Designation: E 220 – 86 (Reapproved 1996)$^{\epsilon 1}$

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

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^1 NOTE—Keywords were added editorially in November 1996.

1. Scope

1.1 This test method covers the techniques of thermocouple calibration based upon comparisons of thermocouple indications 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. Calibrations are covered over temperature ranges appropriate to the individual types of thermocouples within an over-all range from about − 180 to 1700°C (−290 to 2660°F).

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

2. Referenced Documents

2.1 *ASTM Standards:*

E 1 Specification for ASTM Thermometers[2]

E 77 Test Method for Inspection and Verification of Thermometers[2]

E 230 Specification for Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[2]

E 344 Terminology Relating to Thermometry and Hydrometry[2]

E 563 Practice for Preparation and Use of Freezing Point Reference Baths[2]

2.2 *ANSI Standard:*

C 100.2 Direct-Current Ratio Devices: High Precision Laboratory Potentiometers[3]

2.3 *NIST Publications:*

Circular 590—Methods of Testing Thermocouples and Thermocouple Materials [4]

Monograph 126—Platinum Resistance Thermometry[4]

Monograph 150—Liquid-in-Glass Thermometry[4]

3. Terminology

3.1 *Definitions*—The definitions given in Terminology E 344 shall be considered as applying to the terms used in this method.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *type of thermocouple*—the type of a thermocouple is represented by a letter designation as defined in accordance with Specification E 230.

3.2.2 *reference thermometer*—a thermometer whose calibration is known within a certain specified accuracy.

4. Summary of Test Method

4.1 By this test method a thermocouple is calibrated by comparing its indications with those of a reference thermometer at 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 of accuracy required, or other considerations.

4.2 Since the success of the test method depends largely upon the ability to bring the thermocouple and 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 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. The details of the test method, therefore, aim to provide assurance that the emf measured is actually the emf output of the thermocouple at the temperature of test and is not influenced by emf's arising from other sources.

5. Significance and Use

5.1 For users or manufacturers of thermocouples, the test method provides a means of confirming the acceptability of the material in the assembled state. Typically wire producers provide calibration of the individual thermocouple legs.

[1] This method is under the jurisdiction of ASTM Committee E-20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.04 on Thermocouples.

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[2] *Annual Book of ASTM Standards*, Vol 14.03.

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

[4] Available from National Institute of Standards and Technology, U.S. Department of Commerce, Washington, DC 20234.

5.2 The test method provides for certifications to temperature tolerances for specifications such as 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. Apparatus

6.1 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 Baths and Furnaces*—A controlled comparator 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.

6.2.1 *Liquid Baths*—In the range from − 160 to 630°C (−250 to 1170°F) the comparator bath shall usually consist of a well stirred, insulated liquid bath provided with controls for maintaining the temperature constant. Suitable types are described in the appendix to Test Method E 77. Laboratory type tube furnaces may be used above ambient temperature but are not recommended for the most accurate work in this temperature range.

6.2.2 *Fluidized Powder Baths*—In the range from − 70 to 980°C (−100 to 1800°F) the comparator bath may consist of an air-fluidized [5] bath of aluminum oxide or similar powder. Such a bath should be monitored to ensure consistency and uniformity of temperature.

6.2.3 *Tube Furnaces*—At temperatures above approximately 620°C (1150°F) an electrically heated tube furnace shall usually constitute the comparator bath. 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 be provided to control the furnace at a constant temperature for short lengths of time (approximately 10 min) at any temperature in the range over which the furnace is to be used.

6.2.3.2 There should be a zone of uniform temperature into which the thermocouple measuring junctions may be inserted, and the length of the furnace tube must be adequate to permit a depth of immersion sufficient to assure that the measuring junction temperature is not affected by temperature gradients along the thermocouple wires.

NOTE 1—Further discussions of suitable tube furnaces are given in X1.1 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 *Reference Junction Temperatures*—A controlled temperature bath must be provided in which the temperature of the reference junctions is maintained constant at a chosen 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 reference junction temperature should be controlled to a better accuracy than that expected from the thermocouple calibration to minimize this temperature variation as a source of error. An acceptable method for utilizing 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. This system avoids thermal loading of the ice bath by a large number of thermocouple wires and copper connecting wires.

6.3.2 Minimum error can be achieved only by running the thermocouple wires, without splices, from the measuring junction to the reference junction. Any splice represents an inhomogeneity in the circuit due to the mismatch of nominally similar alloys. The magnitude of the error due to the mismatch will depend on the temperature gradients existing.

