Standard Test Method for Mean Specific Heat of Thermal Insulation[1]

This standard is issued under the fixed designation C 351; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of mean specific heat of thermal insulating materials. The materials must be essentially homogeneous and composed of matter in the solid state.

1.2 This test method employs the classical method of mixtures. This provides procedures and apparatus simpler than those generally used in scientific calorimetry, an accuracy that is adequate for most thermal insulating purposes, and a degree of precision that is reproducible by laboratory technicians of average skill. While this test method was developed for testing thermal insulations, it is easily adaptable to measuring the specific heat of other materials.

1.3 The test procedure provides for a mean temperature of approximately 60°C (100 to 20°C temperature range), using water as the calorimetric fluid. By substituting other calorimetric fluids the temperature range may be changed as desired.

1.4 The values stated in SI units are to be regarded as the standard.

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

2. Referenced Documents

2.1 *ASTM Standards:*
E 1 Specification for ASTM Thermometers[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *mean specific heat*—the quantity of heat required to change the temperature of a unit mass of a substance one degree, measured as the average quantity over the temperature range specified. (It is distinguished from true specific heat by being an average rather than a point value.) The unit of measurement is J/kg·K.

3.1.2 *thermal capacity*—the amount of heat necessary to change the temperature of the body one degree. For a homogeneous body it is the product of mass and specific heat. For a nonhomogeneous body it is the sum of the products of mass and specific heat of the individual constituents. Thermal capacity has the units of J/K.

3.1.3 *thermal diffusivity*—the ratio of thermal conductivity of a substance to the product of its density and specific heat. Common unit for this property is m²/s.

3.1.4 *water equivalent*—the mass of water that requires the same amount of heat as the given body in order to change its temperature by an equal amount.

4. Summary of Test Method

4.1 The method of mixtures used in this test method consists essentially of adding a known mass of material at a known high temperature to a known mass of water at a known low temperature and determining the equilibrium temperature that results. The heat absorbed by the water and the containing vessel can be calculated and this value equated to the expression for the heat given up by the hot material. From this equation[3] the unknown specific heat can be calculated.

5. Significance and Use

5.1 Mean specific heat is an essential property of a thermal insulating material when the latter is used under conditions of unsteady or transient heat flow. It is a part of the parameter, thermal diffusivity, which governs the rate of temperature diffusion through insulation. It is a basic thermodynamic property of all substances, the value of which depends upon chemical composition and temperature.

NOTE 1—Specific heat of insulations, as measured by this test method, using small specimens of a multi-component composite or of a low-density product that has to be highly compressed, may not be directly applicable for use in calculations involving transient thermal response. The applicability of the results will depend upon a system being analyzed, the desired accuracy, and the relative amounts, and specific heats of the various solid or fluid components, or both, of the thermal insulation.

6. Apparatus

6.1 The typical apparatus is shown schematically in Fig. 1. It consists of the following:

6.1.1 *Calorimeter and Accessories*—The calorimeter shall be an unlagged Dewar flask with a maximum capacity of not less than 500 mL nor more than 750 mL. The flask shall have

---

[1] This test method is under the jurisdiction of ASTM Committee C-16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurements.

Current edition approved Aug. 15, 1992. Published October 1992. Originally published as C 351 – 54 T. Last previous edition C 351 – 92a.

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

[3] Weber, R. L., *Heat and Temperature Measurement,* Prentice-Hall, New York, NY, 1950.
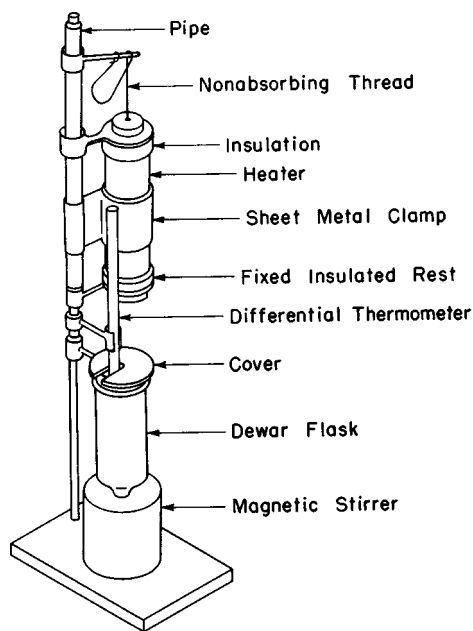
FIG. 1 Specific Heat Calorimeter

an insulated cover or stopper. Other accessories shall include a magnetic stirrer equipped with a speed-regulating device.

