



# Standard Test Method for Inspection and Verification of Thermometers<sup>1</sup>

This standard is issued under the fixed designation E 77; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

## 1. Scope

1.1 This test method covers visual and dimensional inspection, test for permanency of pigment, test for bulb stability, and test for scale accuracy to be used in the verification of liquid-in-glass thermometers as specified in Specification E 1. However, these procedures may be applied to other liquid-in-glass thermometers.<sup>2</sup>

NOTE 1—The use of NIST SP250-23<sup>2</sup> is recommended.

1.2 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

E 1 Specification for ASTM Thermometers

E 344 Terminology Relating to Thermometry and Hydrometry

## 3. Terminology

### 3.1 Definitions:

3.1.1 The definitions given in Terminology E 344 apply. Some that are considered essential to this standard are given below.

3.1.2 *complete-immersion thermometer, n*—a liquid-in-glass thermometer, not specified in ASTM documents, designed to indicate temperature correctly when the entire thermometer is exposed to the temperature being measured.

3.1.3 *partial-immersion thermometer, n*—a liquid-in-glass thermometer designed to indicate temperature correctly when the bulb and a specified part of the stem are exposed to the temperature being measured.

3.1.4 *total-immersion thermometer, n*—a liquid-in-glass thermometer designed to indicate temperature correctly when just that portion of the thermometer containing the liquid is exposed to the temperature being measured.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration, n*—the determination of the indications of a thermometer with respect to temperatures established by a standard resulting in scale corrections to be applied when maximum accuracy is required.

3.2.2 *reference point, n*—a temperature at which a thermometer is checked for changes in the bulb volume.

3.2.3 *verification, n*—the process of testing a thermometer for compliance with specifications.

3.2.4 *verification temperatures, n*—the specified temperatures at which thermometers are tested for compliance with scale error limits.

3.2.5 Other descriptions of terms relating to thermometers are included in Sections 3 and 17 of Specification E 1.

## 4. Significance and Use

4.1 The test method described in this standard will ensure that the thermometers listed in Specification E 1 will indicate temperatures within the maximum scale errors listed, be compatible with the apparatus, and serve the purpose for which they were designed. Fig. 1

4.2 Thermometers that do not pass the visual and dimensional inspection tests may give erroneously high or low temperature readings, or may not fit into existing equipment used in ASTM methods. If the pigment in the scale etchings washes out or fades, the thermometer will be difficult to read. Improper annealing of the bulb, as determined by the bulb stability test, will result in thermometer readings rapidly changing with time and use. For accurate temperature measurements the scale readings of the thermometer should be verified as described in this test method.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E20 on Temperature Measurement and is the direct responsibility of Subcommittee E20.05 on Liquid-in-Glass Thermometers and Hydrometers.

Current edition approved Nov. 1, 2003. Published November 2003. Originally approved in 1949. Last previous edition approved in 1998 as E 77 – 98.

<sup>2</sup> “Liquid-in-Glass Thermometer Calibration Service,” NIST Special Publication 250-23, 1988, Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

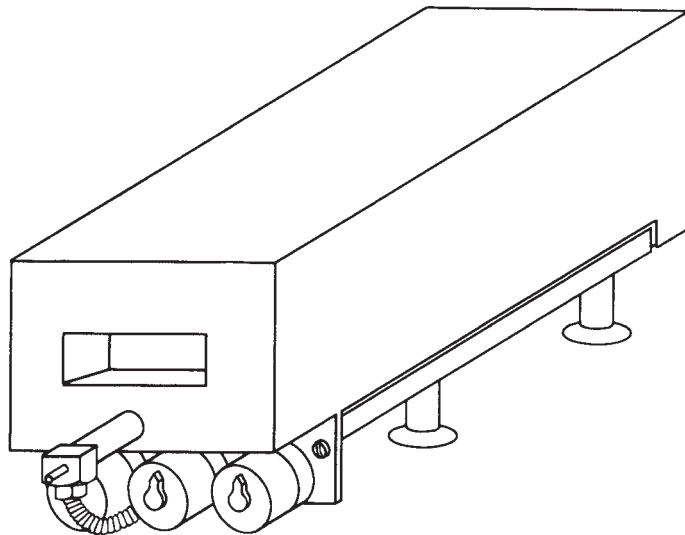
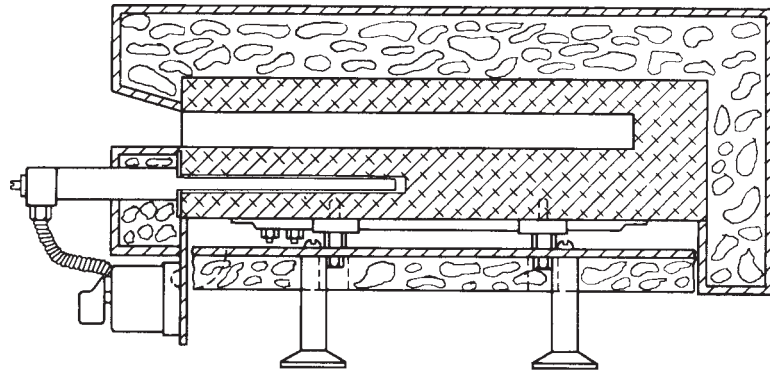


FIG. 1 Oven for Permanency of Pigment Test

## 5. Apparatus

5.1 *Graduated Metal Scales or Templates*—Maximum and minimum specified linear dimensions are measured with graduated metal scales and templates on which lines are ruled at suitable distances from reference points corresponding to the maximum and minimum values of the several specified dimensions.

5.2 *Micrometers and Ring Gages*—Specified diameters of ASTM thermometers are checked using micrometers, or more conveniently with ring gages consisting of metal plates in which holes have been formed corresponding to the maximum and minimum values of the several specified dimensions. The thickness of such gages should approximate the diameters of the holes to minimize errors resulting from the axis of the thermometer stem being other than normal to the plane of the gage. When specified, diameters may also be checked with conventional snap gages having plane parallel working faces.

5.3 *Comparators*—Comparators are required for verification of scale accuracy of liquid-in-glass thermometers. Suitable types are described in Appendix X1.

5.4 *Oven*—The test for permanency of pigment may be conducted with any suitable oven, such as the type shown in Fig. 1.

5.5 *Metal Block Baths*—The bulb stability test may be conducted with a variety of devices. Metal block baths and the salt and tin comparator baths, described in Appendix X1, are examples of the type of equipment that has been found to be suitable for this purpose.

5.6 *Primary Standard Thermometer*—The primary standard thermometer in the range from  $-183$  to  $630$  °C ( $-297$  to  $1166$  °F) is the platinum-resistance thermometer. 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) or an AC resistance bridge. Temperatures may then be calculated using suitable resistance-temperature equations. In order that it shall be satisfactory for such use, the thermometer should meet the requirement that the ratio of resistances at the steam and ice points shall be greater than 1.3925. More complete information on the construction and use of primary standard thermometers may be obtained from NIST SP250-22.<sup>4</sup>

<sup>4</sup> "Platinum Resistance Thermometer Calibrations," NIST Special Publication 250-22, Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325.

**5.7 Secondary Standard Thermometers**—Secondary standard thermometers are more suitable for routine work, and may be of various types as described below. They are simpler to use than a primary standard thermometer with its accessory equipment, the latter being capable of an order of precision and accuracy far in excess of that attainable with liquid-in-glass thermometers. The choice of a secondary standard will be governed by various factors. The following criteria should, in so far as possible, be satisfied: The standard should be a calibrated thermometer of equal or preferably higher sensitivity than the thermometer to be verified, and it should be capable of giving results of an equal or preferably higher order of accuracy and also of an equal or preferably higher order of reproducibility or precision. Scale corrections should always be applied in the use of these standards. Secondary standards may be of the following types.

