



Standard Test Method for Unsteady-State Heat Transfer Evaluation of Flame Resistant Materials for Clothing with Burn Injury Prediction¹

This standard is issued under the fixed designation F2703; 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 measures the non-steady state heat transfer through flame resistant materials for clothing subjected to a combined convective and radiant heat exposure.

1.1.1 This test method is not applicable to materials that are not flame resistant.

NOTE 1—The determination of a material's flame resistance shall be made prior to testing and done in accordance with the applicable performance or specification standard, or both, for the material's end-use.

1.1.2 This test method accounts for the thermal energy contained in an exposed test specimen after the standardized combined convective and radiant heat exposure has ceased and is used to estimate performance to a predicted second-degree skin burn injury.

1.2 This test method is used to measure and describe the response of materials, products, or assemblies to heat under controlled conditions, but does not by itself incorporate all factors required for fire hazard or fire risk assessment of the materials, products, or assemblies under actual fire conditions.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound or other units that are commonly used for thermal testing.

1.4 *This standard does not purport to address 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:²

D123 Terminology Relating to Textiles

D1776 Practice for Conditioning and Testing Textiles

D1777 Test Method for Thickness of Textile Materials

D3776 Test Methods for Mass Per Unit Area (Weight) of Fabric

E457 Test Method for Measuring Heat-Transfer Rate Using a Thermal Capacitance (Slug) Calorimeter

F1494 Terminology Relating to Protective Clothing

3. Terminology

3.1 Definitions:

3.1.1 *breakopen, n*—in testing thermal protective materials, a material response evidenced by the formation of a hole in the test specimen during the thermal exposure that may result in the exposure energy in direct contact with the heat sensor.

3.1.1.1 *Discussion*—The specimen is considered to exhibit breakopen when a hole is produced as a result of the thermal exposure that is at least 3.2 cm² (0.5 in.²) in area or at least 2.5 cm (1.0 in.) in any dimension. Single threads across the opening or hole do not reduce the size of the hole for the purposes of this test method.

3.1.2 *charring, n*—the formation of a carbonaceous residue as the result of pyrolysis or incomplete combustion.

3.1.3 *dripping, n*—a material response evidenced by flowing of the polymer.

3.1.4 *embrittlement, n*—the formation of a brittle residue as a result of pyrolysis or incomplete combustion.

3.1.5 *heat flux, n*—the thermal intensity indicated by the amount of energy transmitted divided by area and time; kW/m² (cal/cm²·s).

3.1.6 *ignition, n*—the initiation of combustion.

3.1.7 *melting, n*—a material response evidenced by softening of the polymer.

3.1.8 *unsteady state heat transfer value, n*—in testing of thermal protective materials, a quantity expressed as the time-dependent difference between the incident and exiting thermal energy values normal to and across two defined parallel surfaces of an exposed thermal insulative material.

3.1.9 *thermal performance estimate (TPE), n*—in testing of thermal protective materials, the cumulative amount of energy identified by the intersection of a measured time-dependent

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

heat transfer response through a subject material to a time-dependent, empirical predicted second-degree skin burn injury performance curve³, expressed as a rating or value; J/cm² (cal/cm²).

3.1.10 *response to heat exposure, n*—in testing the resistance to heat transfer of thermal protective materials, the observable response of the material to the energy exposure as indicated by break-open, melting, dripping, charring, embrittlement, shrinkage, sticking, and ignition.

3.1.11 *second-degree burn injury, n*—in testing of thermal protective materials, reversible burn damage at the epidermis/dermis interface in human tissue.

3.1.12 *shrinkage, n*—a decrease in one or more dimensions of an object or material.

3.1.13 *sticking, n*—a material response evidenced by softening and adherence of the material to the surface of itself or another material.

3.1.14 *sample test suite, n*—any number of test specimens used to derive a single thermal performance estimate value.

3.1.14.1 *Discussion*—the determination of a single thermal performance estimate value requires exposing a number of specimens under varying exposure conditions so that the thermal energy stored in the sample after the heat source is removed is considered and accounted for when determining performance against a burn injury prediction.

3.1.15 For the definitions of protective clothing terms used in this method, refer to Terminology F1494, and for other textile terms used in this method, refer to Terminology D123.

4. Summary of Test Method

4.1 A horizontally positioned test specimen is exposed to a combined convective and radiant heat source with an exposure heat flux of $84 \pm 2 \text{ kW/m}^2$ ($2 \pm 0.05 \text{ cal/cm}^2\text{s}$).

NOTE 2—Other exposure heat flux values are allowed, however different exposure conditions have the potential to produce different results. The test facility shall verify the stability of other exposure levels over the material's exposure time interval (used to determine the thermal performance estimate value) and include this in the test results report.

4.2 The unsteady-state transfer of heat through the test specimen is measured using a copper slug calorimeter. The change in temperature versus time is used, along with the known thermo-physical properties of copper, to determine the respective thermal energy passed through the test specimen.

4.3 A Thermal Performance Estimate value of the test specimen is determined iteratively as the intersection of the time-dependent cumulative heat response as measured by the calorimeter to a time-dependent, empirical predicted second-degree skin burn injury performance curve identified in 10.4.1.5, Eq 1).

4.4 Observations of the thermal response of the specimen resulting from the exposure are optionally reported.

³ Derived from: Stoll, A.M. and Chianta, M.A., "Method and Rating System for Evaluations of Thermal Protection", Aerospace Medicine, Vol 40, 1969, pp. 1232-1238 and Stoll, A.M. and Chianta, M.A., "Heat Transfer through Fabrics as Related to Thermal Injury", Transactions – New York Academy of Sciences, Vol 33 (7), Nov. 1971, pp. 649-670.

5. Significance and Use

5.1 This test method is intended for the determination of a thermal performance estimate value of a material, a combination of materials, or a comparison of different materials used in flame resistant clothing for workers exposed to combined convective and radiant thermal hazards.

5.2 This test method evaluates a material's heat transfer properties when exposed to a heat exposure at a constant value and specific duration. Air movement at the face of the specimen and around the calorimeter can affect the measured heat transferred due to forced convective heat losses. Minimizing air movement around the specimen and test apparatus will aid in the repeatability of the results.

5.3 This test method accounts for the thermal energy stored in the exposed test specimen after the heat exposure has ceased. Higher values of Thermal Performance Estimate ratings determined in this test associate to higher values of thermal (convective and radiative) energy protection against a predicted skin burn injury.

5.4 This test method maintains the specimen in a static, horizontal position and does not involve movement except that resulting from the exposure.

5.5 This test method specifies a standardized $84 \pm 2 \text{ kW/m}^2$ ($2 \pm 0.05 \text{ cal/cm}^2\text{s}$) exposure condition. Different exposure conditions have the potential to produce different results. Other exposure conditions representative of the expected hazard are allowed but shall be reported with the results along with a determination of the exposure energy level stability.

5.6 This test method contains optional provisions for conducting certification testing against a prescribed Thermal Performance Estimate value.

6. Apparatus and Materials

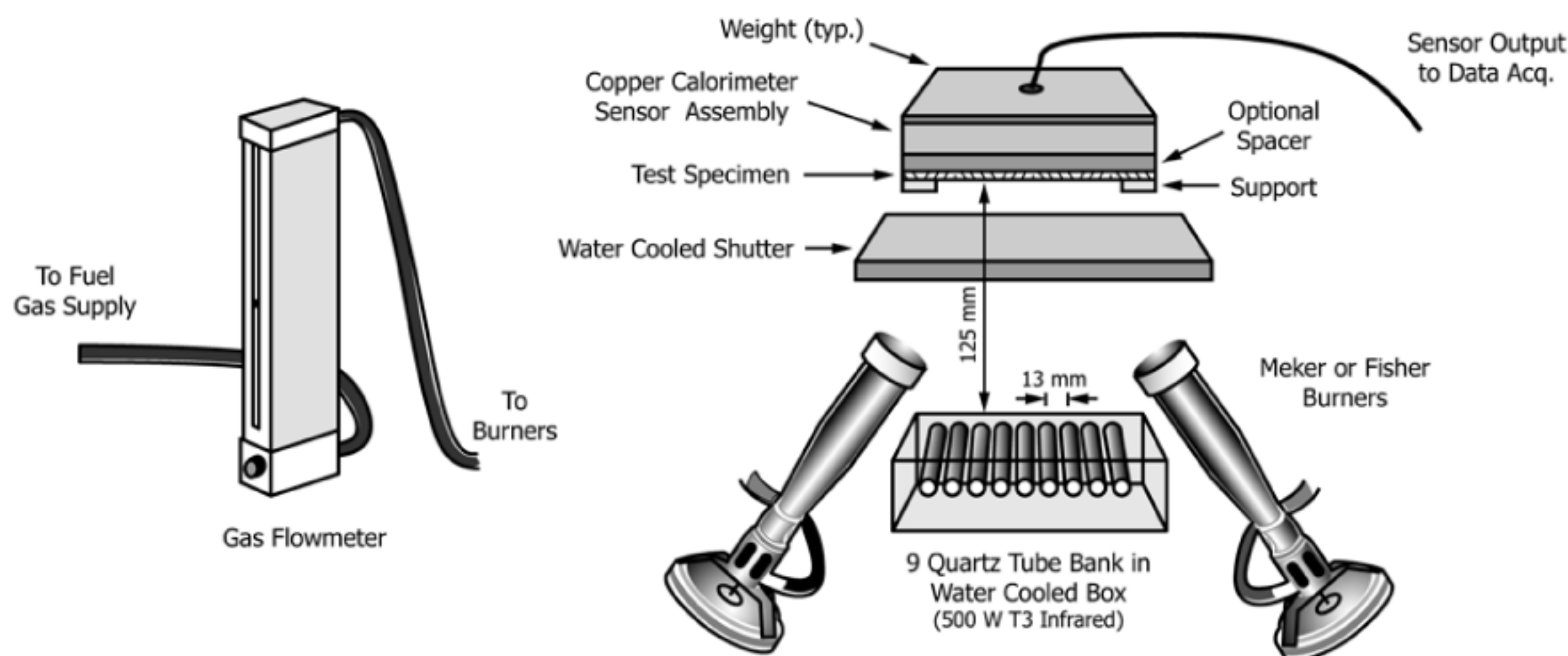
6.1 *General Arrangement*—The measurement apparatus configuration consists of a combined convective and radiant energy heat source, a water cooled shutter for exposure control, a specimen and sensor support structure, a specimen holder assembly, a copper calorimeter sensor assembly, and a data acquisition/analysis system. Automation of the apparatus for execution of the measurement procedure is allowed. The general arrangement of the test apparatus configuration is shown in Fig. 1.

6.2 *Gas Supply*—Propane (commercial grade or better) or Methane (technical grade or better).

6.3 *Gas Flowmeter*—Any gas flowmeter or rotometer with range to give a flow equivalent of at least 6 L (0.21 ft³)/min air at standard conditions.

6.4 Thermal Energy Source

6.4.1 Two each, Meker or Fisher burners jetted for the selected fuel gas (propane or methane) with a 38 mm (1.5 in.) diameter top arranged so that the bodies (top section) do not obstruct the quartz lamps and their flame profiles overlap. Dimension tolerances are $\pm 5 \%$.



