



Designation: C 745 – 92 (Reapproved 1999)

Standard Test Method for Heat Flux Through Evacuated Insulations Using a Guarded Flat Plate Boiloff Calorimeter¹

This standard is issued under the fixed designation C 745; 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, from cryogenic to near room temperatures, of heat flux through evacuated insulations (Note 1) within the approximate range from 0.3 to 30 W/m². Heat flux values obtained using this method apply strictly only to the particular specimens as tested.

NOTE 1—This test method is primarily intended for use to assess heat flux through evacuated *multilayer* insulations which are highly anisotropic by nature. Characteristically, multilayer insulations exhibit apparent thermal conductivity values one or two orders of magnitude lower than the best available powder, fiber, or foam insulations. Although this test method is also technically applicable to these latter insulations, other ASTM methods with less stringent requirements are equally applicable and much more economical and practical for such materials.

1.2 This shall be a primary test method for measuring heat flux through evacuated insulations (Note 2), since calibration of the apparatus depends on measurement standards traceable to the National Institute of Standards and Technology (NIST) for length, force, temperature, time, etc. Traceable standards are not yet available for heat flux through standard evacuated reference specimens or transfer standards.

NOTE 2—Values of heat flux for the same materials and environments specified in this method may also be obtained by measuring electrical energy dissipation using a guarded hot plate (Test Method C 177) (1, 2)² or a guarded cylindrical apparatus (3, 4), or by measuring transient thermal response (5).

1.3 Specimens to be tested using this method shall be flat and may be either a circular or a rectangular configuration, as appropriate for the particular apparatus being used (Note 3). Contoured specimens or those of other shapes must be tested by other methods which are outside the scope of this standard. Specimen sizes and thicknesses shall conform to the limitations specified in Section 7.

NOTE 3—Existing guarded flat plate boil-off calorimeters require cir-

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 Measurement.

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² The boldface numbers in parentheses refer to the list of references at the end of this test method.

cular specimens. For highly anisotropic multilayer insulations, this configuration somewhat simplifies heat transfer calculations, since the resulting heat flow is two-dimensional rather than three-dimensional as it would be for a rectangular specimen.

1.4 Environmental and other parameters that can be varied in the application of this method are (1) the hot and cold boundary temperatures, (2) the boundary temperature at the exposed edge of the specimen, (3) the mechanical compressive pressure to be imposed on the specimen, and (4) the species and partial pressure of the gas occupying the interlayer cavities of the specimen and the test chamber (Note 4). Hot boundary temperature can be varied within the approximate range from 250 to 670 K, while cold boundary temperature can be varied from approximately 20 to 300 K (Note 5). Selection of boundary temperatures to be imposed at the hot and cold surfaces and at the edge of the specimen shall be subject to the limitations specified in Section 5. Mechanical compressive pressure values to be imposed using this method can vary in the approximate range from 5 to 10 kPa (Note 6).

NOTE 4—Although this test method is primarily intended for use to measure heat flux through evacuated insulations, it is also applicable for measurements where the specimen contains air or other gases at pressures ranging from fully evacuated to atmospheric. However, where measurements are to be made on a specimen that is not evacuated to a pressure of 1 mPa or less, the apparatus shall be provided with a low-conductivity pressure diaphragm to maintain high-vacuum conditions in the annular space between the measuring and guard vessels.

Heat transfer through evacuated multilayer insulations can vary significantly from specimen to specimen or from test to test due to the presence of minute but unknown quantities of outgas components (primarily water vapor) within the interstitial cavities. This effect can be minimized with preconditioning of the specimen by extended evacuation at room temperature or by a combination of heat and evacuation over a much shorter time span (see 9.2).

NOTE 5—Cold boundary temperatures down to that of liquid hydrogen (20 K) can be achieved using existing apparatus. Temperatures to approximately 4 K could be achieved with development of an apparatus suitable for use with liquid helium.