**5.7.1 Direct-Reading Resistance Thermometers**—Direct-reading resistance thermometers are available commercially, are very convenient to use, and have the advantage over the primary type that temperature indications are given directly in the instrument reading. They should be completely recalibrated every 6 to 12 months, depending upon the temperatures of usage. Ice points should be taken every 3 months.

**5.7.2 Liquid-in-Glass Thermometers**—Liquid-in-glass thermometers, when used as secondary standards, may be classified into two groups, those intended for testing general purpose total or partial-immersion thermometers, and those for testing special use partial-immersion thermometers.

**5.7.2.1 Total-Immersion Thermometers**—In the case of general purpose total-immersion thermometers, the sensitivity of the thermometers to be tested will govern the choice of standard. For thermometers graduated in 1, 2, or 5° divisions, a set of well-made thermometers will be adequate when calibrated and used with applicable corrections. For fractionally graduated thermometers a calibrated set of the following thermometers is recommended. Specifications for these ASTM Precision Thermometers appear in Specification E 1.

ASTM Ther- mometer Number	Range	Celsius	Divisions	Length, mm
62C	-38 to	+2°C	0.1°C	380
63C	-8 to	+32°C	0.1°C	380
64C	25 to	55°C	0.1°C	380
65C	50 to	80°C	0.1°C	380
66C	75 to	105°C	0.1°C	380
67C	95 to	155°C	0.2°C	380
68C	145 to	205°C	0.2°C	380
69C	195 to	305°C	0.5°C	380
70C	295 to	405°C	0.5°C	380

ASTM Ther- mometer Number	Range	Fahrenheit	Divisions	Length, mm
62F	-36 to	+35°F	0.2°F	380
63F	18 to	89°F	0.2°F	380
64F	77 to	131°F	0.2°F	380
65F	122 to	176°F	0.2°F	380
66F	167 to	221°F	0.2°F	380
67F	203 to	311°F	0.5°F	380
68F	293 to	401°F	0.5°F	380
69F	383 to	581°F	1.0°F	380
70F	563 to	761°F	1.0°F	380

The foregoing set is calibrated for total immersion. With the exception of the first two, each thermometer is provided with an auxiliary scale including 0 °C (32 °F), thus providing means for checking at a fixed point, which should be done each time the thermometer is used. The change in ice-point reading should then be applied to all readings. It is only necessary to have a liquid-in-glass thermometer completely calibrated one time. Recalibration is performed as described in 6.5.8.

**5.7.2.2 Partial-Immersion Thermometers**—General purpose partial-immersion thermometers, as commonly listed in manufacturers' catalogs according to their own specifications, are normally bought and sold without specification of the temperatures of the emergent column for the various temperature indications of the thermometers. In such cases, verification is usually carried out for the emergent column temperatures prevailing with the verification equipment being employed.

**5.7.2.3 Special Use Partial-Immersion Thermometers**—Special use partial-immersion thermometers, such as those covered in Specification E 1, have specified emergent mercury columns or stem temperatures. These thermometers can be used as standards to calibrate other thermometers similar in all details of construction above the immersion point, but may differ below the immersion point to the extent of including an auxiliary ice point scale.

**5.8 Engraving Date on ASTM Thermometers**—If a thermometer's specification was changed, the year that it was changed is engraved on the back of the thermometer after the ASTM designation. For example, "12C-98."

## 6. Procedure

### 6.1 Visual Inspection:

**6.1.1 Gas Bubbles and Separations**—Gas bubbles are readily detected and are more likely to occur in shipment than during service. No method has been discovered that will entirely prevent such displacement of the gas. If bubbles are observed in the bulb, they can generally be removed by cooling the bulb with dry ice or other convenient coolant until all the liquid is drawn into the bulb. Gentle tapping of the thermometer while held upright will cause the bubbles to rise to the surface. It is very important that, if the bulb is cooled in this process below the freezing point of the liquid, care should be exercised to warm the stem sufficiently during the melting process so that no solidification occurs in the stem; otherwise the bulb may burst or the capillary may split internally because of the expansion forces generated in the bulb.

**6.1.1.1** If a mercury separation is observed in the stem, several different ways are suggested for joining the columns, depending on the construction of the thermometer and the type of separation. If a small portion of the liquid has separated at the top of the column and the thermometer is provided with an expansion chamber, the liquid usually can be joined by carefully and slowly heating the bulb until the separated portion is driven into the expansion chamber. Never heat the bulb in an open flame. When the column itself follows into the chamber, the separated portion usually will join onto the main column. A slight tapping of the thermometer against the palm of the hand will facilitate this joining. This method should not be employed for high-temperature thermometers (above 260 °C or 500 °F), because the heating of the bulb, which is

necessary to drive the liquid into the expansion chamber, may overheat the glass and either break the bulb, because of the pressure of the gas, or destroy the accuracy of the thermometer by expanding the bulb. Thermometers that have a contraction chamber below the lowest graduation are likely to develop separations either in the chamber or above it. It is frequently possible to join such separations by cooling the thermometer so that the separated portion as well as the main column both stand in the chamber. Tapping the tube against the hand or the bulb on a soft spongy material, such as a rubber stopper, usually will bring the liquid together. For more stubborn separations it may be necessary to cool the bulb in dry ice to a point low enough to bring all of the liquid into the bulb itself. By softly tapping on a soft spongy material or against the hand it usually is possible to bring the liquid together in the bulb. The bulb should be allowed to warm up slowly. The liquid should emerge into the bore with no separation.

6.1.1.2 In organic-liquid-filled thermometers distillation may occur, with subsequent condensation of the colorless parent liquid in the upper part of the thermometer. Such thermometers should always be inspected for these separations, which can be repaired by the procedures described above. If the thermometer has an expansion chamber that is observed to be filled with liquid, the column can be reunited by very careful heating of the chamber to drive the liquid into the bore where it can be rejoined to the main body as described above.

6.1.1.3 Organic liquids as used in thermometers, in contrast to mercury, wet the glass. Sufficient time should always be allowed for drainage to occur, particularly when using or verifying such thermometers below 0 °C (32 °F). It is frequently a good practice to immerse only the bulb of the thermometer. This keeps the viscosity of the liquid in the capillary low and aids in hastening drainage.

6.1.2 *Globules of Liquid*—Globules of liquid in the stem, which result from mechanical separation, can normally be rejoined by heating the bulb until the liquid column merges with the globules. If such globules appear to unite and then reappear on cooling the bulb, they are indicative of oxidation of the mercury or the presence of obstructions in the bore, and should result in rejection of the thermometer.

6.1.3 *Foreign Matter*—Foreign matter in the bore can sometimes be detected with the unaided eye, but it is generally convenient to use a magnifying glass of low power for this examination. A magnifier of 10× is recommended for visual examination. The most common types of foreign matter which should be cause for rejection are glass chips, particles of dirt or lint, oxide of mercury (either red, yellow, or black), products of glass weathering commonly called white deposit, and stones or iron spots traceable to faulty glass fabrication.

6.1.3.1 Where a specific gas is specified to be used as filling above the liquid, any other gas present may be treated as foreign matter. The most common example is the use of air instead of nitrogen in mercury-in-glass thermometers, which may have been introduced by accident or in violation of the specifications. The presence of air can readily be detected by exposing the gas-filled portion of the thermometer and a short section of the liquid column to a temperature of 340 to 370 °C (644 to 698 °F), using an oven of the type illustrated in Fig. 1.

Under these conditions oxidation of the mercury will occur and will normally be evidenced by the production of crystals of red oxide of mercury after 10 to 12 h of exposure.