6.1.2 *Differential Temperature Sensor*—An appropriate temperature difference sensor, such as a Beckmann differential thermometer or a suitable equivalent, preferably with a magnifier, and having a range of at least 5°C and a sensitivity of no less than 0.01°C shall be used to determine the rise in temperature of the calorimetric fluid during test. Where a differential thermometer is used, it shall be set with its lowest temperature division at the approximate room temperature, and the setting point checked with a precision-type temperature sensor (such as Thermometer 632C, preferably with a magnifier, described in Specification E 1).

6.1.3 *Heater*—The heater shall be of the open-end radiation type similar to the cylindrical device shown in Fig. 1. It may be heated by electricity or steam. The relative dimensions of the heater and the capsule shall be such that the specimen will be heated to a uniform and constant temperature as required. A maximum variation of ± 1°C over the length of the heater is permitted. The heater shall be provided with an insulated removable top cover designed both to permit passage of the leads of the temperature sensor and to suspend the capsule. The bottom shall be closed with a removable insulated cover to permit free dropping of the capsule. The heater assembly shall be mounted so it can be swung quickly into place over the calorimeter.

6.1.3.1 A convenient form of electric heater can be constructed by covering a 254-mm length of 38-mm diameter brass pipe with asbestos paper, winding about 70 turns of 22-gage (0.64 mm) Nichrome wire over the paper, and insulating the assembly with 25.4-mm thick pipe insulation. It is necessary that the end turns be closer together than those over the center portion of the heater to compensate for end heat loss. The heater temperature is controlled by regulating the electric current to the heater with a variable transformer or resistor. A constant voltage source of power within ±1 % maximum

voltage variation is necessary to minimize temperature fluctuations.

6.1.4 *Capsule*—The capsule shall comprise a hollow cylinder of brass approximately 25.4 mm in diameter by 50.8 mm long. It shall have a removable cap and a thermocouple well extending into the cavity space to accommodate the temperature sensor. It is imperative that the capsule assembly be absolutely watertight, as no leakage can be tolerated. The completed capsule, including cap, gasket, and suspension loop, shall have a thermal capacity not exceeding 10.5 J/K. A capsule design meeting the above requirements is shown in Fig. 2.

6.1.5 *Temperature Sensor*—A suitable, calibrated temperature sensor and associated read-out equipment of suitable range and precision to permit reading temperatures to an accuracy of 0.1°C shall be used. If a thermocouple is used, the wire size should be small to limit the error caused by thermal conductance losses along the length. Thermocouples can be made from any of the standard pairs registered with the National Institute of Standards and Technology.[4] A particular suitable thermocouple is chromel/constantan, fabricated from wires having a diameter no greater than No. 30 B & S gage (0.265 mm). The pair combines the attributes of reduced heat leakage and a higher emf than copper/constantan.

6.1.6 *Test Room*—The temperature of the room in which the test is conducted shall be reasonably constant during the test period. The test room temperature control is satisfactory if the time-temperature curve is a straight line (within the allowable experimental error) for a 10-min period before the dropping of the capsule and for a 10-min period just prior to the termination of the test.

6.1.7 *Specific Heat Standard*—Electrolytic copper (commercial electrical bus bar copper) shall be used as a specific heat standard. A standard specimen as shown in Fig. 3 shall be used to determine the water equivalent of the calorimeter flask and its accessories. For the temperature range covered (between 100 and 20°C) the mean specific heat of copper shall be taken as 390.0 J/(kg·K).

7. Test Specimens

7.1 Specimens shall be selected at random as required to provide test material representative of the lot sampled. The number of specimens may be determined by agreement but shall be not less than three.

7.2 The specimens shall be tested in the compressed form, since it is desirable to have as large a mass as possible. A specimen press consisting of a hollow cylinder with a close-fitting plunger used in conjunction with a bench vise or small hydraulic press has been found convenient.

7.3 Prior to test, all specimens shall be dried to constant mass in an oven at a temperature of 102 to 120°C. If the specimen is likely to be damaged by this temperature, it may be dried in a desiccator at a lower temperature. If the specimens are dried at temperatures lower than 102°C, this fact shall be reported.

