Designation: F 955 – ~~96~~03

Standard Test Method for Evaluating Heat Transfer through Materials for Protective Clothing Upon Contact with Molten Substances[1]

This standard is issued under the fixed designation F 955; 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 evaluation of materials' thermal resistance to molten substance pour by describing means of measuring heat transfer.

NOTE 1—As used in this test method, the term *molten substance* refers to the three compositions (aluminum, brass, and iron) for which the procedure was validated. The test design may be adapted for use with other substances not validated as part of the test method.

1.2 This test method is applicable to materials from which finished protective apparel articles are made.

1.3 This test method does not measure the flammability of materials, nor is it intended for use in evaluating materials exposed to any other thermal exposure exclusive of the molten substance itself (see Note 1).

1.4 This test method should be used to measure and describe the properties of materials, products, or assemblies in response to molten substance pour under controlled laboratory conditions and should not be used to describe or appraise the thermal hazard or fire risk of materials, products, or assemblies under actual conditions. However, results of this test may be used as elements of a thermal risk assessment which takes into account all the factors that are pertinent to an assessment of the thermal hazard of a particular end use.

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.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]

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[1] This test method is under the jurisdiction of ASTM Committee F~~-~~23 on Protective Clothing and is the direct responsibility of Subcommittee F23.80 on Molten Substances.
Current edition approved ~~May~~ August 10, ~~1996.~~ 2003. Published ~~July 1996.~~ October 2003. Originally ~~published as F 955 – 85.~~ approved in 1985. Last previous edition ~~F 955 – 85 (1989) ϵ1.~~ approved in 1996 as F 955 – 96.

E 457 Test Method for Measuring Heat-Transfer Rate Using a Thermal Capacitance (Slug) Calorimeter[3]
F 1494 Terminology Relating to Protective Clothing[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *break-open*—in testing thermal protective material, a response evidenced by the formation of a hole in the material which allows the molten substance to pass through the material.

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

3.1.3 *dripping*—in testing thermal protective material, a response evidenced by flowing of the fiber polymer.

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

3.1.5 *heat flux*—the thermal intensity indicated by the amount of energy transmitted per unit divided by area and per unit time time, W/m^2 (cal/cm 2 s) (watts/cm^2 s).

3.1.6 *human tissue burn tolerance*—in the testing of thermal protective materials, the amount of thermal energy predicted to cause a second-degree burn in human tissue.

3.1.7 *ignition*—the initiation of combustion.

3.1.8 *melting*—in testing thermal protective material, a response evidenced by softening of the material, resulting in a nonreversible change.

3.1.9 *response to molten substance pour*—in testing thermal protective material, the observed effect of molten substance contact on textile properties or deterioration of the material.

3.1.10 *shrinkage*—a decrease in one or more dimensions of an object or material.

3.1.11 *thermal end point*—in testing of thermal protective materials, the point of where the copper slug calorimeter sensor response on the recorder chart (heat energy measured) intersects the human tissue with a predicted skin burn tolerance criteria overlay. injury model.

3.2 For definitions of other textile terms used in this test method, refer to Terminology D 123.

4. Summary of Test Method

4.1 The test exposes a

4.1 A material used in the fabrication of protective apparel specimen is mounted on a vertical incline and is exposed to a molten substance under standardized conditions pour of prescribed minimum temperature, volume, pour rate, and measures with a copper calorimeter the vertical height.

4.2 The amount of heat energy that is transmitted through the test specimen.

4.2 Comparison of data from this test method with related information on specimen during and after the molten substance exposure is measured using two copper slug calorimeters. The heat transport response is assessed versus the Stoll curve, an approximate human tissue tolerance predictive model that projects the onset of human a second-degree skin to heat absorption permits estimation burn injury as manifested by a blister (See 12.2).

4.3 A specific set of subjective evaluations is performed on the protective capacity of test specimen response to the material tested.

4.3 The effect of the molten substance contact with the specimen is observed and recorded. exposure using a standardized rating scale for appraisal (found in Table X.1).