6.1.4 *Glass Faults*—Glass faults may be of various types. Any stones or striae that distort the bore or its appearance should be cause for rejection. Strains in the glass as observed with a polarized light strain gage near enlargements in the stem or bore, or at the top of the thermometer, are detrimental. If so, severe fire cracks may later occur. Strains near the bulb are indicative of incomplete glass stabilization and are particularly objectionable in thermometers for use above 150 °C (302 °F). The test for bulb stability will normally serve to reject high range thermometers in which this defect is most significant.

#### 6.2 *Dimensional Inspection:*

6.2.1 Determine compliance with lineal dimensional specifications by comparison of the thermometer with the appropriate scale or template described in 5.1. In the case of diameter measurements, the bulb or other portions of the thermometer being tested should not enter the smaller of the holes in the ring gage (see 5.2), or fail to enter the larger of the holes. In the case of ASTM thermometers, the diameter limits apply over the entire length of the section of the thermometer being tested.

6.2.2 Inspect the thermometers for uniformity of graduation spacing. Normally, the effect of taper in the capillary or nonlinearity in the expansion characteristics of organic liquids is manifested by a gradual change in the spacing of the graduations. Discontinuities in the spacing are evidence of faulty graduating. Inspection for uniformity of spacing can generally be accomplished by eye, but for greater accuracy a pair of dividers may be used to advantage. Adjust the dividers to extend over 10 or 20 graduations, and successive intervals can be intercompared rapidly. Where marked changes in spacing are noted, it is well to include in the verification several temperatures in that region.

#### 6.3 *Permanency of Pigment:*

6.3.1 The test for permanency of pigment is designed to determine the ability of the pigment material to withstand the exposure conditions encountered in use without being obliterated.

6.3.2 Place any convenient portion of the scale section of the thermometer to be tested in an oven of the type shown in Fig. 1. Heat for 3 h at approximately 260 °C (500 °F). Allow to cool slowly. Inspect the thermometer for differences in appearance of the tested and untested sections of the scale portion. Burning out, loosening, chalking, or fading of the pigment shall be cause for rejection.

#### 6.4 *Bulb Stability:*

6.4.1 The test for bulb stability is designed to determine the adequacy of the stabilizing heat treatment accorded the thermometer bulb during manufacture. An inadequately stabilized bulb will undergo shrinkage with time which may be significant particularly in higher temperatures.

6.4.1.1 Heat the thermometer for 5 min at the temperature specified in 12.1 of Specification E 1 in a preheated bath which may be of the type shown in Fig. 2. Immersion must be sufficient that all of the thermometer bulb is at the specified temperature. Allow the thermometer to cool, either naturally in still air, or slowly in the test bath at a specified rate, to a span

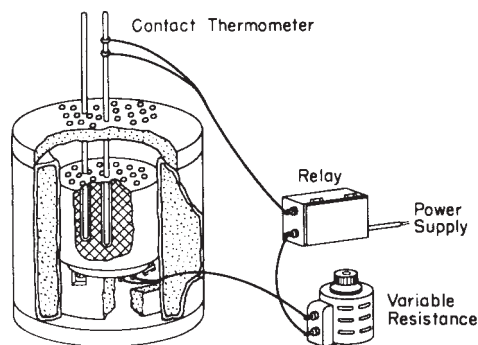


FIG. 2 Air Bath for Bulb Stability Test

of 20° on the Celsius scale (36° on the Fahrenheit scale) above ambient temperature or to 50 °C (122 °F), whichever is the lower, and then determine the reading at some reference point, such as the ice point. If natural cooling in still air is used, determine the reference reading within 1 h. Return the thermometer to the bath, preheated to the temperature of test, and heat for 24 h. Allow the thermometer to cool, at the same rate as at the start of the test, to the temperature referred to above, and redetermine the reference reading under the same conditions as before. The magnitude of any change in this reference reading as a result of the 24-h heating period is a measure of the quality of the bulb glass and the adequacy of the previous heat treatment accorded the thermometer in manufacture.

6.4.2 For kinematic viscosity thermometers used for measuring temperatures at and above 50 °C (122 °F), the following bulb stability test is used.

6.4.2.1 Heat the thermometer to the selected reference point on the main scale, maintain the temperature for at least 15 min, and determine the scale correction at this point.

6.4.2.2 Allow the thermometer to cool slowly in the test bath (or naturally in still air) to at least a span of 20° on the Celsius scale (36° on the Fahrenheit scale) above ambient or to 50 °C (122 °F), whichever is the lower, and then determine the correction after at least 15 min at the ice point. If natural cooling is used, the correction should be determined within 1 h.

6.4.2.3 Heat the thermometer again to the selected reference point on the main scale, keep it at this temperature for 168 h. Allow the thermometer to cool as described in 6.4.2.2 and then repeat the procedures described in 6.4.2.1 and 6.4.2.2. It must be emphasized that to obtain meaningful results, the procedure adopted after the 168-h period of heating must be identical to that used in the original calibration.

#### 6.5 Verification of Scale Accuracy:

6.5.1 *Reading Thermometers to Avoid Parallax*—The error due to parallax may be eliminated by taking care that the reflection of the scale can be seen in the mercury thread, and by adjusting the line of sight so that the graduation of the scale nearest the meniscus exactly hides its own image; the line of sight will then be normal to the stem at that point. In reading thermometers, account must be taken of the fact that the lines are of appreciable width. The best practice is to consider the position of the lines as defined by their middle parts.

6.5.2 *Depth of Immersion for Total-Immersion Thermometers*—Although by definition total-immersion thermometers should have the bulb and stem containing the

mercury immersed both in use and in verification, it is frequently inconvenient to do so. If any portion of the stem containing mercury is exposed, the emergent mercury column correction should be determined. No correction need be applied if it is found to be less than one fifth of the verification error; otherwise, the proper correction should be applied. In some instances total-immersion thermometers may be used under conditions of complete immersion. With thermometers such as the kinematic-viscosity thermometer 30F, a significant error will be introduced unless the thermometer is verified under the same conditions as those in use. This is due to the effect of increased gas pressure above the column producing a distortion of the bulb.

6.5.3 *Depth of Immersion for Partial-Immersion Thermometers*—Thermometers of this type shall be verified by one of the methods described in 6.5.7.2, 6.5.7.3, and 6.5.7.4 using the appropriate comparators. If the thermometer is of the general-purpose type, it should be verified by immersion to the specified depth and the readings compared with those of the secondary standard.

#### 6.5.4 Verification at the Ice Point:

6.5.4.1 Select clear pieces of ice or ice made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the crushed ice and add sufficient distilled, preferably precooled, water to form a slush, but not enough to float the ice. As the ice melts it will be necessary to drain off some of the water and add more crushed ice. Insert the thermometer, packing the ice gently about the stem, to a depth approximately one scale division below the 0 °C (32 °F) gradation.

6.5.4.2 After at least 3 min have elapsed, tap the stem gently and observe the reading. Successive readings taken at least 1 min apart should agree within one tenth of a division. It may be necessary to repack the ice around the thermometer because of melting.

#### 6.5.5 Verification at the Steam Point:

6.5.5.1 If the range of the thermometer is such that the ice point is not included, the steam point at 100 °C (212 °F) can be used. For a description of the equipment and techniques best suited to the purpose, reference may be made to NIST SP250-23.<sup>2</sup>

#### 6.5.6 Verification of Total-Immersion and General Use Partial-Immersion Thermometers at Temperatures Other Than Fixed Points:

6.5.6.1 Determine the ice point of the primary or secondary standard and the thermometer to be tested as described in 6.5.4. If the secondary standard is of the liquid-in-glass type, it should be held at room temperature for at least 72 h before this determination unless the ice point was originally determined immediately after heating to a specified temperature. The thermometer to be tested should be treated in similar manner.