---

[4] "Reference Table for Thermocouples," *Circular No. 508*, National Institute of Standards and Technology, Gaithersburg, MD 20899.
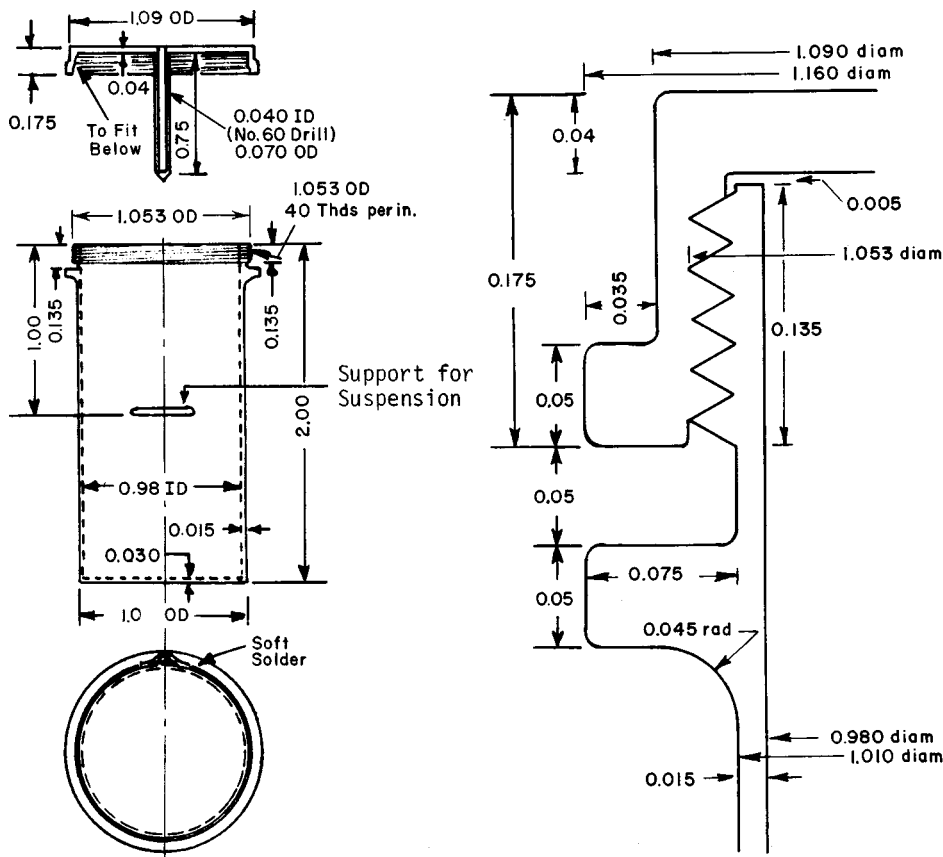
All Dimensions in Inches

(a) Capsule Assembly		(b) Screw-Cap Assembly (Enlarged)	
Inch-Pound Units	SI Equivalents	Inch-Pound Units	SI Equivalents
0.005 in.	0.13 mm	0.175 in.	4.45 mm
0.015 in.	0.38 mm	0.75 in.	19.1 mm
0.030 in.	0.76 mm	0.98 in.	25.1 mm
0.035 in.	0.89 mm	1.00 in.	25.4 mm
0.040 in.	1.0 mm	1.010 in.	25.65 mm
0.045 in.	1.14 mm	1.053 in.	26.75 mm
0.05 in.	1.3 mm	1.090 in.	27.68 mm
0.070 in.	1.8 mm	1.160 in.	29.46 mm
0.135 in.	3.43 mm	40 thds/in.	1.575 thds/mm

Note 1—In addition to the dimensional requirements shown on the drawing, the capsule assembly shall conform to the following requirements:
Material—Brass, (nominal composition 65 to 75 % Cu, 35 to 25 % Zn) except as noted on drawing.
  Mass (total)–less than 26 g
  Volume–3.084 cm^3
  Specific heat of brass–410 J/kg·K. M_cC_c 10.66 J/K

FIG. 2 Capsule

8. Calibration

8.1 Determine the water equivalent of the calorimeter and its accessories, following the same procedure as described for the specific heat determination in Section 9, except to substitute the specific heat standard (6.1.7) for the capsule and specimen. Determine the thermal capacity of the capsule assembly by the same procedure except to leave the capsule empty (Note 2). It is extremely important that all the test details and the procedure followed be precisely the same in all particulars for the specific heat determinations, the water equivalent determinations, and the thermal capacity determinations. Capsule transit time, radiation losses, and evaporation must be held constant by strict routines, and their evaluations will thus be included in the water equivalent and the heat capacity.

Note 2—It may be desirable to weight the empty capsule with a known quantity of bus bar copper to facilitate the transfer to the calorimeter. If this is done it must be taken into account in the subsequent calculations.