5. Significance and Use

5.1 Workers may be exposed to contact with molten substances. The clothing used should provide some protection. Whether personal injury results from such contact depends on the resistance of the material from which the clothing is made to molten substance contact and the amount of heat transferred through the material to the wearer.

5.2 This test method rates materials, that are intended for protective clothing against potential molten substance contact, for their thermal insulating properties and their reaction to the test exposure.

5.3 The protective performance, as determined by this test method, will relate to the actual end-use performance only to the degree that the end-use exposure is identical to the exposure used in the test method.

5.4 Visual inspection of the specimen subjectively notes the material's resistance to molten substance contact.

6. Apparatus

6.1 The test apparatus consists of four major components as follows:

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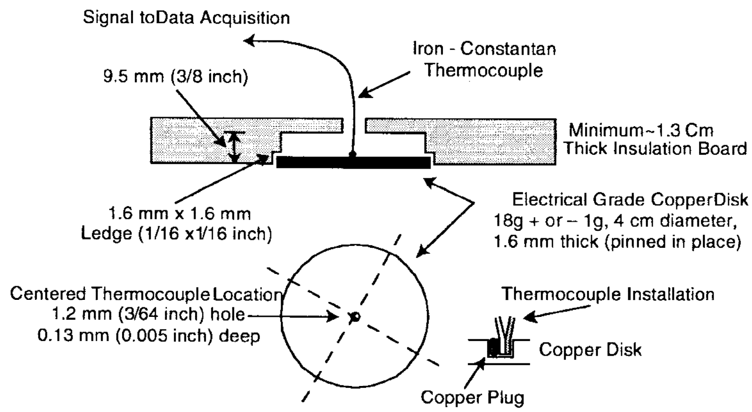
[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] Marinite which is available from Manville Corp., P.O. Box 5108, Denver, CO 80217 has been found satisfactory.

[3] *Annual Book of ASTM Standards*, Vol 15.03.

[4] Supporting data are available from

[4] *Annual Book of* ASTM Headquarters. Request RR:F23-1000. *Standards*, Vol 11.03.



NOTE 1—Sensor Construction—Place a straight, bare tip thermocouple wire into the hole located in the center of the copper disk. Place a short "copper plug" into the center hole with the thermocouple junction by clipping a section of bare copper wire of appropriate diameter that, when combined with the thermocouple wire, will just fit into the center hole. Use a center punch to mechanically wedge the wire bundle into place. Peen around the hole to further secure the thermocouple into the copper disk. Take care not to cut the thermocouple wire during this assembly process. Check the electrical continuity and thermal response prior to using the completed copper slug calorimeter.

FIG. 1 Sensor Construction

6.1.1 An inclined specimens mounting and exposure board with two copper slug calorimeter thermal sensors and support stand, supports,

6.1.2 A pouring crucible and pouring mechanism,

6.1.3 A furnace for melting the test metal, and

6.1.4 Instruments for measuring test conditions and test results.

6.2 *Sensor— Construction*

6.2.1 Each copper slug calorimeter mounted in an insulating block shall be 18 ± 1 g, 40 mm diameter and 1.6 mm thick and shall be constructed from electrical grade copper with a single 30 gage, iron/constantan thermocouple wire and shall be installed as described identified in Figs. 1 and 2. figure 1.

6.2.2 The exposed surface of each copper slug calorimeter shall be painted with a thin coating of a flat black high temperature spray paint with an emissivity of 0.9. The painted sensor shall be dried before use and present a uniformly applied coating (no visual thick spots or surface irregularities). Note that an external heat source, for example, an external heat lamp, has been used to completely drive off any remaining organic carriers on a freshly painted surface.

6.3 *Specimen/Sensor Board*—F

6.3.1 The specimen/sensor board shall be nominally 250 mm by 406 mm [10 in. by 16 in.] and fabricated of a flame and heat resistant material with two a thermal conductivity value of ≤ 0.15 W/m·K, high temperature stability, and resistance to thermal shock. The board shall be nominally 13 mm [0.5 in.] or greater in thickness.