NOTE 1—Note the exposure heat source incorporates two Meker burners and nine quartz infrared lamps

FIG. 1 Apparatus used to Measure Heat Transfer Performance of Textile Materials

6.4.2 Nine 500W T3 translucent quartz infrared lamps⁴, connected to a variable electrical power controller, arranged as a linear array with 13 ± 0.5 mm center-to-center spacing set 125 ± 10 mm from the specimen surface.

6.4.2.1 Use of a water-cooled housing for the quartz infrared lamp bank is recommended. This helps to avoid heating adjacent mechanical components and to shield the operator from the radiant energy.

6.5 Thermal Sensor

6.5.1 The transmitted heat sensor is a 4 ± 0.05 cm diameter circular copper slug calorimeter⁵ constructed from electrical grade copper with a mass of 18 ± 0.05 g (prior to drilling) with a single ANSI type J (Fe/Cu-Ni) or ANSI type K (Ni-Cr/Ni-Al) thermocouple wire bead (0.254 mm wire diameter or finer—equivalent to 30 AWG) installed as identified in 6.5.2 and shown in Fig. 2. The sensor holder shall be constructed from non-conductive heat resistant material with a thermal conductivity value of ≤ 0.15 W/m•K, high temperature stability, and resistance to thermal shock. The board shall be nominally 1.3 cm (0.5 in.) or greater in thickness. The sensor is held into the recess of the board using three straight pins, trimmed to a nominal length of 5 mm, by placing them equidistant around the edge of the sensor so that the heads of the pins hold the sensor flush to the surface.

6.5.1.1 Paint the exposed surface of the copper slug calorimeter with a thin coating of a flat black high temperature spray paint with an absorptivity of 0.9 or greater⁶. The painted sensor must be dried and cured, in accordance with the manufacturers instructions, before use and present a uniformly applied coating (no visual thick spots or surface irregularities). In the absence of manufacturer's instructions, an external heat source, for example, an external heat lamp, shall be used to

completely drive off any remaining organic carriers in a freshly painted surface before use.

NOTE 3—Emissivity of painted calorimeters is discussed in the ASTM Report, "ASTM Research Program on Electric Arc Test Method Development to Evaluate Protective Clothing Fabric; ASTM F18.65.01 Testing Group Report on Arc Testing Analysis of the F1959 Standard Test Method—Phase 1"⁷

6.5.2 The thermocouple wire bead is installed in the calorimeter as shown in Fig. 2.

6.5.2.1 The thermocouple wire bead shall be bonded to the copper disk either mechanically or by using high melting point (HMP) solder.

(1) A mechanical bond shall be produced by mechanically deforming the copper disk material (utilizing a copper filling slug as shown in Fig. 2) around the thermocouple bead.

(2) A solder bond shall be produced by using a suitable HMP solder with a melting temperature $>280^\circ\text{C}$.

NOTE 4—HMP solders consisting of 5 %Sb-95 %Pb ($\sim 307^\circ\text{C}$ melting point) and 5 %Sb-93.5 %Pb-1.5 %Ag ($\sim 300^\circ\text{C}$ melting point) have been found to be suitable. The 280°C temperature minimum identified above corresponds to the point where melting of the solder bond would be experienced with an ~ 17 second exposure of an 84 kW/m^2 heat flux to a prepared copper calorimeter with a surface area of 12.57 cm^2 and a mass of 18.0 g. A careful soldering technique is required to avoid "cold" solder joints (where the solder has not formed a suitable bond of the thermocouple to the copper disk).

6.5.3 Weight the sensor board assembly so that the total mass is 1.0 ± 0.01 kg and the downward force exhibited by the copper slug sensor surface is uniform.

NOTE 5—Any system of weighting that provides a uniformly weighted sensor is allowed. An auxiliary stainless steel plate affixed to or individual weights placed at the top of the sensor assembly, or both have been found to be effective.

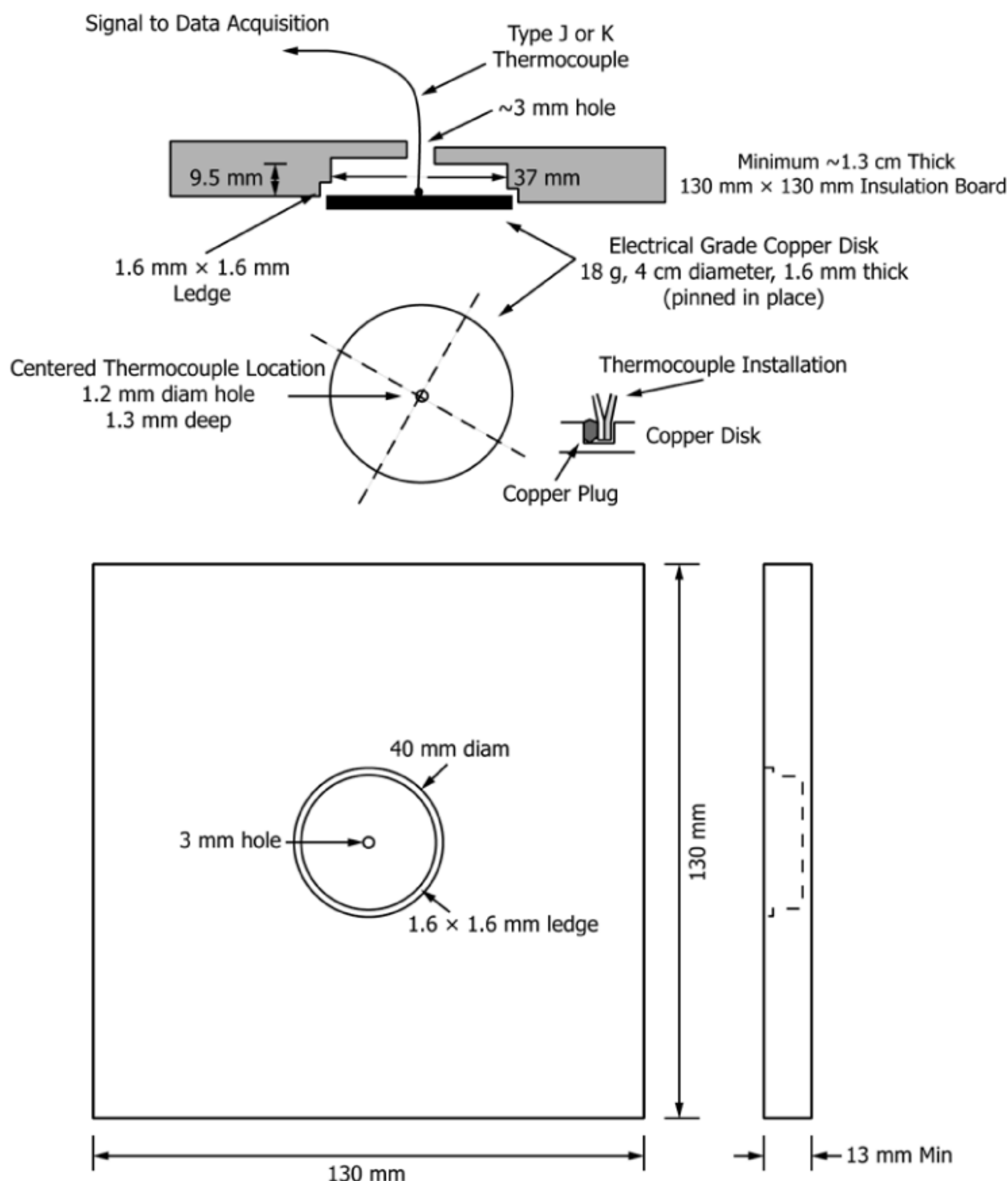
6.6 Data Acquisition/Analysis System—A data acquisition/analysis system is required that is capable of recording the

⁴ A500 Watt T3 120VAC quartz infrared heat lamp, product number 21651-1 from Philips Lighting Company has been used successfully in this application.

⁵ See Test Method E457 for information regarding slug calorimeters.

⁶ Zynolyte #635 from Aervoe Industries has been found suitable. Zynolyte is a registered trademark of the Glidden Company.

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:F18-1001.



NOTE 1—Secure sensor into supporting insulation board with three sewing pins cut to a nominal 5 mm. All dimensional tolerances are $\pm 1\%$.

FIG. 2 Copper Calorimeter Sensor Detail

calorimeter temperature response, calculating the resulting thermal energy, and determining the test endpoint by comparing the time-dependent thermal energy transfer reading to an empirical performance curve.

6.6.1 The data acquisition component shall have a minimum sampling rate of four samples per second for temperatures to 250°C with a minimum resolution of 0.1°C and an accuracy of $\pm 0.75^\circ\text{C}$. It must be capable of making cold junction corrections and converting the millivolt signals from either the type J or K thermocouple to temperature (see NIST Monograph 175 or ASTM MNL 12⁸ *Manual on the Use of Thermocouples in Temperature Measurement*).

6.7 *Solvents*, alcohol or petroleum solvent for cleaning the copper slug calorimeter.

6.8 *Paint*, flat-black, spray type with an absorptivity value > 0.90 .

6.9 *Specimen Holder Assembly*—See Fig. 3. Three complete assemblies are desirable for testing efficiency. Alteration is allowed to provide for mechanically restraining a specimen in the holder (see 10.3.2.1).

NOTE 6—The upper specimen mounting plate is designed so that the copper calorimeter assembly fits into the center cutout. An optional spacer component is also designed to fit into the center cutout with the copper calorimeter positioned on top of it. Tolerances for all dimensions are $\pm 1\%$ to accommodate these arrangement requirements.

6.10 *Shutter*—A manual or computer-controlled shutter is used to block the heat flux from the burner (placed between the specimen holder and the burner). Water-cooling is recommended to minimize radiant heat transfer to other equipment components and to prevent thermal damage to the shutter itself.

⁸ Available from ASTM Headquarters.