6.5.6.2 Insert the thermometer to be tested and the standard in the thermometer holder, and adjust the temperature of the comparator to a value approximately 5 °C (10 °F) below the verification temperature. It may be advantageous to use two

secondary standard liquid-in-glass thermometers, since observational errors of the standard may then be readily detected. Apply sufficient heat to raise the temperature at a slow uniform rate. At the verification point the rate should not exceed one scale division in 3 to 10 min. Fulfillment of this requirement will ensure that any exposed portion of the stem will have attained thermal equilibrium before readings are taken.

6.5.6.3 When the proper rate of temperature rise has been established, read the thermometers in the following order at equal time intervals: standard, thermometer or thermometers to be tested, standard, thermometers to be tested in reverse order, standard. The average of the first and third readings of the standard should agree with the second. A comparison of differences in successive readings will also indicate if the rate of rise has been uniform.

6.5.6.4 Calculate the average readings for all thermometers. Apply the appropriate corrections to the reading of the standard. Calculate the corrections to be applied to the thermometers under test.

6.5.6.5 For verification purposes, one series of readings is usually sufficient. If the thermometer is being calibrated, check determinations should be made. The results of each of at least three series should agree with the mean of the series within plus or minus one tenth of a division.

6.5.6.6 Unless otherwise specified, tests should be made not less than 40 nor more than 100 divisions apart throughout the range of the thermometer.

6.5.6.7 Comparisons may be made at constant temperature in preference to the use of slowly rising temperatures. Such a procedure is satisfactory if the bath temperature does not vary more than the precision of reading and if the thermometer is jarred, as by tapping, before taking a reading in order to overcome any sticking of the mercury to the glass. Such tapping is particularly important with thermometers having a capillary diameter of the order of 0.1 mm or less and may be employed to advantage in the rising temperature method of test as well. Sufficient time should elapse before taking readings to ensure that all portions of the thermometer have attained thermal equilibrium. This is particularly important at test temperatures remote from room temperatures.

6.5.7 *Verification of Partial-Immersion Thermometers With Specified Stem Temperatures:*

6.5.7.1 Partial-immersion thermometers having specified stem temperatures, such as those in Specification E 1, can be verified in one of three ways.

6.5.7.2 One method involves comparison of the thermometers at total immersion with total-immersion standards. The number of degrees of scale, which will be in the emergent column when in actual use, shall then be measured. From these data the corrections under the specified emergent column temperatures may then be calculated (7.2). In the case of organic liquid-filled thermometers such as ASTM Low Cloud and Pour, 6 °C and 6 °F, the coefficient of expansion of the liquid should be obtained by experiment or from the manufacturer in order to perform these computations. This method has the advantage that the standard may be selected to have greater sensitivity than the thermometer being tested, thus increasing the accuracy of measurement.

6.5.7.3 A second method, which is the one best suited to large-quantity testing, involves comparison of the thermometers with standards similar in all details of construction above the immersion point, but may differ below the immersion point to the extent of including an auxiliary ice-point scale. Such thermometers, when completely calibrated, as by the National Institute of Standards and Technology, may then be employed indefinitely for verification purposes if periodic ice-point checks are made. The particular field of application of this second method is evidenced by the fact that many of the ASTM thermometers include the ice point in the specified range, but the following fractionally graduated thermometers are not provided with an ice-point scale:

Paraffin Wax Melting Point: 14C, 14F

High Softening Point: 16C, 16F

Saybolt Viscosity: 17C, 17F, 18C, 18F, 19C, 19F, 20C, 20F, 21C, 21F, 22C, 22F, 77F to 81F, 108F, 109F

Engler Viscosity: 23C, 24C, 25C

Stability Test of Soluble Nitrocellulose: 26C

Turpentine Distillation: 27C

Aniline Point: 34C, 34F, 35C, 35F

Solvents Distillation: 38C, 39C, 40C, 41C, 42C, 102C, 103C, 104C, 105C, 106C, 107C

Stormer Viscosity: 49C

Gas Calorimeter Inlet: 50F

Gas Calorimeter Outlet: 51F

Congealing Point: 54F

Petrolatum Melting Point: 61C, 61F

Antifreeze Freezing Point: 76F

Solidification Point: 91C to 96C, 100C, 101C

Tar Acids Distillation: 111C

Brookfield 122C, 123C, 124C, 125C

The ice point was omitted from the above listed thermometers as a matter of necessity. In order to supply an ice point, when not included in the range of the thermometer, it is necessary to blow an enlargement or contraction chamber below the first graduation to take care of the volume of mercury between the ice point and this first graduation. This contraction chamber may introduce an error in the readings unless it is entirely immersed in or is at the same temperature as the medium the temperature of which is to be measured. This immersion requirement may be difficult if not impossible to attain in the apparatus in which the thermometers are used. Such limitations of space prevented the inclusion of ice points in the above group of thermometers. However, by increasing the lengths of the thermometers, ice-point scales can be incorporated in the thermometers to be used as standards. For those in the group that are partial immersion, the dimensions above the immersion point should be as prescribed in the specifications for the corresponding ASTM thermometer. For the total-immersion thermometers this requirement may be waived.

6.5.7.4 A third method uses total-immersion standards, measuring stem temperature of the emergent liquid column in the partial-immersion thermometer with a Faden thermometer or small auxiliary thermometers. With this data, stem temperature adjustments can be made. See NIST SP250-23<sup>2</sup> for additional details.

### 6.5.8 Recalibrate by Checking Changes in Bulb Volume:

6.5.8.1 The verification procedure described above can be used to obtain corrections for the calibration of thermometers.

6.5.8.2 Small changes in the volume of the thermometer bulb during use, especially significant for large bulb and relatively short range thermometers graduated in fractional degrees, may occur, even though the thermometer has been carefully annealed or aged, and will result in changes in the indications as well as the corrections. Usually the bulb contracts slowly, and, as a result, the thermometer will read higher after it has been placed in service. Less frequently the bulb expands and the thermometer will read lower after use.

6.5.8.3 Two procedures are followed for using the ice point to determine the change in bulb volume and to recalibrate a liquid-in-glass thermometer. One method requires that the thermometer remain at approximately room temperature for 72 h before the ice point is taken. The other method stipulates that the ice point reading be taken immediately or within a definitely stated short period of time after heating to the test temperature. Changes in the corrections of the thermometer may be observed by determining the changes in the ice point by either method.

6.5.8.4 Record the ice-point reading and compare it with the previous reading. If the ice-point reading is found to be higher or lower than the ice-point reading of the previous calibration, readings at all other temperatures will be correspondingly increased or decreased. If the method involving the taking of the ice point immediately after heating to a specified temperature is used, such as with most kinematic viscosity thermometers, a note should appear in the table of corrections for the thermometer as follows:

NOTE 2—The tabulated corrections apply for the condition of immersion indicated, provided the ice-point reading taken after heating to \_\_\_ for not fewer than 3 min is \_\_\_. If the ice-point reading (taken in not fewer than 2 min and not more than 1 h after removal of the thermometer from the heated bath) is found to be higher (or lower) than stated, all other readings will be higher (or lower) to the same extent.

6.5.8.5 When the thermometers are first received, make a monthly check of the ice point: later these time intervals may be lengthened if the changes are found to be insignificant.

6.5.8.6 High-quality liquid-in-glass thermometers require only one complete calibration in their lifetime, and it is possible to avoid the usual requirement for complete recalibration of the instrument by the recalibration of a single previously calibrated temperature. The need for recalibration of properly manufactured liquid-in-glass thermometers is due to the gradual relaxation of residual mechanical strains in the glass that have a significant effect on the volume of the bulb. The recalibration of a single point provides a reliable indication of the effect of this change in volume and provides a means for the accurate adjustment of the remainder of the scale.<sup>5</sup>

## 7. Treatment of Data

7.1 From the corrections at the ice point and the other verification temperatures, a table of corrections can be prepared for each thermometer. If the thermometer is to be used frequently at temperatures other than those of calibration, a correction curve will be found more convenient than interpolation from a table. In such a curve the corrections are plotted against the temperature readings.