NOTE 6—The lower limit of mechanical compressive pressure that can be achieved for any particular specimen is the *self-compression* value due to the weight of the specimen within the earth's gravitational field.

1.5 Stating that test results were obtained using this specific method requires that all of the variables must be controlled, measured, and recorded as specified herein.

1.6 Details of construction of the calorimeter cannot be covered entirely by this specification since some technical knowledge is required regarding the compatibility of materials with the fluids used, temperature extremes that will be encountered, practical limitations in achieving and controlling the mechanical compressive pressure, and other contingencies. However, existing types of construction and measuring techniques were considered as a guide for this specification and are presented herein as requirements with the realization that developments and improvements can always be made.

1.7 SI units are to be regarded as standard in this test method. Conversion factors for use to obtain imperial equivalents are presented in Table A1.1.

1.8 *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.* Specific precautionary statements are given in Section 6.

2. Referenced Documents

2.1 ASTM Standards:

C 177 Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded Hot-Plate Apparatus³

2.2 Military Specifications:

MIL-SPEC-P-27201C Propellant, Hydrogen⁴

MIL-SPEC-P-27401B Propellant Pressurizing Agent, Nitrogen⁴

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *heat flux*—for the purposes of this standard, heat flux is defined as the time rate of heat flow, under steady-state conditions, through unit area, in a direction perpendicular to the isothermal shield and spacer surfaces. It is calculated using the equations presented in Section 11.

3.1.2 *multilayer insulations*—for the purposes of this test method, multilayer insulations are defined as those composed of multiple radiation shields mechanically separated to reduce conductive heat transfer. In most applications, the radiation shields are thin plastic membranes (usually polyester or polyimide films) coated on one or both sides with a low-emittance, vapor-deposited metal (usually aluminum, gold, or silver), but they can be thin metal foil membranes. Separation of the shields can be accomplished by (1) alternating thin layers of low-density, low-conductivity materials such as woven fabric net, fibrous paper, powder insulation, or sliced foam spacers with the radiation shields; (2) bonding low-density, low-conductivity filaments to one side of the radiation shields; or (3) mechanically crinkling, dimpling, or embossing the radiation shields themselves. Where the latter technique is employed, the radiation shields are commonly metallized on one side only to achieve minimum conductive heat transfer.

³ Annual Book of ASTM Standards, Vol 04.06.

⁴ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

4. Significance and Use

4.1 The thermal performance of multilayer insulations will vary from specimen to specimen due to differences in the material properties, such as the emittance of the reflective shields. In addition, it can vary due to environmental conditioning and the presence of foreign matter such as oxygen or water vapor. Finally, it can vary due to aging, settling, or exposure to excessive mechanical pressures which could wrinkle or otherwise affect the surface texture of the layers. For these reasons, it is imperative that specimen materials be selected carefully to obtain representative samples. It is recommended that several specimens of any one material be tested and no less than four data points obtained for each. For specimens where heat transfer measurements under high-vacuum conditions are required, a preconditioning procedure should be employed to remove water vapor and other outgas components from the multilayer materials.

5. Calorimeter

5.1 In this device, thermal energy transferred through an insulation specimen is measured by a boiloff calorimeter method (6). Ideally, all of the energy crossing the cold boundary in a direction normal to the plane of the insulation layers in the central portion of a circular or rectangular specimen is intercepted by a boiling fluid maintained at constant saturation conditions. Calorimeter fluids selected for use with this method shall meet the requirements for purity specified in MIL-SPEC-P-27201C and MIL-SPEC-P-27401B. This energy is absorbed totally by vaporization of the calorimetric fluid that is subsequently vented. Heat flux is calculated from thermodynamic properties of the fluid and the measured boiloff flow rate. Measurements of the mechanical compressive force applied to the specimen and the separation between hot and cold boundary surfaces in contact with the insulation also are obtained. Minimum requirements for a flat-plate calorimeter (FPC) that is suitable for use with this method are described in Annex A1. Particular design features required for safety are discussed in Section 6. A typical FPC design is shown in the cross-section drawing of Fig. A1.1.