6.4 *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 emf ranges suitable for use with thermometers. The first two groups are manually balanced potentiometers that are not self-contained and that require 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 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 of this group have no slide wires, all settings being made by means of dial switches. All design features will be consistent with the attainment of the highest accuracy. Such instruments shall have a limit of error of 0.2 μV at 1000 μV and 5 μV or better at 50 000 μV in accordance with ANSI C100.2.

6.4.2 *Group B Potentiometers* will normally be sufficiently accurate for most work. Such potentiometers may contain a slide wire, but all design features shall be directed toward high accuracy. Instruments of this class shall have limits of error of 1 μV at 1000 μV and 12 μV at 50 000 μV.

6.4.3 *Group C Instruments* include electronic digital voltmeters and analog-to-digital converters of potentiometric or other high-impedance design. Instruments of this class have limits of error similar 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 with time.

[5] Callahan, J. T., "Heat Transfer Characteristics in Air Fluidized Solids up to 900°F," ASME Paper 70W A/Temp 3, *Journal of Basic Engineering*, 1971.

6.5 *Connecting Wire Assembly*—Connecting wires from the reference junction to the potentiometer are of insulated copper and should be run in a grounded conduit or braided cable if they are subject to electrical pickup.

6.5.1 Selector switches may be used to switch between different thermocouples being calibrated and the standard thermocouple. Such switches should 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 the potentiometer. The switches should be constructed with copper contacts, connections, and terminals and must be located in the copper portion of the circuit to preserve the all-copper circuit from the reference junction to the potentiometer. Precautions should be taken to protect the switches from temperature fluctuations due to air currents or radiation from hot sources.

6.5.2 Terminal blocks may be used in the connecting circuit, if convenient, but should be provided with copper binding posts and should be protected against the development of temperature gradients in the blocks.

6.6 *Thermocouple Insulation and Protection Tubes*—Two-hole ceramic 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 material which will not contaminate the thermocouple and which will provide the necessary electrical insulation at the highest temperature of the calibration. 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 diameters that provide a loose fit for the thermocouple wires without binding. During the test, the thermocouples may be inserted in a protection tube which should be resistant to thermal shock, noncontaminating to the thermocouple materials, and gastight.

6.6.1 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. 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. Reference Thermometers

7.1 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 of the calibration, or in cases where more than one type of thermometer will suffice, the convenience or preference of the calibrating laboratory.

7.2 *Platinum Resistance Thermometers*—The standard platinum resistance thermometer is the most accurate reference thermometer for use in stirred liquid baths at temperatures from approximately − 180 to 630°C (−300 to 1170°F). 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), there are few alternatives to the use of resistance thermometers as references. Standard resistance thermometers are described in X2.1 and X2.2.

7.3 *Liquid-in-Glass Thermometers*—This type of thermometer may be used from − 180°C (−300°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 of different types of liquid-in-glass thermometers are given in X2.3. Specifications for ASTM thermometers are given in Specification E 1.

7.4 *Types R and S Thermocouples (Platinum-Rhodium/Platinum)*—The platinum-10 % rhodium/platinum (Type S), or the platinum-13 % rhodium/platinum thermocouple (Type R) of 24-gage (0.51-mm) wire is recommended as the reference thermometer for temperatures from 630°C (1170°F) to 1200°C (2190°F). Their use may also be extended down to room temperature. Accuracies attainable with careful use are given in Table 1. 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/platinum6 % 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

TABLE 1 Calibration Uncertainties in Calibrating Thermocouples by the Comparison Method—Temperatures in Degrees Celsius[A]

Thermocouple Type[B]	Temperature Range	Calibration Points[C]	Uncertainty[D] At Observed Points	Uncertainty[D] Of Interpolated Values[E]
E	0 to 870[F]	every 100	0.5	1
	0 to 870[F]	300, 600, and 870	0.5	2
	0 to 350[G]	every 100	0.1	0.5
	−160 to 0[G]	every 50	0.1	0.5
J	0 to 760[F]	100, 300, 500, and 750	0.5	1
	0 to 350[G]	every 100	0.1	0.5
K	0 to 1250[F]	every 100	0.5	1
	0 to 1250[F]	300, 600, 900, and 1200	0.5	2
	0 to 350[G]	every 100	0.1	0.5
	−160 to 0[G]	every 50	0.1	0.5
R and *S*	0 to 1450[F]	every 100	0.3	0.5 to 1100 and 2 at 1450
	0 to 1450[F]	600 and 1200	0.3	1 to 1100 and 3 at 1450
B	0 to 1700[F]	every 100	0.3	0.5 to 1100 and 3 at 1700
		600 and 1200	0.3	1 to 1100 and 5 at 1700
T	0 to 370[G]	every 100	0.1	0.2
	0 to 100[G]	50 and 100	0.05	0.1
	−160 to 0[G]	every 60	0.1	0.2