7.2 Where corrections are to be calculated for emergent columns, the following equation shall be used:

$$\text{Emergent stem correction} = kn(T - t) \quad (1)$$

where:

- $k$  = differential expansion coefficient of the liquid and the glass of which the thermometer is made; for Celsius mercury-in-glass thermometers the value of  $k$  is 0.00016, for Fahrenheit mercury-in-glass thermometers it is 0.00009, for Celsius organic liquid-in-glass thermometers it is 0.001, and for Fahrenheit organic liquid-in-glass thermometers it is 0.0006.
- $n$  = number of degrees emergent from the bath,
- $T$  = temperature of the bath, and
- $t$  = average temperature of the liquid column of  $n$  degrees.

7.2.1 The use of more accurate values of  $k$  is not justified, because of the uncertainty of the true average temperature of the liquid thread not exposed to the temperature being measured.

7.2.2 For emergent columns of partial-immersion thermometers, where the column temperature is other than specified in Table 2 of Specification E 1, calculate the correction to the observed reading as follows:

$$\text{Emergent stem correction} = kn(t_s - t_o) \quad (2)$$

where:

- $k$  = same as in 7.2,
- $n$  = the number of scale degrees from the immersion mark to the top of the mercury column. The ungraduated length between the immersion mark and the first graduation on the scale shall be evaluated in terms of scale degrees and be included,
- $t_s$  = temperature specified in Table 2 of Specification E 1,
- $t_o$  = observed average temperature of the liquid column of  $n$  degrees.

7.3 The limit of precision to which the corrections at the calibration temperatures shall be recorded, to which correction tables or charts shall be made, or to which any temperature measured by the thermometer shall be reported, is a measure of the sensitivity of the thermometer and is generally described by the phrase, *corrections stated to*.

7.4 The limit of reliability of any temperature measurement made with a calibrated thermometer after all corrections, determined as carefully as possible, have been applied is termed the *uncertainty in degree* of the thermometer.

7.5 For compliance with specifications, the errors in the temperature indications of ASTM thermometers being verified shall not exceed the *scale errors* as expressed in Table 1 of

<sup>5</sup> Wise, Jacquelyn, "A Procedure for the Effective Recalibration of Liquid-in-Glass Thermometers," NIST Special Publication 819, 1991. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325.

Specification E 1. Unless otherwise expressed, it is understood that such thermometers are to be used without application of corrections.

## 8. Precision and Bias

8.1 No statement is made about either precision or bias of Test Method E 77 for visual and dimensional inspection, permanency of pigment, or bulb stability, since the results merely state whether there is conformance to the criteria for success or failure specified in the procedure.

### 8.2 Precision of Verification of Scale Accuracy

8.2.1 It is not practicable to specify the precision of the verification of the scale accuracy because of the wide variety of thermometers listed in Specification E 1. Of the 129 different kinds of liquid-in-glass thermometers, they are all either for use at total immersion or partial immersion.

8.2.2 The verification of total-immersion thermometers will produce more repeatable results because the temperature of the stem containing the mercury is at the same temperature as the bulb, when the thermometer is immersed correctly. The temperature of the emergent stem of partial-immersion thermometers can vary depending on the temperature of the environment and this will affect the repeatability of the data obtained. The method used for measuring the temperature of the emergent mercury column, when stem-temperature measurements are made, has an effect on the repeatability of the data. Generally, total-immersion thermometers have an uncertainty of approximately ten times better than partial-immersion thermometers.

8.2.3 Most thermometers contain mercury as the actuating liquid. Some thermometers, especially those for use at temperatures below  $-56^{\circ}\text{C}$  ( $-69^{\circ}\text{F}$ ), use an organic liquid. Unlike mercury, the organic liquid will wet the wall of the capillary. Adequate time must be given for complete drainage to occur. This can take up to 1 h and can have an influence on the precision of the measurement. Thermometers containing organic fluids are less accurate than those containing mercury or mercury thallium.

8.2.4 Thermometers are graduated in intervals of  $0.01^{\circ}$ ,  $0.02^{\circ}$ ,  $0.05^{\circ}$ ,  $0.1^{\circ}$ ,  $0.2^{\circ}$ ,  $0.5^{\circ}$ ,  $1.0^{\circ}$ ,  $2.0^{\circ}$ , and  $5.0^{\circ}$ . The ability to read these thermometers will have an influence on the precision of the measurements. If the thermometers are read with a

telescope, thermometers graduated in intervals of  $0.01^{\circ}$  or  $0.02^{\circ}$  can be read to the nearest  $0.001^{\circ}$ ; in  $0.05^{\circ}$  or  $0.1^{\circ}$  to the nearest  $0.005^{\circ}$ ; in  $0.2^{\circ}$  to the nearest  $0.01^{\circ}$ ; in  $0.5^{\circ}$  or  $1.0^{\circ}$  to the nearest  $0.05^{\circ}$ ; in  $2.0^{\circ}$  to the nearest  $0.1^{\circ}$ ; and in  $5.0^{\circ}$  to the nearest  $0.5^{\circ}$ .

8.2.5 Each laboratory can determine the precision of the verification of scale accuracy or the calibration of liquid-in-glass thermometers using the above information as a guideline. A statistical analysis of data collected on the various types of thermometers verified could be used to obtain a value for the random error.

### 8.3 Bias

8.3.1 Like precision, it is very difficult to determine a bias statement for the verification of the scale accuracy because of the systematic error from the choice of standard used and the gradients in the comparators in each laboratory.

8.3.2 If a standard platinum resistance thermometer is used as the standard, it can be capable of reproducing temperatures to approximately  $0.001$  to  $0.003^{\circ}\text{C}$ . If total-immersion liquid-in-glass thermometers, such as the ASTM set of  $62^{\circ}\text{C}$  ( $62^{\circ}\text{F}$ ) through  $70^{\circ}\text{C}$  ( $70^{\circ}\text{F}$ ), are used as standards, the uncertainty at the calibration points will range from  $0.03^{\circ}\text{C}$  ( $0.05^{\circ}\text{F}$ ) to  $0.5^{\circ}\text{C}$  ( $1.0^{\circ}\text{F}$ ). The uncertainty of partial-immersion thermometers will range from approximately  $0.3^{\circ}\text{C}$  ( $0.5^{\circ}\text{F}$ ) to  $2.0^{\circ}\text{C}$  ( $3.0^{\circ}\text{F}$ ). This uncertainty will be less if special use partial-immersion thermometers are used as standards where the readings are adjusted for a specified stem temperature. In all cases the average of two standard liquid-in-glass thermometers will give a more accurate value of the temperature of the medium of the comparator.

8.3.3 The error due to gradients in the comparators must be determined in each laboratory. These gradient errors will usually increase where comparisons are made in comparators at higher temperatures.

8.3.4 The precision and bias for the verification of scale accuracy must be calculated for each laboratory. An example of how this can be done is shown in NIST SP250-23.<sup>2</sup>

## 9. Keywords

9.1 calibration; liquid-in-glass thermometers; mercury separation; organic liquids; verification

## APPENDIX

### (Nonmandatory Information)

#### X1. DISCUSSION OF APPARATUS FOR VERIFICATION OF LIQUID-IN-GLASS THERMOMETERS

X1.1 An *oven* suitable for the test for foreign matter in the bore (see 6.1.3.1) and permanency of pigment (6.3) is illustrated in Fig. 1.

X1.2 An *air bath* suitable for the bulb stability test (6.4) is illustrated in Fig. 2.