6. Safety Precautions

6.1 Prior to operation of the FPC with any potentially hazardous fluid such as natural gas (LNG) or hydrogen (LH₂), a complete review of the design, construction, and installation of all systems shall be conducted. Safety practices and procedures regarding handling of hazardous fluids have been extensively developed and proven through many years of use (7, 8, 9, 10). Particular attention shall be given to ensure (1) adequate ventilation in the test area, (2) prevention of leaks, (3) elimination of ignition sources, (4) failsafe design, and (5) redundancy provisions for fluid fill and vent lines.

7. Test Specimens

7.1 Prepare test specimens from previously selected materials. Cut spacers to the diameter or width of the hot and cold boundary plates. Cut the radiation shield to a diameter that is approximately 10 mm less. The maximum specimen thickness to be tested using this test method shall be 0.05 times the guard width.

NOTE 7—The maximum specimen thickness that can be tested for a fixed guard width will increase as the degree of anisotropy of the insulation decreases for any specified allowable tolerance on measured heat flux values. However, a constant thickness-to-guard ratio is specified for this method since no valid criteria currently exist to determine the maximum allowable thickness based on material properties that are readily available prior to test. The specified ratio of 0.05 is based on tests conducted for one highly anisotropic multilayer insulation using an existing apparatus and allowing a tolerance of $\pm 5\%$ on the measured heat flux value. It is conservative for more isotropic insulations, and can be increased for use with this method provided sufficient analytical or test data are obtained to show that the error in measured heat flux does not exceed $\pm 5\%$. Heat transfer mechanisms that result in the edge effect error include parallel conductivity, primarily through the radiation shield metallic coatings, and radiation tunneling between the multilayer shield and spacer material. Calculation of the edge effects is made complex by interaction of the parallel conduction and radiation mechanisms and by the variable temperature difference between the edge guard radiation shield and the multilayers.

7.2 Unless otherwise specified, the following procedures shall be used to prepare specimens for test using this method:

7.2.1 Visually inspect multilayer shield and spacer materials and cut the test specimens from material that is free of tears, abnormal creases, or other defects. Clean gloves shall be worn when handling materials and specimens.

7.2.2 Use a template to ensure that each layer of shield and spacer material is cut uniformly to the desired dimensions within ± 5 mm. Clean the template with a suitable degreasing solvent and take care to avoid touching the template or the multilayers with bare fingers or soiled gloves.

7.2.3 Determine within $\pm 10\%$ the total hemispherical emittance at room temperature for one or more of the specimen shields. This may be accomplished either by direct measurement using a calorimetric emittance apparatus or by calculation based on a measurement of the near-normal infrared reflectance. An equation relating the near-normal infrared reflectance to total emittance is presented in Section 11.

7.2.4 Install specimens consisting of reflective shield metallized on one side only into the FPC with the metallized surfaces oriented toward the hot boundary plate. Assemble and install with spacer layers at each of the hot and cold boundary surfaces those specimens that consist of separate reflective shields and spacers.

7.2.5 Weigh assembled specimens and measure their free-stacked thicknesses prior to installation into the FPC. Record the mass and thickness values.

8. Calibration

8.1 Prior to each test or every 3 months if in continuous use, leak check the measuring vessel and all associated plumbing lines using helium gas. Calibrate the FPC instrumentation and data recording equipment to ensure that the required measurement accuracies are being achieved. The maximum permissible leakage rate shall be 1×10^{-6} standard cm^3/s of helium gas.

8.2 Calibrate the temperature sensors that are used to measure the temperature of the hot boundary plate, the edge guard radiation shield, and other components by comparison of outputs with secondary standards traceable to NIST at several temperature values over the full operating range.

8.3 Calibrate the load cell or equivalent device that is used to measure mechanical compressive pressures imposed on the

test specimen over the full operating load range using secondary standard cells or dead weights traceable to NIST. Perform this calibration with the load cell at the anticipated test temperature.

