however, for the furnace described in X1.2. With this furnace the central 150 mm (6 in.) of the 600-mm (24-in.) long tube has been found to be practically at a uniform temperature. If no more than two thermocouples of 8-gage (3.26-mm) wire and a 24-gage (0.51-mm) Type S reference thermocouple are mounted in separate two-hole porcelain insulators and bundled together in a ceramic protection tube, as described in Section 10, immersion to the center of the furnace tube 300 mm (12 in.) has been found adequate. tube.

##### X1.2 Tube-Type Heating Ceramic Element Electric Tube Furnaces

X1.2.1 AFor temperatures above 1200 °C (2200 °F), laboratory tube furnaces with heating element furnace may be found convenient when Procedure A (Section 9.2) is to be used. One successful design elements of furnace is shown schematically in Fig. X1.1. The heating element consists of a nickel (80)-chromium (20) tube clamped between two water-cooled terminals. The tube, which is 22 mm (7/8 in.) in inside diameter, 31 mm (1¼ in.) in outside diameter and 600 mm (24 in.) long, is heated electrically, the tube itself serving materials such as the heating element silicon carbide (SiC) or resistor. The large current necessary to heat the tube is obtained from a transformer. A radiation shield and a furnace jacket are mounted around the heating tube, molybdenum disilicide (MoSi<sub>2</sub>) may be used as shown in Fig. X1.1 an alternative to minimize temperature gradients along the tube platinum wire. Silicon carbide furnaces operate upto 1500 °C or 1600 °C (2700 °F or 2900 °F) and molybdenum disilicide to reduce radiation losses. To minimize the time required for heating and cooling of the furnace, no thermal insulation is used between the heating tube, the radiation shield, and the furnace jacket. This furnace responds rapidly, both in heating and cooling; but is not recommended for procedures requiring the furnace to stabilize at a particular temperature for a period of time, as is the case when the reference approximately 1700 °C (3100 °F). For thermocouple and the unknown must calibration purposes, these furnaces should always be read alternately fitted with a single potentiometer. suitable ceramic tube.

## X2. REFERENCE THERMOMETERS

### X2.1 Standard Platinum Resistance Thermometers

X2.1.1 The standard platinum resistance thermometer is the specified instrument for interpolation between fixed points on the International Practical Temperature Scale of 1968 in the range from  $-260$  to  $+630^{\circ}\text{C}$ . Temperatures thermometers (SPRTs) are not measured directly with this instrument. Its electrical resistance is determined by comparison with a standard resistor using a potentiometer, a Kelvin-type double bridge, or a Wheatstone bridge, preferably of the Mueller type. Temperatures are then calculated using prescribed resistance-temperature formulas. Such resistance most accurate reference thermometers should be so designed and constructed that are used in defining the wire of the platinum resistor ITS-90 from approximately  $-259$  to  $962^{\circ}\text{C}$ . The SPRT sensing element is as strain-free as practicable made from pure platinum and will remain so during use. In addition, certain restrictions are imposed on the electrical and physical properties supported essentially strain-free. Because of the wire of which delicate construction, the resistor SPRT is formed. For use at temperatures up easily damaged by mechanical shock and must be handled carefully to  $500^{\circ}\text{C}$  ( $930^{\circ}\text{F}$ ), these instruments are usually made with a borosilicate glass protection tube. This range is extended to  $630^{\circ}\text{C}$  ( $1160^{\circ}\text{F}$ ) through retain its calibration.

NOTE X2.1—No single SPRT covers the use entire temperature range of silica glass protection tubes. More details may be obtained from publications from the National Bureau of Standards, Monograph 126,  $-260$  to  $962^{\circ}\text{C}$ .

### X2.2 Direct-Reading Industrial Platinum Resistance Thermometers

#### X2.2.1 Direct-reading

X2.2.1 Certain industrial platinum resistance thermometers are available commercially specially manufactured and may be used when the highest accuracy is not required. subjected to special heat treatment and calibration to establish their measurement uncertainty. These instruments have the advantage 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 temperature. Method E 644.

### 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

X2.34.1 The liquid-in-glass thermometers can be used as a relatively simple and accurate temperature references over a wide range range of moderate temperatures when good usage techniques are followed. The uncertainties given in Tables X2.1-X2.3 apply to well designed and carefully manufactured thermometers which have been calibrated and all corrections have been applied. Discussions of the calibration and use of liquid-in-glass thermometers are given in the National Institute of Standards Ref (5,6) and Technology Monograph 150 and Test Method E 77. E 77.

### 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 ~~stray thermal~~ 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 or Fig. 2, 1, remove one of the ends of the test thermocouple from the reference junction bath and connect a short piece of copper wire ~~between~~ across the pools of mercury in the two glass tubes: measurement terminals. This copper link will complete the circuit through the connecting wires to the ~~potentiometer, voltmeter,~~ which will now indicate any emf originating in the selector switch, scanner system, binding posts, or other system c-omponents, as well as ~~stray~~ 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. ~~A simpler alternative~~ 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 ~~remove one~~ 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 insert it into 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 de-~~termine~~ terminate 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 w (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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