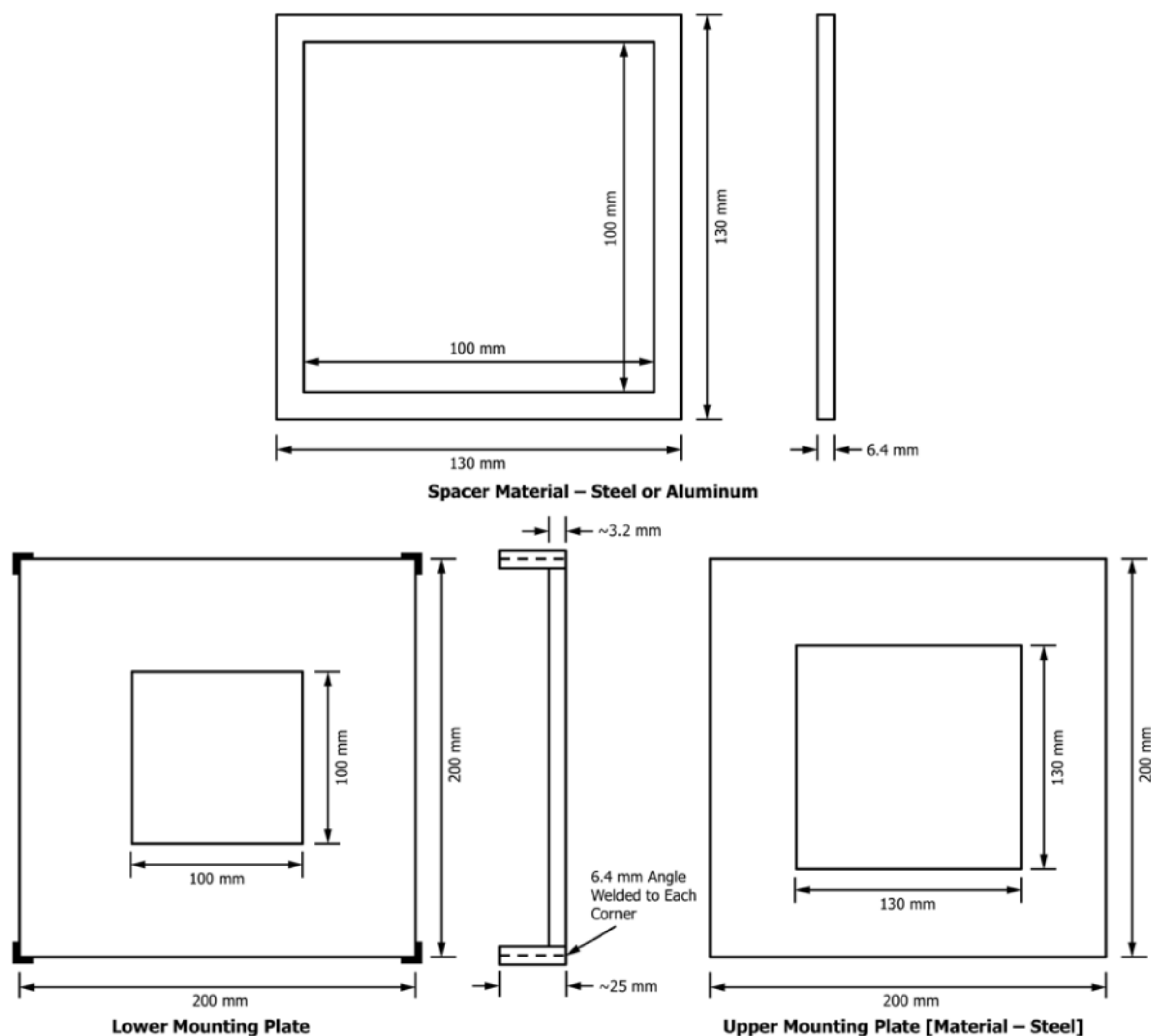


FIG. 3 Details of Specimen Holder Construction, Specimen Holder Parts

NOTE 7—Opening and closing times of the shutter are a source of measurement variability. Accounting for these times, either manually or via computer control in the exposure duration has been shown to improve measurement precision.

7. Hazards

7.1 Perform the test in an appropriate exhaust hood that is designed to contain and carry away combustion products, smoke, and fumes. Shield the apparatus or turn off the hood while running the test; turn the hood on to clear the fumes. Maintain an adequate separation between the burner and combustible materials.

7.2 The specimen holder and calorimeter assembly become heated during testing. Use protective gloves when handling these hot objects.

7.3 Use care when the specimen ignites or releases combustible gases. Remove the burner using gloves and allow the sample to burn out, or smother it with a flat plate if necessary.

7.4 Refer to manufacturer's Material Safety Data Sheets (MSDS) for information on handling, use, storage, and disposal of materials used in this test method.

7.5 Refer to local codes for compliance on the installation and use of the selected fuel gas (propane or methane).

8. Sampling and Specimen Preparation

8.1 *Laboratory Sample*—Select a minimum of a 1.0 m² (1.2 yd²) sample size from the material to be tested. Individual test specimens will be produced from this sample.

8.2 Laundering of Laboratory Sample:

8.2.1 For specimens submitted without explicit test laundering specifications, launder the laboratory sample for one wash and dry cycle prior to conditioning. Use laundry conditions of AATCC Test Method 135, (1, V, A, i).

8.2.1.1 Stitching the edges of the laboratory sample is allowed to minimize unraveling of the sample material.

8.2.1.2 Restoring test specimens to a flat condition by pressing is allowed.

8.2.1.3 If an alternative laundry procedure is employed, report the procedure used.

8.2.2 For those materials that require cleaning other than laundering, follow the manufacturer's recommended practice

using one cleaning cycle followed by drying and note the procedure used in the test report.

8.2.3 Record the procedure used in the test report for materials that are submitted with explicit laundering instructions.

8.2.4 Materials designated by the manufacturer not to be laundered or cleaned shall be tested as received.

8.3 *Test Specimens*—Cut the required test specimens from each swatch in the laboratory sample. Make each test specimen 150 by 150 ± 5 mm (6 by 6 ± 3/16 in.) with (a) two of the sides of the specimen parallel with the warp yarns in the woven material samples; (b) the wales in knit material samples; or (c) the length of the material in batts or nonwovens. Do not cut samples closer than 10 % of the material width from the edge; arrange the specimens diagonally across the sample swatch so as to obtain a representative sample of all yarns present.

8.3.1 A minimum of five sample suites is required for testing. The number of specimens in each suite will depend on the measurement response.

NOTE 8—Experience has shown that the first sample suite typically requires five to seven test specimens (especially if no prior knowledge of the material's response is known), the remaining four suites will on average require two to four test specimens each.

8.3.2 If the laboratory sample edges have been stitched to reduce unraveling (see 8.2.1.1), test specimens shall be cut so they do not incorporate the stitching material.

8.3.3 Three independent test specimens from those identified above are required for determining average thickness and average surface density (see 8.5 and 8.6).

8.4 *Conditioning*—Condition each test specimen for at least 24 h at 21 ± 2°C (70 ± 5°F) and 65 ± 5 % relative humidity. The specimens shall be tested within 30 min of removal from the conditioning area.

8.4.1 If any specimens removed from conditioning cannot be tested within 30 min, return them to the conditioning area or seal them in polyethylene bags (or other material with low water vapor permeability) until immediately prior to testing.

8.4.2 Bagged specimens have a four hour storage limit and are required to be tested within 20 min after removal from the bag.

8.4.3 Bagged specimens that exceed the four hour storage limit shall be removed from their bag and reconditioned in accordance with 8.4 prior to testing.

8.5 *Determination of Test Specimens Average Thickness*—Determine the three specimens' average thickness identified in 8.3.3 following ASTM Standard Test Method D1777. Save these specimens for determining average surface density.

8.6 *Determination of Test Specimens Average Surface Density*—Following the average thickness determination, use the same three specimens to establish an average surface density (mass divided by surface area) following ASTM Standard Test Method D3776.

9. Preparation, Calibration, and Maintenance of Apparatus

9.1 Remove the sensor assembly and any specimens from the specimen holder and place the apparatus in its measurement

position (sample holder directly over the heat source). Position the two Meker or Fisher burners so that the center of each burner head surface is separated by 125 ± 10 mm, located 65 ± 10 mm beneath the specimen holder assembly opening, and subtending an approximate 45-degree angle from the vertical so that the resulting flames converge at a point immediately beneath the specimen.

9.2 *Heat Flux Calibration*—Calibrating the dual burner/quartz lamp heat source heat flux value is an iterative process that begins with the quartz infrared lamp assembly. After the lamp assembly heat flux is fixed, the burners are adjusted to obtain an 84 ± 2 kW/m² (2.0 ± 0.05 cal/cm² s) value for testing. Several calibration passes of both heat source components are typically required to establish the standard value for testing within the specifications described below.

9.2.1 Set the output of the quartz infrared lamp assembly after a minimum 15 min warm-up period to 13 ± 4 kW/m² (0.3 ± 0.1 cal/cm² s), as measured by an independent NIST traceable Schmidt-Boelter or Gardon type radiant heat flux sensor, positioned in the same geometry as the copper calorimeter sensor in the apparatus, using the lamp's variable power control.

NOTE 9—Fixing the NIST traceable Schmidt-Boelter or Gardon type radiant heat flux sensor into an unused sensor supporting insulation board (Fig. 2) has proven effective in calibration. Also note that the use of two properly adjusted Meker or Fisher burners and a quartz lamp bank (heat flux output set to 13 kW/m²) establishes an approximately 50 % radiant, 50 % convective heat flux at 84 kW/m² for testing.

9.2.2 *Burner Gas Supply*—Reduce the pressure on the gas supply to about 55 kPa (8 psig) to allow for proper flame adjustment. Remove the Schmidt-Boelter or Gardon type radiant heat flux sensor from the specimen holder (calibration of the quartz lamp assembly is complete).

9.2.3 Leave the calibrated quartz lamp bank on and start the two burners at a low gas flow rate (low setting on the gas flowmeter/rotometer). Adjust the burner needle valves so that the flames from each burner converge just below the center of the specimen holder (hottest portion of the flames). Adjust the combustion air control at the base of each burner so that the inner flame profile on the burner grids has clearly defined stable blue tips and the larger converging diffuse flames are blue.

9.2.4 Once the flame geometry in 9.2.3 is established, the heat flux calibration is completed by increasing or decreasing the gas flow to the burners using the flowmeter/rotometer. Do not adjust the quartz lamp assembly once it has been calibrated. Minor burner needle valve and air flow adjustments are allowed as required to maintain the converged flame profile characteristics.

9.2.5 Verify that the copper calorimeter sensor is at room temperature. Ensure the sensor has a clean, black surface without any accumulation of deposits. Otherwise, recondition the sensor surface as described in 9.3.2. Calibration shall not proceed until the sensor temperature has stabilized (less than 1°C temperature change for a 1 min duration).

9.2.6 With the heat source active, start the data acquisition system then place the sensor onto the specimen holder.

9.2.7 Expose the copper calorimeter to the heat source for at least 10 s.

9.2.8 Stop the data acquisition system and remove the sensor from the holder, placing it away from the apparatus where it is allowed to cool to room temperature.

NOTE 10—Use protective gloves when handling the hot copper calorimeter sensor.

NOTE 11—Using the shutter to control the heat flux calibration exposure in 9.2.6 – 9.2.8 is allowed but not required

9.2.9 Calculate the average exposure heat flux value using a sampling interval that starts with the temperature measured at time = 0 (data sample taken just as the sensor is placed onto the sample holder) and ends with the temperature measured at exposure time = 10 s using the computational method identified in 11.1 (Sensor response). This value is the measured heat flux.

9.2.10 If the heat flux value determined in 9.2.9 is within the specifications of $84 \pm 2 \text{ kW/m}^2$ ($2.0 \pm 0.05 \text{ cal/cm}^2\text{s}$), the system is considered calibrated. The actual measured value shall be recorded as the incident heat flux value and shall be used for the determination of the Thermal Performance Estimate value in 10.4. If the heat flux value is outside the specifications, adjust the flowmeter / rotometer in the direction required and repeat the calibration process (see 9.2.5 – 9.2.9).

9.2.11 When the correct heat flux is achieved, note the flowmeter / rotometer reading (as well as all other settings for the specific apparatus configuration) as a guide for subsequent adjustments.

9.3 Sensor Care:

9.3.1 *Initial Temperature*—Cool the sensor after an exposure with a jet of air (or contact with a cold surface) to room temperature, approximately 21°C (70°F), prior to positioning the sensor onto the test specimen holder. A measurement shall not proceed until the sensor temperature has stabilized (less than 1°C temperature change for a 1 min duration).