[A] Values given in this table are extracted from National Bureau of Standards Circular 590.
[B] See 3.2.
[C] Approximate calibration points.
[D] With homogeneous thermocouples and reasonable experimental care.
[E] Using difference curve from reference table.
[F] In tube furnaces, by comparison with a calibrated Type S thermocouple.
[G] In stirred liquid baths, by comparison with a standard platinum resistance thermometer.

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.

9.2 *Procedure A* is applicable when a standard thermocouple and two potentiometers can be employed. The method is particularly adapted to the calibration of the thermocouples at any number of selected points when the same furnace is to be used over a wide temperature range, its temperature being changed for each point. Through the use of two potentiometers it is possible to make simultaneous readings of a standard thermocouple and a thermocouple being calibrated without waiting for the furnace or bath to stabilize at each temperature. 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 with a reflecting

TABLE 2 Calibration Uncertainties in Calibrating Thermocouples by the Comparison Method—Temperatures in Degrees Fahrenheit[A]

Thermocouple Type[B]	Temperature Range	Calibration Points[C]	Uncertainty[D] At Observed Points	Uncertainty[D] Of Interpolated Values[E]
E	32 to 1600[F]	every 200	1	2
	32 to 1600[F]	600, 1100, and 1600	1	4
	32 to 650[G]	every 200	0.2	1
	−256 to + 32[G]	every 100	0.2	1
J	32 to 1400[F]	300, 600, 1000, and 1400	1	2
	32 to 650[G]	every 200	0.2	1
K	32 to 2300[F]	every 200	1	2
	32 to 2300[F]	600, 1200, 1800, and 2300	1	4
	32 to 650[G]	every 200	0.2	1
	−256 to + 32[G]	every 100	0.2	1
R and *S*	32 to 2700[F]	every 200	0.5	1 to 2000 and 4 at 2700
	32 to 2700[F]	1100 and 2200	0.5	2 to 2000 and 5 at 2700
B	0 to 3100[F]	every 200	0.5	1 to 2000 and 5 at 3100
		1100 and 2200	0.5	2 to 2000 and 9 at 3100
T	32 to 700[G]	every 200	0.2	0.4
	32 to 200[G]	110 and 200	0.1	0.2
	−256 to + 32[G]	every 100	0.2	0.4

[A] 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.
[B] See 3.2.
[C] Approximate calibration points.
[D] With homogeneous thermocouples and reasonable experimental care.
[E] Using difference curve from reference table.
[F] In tube furnaces, by comparison with a calibrated Type S thermocouple.
[G] In stirred liquid baths, by comparison with a standard platinum resistance thermometer.

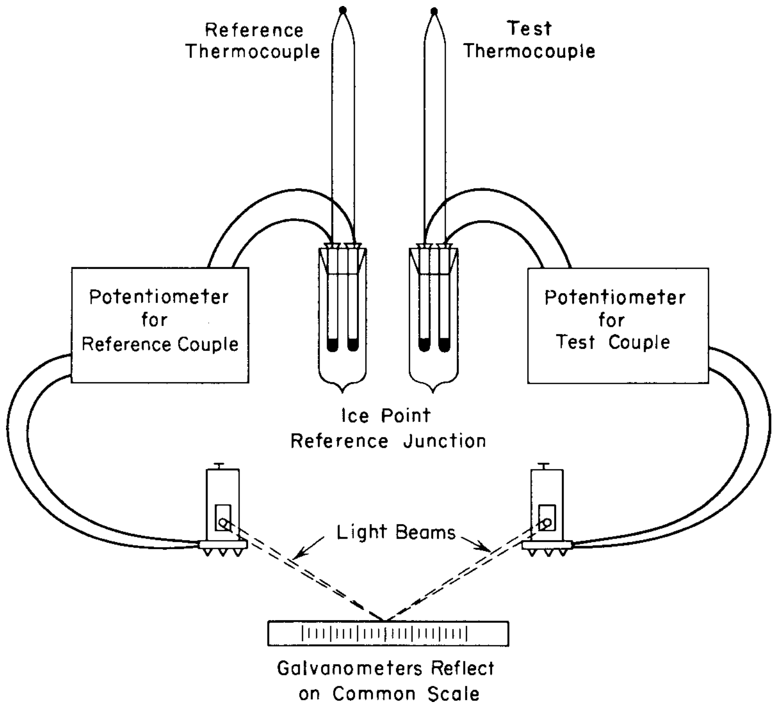

FIG. 1 Schematic Arrangement for Two-Potentiometer Method

galvanometer. The spots 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 in turn.