X1.3 *Comparators, General Requirements*—Comparators

for use in verification are of two types, fixed-point or variable. Certain general requirements should be met by all such comparators and their accessory equipment. All readings should be observed, using an optical device suitable for observing vertical motions. Focusing range should start from not more than 20 cm, magnification should be about 5 to 10 diameters, and the field about 1.5 cm. The eyepiece should be



provided with 90° crosshairs and may include an erecting prism. Vertical movement should total approximately 30 cm and may be obtained with both rough and delicate adjustment. A suitable instrument is illustrated in Fig. X1.1; component parts are available commercially.

X1.3.1 Heat input should be capable of accurate continuous control to meet the requirement of a very slowly rising temperature at the test point. For best results an ac power supply is recommended in conjunction with variable transformers. Such transformers are available commercially in various capacities. Other types of voltage regulators, such as the so-called induction type, may be used. While generally more expensive, they are capable of finer adjustment.

X1.3.2 In all test baths a properly located well or other suitable provision should be made for using a platinum resistance thermometer as ultimate primary standard.

#### X1.4 Comparators, Fixed-Point:

X1.4.1 The most common and also most useful of the fixed-point type comparators is the ice-point apparatus. Fig. X1.2 illustrates a typical setup, consisting of a Dewar flask, a thermometer holder, a viewing telescope of 10× magnification, and the necessary supports and siphon tube for withdrawing excess water. A suitable ice-shaving machine is a desirable adjunct. For readings to one tenth of a division, the viewing telescope is necessary, but it can be omitted if lower precision is desired.

X1.4.2 For the steam point see NIST SP250-23.<sup>2</sup>

#### Comparators, Variable-Temperature

X1.5 For verification at other than fixed points, the variable-type comparators are used. Figs. X1.3-X1.7 illustrate various types of such units for use in the range from -160 to +621 °C (-256 to +1150 °F). All of these units basically consist of a well-stirred, insulated liquid bath provided with suitable controls for maintaining the temperature either constant or uniformly increasing. As with the ice-point equipment, a viewing telescope is necessary for precision of one tenth of a scale division. The liquids used in the comparators are chosen to fulfill the following requirements: low viscosity, nonflammability, nontoxicity, and freedom from other offensive characteristics in the various temperature ranges in which they are to be used.

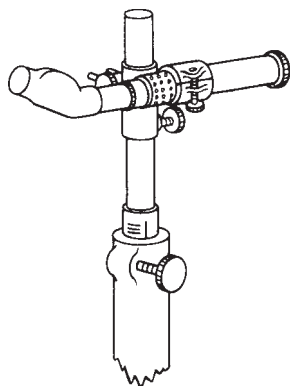


FIG. X1.1 Magnifier for Reading Thermometers

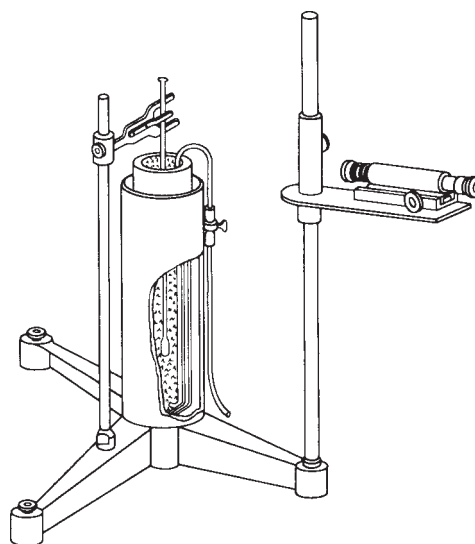


FIG. X1.2 Apparatus for Calibration at Ice Point

X1.6 Comparators for the Range from -160 to -75 °C (-256 to -103 °F)—For comparison in this range, which is below the sublimation point of solid carbon dioxide, liquid nitrogen is used as the cooling medium for an isolated bath filled with some low-boiling hydrocarbon such as isopentane. The dimensions given below are satisfactory but are not mandatory. The following individual items are necessary.

X1.6.1 *Aluminum Block*, 28 mm in diameter by 150 mm in length. Four holes 10 to 11 mm in diameter may be drilled in the center of the block, with the holes overlapping slightly so that the final pattern resembles a cloverleaf. The holes shall be deep enough so that the liquid index of the thermometers under test may be completely immersed. This block rests on a cushion of glass wool on the bottom of the test tube, the cushion being of convenient height.

X1.6.2 *Test Tube*, 33 mm in diameter by 235 mm in length with a resistance coil (165 Ω) on the outside. This is an ordinary test tube with a spiral of resistance wire wrapped around the outside. The wire may be held in place by glass studs or by cellulose tape. The resistance shall be such that the wire will not become dangerously hot if the full voltage of a variable transformer is accidentally sent through. The test tube is in turn supported in the evacuable jar by a cushion of glass wool.

X1.6.3 *Vacuum Pump*.

X1.6.4 *Evacuatable Vacuum Jar*, unsilvered, with attached side arm containing a stopcock, 50 mm in inside diameter by 300 mm in inside depth. An ordinary unsilvered Dewar flask is fitted with a side arm containing a tightly fitting stopcock.

X1.6.5 *Variable Transformer*, 0.3 kVA.

X1.6.6 *Vacuum Jar*, silvered, without side arm, 80 mm in inside diameter by 350 mm in inside depth.

X1.6.7 *Platinum Resistance Thermometer* and accessories, or suitable liquid-in-glass secondary standard.

X1.6.8 *Cardboard Cover*:

(not shown) to fit over the top of the test tube.

X1.6.9 *Glass Wool*.

X1.6.10 *Liquid Nitrogen*.

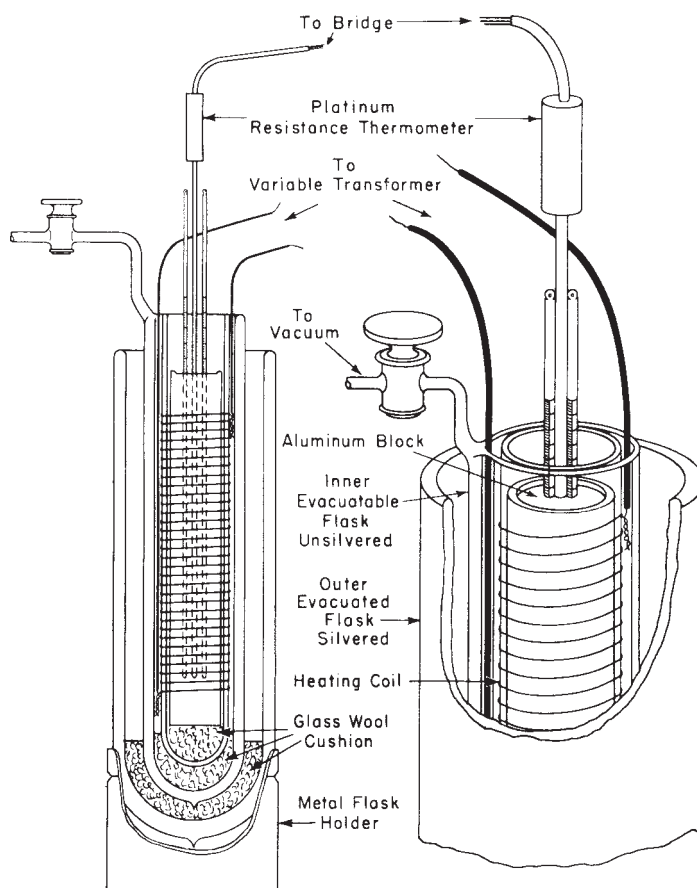


FIG. X1.3 Comparator for Temperature Range from  $-160$  to  $-75$  °C ( $-256$  to  $103$  °F)

#### X1.6.11 Isopentane.

X1.6.11.1 In use the apparatus is assembled as shown in Fig. X1.3. The evacuatable flask is positioned in the larger container by three cork wedges strung on a wire. It must extend above the nitrogen container enough so that nitrogen may be added without spilling into the isopentane. The aluminum block is completely covered with isopentane and the space between the two Dewar flasks filled with liquid nitrogen. A loose fitting cardboard shield with proper holes is placed over the top to reduce condensation and convection. When the system has cooled to a few degrees above the desired temperature, the inner flask is evacuated by the pump to stabilize the temperature equilibrium in the test tube. (When testing in the range of  $-157$  °C ( $-251$  °F), this evacuation is not necessary). At the desired test temperature, heat transfer is exactly balanced by means of the heating coil. It will be necessary to agitate the isopentane by raising and lowering the thermometer, but any vigorous stirring at low temperatures will generate enough heat to make difficult the maintaining of proper equilibrium.