9.3.2 *Surface Reconditioning*—Wipe the sensor face with a nonabrasive material immediately after each exposure, while hot, to remove any decomposition products that condense on the sensor since these could be a source of error. If a deposit collects and appears to be irregular or thicker than a thin layer of paint, the sensor surface requires reconditioning. Carefully clean the cooled sensor with solvent, making certain there is no ignition source nearby. If bare copper is showing on the sensor surface, completely clean it to bare copper (remove any remaining paint on the surface) and repaint the copper sensor with a thin layer of flat black high temperature spray paint identified in 6.5.1.1. Repeat the calibration process (see 9.2.5 – 9.2.9) with the resurfaced sensor before continuing.

9.4 *Specimen Holder Care*—Use dry specimen holders at ambient temperature for test runs. Alternate with several sets of holders to permit cooling between runs, or force cool with air or water. Clean the holder with a non-aqueous solvent if it becomes coated with tar, soot, or other decomposition products.

10. Procedure

10.1 A minimum of five sample test suites is required for determination of a Thermal Performance Estimate value. If

additional specimen suites are taken from the laboratory sample and exposed, they shall be included in the determination of the thermal resistance performance rating. Follow 10.6 for optional certification testing.

10.1.1 *Sample Test Suite*—The determination of a single sample test suite Thermal Performance Estimate value requires multiple sample specimens and an iterative exposure technique.

10.2 *Calibrate the heat source*—Calibrate the system as described in 9.1 and 9.2. Then carefully move the specimen holder assembly and burner away from each other to allow setting up the specimens and sensor in the apparatus for exposure.

10.3 *Specimen Mounting*—Single layer specimens are mounted either restrained, to restrict heat shrinkage, or relaxed, to permit heat shrinkage. Choose restrained mounting to evaluate barrier performance such as break-open resistance. Choose relaxed mounting for material shrinkage during exposure. Multiple-layer samples are tested relaxed with the sensor in contact with the back surface of the specimen, unless otherwise specified.

10.3.1 *Optional Spacer*—The optional 6.4 mm ($\frac{1}{4}$ in.) spacer, if used, is placed between the sensor assembly and the back surface of the specimen. See Fig. 1 for a graphical representation of the appropriate arrangement of the specimen holder (with specimen), spacer, and sensor assembly.

10.3.2 *Restrained Single Layer*—Center the specimen on the lower mounting plate with the surface that will be worn next to the skin facing up and secure all four edges with pressure-sensitive tape of at least 12.7 mm (0.5 in.) width. Attach one edge of the specimen to the plate and then attach the opposite edge of the specimen, using slight tension to remove any sags or wrinkles. Do not pull enough to remove weave crimp or distort a knit fabric or nonwoven structure. Similarly, attach the other two sides with slight tension. The securing tapes will then contact the upper or inside face of the fabric. Place the upper mounting plate on top of the secured specimen.

10.3.2.1 A specimen holder with upper or lower, or both plate pins⁹ or other mechanical restraints is allowed for use in lieu of the pressure-sensitive tape.

10.3.3 *Relaxed Single Layer* (heat shrinkage permitted)—Center the specimen on the lower mounting plate, with the surface to be worn next to the skin facing up. Place the upper mounting plate on top of the specimen. Do not restrain with tape or other mechanical means.

10.3.4 *Multiple Layer Samples*—Place the surface of the material to be used as the outside of the garment face down on the lower mounting plate. Place the subsequent layers on top of each other in the order used in the garment, with the surface to be worn toward the skin facing up. Place the upper mounting plate on top of the layered specimen.

NOTE 12—*Multiple Layer Optional Spacer Use*. The optional spacer is typically used to simulate the average air layer between the inner surface of a worn garment and the wearer. On some multilayer systems, use of the

⁹ An example of a lower mounting plate employing pins can be found in Canadian General Standards Board CAN/CGSB-155.20-200 Workwear for Protection Against Hydrocarbon Flash Fire.

optional spacer can produce test conditions that exceed the generally accepted range of applicability of the literature derived empirical exposure reference model (see 10.4.1.5, Eq 1) used in this test method. This occurs when exposure times exceed ~60 s. The use of the spacer is not recommended for multilayer systems exceeding 60 s exposure times in this configuration.

NOTE 13—The 60 second limit is a derived value based on an extrapolation of the curve identified in the cited literature reference (see Footnote 5).

10.4 Test Exposure—Follow the procedure outlined in 10.4.1 for samples with an unknown thermal performance estimate value. Follow the procedure outlined in 10.4.2 for samples where the approximate thermal performance estimate value is known (for example, repeats of sample test suites as identified in 10.1).

10.4.1 Test Exposure of samples with unknown thermal performance estimate values—A method of successive halving is employed to determine the thermal performance estimate value.

10.4.1.1 Mount the specimen in the holder in accordance with 10.3.

10.4.1.2 Ensure that the sensor that has a clean, black surface without any accumulation of deposits otherwise recondition the sensor surface as described in 9.3.2.

10.4.1.3 Place the copper calorimeter sensor assembly onto the specimen holder plate (with or without the spacer as selected in 10.3). The black copper slug shall always be facing downward towards the back of the specimen.

10.4.1.4 Place the shutter over the calibrated heat source to block the exposure radiant and convective thermal energy. Center the combined sensor assembly/prepared specimen holder plate over the blocked heat source essentially matching the position used for calibrating the sensor. Remove the shutter to expose the specimen to the heat source and simultaneously start the data acquisition system (sensor data collection).

NOTE 14—Variations using a static sensor assembly and specimen holder (with shutter) with a movable heat source are allowed. Either sequence of events can be manually functioned or computer controlled. Data acquisition initiation starts when the shutter completely unblocks the heat source.

NOTE 15—Use protective gloves when handling the hot shutter if a manual option is used.

NOTE 16—Opening and closing times of the shutter are a source of measurement variability. Accounting for these times, either manually or via computer control in the exposure duration has been shown to improve measurement precision.

10.4.1.5 Terminate the heat exposure to the specimen holder / calorimeter assembly by inserting the heat blocking shutter and stop the data acquisition after the total accumulated thermal energy as measured by the calorimeter (see 11.1) meets/exceeds the following empirical predicted second-degree skin burn injury performance curve criteria:

$$J/cm^2 = 5.0204 \times t_i^{0.2901} \text{ (cal/cm}^2 = 1.1991 \times t_i^{0.2901}) \quad (1)$$

where t_i is the time value in seconds of the elapsed time since the initiation of the thermal exposure (shutter fully opened). Assign the measured exposure time value t_{max} equal to the time where the measured cumulative heat exposure value of the test specimen intersects the empirical performance curve of Eq 1. This represents an approximate second-degree predicted burn

injury point for the continuous heating of the sample specimen without accounting for heat remaining in the specimen.

10.4.1.6 Allow the specimen holder and calorimeter assembly to cool to room temperature before disassembling and removing the exposed specimen.

NOTE 17—Use protective gloves when handling the hot shutter and specimen/copper calorimeter assembly.

10.4.1.7 Determine the exposure time trial value for the next iterative exposure by dividing t_{max} (determined in 10.4.1.5) by two,

$$\text{trial exposure time, } t_{trial} = t_{max}/2$$

10.4.1.8 Prepare another test specimen as outlined in 10.3.

10.4.1.9 Repeat 10.4.1.2 – 10.4.1.4 to initiate another exposure.

10.4.1.10 At t_{trial} seconds, terminate the heat exposure to the specimen holder/calorimeter assembly by inserting the heat blocking shutter and separating the heat source and the specimen holder/sensor apparatus. Use care to minimize disturbing the specimen holder/calorimeter assembly during the continuing data acquisition period.

NOTE 18—Avoid uncontrolled air flows and other sources of forced convection around the exposed specimen holder/sensor apparatus during data acquisition to minimize measurement variation.

10.4.1.11 Acquire calorimeter data for at least 30 seconds after terminating the heat exposure to the specimen and until the thermal energy stored in the specimen has been released (into the calorimeter and environment). Data acquisition is terminated when the cumulative energy as measured by the sensor begins to decrease. Acquisition times greater than 30 seconds after removal are possible on heavy single and multilayer specimens.

10.4.1.12 From the measured calorimeter response, determine if a predicted second-degree burn injury occurred by comparing the time-dependent cumulative heat response to the empirical second-degree burn injury performance curve, Eq 1 (see 11.1 for determining sensor response).

(1) If a second-degree burn injury is not predicted (the measured heat response did not intersect the burn injury performance curve), determine a new exposure time value that is half way between the just completed t_{trial} value and the higher previous exposure time value (for the first time through, the higher previous exposure time value will be t_{max}). Assign t_{trial} time to this value and repeat 10.4.1.8 – 10.4.1.12.

(2) If a second-degree burn injury is predicted, determine a new exposure time value that is half way between the just completed t_{trial} value and lower previous exposure time value (for the first time through, the lower previous exposure time value will be zero). Assign t_{trial} time to this value and repeat 10.4.1.8 – 10.4.1.12.

(3) If the difference between the current t_{trial} and the previous t_{trial} is ≤ 0.5 seconds, then the thermal performance estimate value for this sample test suite is

$$\begin{aligned} \text{thermal performance estimate value, } J/cm^2 = \\ \text{current } t_{trial}, \text{ seconds} \times \text{exposure heat flux value, } kW/m^2/10 \\ (\text{thermal performance estimate value, } cal/cm^2 = \\ \text{current } t_{trial}, \text{ seconds} \times \text{exposure heat flux value, } cal/cm^2s) \end{aligned}$$

(4) Subjective information observed during testing is optionally recorded with each exposure (see [Appendix X1](#) and [Appendix X2](#)).

10.4.2 Test Exposure of samples with approximately known thermal performance estimate values—A method of successive halving is employed to determine the thermal performance estimate value.

10.4.2.1 Assign the t_{trial} value as

$$t_{\text{trial}} \text{ value, } s = 1.2 \times \text{approx thermal performance estimate value, } J/cm^2 \times 10/\text{radiant heat flux, } kW/m^2$$

$$(t_{\text{trial}} \text{ value, } s = 1.2 \times \text{approx thermal performance estimate value, } cal/cm^2/\text{radiant heat flux, } cal/cm^2s)$$

and a previous t_{trial} value as

$$\text{previous } t_{\text{trial}} \text{ value, } s = 0.8 \times \text{approx thermal performance estimate value, } J/cm^2 \times 10/\text{radiant heat flux, } kW/m^2$$

$$(\text{previous } t_{\text{trial}} \text{ value, } s = 0.8 \times \text{approx thermal performance estimate value, } cal/cm^2/\text{radiant heat flux, } cal/cm^2s)$$

NOTE 19—with an approximately known thermal performance estimate value, the successive halving trial range can be reduced to conserve specimens and speed the determination of a measured value for this sample suite. Narrowing the trial range to $\pm 20\%$ of the approximately known value has demonstrated measurement convergence of a sample suite's thermal performance estimate value in two to three exposure trials.