9.3 *Procedure B* is applicable when only one potentiometer is used. The arrangement when several thermocouples are being calibrated using a thermocouple thermometer as a reference is shown 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 to the potentiometer in sequence by means of the selector switch, as shown. The reference thermocouple should be read just before and just after the reading of the emf of each thermocouple. After measuring the emf 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 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 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 of this method would include the use of a platinum resistance thermometer or liquid-in-glass thermometer as reference in a stirred liquid bath.

9.4 *Procedure C*

9.4.1 When the thermocouple being calibrated is of the same type as the reference, as, for example, a Type S thermocouple being calibrated against a Type S reference thermocouple, a convenient variation of the two potentiometer methods may be employed. With this technique, the emf of the reference thermocouple is measured with one potentiometer, as in Procedure A, but in this case the second potentiometer is used to measure simultaneously the relatively small emf difference between the reference thermocouple and the thermocouple being calibrated. At least two distinct advantages may be realized through the use of this technique. First, since the emf differences are a small fraction of the emf, they can be

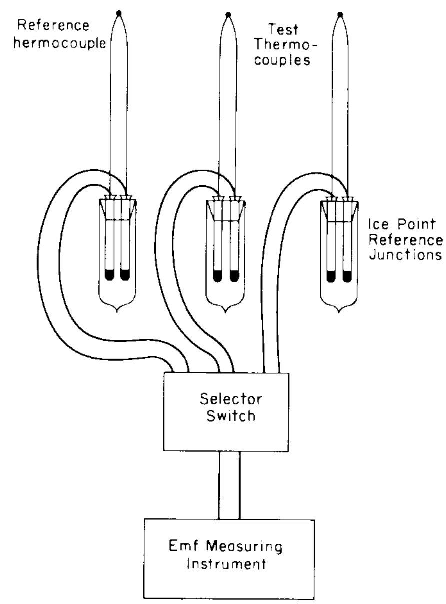

FIG. 2 Schematic Arrangement with a Selector Switch and a Single Measuring Instrument

measured to a higher degree of absolute accuracy (microvolts). Secondly, if the emf differences vary relatively slowly with change in temperature, the actual temperature at the time of a measurement need not be known accurately, and a higher rate of change of furnace temperature can be used than that tolerated when using Procedure A. Because of these two circumstances the method may be readily automated .