X1.6.11.2 Certain safety precautions must be carefully observed. Some of the more important are as follows:

X1.6.11.2.1 Since liquid nitrogen has a lower absolute boiling point than liquid air, substantial quantities of liquid air or atmospheric moisture, or both, will condense within the walls of the evacuatable flask. Therefore, the flask should never be warmed without being vented to the atmosphere or under vacuum and should always be vented through a drying tube.

X1.6.11.2.2 Liquid air or liquid oxygen, or a mixture of them, should never be used as the refrigerant.

X1.6.11.2.3 At all times the level of the pentane should be slightly above the surface of the aluminum block. Whenever fresh chilled pentane is added, care must be taken not to spill any on the heating coil.

X1.6.11.2.4 The heating coil should be attached to the variable transformer by means of thoroughly insulated leads.

X1.7 Comparators for the Range from  $-80$  to  $+5$  °C ( $-112$  to  $+41$  °F)—For temperature testing in this range, as indicated in Fig. X1.4, two baths are recommended: one for thermometers requiring a long depth of immersion and the other for short immersion. In either case the bath shall consist of a Dewar-type vacuum flask, the walls of which may or may not be silvered as required. Normally the unsilvered is preferred. Metal vacuum flasks may also be employed instead of the glass type. The bath tops are not available commercially and must be made of some nonheat-conducting material such as phenolic plastic. The small holes should be approximately 9.0 mm in diameter. The thermometers under test may then be held in place by means of small sections cut from a length of rubber tubing. It is important that the blades of the propeller shall give a downward thrust. If desired, the propeller may be mounted within a hollow tube so as to minimize swirling and improve the agitation of the bath liquid. The individual items needed are as follows:

##### X1.7.1 Materials for Long Bath:

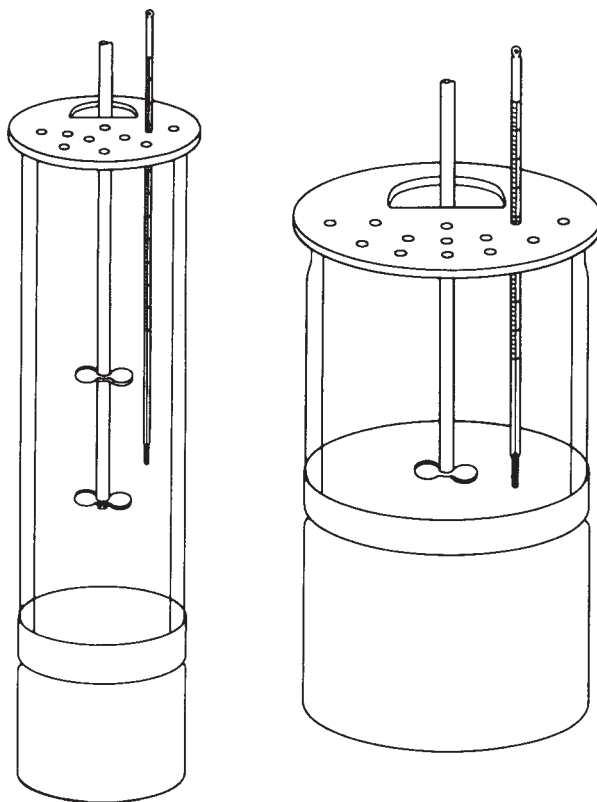


FIG. X1.4 Comparator for Temperature Range from  $-80$  to  $+5$  °C ( $-112$  to  $+41$  °F)

X1.7.1.1 Vacuum insulated flask 12 in. (305 mm) in depth by

X1.7.1.2  $2\frac{3}{4}$  in. (70 mm) in inside diameter,

X1.7.1.3 Phenolic plastic cover, as shown in Fig. X1.4, and

X1.7.1.4 Glass stirrer shaft with two propellers.

X1.7.2 *Materials for Short Bath:*

X1.7.2.1 Vacuum flask  $7\frac{3}{4}$  in. (197 mm) in depth by  $4\frac{5}{8}$  in.

X1.7.2.2 (117 mm) in inside diameter,

X1.7.2.3 Phenolic plastic cover, as shown in Fig. X1.4, and

X1.7.2.4 Glass stirrer with one propeller.

X1.7.3 *Accessories (for either bath):*

X1.7.3.1 Stirring motor,

X1.7.3.2 Sections of rubber tubing,

X1.7.3.3 Solid  $\text{CO}_2$  and a means of crushing it finely, and

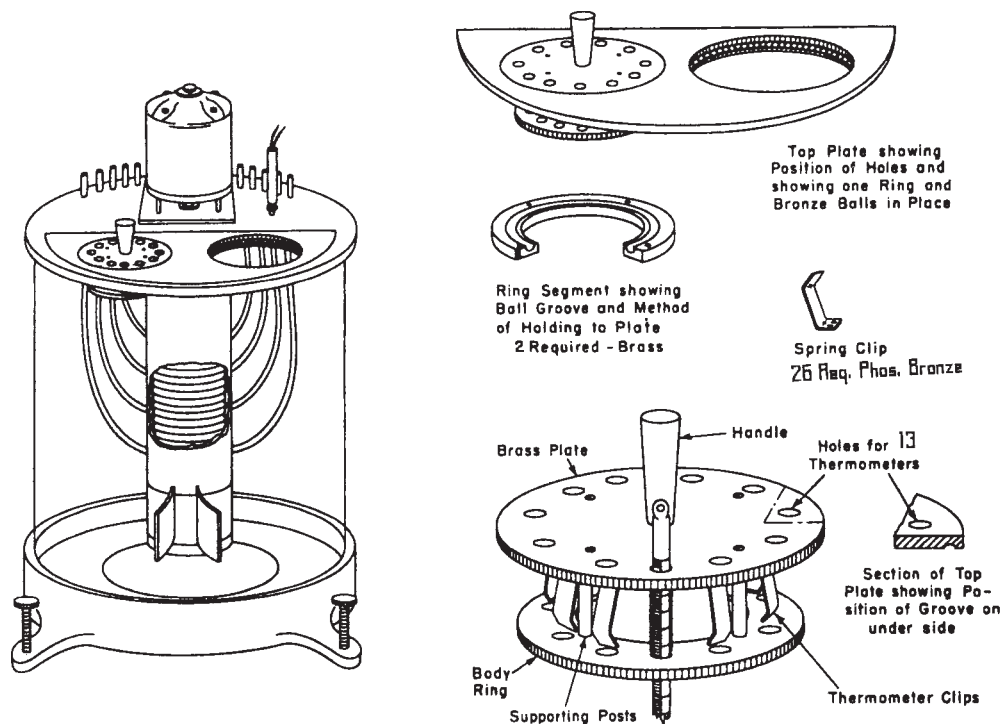
X1.7.3.4 Bath medium. This may be either alcohol, light hydrocarbon, or other organic solvent. If a water-miscible solvent is used, the water content must be not more than 5 %.