9. Procedure

9.1 Place a dried specimen of as large a mass as practical in the capsule and screw the cap on tightly. Determine the mass of the dried specimen, to the nearest 0.001 g, as the difference between the total mass of the specimen plus capsule assembly and the mass of the empty capsule assembly. Suspend the capsule containing the specimen in the heater by means of

3

FIG. 3 Specific Heat Standard
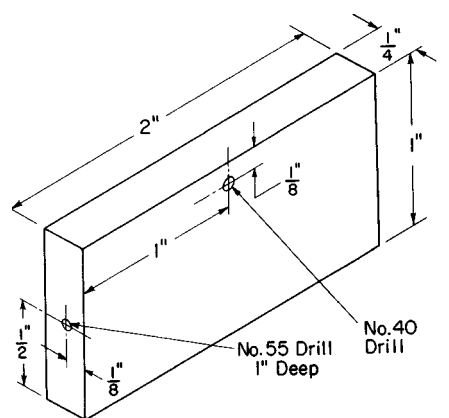
Material — Commercial Bus Bar Copper

Mass — 70 to 75 g

Inch-Pound Units	SI Equivalents	Inch-Pound Units	SI Equivalents
1/8 in.	3.2 mm	2 in.	50.8 mm
1/4 in.	6.4 mm	No. 40 drill	6 mm
1/2 in.	12.7 mm	No. 55 drill	4 mm
1 in.	25.4 mm		

up to 150°C. Where the upper limit of the temperature range is greater than 150°C, a liquid with a high boiling point having a mean specific heat (over the temperature range $T_m - T_c$) known to an accuracy of ± 1 % and a flash point above the temperature of the heated capsule, T_h, must be used. This liquid shall be noncorrosive to the apparatus, essentially nontoxic, and nonirritating to the skin and should have a low vapor pressure to minimize volatilization losses. Where the lower limit of the temperature range is below 0°C, the calorimeter bath must be a low freezing point liquid having a mean specific heat (over the temperature range $T_m - T_c$) known to an accuracy of ± 1 %. It must be noncorrosive to the apparatus, essentially nontoxic, nonirritating to the skin, and resistant to hydrolysis.

9.3 After approximately 10 min, when a constant thermal exchange exists between the calorimeter and the environment, start observations of the temperature of the calorimeter. Since the calorimeter is close to ambient temperature, the rate of thermal exchange will be small. Record the temperature of the calorimeter (estimated to the nearest 0.001°C) at the end of 1-min intervals for a period of 9 min. Immediately before the end of the tenth minute, swing the heater over the top of the calorimeter and remove the cover of the calorimeter. Precisely at the end of the tenth minute, lower the capsule rapidly into the calorimeter (Note 5), replace the calorimeter cover, and swing the heater back into position. Beginning with the eleventh minute, resume temperature readings and continue them at 1-min intervals until the slope of the temperature-time curve is constant over a 10-min interval. Generally, this means through about the thirtieth minute.

NOTE 5—If the transfer of the capsule is not completed precisely at the end of the tenth minute, the actual time shall be noted and used in the extrapolation determination of T_c and T_m.

9.4 Upon completion of a test, reweigh the capsule and specimen. Discard any test run that shows an increase in mass of the specimen greater than 0.001 g due to water leakage into the capsule.

nonabsorbing thread (Note 3), with the temperature sensor inserted to the full depth of the well in the capsule. Care must be taken to see that the leads are not shorted by contact with the capsule or heater. If a thermocouple is used, it is also desirable to keep the lead wires together to minimize Thomson effects. Regulate the heater power so that the temperature of the specimen, when heated, will be between 95 and 100°C. Adjust the total length of the thread so that when the capsule is dropped into the calorimeter flask the thread will hold it suspended entirely submerged in the fluid. It should be noted that the capsule is in a vertical position while in the heater, and horizontal in the flask. This latter position minimizes the amount of water needed in the flask.

NOTE 3—Nylon and orlon acrylic fiber have been found satisfactory for this purpose.

9.2 When the capsule and specimen are in thermal equilibrium in the heater, with the temperature within the capsule changing not more than 0.1°C over three successive 5-min readings, pour 300 $\pm$ 5 g of distilled water (Note 4) at a temperature of approximately 20°C into the flask. Determine the exact mass of water, to the nearest 0.1 g, as the difference between the mass of the flask with water and the mass of the empty flask. Place the flask with the water, and the differential temperature sensor, in position (Fig. 1) and set the stirrer in motion. Keep the flask covered at all times except when the capsule is actually being dropped into the flask. It is also important that a moderate stirring rate be used, note being made of the speed setting, and care taken that the same setting be used during all determinations. An appreciable amount of heat is introduced by the agitation of the water. In order to secure greatest accuracy, the rate of introduction of heat should be as low as possible and particularly, as constant as possible.