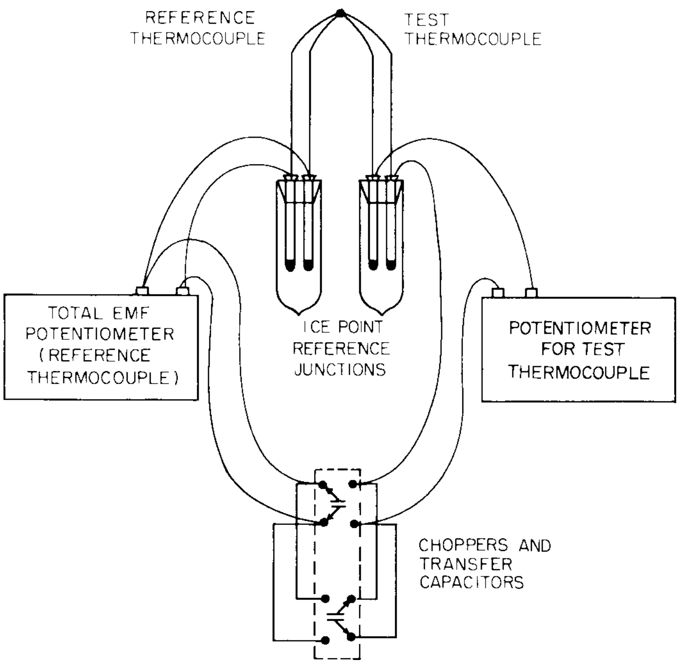

FIG. 3 Schematic Arrangement Showing Basic Isolating Potential Circuit

9.4.2 When comparison measurements are being made by direct subtraction in this manner, there can be no electrical circuit between the various thermocouples except at the point where the difference voltage is measured. In liquid and dry fluidized baths with common media, this requirement presents no problem, provided that the thermocouples do not touch each other. In tube furnaces it may be difficult to keep the various junctions at the same temperature without electric contact. In this situation the electrical isolation can be achieved at the reference end by use of an isolating potential comparator circuit.[6] 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 to the potential of the reference thermocouple and then is moved into series opposition with the emf of the test thermocouple so that the difference potentiometer indicates the difference in emf between the two. The total emf of the reference thermocouple is continuously monitored by the other potentiometer. To obtain the highest sensitivity, two chopper and condenser units are used at 180-deg phase difference, as shown in Fig. 3. The potentiometers may be of the manually balanced type, or if self-balancing potentiometers are used, the results may be presented on a recorder chart or other automatic data systems. With appropriate switching, automatic or manual operation, the number of thermocouples that may be calibrated together will depend principally on the size of the furnace used.

10. Preparation 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 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. Except when using Procedure C (9.4), the wires 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.[7] 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 contact by wrapping them with platinum wire or foil. Slip the insulating tubes down on the thermocouple wires 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. 4. In this process take care not to stress or cold work the wires. Special care must also be taken during handling of the thermocouple wires to avoid contaminating them.

10.2 *Procedures for Types B, R, and S Thermocouples (Platinum-Rhodium/Platinum)*—This procedure for annealing is adapted to the calibration of bare wire platinum-10 % rhodium/platinum (Type S) or platinum-13 % rhodium/platinum (Type R) thermocouples in the temperature range from 0 to 1480°C (32 to 2700°F) and to the calibration of platinum-30 % rhodium/platinum-6 % rhodium (Type B) thermocouples in the range from 0 to 1700°C (32 to 3100°F). 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. Following this anneal, allow 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 temperature is most readily determined by sighting on the platinum leg of the thermocouple with an optical pyrometer. A pyrometer 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.

10.2.1 Following the anneal, thread the thermocouple through its insulating tube and mount, together with a reference

[6] Dauphinne, T. M., "An Apparatus for Comparison of Thermocouples," *Canadian Journal of Physics*, Vol 33, 1955, p. 275.

[7] For information on welding of measuring junctions, see *Manual on the Use of Thermocouples in Temperature Measurement*, *ASTM STP 470 B*, 1981.

thermocouple, in a protection tube as described in 9.1. The reference thermocouple shall be either a Type B, R, or S thermocouple that has been calibrated at fixed points or by comparison with another thermocouple so calibrated. Special care must be exercised during handling of the annealed thermocouple wires to avoid contaminating or stressing them.

10.3 *Procedure for Base-Metal Thermocouples in Laboratory Furnaces*—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 2—This method is intended for use with new wire. Base-metal thermocouples undergo changes with use at high temperature which often render them unfit for recalibration. Therefore, these thermocouples should not be recalibrated.

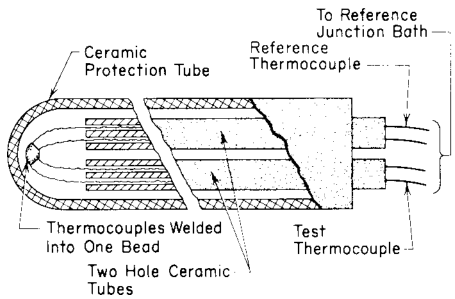

FIG. 4 Thermocouple Assembly in Protection Tube

10.3.1 Thread the thermocouple through its insulating tube and mount with a reference thermocouple, in a protection tube as 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 so calibrated. When calibrating base metal thermocouples against a Type B, S, or R thermocouple, Procedure C (9.4) cannot be used. For Procedures A and B (9.2 and 9.3) protect the reference thermocouple by a two-holed ceramic insulating tube to within a few millimetres of the measuring junction, and seal this end of the ceramic tube to the thermocouple by borosilicate glass or by 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 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 to form a new junction. Examine the seal after each use and remake it if it does not appear to be good.

10.3.2 If the wires 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 probably better practice, however, to weld the measuring junction of the reference thermocouple into the common junction as shown in Fig. 4.

10.3.3 When wires, 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 atmosphere is compatible with the sheath. The precaution discussed in 9.4 must be taken if differential comparison techniques are used.