10.4.2.2 Prepare a test specimen as outlined in 10.3.

10.4.2.3 Initiate an exposure following 10.4.1.2 – 10.4.1.4.

10.4.2.4 At t_{trial} seconds, terminate the thermal exposure by inserting the heat blocking shutter. Separate the specimen holder/calorimeter assembly from the heat source. Use care to minimize disturbing the specimen holder/calorimeter assembly during data acquisition.

10.4.2.5 Acquire calorimeter data for at least 30 seconds after terminating the thermal exposure and until the heat stored in the specimen has been released (into the calorimeter and environment). Data acquisition is stopped when the cumulative energy as measured by the sensor begins to decrease. Acquisition times greater than 30 seconds after removal are possible on heavy single and multi-layer specimens.

10.4.2.6 From the measured calorimeter response, determine if a predicted second-degree burn injury occurred by comparing the time-dependent cumulative heat response to the empirical second-degree burn injury performance curve, Eq 1 (see 11.1 for determining sensor response).

(1) If a second-degree burn injury is not predicted, determine a new exposure time value that is half way between the just completed t_{trial} value and the higher previous exposure time value (for the first time through, select a higher previous exposure time value as 50 % of the approximately known value, or $1.5 \times t_{\text{max}}$). Assign t_{trial} time to this value and repeat [10.4.1.2 – 10.4.1.6](#)

(2) If a second-degree burn injury is predicted, determine a new exposure time value that is half way between the just completed t_{trial} value and lower previous exposure time value (for the first time through, the lower previous exposure time value will be the previous t_{trial} value determined in [10.4.2.1](#)). Assign t_{trial} time to this value and repeat [10.4.1.2 – 10.4.1.6](#).

(3) If the difference between the current t_{trial} and the previous t_{trial} is ≤ 0.5 seconds, then the thermal performance estimate value for this sample test suite is

$$\text{thermal performance estimate value, } J/cm^2 = \text{current } t_{\text{trial}} \text{ seconds} \times \text{exposure heat flux value, } kW/m^2/10$$

$$(\text{thermal performance estimate value, } cal/cm^2 = \text{current } t_{\text{trial}} \text{ seconds} \times \text{exposure heat flux value, } cal/cm^2s)$$

(4) Subjective information observed during testing is optionally recorded with each exposure (see [Appendix X1](#) and [Appendix X2](#)).

10.5 Prepare and test enough specimens as outlined in 10.4 until five complete sample suites of values are obtained.

10.6 Optional Certification Testing Procedure—The following optional procedure is allowed for certification testing where a validation of a previously determined thermal performance estimate value is desired.

10.6.1 Certification testing requires a minimum of five individual test specimens, a defined thermal performance estimate (TPE) value, and a specification for the number of specimens that are allowed to have a predicted burn injury performance using this test method.

NOTE 20—A statistical variation about the mean thermal performance estimate value will be observed using this optional test procedure. As this test method establishes a nominal 50 % probability for predicted burn injury, a number of specimens tested at certification exposure times (calculated from the thermal performance estimate defined for certification testing using [10.6.2](#)) that match the measured TPE value will predict burn injury. A statistical analysis is recommended to establish the requirements for the number of specimens allowed to predict burn injury for certification.

10.6.2 Determine the Certification Exposure Time using the following:

$$\text{Certification Exposure Time, } s = TPE_{\text{certified}} J/cm^2 \times 10/\text{exposure heat flux value, } kW/m^2$$

$$(\text{Certification Exposure Time, } s = TPE_{\text{certified}} cal/cm^2 / \text{exposure heat flux value, } cal/cm^2s)$$

where $TPE_{\text{certified}}$ is the thermal performance estimate value required for certification and the exposure heat flux value is the calibrated value of the apparatus (from Section 9).

10.6.3 Calibrate the heat source—Calibrate the system as described in [9.1](#) and [9.2](#). Then carefully separate the specimen holder assembly and heat source to allow setting up the specimens and sensor in the apparatus for an exposure.

10.6.4 Prepare a test specimen as outlined in 10.3.

10.6.5 Initiate an exposure following 10.4.1.2 – 10.4.1.4.

10.6.6 At the Certification Exposure Time calculated in 10.6.2, terminate heating of the specimen holder/calorimeter assembly. Insert the heat blocking shutter and separate the specimen holder/sensor apparatus from the heat source so that it no longer contributes heat to the specimen. Use care not to disturb the specimen holder/calorimeter assembly during data acquisition.

10.6.7 Acquire calorimeter data for at least 30 seconds after terminating the thermal exposure and until the heat stored in the specimen has been released (into the calorimeter and environment). The data acquisition is terminated when the cumulative energy as measured by the sensor begins to

decrease. Acquisition times greater than 30 seconds after heat source removal are possible on heavy single and multilayer specimens.

10.6.8 From the measured calorimeter response, determine if a predicted second-degree burn injury occurred by comparing the time-dependent cumulative heat response to the empirical second-degree burn injury performance curve, **Eq 1** (see **11.1** for determining sensor response). Record if a predicted second-degree burn injury occurred or did not occur.

10.6.9 Repeat **10.6.4 – 10.6.8** for the remaining four test specimens.

10.6.10 If a second-degree burn injury is predicted (the measured heat response intersected the burn injury performance curve, **Eq 1**) in no more than the maximum allowed number of specimens, the sample shall be considered to meet the certification requirements for the specified thermal performance estimate value. If a second-degree burn injury is predicted in more than the allowed number of specimens, the sample shall not be considered to meet the certification requirements for the specified thermal performance estimate value.

10.6.11 Subjective information observed during testing is optionally recorded with each exposure (see **Appendix X1** and **Appendix X2**).

11. Calculation of Results

11.1 *Sensor Response*—The sensor response is determined shortly before and all during the heat exposure to the test specimen.

11.1.1 The temperature value just prior to exposing the specimen marks the sampling time initiation point, or $t = 0$ value.

11.1.2 The heat capacity of each copper slug at the initial temperature is calculated using:¹⁰

$$C_p = \frac{4.1868 \times (A + B \times t + C \times t^2 + D \times t^3 + E/t^2)}{63.546 \text{ g/mol}} \quad (2)$$

where

- t = (measured temperature °C + 273.15) / 1000
- A = 4.237312
- B = 6.715751
- C = -7.46962
- D = 3.339491
- E = 0.016398

NOTE 21—The heat capacity of copper in J/g°C at any temperature between 289 K and 1358 K is determined via **Eq 2** (Shomate Equation with coefficients from NIST)

11.1.3 The time-dependent cumulative energy values are determined from the temperatures at the beginning and end of the sampling intervals.

11.1.3.1 The copper slug heat capacity is determined at each data acquisition interval. This is done by calculating an average heat capacity for each sensor from the initial heat capacity, determined in **11.1.2**, and the measured temperature at the time interval of interest,

$$\bar{C}_p = \frac{C_p @ Temp_{initial} + C_p @ Temp_{final}}{2} \quad (3)$$

11.1.3.2 The measured cumulative energy exposure value at any exposure time duration is determined in J/cm² by using the relationship,

$$\text{Cumulative heat exposure, } Q = \frac{\text{mass} \times \bar{C}_p \times (Temp_{final} - Temp_{initial})}{\text{area}} \quad (4)$$

where

- Q = Cumulative energy detected by the calorimeter, J/cm²,
- mass = mass of the copper disk/slug (g),
- \bar{C}_p = Average heat capacity of copper during the temperature rise (J/g°C),
- $temp_{final}$ = Temperature of copper disk/slug at time interval of interest (°C),
- $temp_{initial}$ = Initial temperature of the copper disk/slug at time = 0 (°C),
- area = Area of the exposed copper disk/slug (cm²).

11.1.3.3 For a copper disk/slug that has a mass of 18.0 g and exposed area of 12.57 cm², the determination of cumulative energy to the sensor at any time interval reduces to:

$$\text{Cumulative thermal energy, } Q = 1.432 \times \bar{C}_p \times (Temp_{final} - Temp_{initial}) \text{ J/cm}^2 \quad (5)$$

NOTE 22—If a copper disk/slug with a different mass or exposed area, or both is used, the constant factor in **Eq 5** must be adjusted correspondingly. If required, the value in cal/cm² can be determined by multiplying the cumulative thermal energy in **Eq 6** by the conversion factor 1/4.1868 cal/J.

11.1.3.4 Calculating Heat Flux for Sensor Calibration

(1) Incident heat flux to the copper calorimeter can be calculated over any time interval using:

$$\text{Incident heat flux, } q = \frac{\text{mass} \times \bar{C}_p \times (Temp_{final} - Temp_{initial})}{\text{absorptivity} \times \text{area} \times (time_{final} - time_{initial})} \quad (6)$$

where the absorptivity is the value for the black paint used for the calorimeter surface (typically ~0.9).

(2) For a copper disk/slug that has a mass of 18.0 g, an exposed area of 12.57 cm², a paint absorptivity of 0.9, and a 10 second calibration sampling interval the determination of incident heat flux reduces to:

$$\text{Incident heat flux, kW/m}^2 = 1.591 \times \bar{C}_p \times (Temp_{t=10s} - Temp_{t=0s}) \quad (7)$$

NOTE 23—If a copper disk/slug with a different mass or exposed area, or both, is used, or the calibration time interval is changed from 10s the constant factor in **Eq 7** above must be adjusted correspondingly. If required, the value in cal/cm²s can be determined by multiplying the incident heat flux in kW/m² by the conversion factor 0.02389 cal m²/kW cm² s.

11.2 Determination of Thermal Performance Estimate Rating

11.2.1 *Thermal Performance Estimate Values*—Take the average of at least five sample test suite thermal performance estimate values determined in Section **10** and report this value as the specimen Thermal Performance Estimate (TPE) rating, J/cm² [cal/cm²]. Any additional sample suites tested from the laboratory sample shall be included in the averaged value.

¹⁰ **Eq 2** represents the Shomate Equation for temperature dependent heat capacity. The listed coefficients are from NIST.

TABLE 1 Single Laboratory Precision of the Test Method (TPE values in cal/cm²)

NOTE 1— s_r = repeatability standard deviation (pooled within-laboratory standard deviation)

Test	Fabric A (plain weave) 4.5 oz/yd ²	Fabric B (plain weave) 4.5 oz/yd ²	Fabric C (plain weave) 6.0 oz/yd ²	Fabric D (plain weave) 7.0 oz/yd ²
	(spaced, relaxed mounting)	(spaced, relaxed mounting)	(spaced, relaxed mounting)	(spaced, relaxed mounting)
1	7.0	8.0	8.2	5.8
2	7.0	8.0	8.2	6.2
3	7.0	8.0	8.2	6.0
Average	7.0	8.0	8.2	6.0
s_r	-	-	-	0.20
%CV	-	-	-	3.3

12. Report

12.1 State that the test has been performed as directed in Test Method F2703.

12.2 Describe the material sampled and the method of sampling used. In the material description, include:

- 12.2.1 Sample identification and Lot information.
- 12.2.2 Number and ordering of layers in the specimen.
- 12.2.3 Description of each material used to make up the specimen including type of material, construction, thickness, average surface density (basis weight), and color.
- 12.2.4 Number of wash/dry or dry cleaning cycles or specified laundry conditions

12.3 Report the following information:

- 12.3.1 Conditions of test, including:
 - 12.3.1.1 Calibrated exposure energy.
 - 12.3.1.2 Gas used
 - 12.3.1.3 Number of layers tested—single or multiple with the order of lay-up.
 - 12.3.1.4 Specimen mounting—restrained or relaxed
 - 12.3.1.5 Position of the sensor to the specimen—contact or spaced
- 12.3.2 For certification testing, report the Thermal Performance Estimate value that is being certified, the number of specimens that predicted a second degree burn injury, and if the certification requirements were met (10.6.10).
- 12.3.3 For testing other than certification:
 - 12.3.3.1 Report the thermal performance estimate values from each tested specimen suites.
 - 12.3.3.2 Report the average of the individual thermal performance estimate values from each tested specimen suites from the laboratory sample as the Thermal Performance Estimate value (11.2) of the sample.
- 12.3.4 Optional subjective observations (see Appendix X2).