8.4 Calibrate the system used to measure the hot and cold boundary plate separation by placing precision-machined gage blocks between the plate surfaces. Then raise the lower plate until solid contact has been established between the gage blocks and the upper plate. Perform this calibration with hot and cold boundary surface temperatures initially representative of those to be used in actual testing.

8.5 Calibrate the flowmeter by comparison of its output at flow rate over the entire anticipated range, using the gas to be measured during the test with that from a secondary standard flowmeter traceable to NIST, or by using a calibrated gas prover.

8.6 Calibrate the entire FPC assembly by introducing known quantities of thermal energy which simulate heat flux values over the anticipated range for actual tests and by comparing these energy values with those calculated from steady-state boiloff data. During this calibration, isolate thermally the cold boundary plate of the measuring vessel from all surrounding heat sources except those associated with the guard vessel. Accomplish thermal isolation of the cold plate by use of the FPC configuration shown schematically in Fig. A1.2, or an equivalent configuration.

8.7 When the configuration shown in Fig. A1.2 is used, attach the resistor to the midpoint of the calorimeter surface plate with a silver-filled epoxy or an equivalent high-conductance cement. Cover its exposed surfaces with an aluminum tape to minimize radiative losses to the surroundings. Select this resistor to provide a relatively small resistance value compared to the total resistance of the voltage divider in order to minimize the potential error in determining the power measurement. Attach a dished, polished copper plate to the inner guard surface, using indium wire to increase heat transfer, in order to provide an enclosure at guard temperature. Insulate the lower surface of the plate with not less than 30 mm of multilayer insulation, and maintain the hot boundary at the same temperature as the guard vessel. Under these conditions, heat transfer from the copper plate to the test section will occur by radiation only. By connecting the plate to the guard thermally and by insulating it from any higher temperature surroundings, it remains uniformly at the temperature of the guard, which will be approximately 0.03 K higher than the calorimeter temperature. Under these conditions, the only heat input into the calorimeter will be that from the resistor plus any heat leaks from the inner guard to the calorimeter or down the fill and vent tubes which are not intercepted by the radiation baffle and guard.

9. Specimen Loading and Preconditioning Procedure

9.1 Prior to the placement of an assembled test specimen into the FPC, check the operation of the hot boundary plate height adjustment mechanism and the load cell or equivalent force-measuring device. Place a known dead weight equivalent to a compressive pressure of approximately 35 Pa in the center of the hot boundary plate. Either record the tare weight of the plate and its supporting mechanism or compensate for it by

zero-adjustment of the load cell readout equipment. Subsequently, move the plate up and down to verify that the cell output is not affected by variations in bearing friction in the plate guides. Then remove the weight and place the insulation specimen on the hot boundary plate. Determine the uncompressed thickness of the specimen by raising the lower plate until the upper insulation surface is observed to contact the cold surface plate. Measure the spacing between plates using the instrumentation provided, and at each of the viewing ports where these are provided. Then adjust the separation between the hot and cold surfaces to at least twice the initial specimen thickness so that the insulation will not be subjected to any compressive loading during evacuation of the apparatus and preconditioning of the specimen.

9.2 Precondition test specimens that are to be evacuated to a pressure of 1 mPa or less during heat transfer testing to remove excess water vapor and other outgas components prior to initiation of the heat transfer testing. The preconditioning can be accomplished by continued vacuum pumping at room temperature for a minimum of 240 h, or by applying heat to the specimen with subsequent vacuum pumping for a much shorter time. It is recommended that a vacuum oven facility be used; however, the preconditioning can be accomplished within the FPC apparatus. The recommended procedure consists of (1) an initial evacuation of the specimen to achieve a chamber pressure of approximately 1 Pa, (2) backfilling the chamber to atmospheric pressure using dry nitrogen or dry helium gas, (3) operating the heaters to maintain a temperature of approximately 375 K for a minimum of 12 h, and (4) evacuation to maintain a chamber pressure of 1 mPa or less for a minimum of 24 h. If a vacuum oven facility is used, the specimen must be backfilled with dry nitrogen or dry helium gas to achieve atmospheric pressure prior to removing it from the oven for immediate installation into the FPC and reevacuation. After preconditioning, fill the fluid reservoirs and raise the hot boundary plate to achieve the desired specimen thickness or compressive pressure, or both, for the heat transfer test.