10.4 *Procedure for Base-Metal Thermocouples in Stirred Liquid Baths*—This procedure is applicable to the calibration 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 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). From 540 to 620°C (1150°F), use vitreous silica or ceramic tubing. 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 wires with electrical insulation over the length inserted in the protection tube. Slip a loose-fitting insulating sheath over 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 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 a thermocouple inserted in the protection tube with the thermocouple being calibrated, or it may be a liquid-in-glass thermometer or resistance 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 Section 7).

11. Calibration Procedures

11.1 The following methods for taking calibration data are applicable to both platinum and base-metal thermocouples.

11.1.1 *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), and assemble the reference junction, connecting wires, and switching appropriate to Procedures A, B, or C described in Section 9, whichever is selected to be used.

11.1.1.1 *Procedure A*—Measurements at the calibration points should start with the lowest temperature and be continued through succeedingly 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 thermocouple for temperature measurement. Read alternately the emf 's of the reference, the test thermocouple, and the reference again. A comparison of the two readings of the reference thermocouple will indicate whether the furnace has 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 readings of the reference thermocouple. 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 emf 1st measurement/Table emf 2nd measurement) where the Table emf is that listed for the test thermocouple at the temperature indicated by the reference thermometer. The two valves of emf for the test couple are then averaged and assigned to the first measurement temperature. Using the standard reference tables given in Specification E 230, corrections can be applied for temperature differences of up to 10°C (18°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 be adopted will depend upon 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 to point out that many of the precautions and practices representing good techniques described above must be observed. The rate of change of furnace temperature may be significantly higher than is permissible with Procedure A, but it probably should not exceed 10°C (18°F)/min for the most accurate work.

11.1.2 *Thermocouples in Stirred Liquid Baths*—The procedure for taking data in the calibration of thermocouples in stirred liquid baths is identical with Procedure B for use with laboratory furnaces described in 11.1.1.2, except that the reference thermometer may be a liquid-in-glass or resistance thermometer instead of a thermocouple. When measurements approaching an accuracy of 0.1°C are to be made. Potentiometers with a limit of error no greater than given in 6.4.1 must be used.

12. Calculations

12.1 Having determined the emf of the thermocouple at a number of calibration points, the calibration is completed by interpolating between the calibration points. Different methods may be used to accommodate special circumstances, but the method using a difference curve from an arbitrary reference table is often simplest to use. To use this method, choose the appropriate reference table in Specification E 230 to establish 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 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 of ΔE as ordinates and values of E as abscissas, plot the values of ΔE for each calibration point and draw a smooth curve through the points. The value of $\Delta E = 0$ at the reference junction temperature is added as an additional point on the curve. Draw the straight line, $\Delta E = 0$, which represents the difference curve for the table values of emf. At any observed value of E add the corresponding value of ΔE from the curve and enter the table at this corrected value of emf to obtain the true temperature. A difference curve typical of platinum-10 % rhodium/platinum is shown in Fig. 5. Note that the values of ΔE take the sign of a correction which is to be added to the observed emf to give a corrected emf with which one can enter the standard table to get the true temperature. For example, in Fig. 5 at 7 mV the correction is – 2 μV. Therefore, with an observed emf of 7 mV enter the table at 7.000 – 0.002 = 6.998 mV to obtain the corresponding temperature.

12.3 When Procedure C is being used, the data may be recorded in any one of a number of ways. When manually balanced potentiometers are used, the data may be used to produce a difference curve from a standard table as described above, or the data may be used to compute a complete table by computer using standard numerical methods for curve fitting.[8] When self-balancing potentiometers are used, the data may be printed on the chart of an *X-Y* recorder, where the emf difference is plotted as a function of the total emf of the reference thermocouple. Still further automation is possible using computerized systems.

13. Report

13.1 Report the calibration results in any convenient form. This may be a table of values of E at a number of temperatures or it may be a table of values of ΔE at selected values of E.

[8] *Manual on the Use of Thermocouples in Temperature Measurements, ASTM STP 470A,* Am. Soc. Testing Mats., 1974.