13. Precision and Bias

13.1 A single operator intra-laboratory test series was performed on four different fabric types to determine method precision using the apparatus and procedure described above.

13.1.1 Four commercially available flame resistant fabrics used in thermal energy personal protective equipment were selected: a) and b) 4.5 oz/yd² plain weave fabrics from different materials and manufacturers, c) a 6.0 oz/yd² plain weave, and d) a 7.0 oz/yd² twill weave. These single layer fabrics were tested with a spacer and are identified in Table 1 as fabrics A,

B, C, and D. All fabrics were tested in the relaxed state in the sample holder assembly (unrestrained). Three separate test suites were conducted on each fabric type using the number of specimens identified in 10.1 above.

13.1.2 The results of single operator intra-laboratory precision study are shown in Table 1 in cal/cm².

13.1.3 Repeatability—Due to this test method's quantized time interval specification for determining the TPE value, an estimation of repeatability, r , has been established based on a propagation of error analysis.

13.1.3.1 The variation for the test method is estimated based on the allowed variations of heat flux and quantized exposure time resolution as shown below:

$$\left[\frac{\Delta TPE}{TPE} \right]^2 \cong \left[\frac{\Delta Q}{Q} \right]^2 + \left[\frac{\Delta Time}{Time} \right]^2 \quad (8)$$

or

$$s_{TPE}^2 \cong \Delta TPE^2 = TPE^2 \times \left[\left(\frac{\Delta Q}{Q} \right)^2 + \left(\frac{\Delta Time}{Time} \right)^2 \right] \quad (9)$$

13.1.3.2 Substitution of the allowed variations in heat flux and the quantized time interval yields:

$$\text{for TPE in J/cm}^2, s_{TPE}^2 \cong 17.64 + 0.04 \times Time^2 \quad (10)$$

$$(\text{for TPE in cal/cm}^2, s_{TPE}^2 \cong 1 + 0.0025 \times Time^2) \quad (11)$$

where,

- Q = Exposure heat flux, 84 kW/m² [2.0 cal/cm²s]
- ΔQ = 2 kW/m² [0.05 cal/cm²s]
- $Time$ = Measured exposure time, s
- $\Delta Time$ = 0.5 s (quantized time interval)
- TPE = $Q \times Time/10$, J/cm² [$Q \times Time$, cal/cm²]

13.1.3.3 A set of tabulated values of estimated repeatability, r , spanning the exposure time values of three to ten seconds are shown in Table 2 (TPE values are in cal/cm²). Based on these estimates, two single test results, obtained in the same laboratory under normal test method procedures that differ by more than this tabulated r must be considered as derived from different or nonidentical sample populations.

13.1.3.4 Estimated repeatability values for other time intervals and TPE values are directly determined using Eq 10 or Eq 11 found in 13.1.3.2.

13.1.4 Reproducibility—The reproducibility of this test method is being determined and will be available on or before December 2008.

TABLE 2 Test Method Propagation of Error Analysis for $2.0 \pm 0.05 \text{ cal/cm}^2\text{s}$ Heat Flux (TPE values in cal/cm^2)

NOTE 1— s^2 = minimum value determined from variation allowed in heat flux ($\pm 0.05 \text{ cal/cm}^2\text{s}$) and quantized time interval ($\pm 0.5 \text{ s}$) – from 13.1.3.2
 r = repeatability = $2.80 s_r$

<i>Exposure Time, s</i>	3	4	5	6	7	8	9	10
<i>TPE, cal/cm²</i>	6	8	10	12	14	16	18	20
<i>s² (variance)</i>	1.02	1.04	10.6	1.09	1.12	1.16	1.20	1.25
<i>%CV</i>	16.9	12.7	10.3	8.7	7.6	6.7	6.1	5.6
<i>r</i>	2.83	2.86	2.89	2.92	2.97	3.02	3.07	3.13

13.2 *Bias*—The value for TPE rating can only be defined in terms of a test method. Within this limitation, this test method has no known bias.

14. Keywords

14.1 apparel; protective clothing; thermal heat protection; thermal performance estimate, flame resistance

APPENDIXES

(Nonmandatory Information)

X1. SPECIMEN RESPONSE TO CONVECTIVE AND RADIATIVE ENERGY EXPOSURE

X1.1 The effect of the thermal energy exposure on the specimen observed in 10.11 can be included in the report. Observe the effect of the exposure on the test specimen, including each of the layers in a multiple layer specimen.

Describe this effect as one or more of the following: break open, charring, dripping, embrittlement, ignition, melting, shrinkage, sticking.

X2. SUBJECTIVE VISUAL EXAMINATION AND EVALUATION OF THE EXPOSED SPECIMEN

X2.1 The subjective observations on the specimen ignition during the exposure can be reported using the rating system below:

X2.1.1 *Ignition:*

- X2.1.1.1 1 = no ignition, no smoke.
- X2.1.1.2 2 = slight ignition, slight smoke.
- X2.1.1.3 3 = moderate ignition, dark smoke.
- X2.1.1.4 4 = significant ignition, thick blackish smoke.
- X2.1.1.5 5 = heavy ignition, thick blackish smoke or flames, or both.

X2.2 Except for the subjective observation on ignition, the exposed specimen can be evaluated in each of the categories as listed in 10.3 on each side of the specimen.

X2.2.1 The surface of the specimen exposed to the heat source shall be identified as the front side.

X2.2.2 The surface facing the heat sensor shall be identified as the back side.

X2.2.3 For visual examination, lay the exposed specimen parallel on a flat surface with proper illumination.

X2.3 Subjective ratings in the categories (10.3) can utilize the 1 to 5 system with 1 = best and 5 = worst behavior. The total value of the assigned ratings for each category will determine the specimen ranking.

X2.4 *Rate each specimen after exposure using the following subjective terms:*

X2.4.1 *Break open:*

- X2.4.1.1 No breakopen.
- X2.4.1.2 Breakopen characteristic (size of hole)

X2.4.2 *Melting:*

- X2.4.2.1 No melting observed
- X2.4.2.2 Melting observed

X2.4.3 *Dripping:*

- X2.4.3.1 No dripping observed
- X2.4.3.2 Dripping observed

X2.4.4 *Charring:*

- X2.4.4.1 1 = no charring.
- X2.4.4.2 2 = slight specimen scorching/discoloration.
- X2.4.4.3 3 = slight specimen charring evident.
- X2.4.4.4 4 = significant specimen chars and embrittlement.
- X2.4.4.5 5 = severe charring, specimen embrittles and has cracks or holes, or both.

X2.4.5 *Embrittlement:*

- X2.4.5.1 1 = no embrittlement.
- X2.4.5.2 2 = slight, specimen starts to harden.
- X2.4.5.3 3 = moderate, small hardened areas.
- X2.4.5.4 4 = significant, specimen completely embrittles.

X2.4.5.5 5 = heavy specimen embrittlement or cracks or holes, or both.

X2.4.6 *Shrinkage:*

X2.4.6.1 No shrinkage.

X2.4.6.2 % Observed shrinkage.

X2.4.7 *Sticking:*

X2.4.7.1 No sticking.

X2.4.7.2 Sticking observed

X2.4.8 The visual ratings of the specimen exposed can be reported using the format of Table X2.1.

TABLE X2.1 Suggested Visual Response Report Form

Categories	Front Side Rating	Back Side Rating
Break open		
Melting		
Dripping		
Charring		
Embrittlement		
Shrinkage		
Sticking		
Ignition		

X3. ITERATIVE DETERMINATION OF THERMAL PROTECTION ESTIMATE VALUE EXAMPLE

The following example provides an overview of this test method's iterative technique to determine a thermal protection estimate from a sample suite using a fictitious sample material.

X3.1 *Step 1*—when the specimen thermal performance estimate value is unknown, run the test identified in 10.4.1 – 10.4.1.12. This involves running the exposure until the sensor response crosses the “Stoll” criteria. The fictitious material response (for this example, assume an 84 kW/m² [2.0 cal/cm²s] heat flux) is shown in Fig. X3.1:

The sensor response, calculated using 11.1 (11.1.3.3) and plotted for each time step, crosses the “Stoll” criteria at a continuous exposure time of 12.5 seconds. This sets $t_{\max} = 12.5$ s (section 10.4.1.5). Note that the cumulative energy (sensor response) continues to climb quite rapidly after the heat source is removed reflecting the thermal energy still in the specimen (being delivered to the copper calorimeter surface).

X3.2 *Step 2* (10.4.1.6 – 10.4.1.12) Calculate a new trial exposure of $t_{\max}/2$ or 6 s (round to the nearest second). Expose the sample for this amount of time then insert the blocking shutter and carefully move the sensor/sample holder away from the heat source and continue to acquire sensor data for at least another 30 seconds (note that the cumulative energy begins to decrease at ~28 s). For the fictitious material, this resulted in a plotted response in Fig. X3.2.

X3.3 *Step 3* (see 10.4.1.12 *Step 1* and 10.4.1.12 *Step 2*)—from the exposure in Step 2 it is apparent from the plot (see Fig. X3.2) that the sensor response did not cross the “Stoll” criteria. So 10.4.1.12 *Step 1* applies and a new trial exposure would be calculated that is half way in between 12.5 s (previous value - t_{\max}) and 6 s (current trial exposure - $t_{\max}/2$) or 9 seconds (rounding to the second). Repeating 10.4.1.6 through 10.4.1.12, expose the sample for the new trial time of 9 s then insert the blocking shutter and carefully move the sensor/sample holder away from the heat source and continue to acquire sensor data for at least another 30 seconds. For the fictitious material, this resulted in a plotted response in Fig. X3.3.

X3.4 *Step 4* (10.4.1.12 *Step 1* and 10.4.1.12 *Step 2*)—from the exposure in Step 3 it is apparent in Fig. X3.3 that the sensor response did cross the “Stoll” criteria. So 10.4.1.12 *Step 2*

applies and a new trial exposure would be calculated that is half way in between 6 s (previous trial value) and 9 s (current trial exposure) or 8 seconds (rounding to the nearest second). Repeating 10.4.1.6 through 10.4.1.12, expose the sample for the new trial time of 8 s then insert the blocking shutter and carefully move the sensor/sample holder away from the heat source and continue to acquire sensor data for at least another 30 seconds. For the fictitious material, this resulted in a plotted response shown in Fig. X3.4.