X1.7.3.5 In use the proper bath is partly filled with the bath medium. Dry ice is added slowly to prevent bubbling over. At the same time another portion of the liquid, sufficient to fill the bath and an extra amount to serve as a precooling bath, is similarly chilled in a second container. When the two are near the proper temperature, the testing bath is filled and brought to temperature. The thermometers to be tested and the standard may be precooled in the standby bath to approximately the desired temperature before testing. If large batches of thermometers are to be tested, this precooling will materially shorten the time required.

X1.8 *Comparators for the Range from 5 to 375 °C (41 to*

$707$  °F)—In the range from 5 to 200 °C (41 to 392 °F) a comparator as illustrated in Fig. X1.5, or its equivalent, may be used. It consists of a heavy wall borosilicate-glass jar suitably supported and equipped with heaters, cooling coil, and stirring equipment. A bath provided with controls to maintain constant or very slowly rising temperature throughout the testing range and with a top cover having a large opening is shown in Fig. X1.5. For thermometer testing an insert equipped with two rotary holders made as shown in Fig. X1.5 is provided. Each of these holders is simply a flat plate resting on a number of ball bearings so that the plate may easily be spun to bring any particular thermometer into view. Positive heat distribution to all parts of the bath and thorough circulation are obtained by a unique assembly of cooling and heating coils in a cylindrical housing which also surrounds the stirring propeller. When testing in the range of 5 to 95 °C (41 to 203 °F), the bath should be filled with distilled water. When testing in the range of 90 to 200 °C (194 to 392 °F), the bath should be filled with a suitable high-flash-point oil. The motor should then be mounted high enough to protect it from the heat of the bath.

X1.8.1 An alternative design is illustrated in Fig. X1.6(b). This type may be used with water or a series of oils of suitable characteristics in the range of 5 to 315 °C (41 to 599 °F). At the higher temperatures great care must be taken to avoid dangerous flash fires which may occur particularly on removal of thermometers or thermometer holder. A solid cover plate to replace the thermometer holder, as well as adequate fire protection equipment of the carbon-dioxide type, should be provided.



in.	mm
2	50.8
4	101.6
18	457.2

in.	mm
2	50.8
4	101.6
18	457.2

FIG. X1.5 Comparator for Temperature Range from 5 to 200 °C (41 to 392 °F)

X1.9 Comparator for the Range from 200 to 621 °C (392 to 1150 °F):

X1.9.1 A satisfactory comparator for this range is a salt bath. One type is illustrated in Fig. X1.7. However, a suitable apparatus may be assembled using a pressed-steel pot as the salt container which is provided with external heaters, insulation, stirrer, and thermometer holder. Welded or riveted pots should under no circumstances be used. External electrical heating is commonly practiced, although gas fired units have been used successfully.

X1.9.2 Care should be exercised, particularly above 400 °C (752 °F), to avoid bringing any organic matter or low-boiling liquid, such as water, in contact with the molten salt, since dangerous fires or explosions may occur. The bath should be

heated up slowly at the start to avoid the formation of pockets of molten salt which have a tendency to blow out the solid mass with disastrous results.

X1.9.3 At the lower temperatures the thermometers may be immersed directly in the salt, but at the higher temperatures attack of the glass may occur. This can be avoided by using steel wells immersed in the molten salt into which the thermometers are placed. Thin-walled steel tubes closed at one end are suitable for this purpose.

X1.9.4 Comparators with molten tin as the bath liquid, as shown in Fig. X1.6(a), have been used successfully in this temperature range. Details of design, construction, and use may be obtained from the National Institute of Standards and Technology.

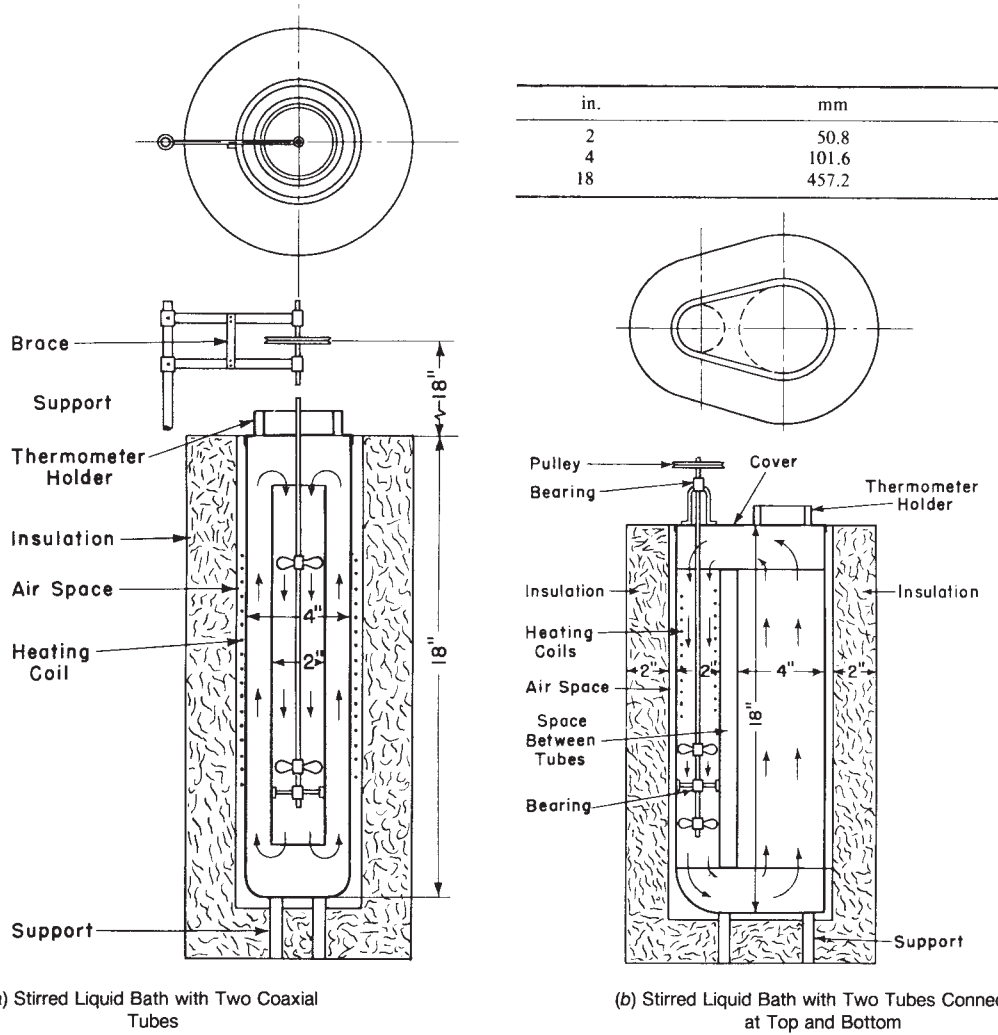
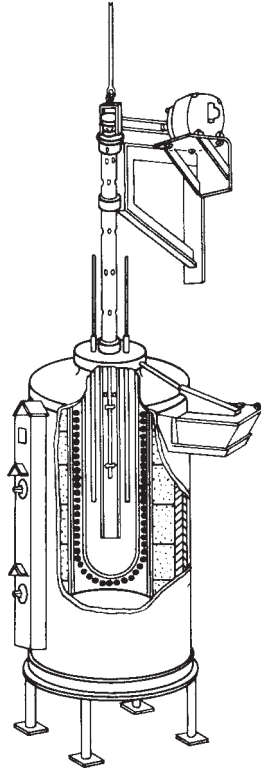


FIG. X1.6 Alternative Designs of Comparators for Temperature Ranges from 300 to 538 °C (572 to 1000 °F) and from 5 to 315 °C (41 to 599 °F)



**FIG. X1.7 Salt Bath Comparator for Temperature Range from 200 to 621 °C (392 to 1150 °F)**

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