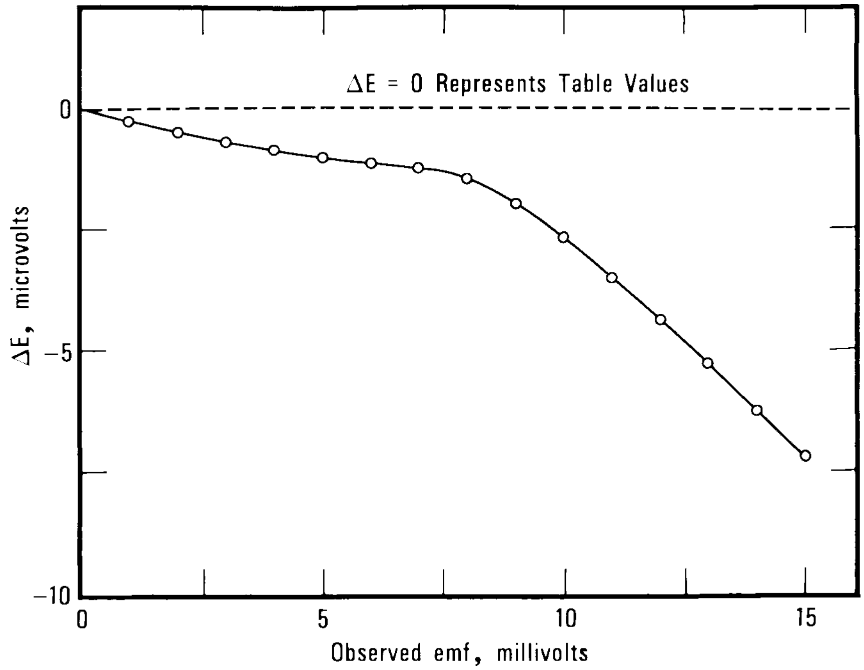

FIG. 5 Difference Curve for a Type S Thermocouple Using an ASTM Reference Table

14. Precision and Bias

14.1 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 are opinions of the committee members and are not supported by interlaboratory testing.

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 *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. The uncertainties that occur at the calibration points for the common types of thermocouples are given in Tables 1 and 2. These uncertainties may result when homogeneous thermocouples are used and when reasonable care is exercised in the work.

14.2.2 *Accuracy of Interpolated Values*—The accuracy of interpolated values will depend upon the number of calibration points and the closeness with which the reference table 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, the fewer the number of calibration points required for a given accuracy. 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 uncertainties of interpolated values using the method of difference curves from table values are given in Tables 1 and 2. These values assume the listed uncertainties at the calibration points.

14.2.3 *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 will cause the emf output of the thermocouple to be dependent upon the particular temperature gradient 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 can arise from this source is not feasible, but the possibilities of such errors should be recognized in assigning accuracies to temperature measurements made with calibrated base-metal thermocouples.

15. Keywords

15.1 comparator baths; comparison calibration; thermocouple

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 equipped with an adjustable rheostat or other means for regulating the current. For temperatures up to about 1150°C (2100°F), a furnace with a heating element of nickel (80)-chromium (20) will suffice. Furnaces with heating elements of platinum or platinum-rhodium are available for higher temperatures. 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.

X1.2 Tube-Type Heating Element Furnace

X1.2.1 A tube-type heating element furnace may be found convenient when Procedure A (Section 9.2) is to be used. One successful design 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 (⅞ in.) in inside diameter, 31 mm (1¼ in.) in outside diameter and 600 mm (24 in.) long, is heated electrically, the tube itself serving as the heating element 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, as shown in Fig. X1.1 to minimize temperature gradients along the tube and 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 thermocouple and the unknown must be read alternately with a single potentiometer.

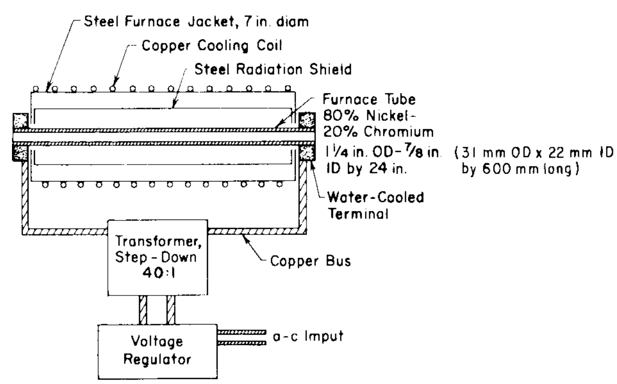

FIG. X1.1 Tube-Type Heating Element Furnace

X2. REFERENCE THERMOMETERS

X2.1 Standard Resistance Thermometer

X2.1.1 The standard 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°C. Temperatures 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 thermometers should be so designed and constructed that the wire of the platinum resistor is as strain-free as practicable and will remain so during use. In addition, certain restrictions are imposed on the electrical and physical properties of the wire of which the resistor is formed. For use at temperatures up to 500°C (930°F), these instruments are usually made with a borosilicate glass protection tube. This range is extended to 630°C (1160°F) through the use of silica glass protection tubes. More details may be obtained from publications from the National Bureau of Standards, Monograph 126.