X3.5 *Step 5* (sections 10.4.1.12 *Step 1* and 10.4.1.12 *Step 2*)—from the exposure in Step 4 it is apparent in Fig. X3.4 that the sensor response did cross the “Stoll” criteria. So 10.4.1.12 *Step 2* applies and a new trial exposure would be calculated that is half way in between 6 s (previous low trial value) and 8 s (current trial exposure) or 7 seconds. Repeating sections 10.4.1.6 through 10.4.1.12, expose the sample for the new trial time of 7 s then insert the blocking shutter and carefully move the sensor/sample holder away from the heat source and continue to acquire sensor data for at least another 30 seconds. For the fictitious material, this resulted in a plotted response that is shown in Fig. X3.5.

X3.6 *Step 6* (10.4.1.12 *Step 1* and 10.4.1.12 *Step 2*)—from the exposure in Step 5 it is apparent in Fig. X3.5 that the sensor response did not cross the “Stoll” criteria. So 10.4.1.12 *Step 1* applies and a new trial exposure would be calculated that is half way in between 8 s (previous high trial value) and 7 s (current trial exposure) or 7.5 seconds. However, note that whatever is obtained for a 7.5 s exposure, it will be 0.5 s away from the previous 7 s and the latter 8 s exposures. So now 10.4.1.12 *Step 3* applies and the thermal performance estimate value would be:

$$7.5 \text{ s} \times 84 \text{ kW/m}^2/10 = 63.0 \text{ J/cm}^2$$

$$[7.5 \text{ s} \times 2 \text{ cal/cm}^2\text{s} = 15 \text{ cal/cm}^2]$$

(One could technically run the 7.5 s exposure and then follow 10.4.1.12 *Step 3* if so inclined and the equipment was capable of resolving a 0.5 s exposure interval).

X3.7 Note that the method of “halving” converges very quickly on an answer. For the remainder of the sample suite measurements, 10.4.2 would apply. From the fictitious case above, one would select the initial starting points (new trial and

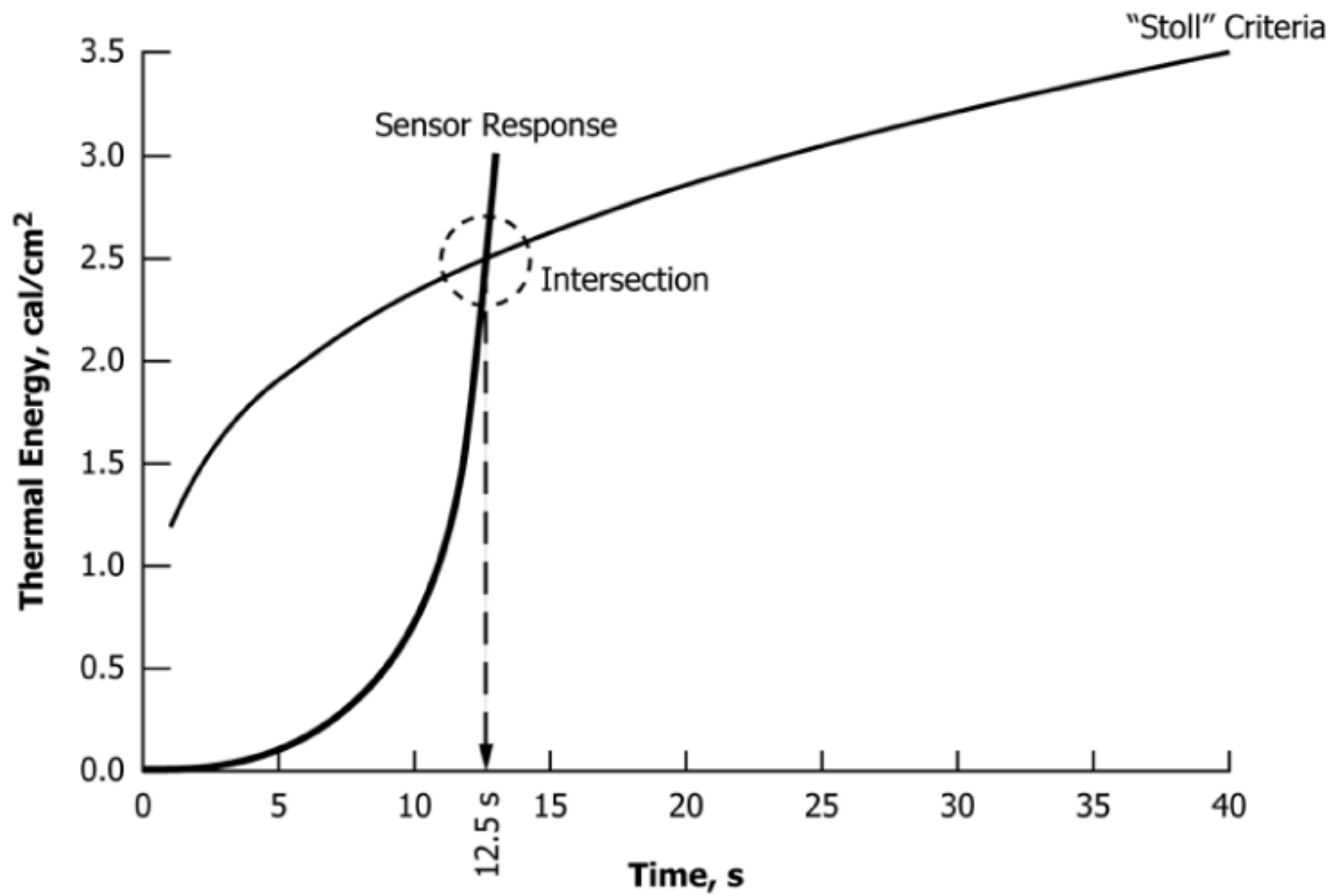


FIG. X3.1 Sensor Response to Continuous Heating Combined Convective and Radiant Heat Exposure

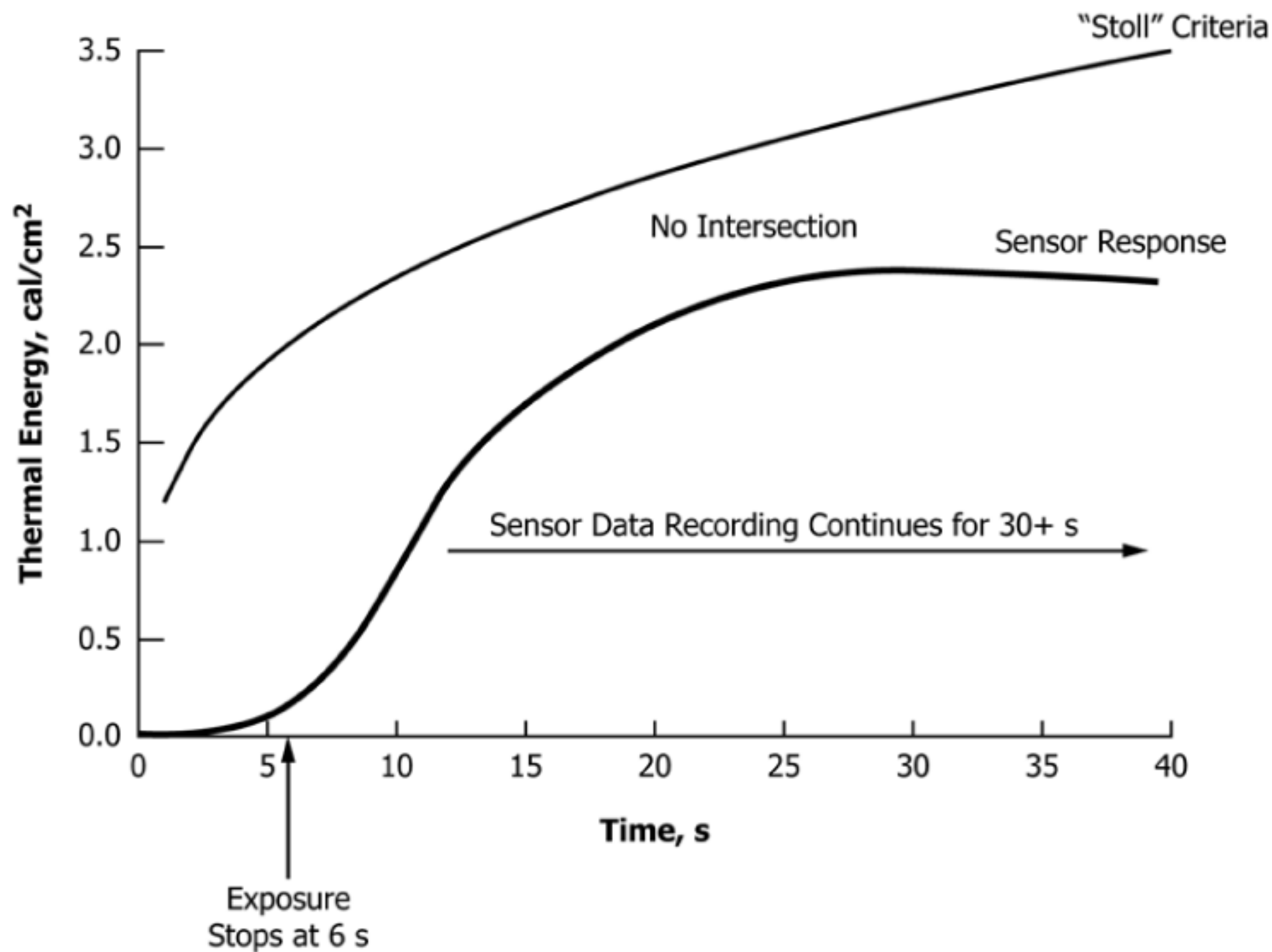


FIG. X3.2 Sensor Response to First Heating Iteration (Second Specimen)

previous value) for the remaining sample suites as (after rounding to the nearest second):

$$t_{\text{trial}} \text{ value, } s = 1.2 \times 63.0 \text{ J/cm}^2 \times 10$$

$$[1.2 \times 15 \text{ cal/cm}^2 / 2.0 \text{ cal/cm}^2 \text{ s}] = 9 \text{ s}$$

$$\text{previous } t_{\text{trial}} \text{ value, } s = 0.8 \times 63.0 \text{ J/cm}^2 \times 10$$

$$[0.8 \times 15 \text{ cal/cm}^2 / 2.0 \text{ cal/cm}^2 \text{ s}] = 6 \text{ s}$$

and repeat the iterative process (10.4.2.2 – 10.4.2.6). Experience shows that subsequent determinations converge quite rapidly – 2 to 3 specimen exposures are all that are required to get subsequent thermal performance estimate values (in this case repeating steps similar to X3.4 – X3.6 to get the values).