NOTE 4—This test method specifies a temperature range of approximately 100°C to 20°C. When specific heat information is needed at other temperatures, a similar test procedure should be followed. In general, distilled water is suitable as a calorimetric fluid for capsule temperatures

10. Calculation

10.1 Plot the temperature of the bath against time, and determine the value of T_m and T_c by extrapolation as illustrated in Fig. 4. The thermal exchange that takes place in the tenth minute is considered instantaneous and is proportional to the temperature difference, $T_m - T_c$, which is read from the graph.

10.2 Calculate the water equivalent, E, of the calorimeter flask and its accessories, to the nearest 0.1 g, as follows:

$$E = [M_r C_r (T_h - T_m)/C_w (T_m - T_c)] - M_w \tag{1}$$

Take as the value of E, the average of at least three satisfactory determinations.

10.3 Calculate the thermal capacity of the capsule, to the nearest 0.05 J/K, as follows:

$$M_c C_c = (M_w + E) C_w (T_m - T_c)/(T_h - T_m) \tag{2}$$

Take as the value of $M_c C_c$, the average of at least three satisfactory determinations.

10.4 Calculate the specific heat of the specimen as follows:

$$C_s = \frac{[(M_w + E) C_w (T_m - T_c)/(T_h - T_m)] - M_c C_c}{M_s} \tag{3}$$

10.5 *Symbols*—The symbols used in Eq 1, Eq 2, and Eq 3 have the following significance:
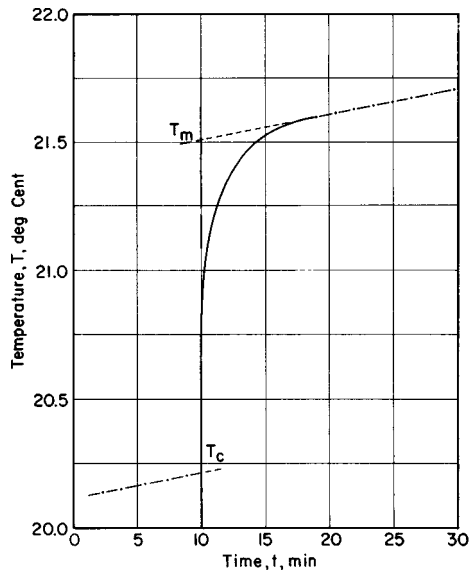
4

FIG. 4 Curve Showing Relation of Temperature to Time for a Typical Specific Heat Determination

E = water equivalent of the calorimeter and its accessories, kg,

M_r = mass of the specific heat standard, kg,

C_r = mean specific heat of the specific heat standard over the temperature range from T_h to T_m, J/(kg·K) (= 390.0 J/(kg·K) for electrolytic copper over the temperature range from 100 to 20°C)

T_h = temperature of the capsule and specimen, capsule, or standard after heating, °C,

T_m = temperature of the mixture extrapolated back to time = 10 min,° C,

C_w = mean specific heat of calorimeter water (Note 6), J/(kg·K),

T_c = temperature of the calorimeter water before capsule or standard is dropped, extrapolated ahead of time = 10 min,° C,

$M_c C_c$ = thermal capacity of the capsule assembly, J/K,

M_w = mass of calorimeter water, kg,

C_s = mean specific heat of test specimen over the temperature range from T_h to T_m, J/(kg·K), and

M_s = mass of the specimen, kg.

NOTE 6—In Eq 1, Eq 2, and Eq 3, the specific heat of distilled water may be taken as 4186 J/(kg·K). If other calorimetric fluids are substituted for distilled water, the calculations must be modified to include the specific heat of the substituted calorimetric fluid.

11. Report

11.1 Report the following information:

11.1.1 Mean specific heat of the material tested, taken as the average of the three determinations on each specimen, and expressed to two significant figures,

11.1.2 Results given in SI units and in inch-pound units (4186 J/(kg·K) = 1 Btu/(lb·°F),

11.1.3 Average temperature range of the test, and

11.1.4 Any deviation from the standard test procedure, including the use of a calorimetric fluid other than distilled water.

12. Precision and Bias

12.1 For the general range and form of thermal insulations currently available, there is a wide variation in the mass-specific heat product. For this reason, it is difficult to state an absolute bias to cover measurement on all materials without possible modification in the apparatus for each material. The results of a limited round-robin study[5] on one material indicated that a bias of ±10 % with comparable precision can be obtained, but that a higher bias is attainable.

13. Keywords

13.1 calorimeter; electrolytic copper; method of mixtures; specific heat; thermal capacity; thermal diffusivity; water equivalent

[5] Spear, Norman, A., "A Proposed Method of Test for Specific Heat of Thermal Insulating Materials," *ASTM Bulletin*, September 1950, pp. 79–82.