X2.2 Direct-Reading Resistance Thermometers

X2.2.1 Direct-reading resistance thermometers are available commercially and may be used when the highest accuracy is not required. These instruments have the advantage that they indicate directly in temperature.

TABLE X2.1 Calibration Uncertainties for Celsius Total-Immersion Mercury Thermometers[A]

Temperature Range, °C	Graduation Interval, °C	Uncertainty, °C
0 up to 150	1.0 or 0.5	0.1 to 0.2
0 up to 150	0.2	0.02 to 0.05
0 up to 100	0.1	0.01 to 0.03
Thermometers Not Graduated Above 300°C		
0 up to 100	1.0 or 0.5	0.1 to 0.2
Above 100 up to 300		0.2 to 0.3
0 up to 100	0.2	0.02 to 0.05
Above 100 up to 200		0.05 to 0.1
Thermometers Graduated Above 300°C		
0 up to 300	2.0	0.2 to 0.5
Above 300 up to 500		0.5 to 1.0
0 up to 300	1.0 or 0.5	0.1 to 0.5
Above 300 up to 500		0.2 to 0.5

[A] Values given in this table are extracted from National Institute of Standards and Technology Monograph 150.

TABLE X2.2 Calibration Uncertainties for Fahrenheit Total-Immersion Mercury Thermometers[A]

Temperature Range, °F	Graduation Interval, °F	Uncertainty °F
32 up to 300	2.0	0.2 to 0.5
32 up to 300	1.0 or 0.5	0.1 to 0.2
32 up to 212	0.2 or 0.1	0.02 to 0.05
Thermometers Not Graduated Above 600°F		
32 up to 212	2 or 1	0.2 to 0.5
Above 212 up to 600		0.5
Thermometers Graduated Above 600°F		
32 up to 600	5	0.5 to 1.0
Above 600 up to 950		1.0 to 2.0
32 up to 600	2 or 1	0.2 to 1.0
Above 600 up to 950		0.5 to 1.0

[A] Values given in this table are extracted from National Institute of Standards and Technology Monograph 150.

X2.3 Liquid-in-Glass Thermometers

X2.3.1 The liquid-in-glass thermometers can be used as a relatively simple and accurate temperature reference over a wide 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 and Technology Monograph 150 and Test Method E 77.

TABLE X2.3 Calibration Uncertainties for Low-Temperature Total-Immersion Thermometers[A]

Temperature Range	Type of Thermometer	Graduation Interval	Uncertainty
Fahrenheit Thermometers			
–35 to + 32°F	Mercury	1 or 0.5°F	0.1 to 0.2°F
–35 to + 32°F	Mercury	0.2°F	0.05°F
–69 to + 32°F	Mercury-thallium	1 or 0.5°F	0.1 to 0.2°F
–69 to + 32°F	Mercury-thallium	0.2°F	0.05°F
–328 to + 32°F	Organic liquid	2 or 1.0°F	0.3 to 0.5°F
Celsius Thermometers			
–35 to 0°C	Mercury	1 or 0.5°C	0.1 to 0.2°C
–35 to 0°C	Mercury	0.2°C	0.02 to 0.05°C
–56 to 0°C	Mercury-thallium	0.5°C	0.1 to 0.2°C
–56 to 0°C	Mercury-thallium	0.2°C	0.02 to 0.05°C
–200 to 0°C	Organic liquid	1.0°C	0.2 to 0.5°C

[A] Values given in this table are extracted from National Institute of Standards and Technology Monograph 150.

X3. TEST FOR STRAY THERMALS IN COPPER CONNECTING WIRES

X3.1 A test for stray thermals 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, remove one of the ends of the test thermocouple from the reference junction bath and connect a short piece of copper wire between the pools of mercury in the two glass tubes. This copper link will complete the circuit through the connecting wires to the potentiometer, which will now indicate any emf originating in the selector switch, binding posts, etc., as well as stray 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 is to remove one of the copper connecting wires from its glass tube and insert it into the other tube with the second copper connecting wire. This may be done without removing either thermocouple wire from its tube.