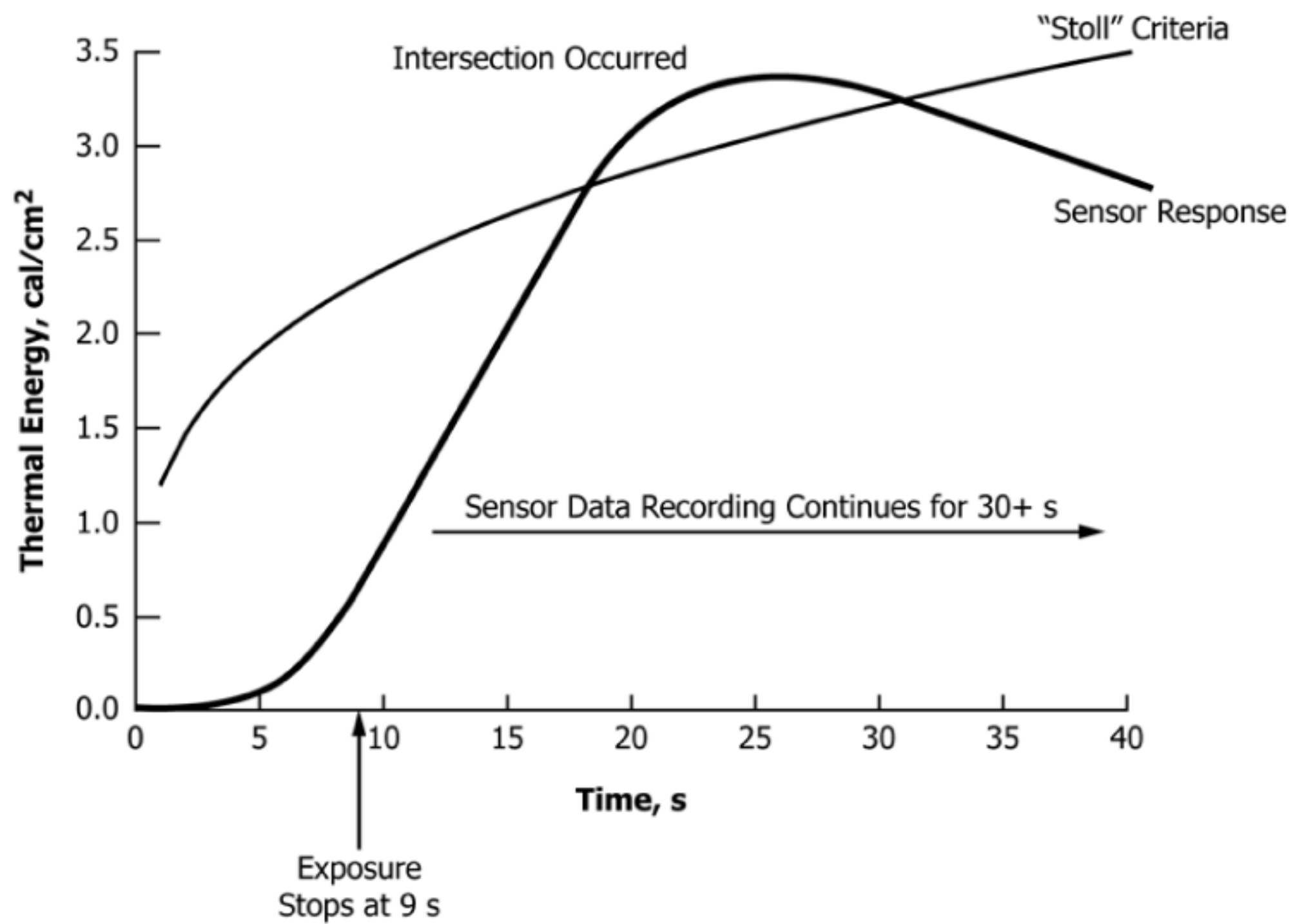


FIG. X3.3 Sensor Response to Second Heating Iteration (Third Specimen)

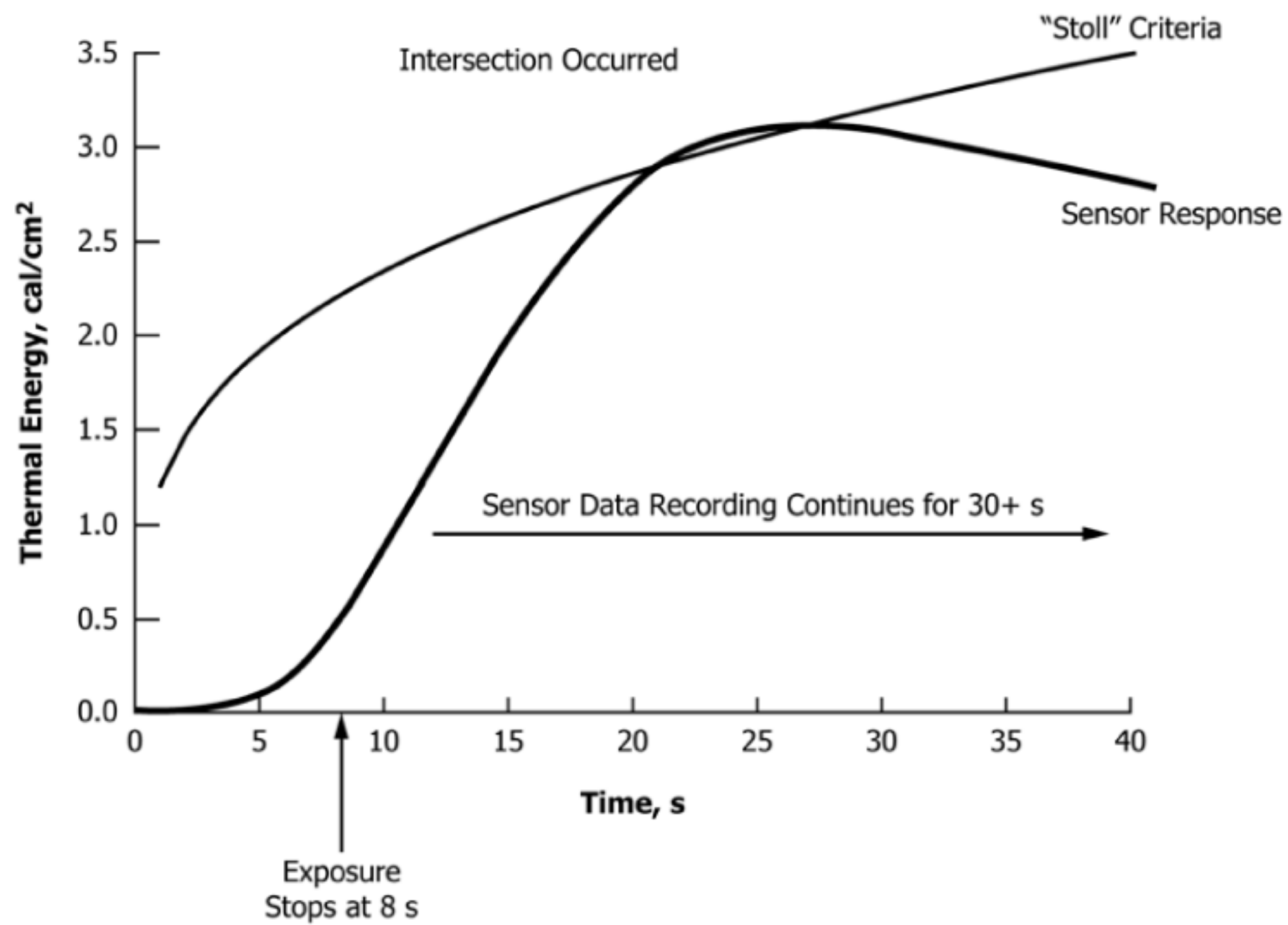


FIG. X3.4 Sensor Response to Third Heating Iteration (Fourth Specimen)

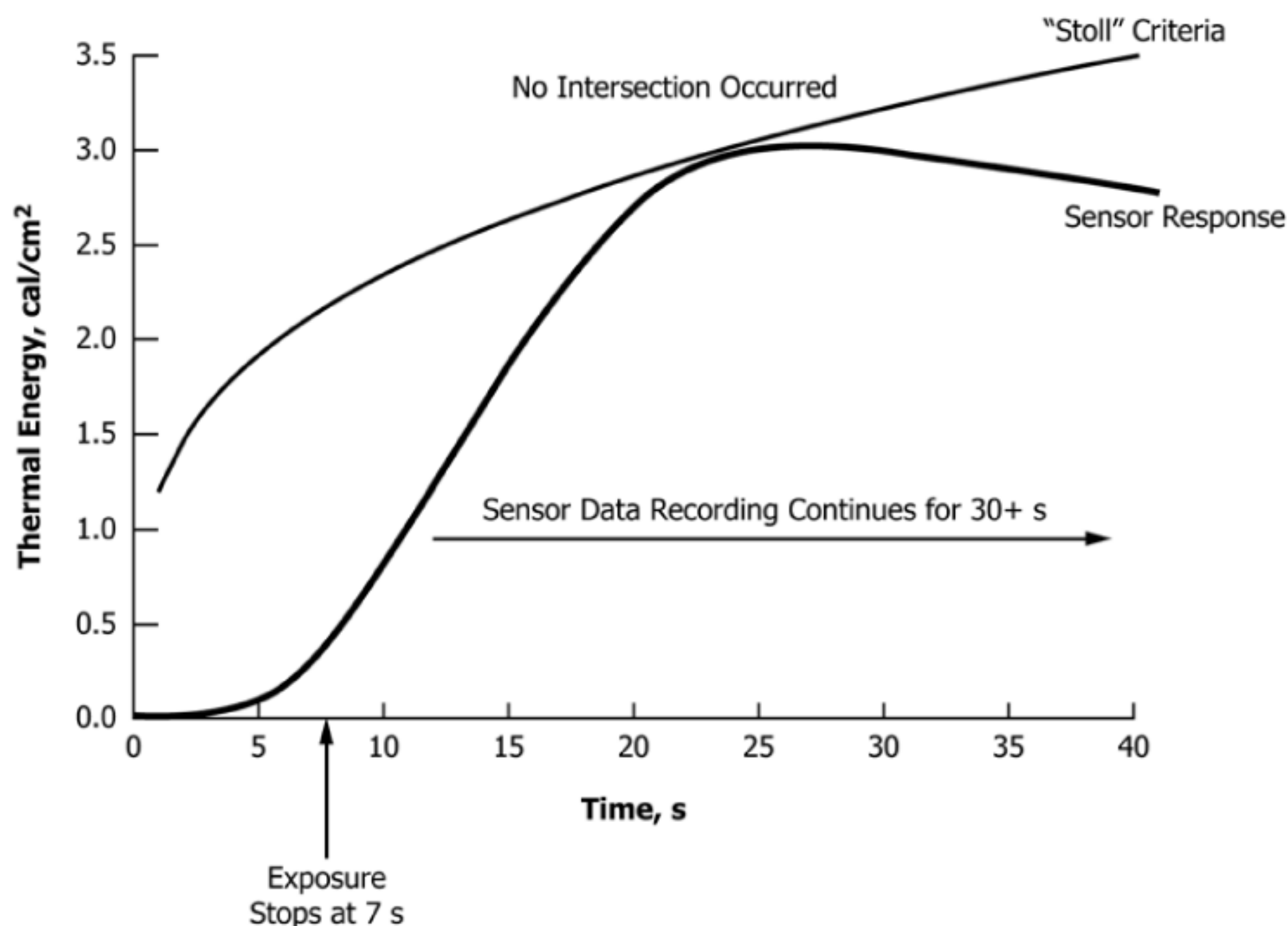


FIG. X3.5 Sensor Response to Fourth Heating Iteration (Fifth Specimen)

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