10. Procedure

10.1 Subsequent to the installation, preconditioning, evacuation, and initial loading of the test specimen as specified in Section 9, set the hot boundary and edge guard radiation shield temperatures to the values desired for the test. In addition, set the pressure controls of both the measuring and the guard vessels and the temperature of the vent gas environment control chamber to the specified values. Continue the test until equilibrium conditions have been effectively achieved (Note 9). Record temperatures at hourly intervals, but record the pressure differential between the calorimeter and primary guard vessels and the load cell output continuously. Record plate separation and flowmeter environmental temperature and pressure at not greater than 4-h intervals throughout the duration of the test. After completion of the test as described above, obtain additional data as required for other hot boundary temperatures, or specimen thicknesses or compressive loads, or a combination thereof.

NOTE 8—The time required to reach equilibrium conditions will vary significantly with the thermal and physical characteristics of the insulation

materials and of the calorimeter fluid used. Good engineering judgment should be exercised in determining the time when equilibrium conditions have been effectively achieved. This can be accomplished best by plotting boiloff flowrate values as a function of time. The slope of the flowrate-time curve will approach zero asymptotically at equilibrium; however, *quasi-equilibrium* can often be achieved many hours before *true equilibrium* has been attained. For purposes of this specification, *quasi-equilibrium* is defined as that condition where the measured boil-off flowrate is within $\pm 5\%$ of the value of true equilibrium.

11. Calculation

11.1 Calculate the net mechanical compressive pressure that is imposed on the test specimen as follows:

$$P_M = (F_A - F_O)/A_s \quad (1)$$

where:

P_M = net mechanical compressive pressure imposed on the test specimen in a direction normal to the multilayers, Pa (= N/m²),

F_A = total compressive force applied to the specimen as measured by the load cell or equivalent measuring device, N,

F_O = incremental force due to the total mass weight of the hot boundary plate and the supporting mechanism, and one half of the specimen mass, N, and

A_s = total surface area of the test specimen at the test temperature which is in contact with the hot and cold boundary plates, m².

Values of F_A shall be obtained from load-cell or equivalent force-measuring system outputs. The value of F_O shall be taken from the calibration data, and the value for A_s shall be that corresponding to the area of the FPC cold boundary surface at the anticipated test temperature.

11.2 Calculate the heat rate through a multilayer test specimen as follows:

$$Q_S = V_G H_V \rho_G - Q_O \quad (2)$$

where:

Q_S = time rate of heat flow through the test specimen in a direction normal to the multilayers, W,

V_G = volume flow rate of the calorimetric fluid boil-off gas at the specified vent pressure and temperature, m³/s,

H_V = latent heat of vaporization of the calorimetric fluid, J/kg,

ρ_G = density of the calorimetric fluid boiloff gas at the specified vent pressure and temperature, kg/m³, and

Q_O = heat leak into the calorimeter, W.

The volume flow rate of the boiloff gas V_G , shall be taken from the flowmeter output at steady-state conditions (Note 9). Values for the latent heat of vaporization and gas density of any calorimetric fluid can be obtained from handbook data. The value of calorimeter heat leak shall be that determined during the system calibration.

NOTE 9—The measured volume flow rate should be corrected to compensate for the boiloff gas remaining in the measuring vessel, occupying the volume initially occupied by liquid, but which never gets measured. This is usually considered negligible for nitrogen, but amounts to approximately a 2% error for hydrogen where total volume flow measurements are obtained over relatively long time increments using a wet test-type flowmeter.