6.3.2 The copper slug calorimeters shall be located in the centerline of the sensor board and mounted as shown in Fig. 3. Figure 1. The sensor board should calorimeters shall be nominally 16 by 10 located 101 mm and 203 mm [4 in. The relationship between the pouring ladle position and 8 in.] respectively, from the calorimeters is critical. Means should be provided for attaching the test materials to the top of the sensor board to the calorimeter centers as indicated in a manner such figure 2.

6.3.3 An attachment method shall be provided that affixes the test specimen will cover to the board such that it covers both sensors and extends at least 25 mm [1 in.] beyond all edges. During tests, the edges.

6.3.4 The sensor board shall be inclined at an angle of 70° from the horizontal. Details of mounting thermocouple leads in horizontal.

6.3.5 The sensor board shall be located so that it meets the copper calorimeters are pour geometry illustrated in Fig. 1 and of Figure 2, where the molten substance is introduced at the centerlinge of the calorimeter in the sensor board in Fig. 2. board.

6.4 *Pouring Crucible*—The pouring crucible shall be suitable for the substance being poured and for handling temperatures up to at least 56°C [100°F] above the pouring temperature. Crucible sizes appropriate for specific substances are shown in Table 1. It The crucible shall be attached to a tilting mechanism capable of turning the ladle at the rate of one revolution per 4.7 ± 0.2 s. The crucible shall be pivoted at a point 25 mm [1 in.] below the top of the crucible on the vertical. When tilted, the tilted action shall be stopped mechanically after a rotation of 10 ± 5° from the vertical (Fig. 3). 2). See also Appendix X2.

6.5 *Furnace*—A suitable furnace for melting and heating the test substance is needed. The furnace and lining must be of a type that the chemistry of the substance is not changed during contact with the furnace nor throughout the term of the test series.

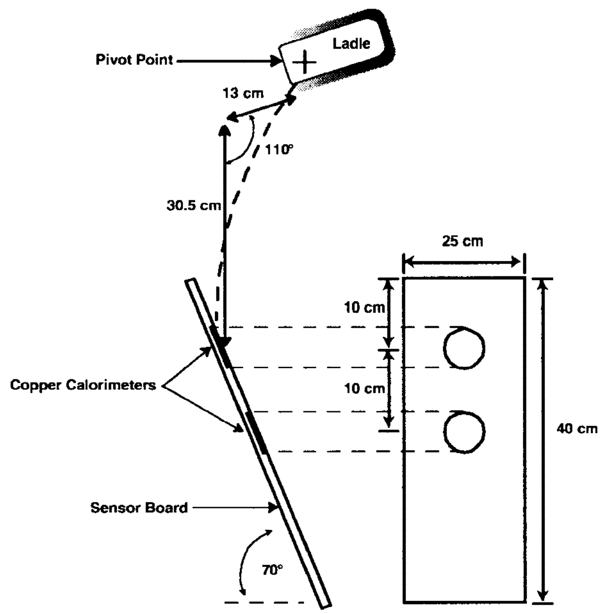


FIG. 2 Schematic of Test Apparatus

TABLE 1 Test Parameters[A]

NOTE 1—When this test method is used for substances other than below, use a pour temperature equal to use temperature or sufficiently above the melting point to give reproducible pouring characteristics.

	Substance	Pour Temperature (min)	Weight of Test Substance	Substance Specification
~~A~~	~~Iron~~	~~2800°F (1538°C)~~	~~2.2 lb (1000 g)~~	~~Eutectic iron~~[B]
A	Iron	2800°F [1538°C]	2.2 lb [1000 g]	Eutectic iron[B]
~~B~~	~~Aluminum~~	~~1400°F (760°C)~~	~~2.2 lb (1000 g)~~	~~Alloy 1100~~
B	Aluminum	1400°F [760°C]	2.2 lb [1000 g]	Alloy 1100
~~C~~	~~Brass~~	~~2100°F (1149°C)~~	~~2.2 lb (1000 g)~~	~~Alloy 875~~[C]
C	Brass	2100°F [1149°C]	2.2 lb [1000 g]	Alloy 875[C]

[A] See Appendix X3.

[B] Carbon equivalent = 4.3± 0.15 %. Carbon equivalent $= \% C + \frac{Si}{4} + \frac{P}{2}$ (approximately 2 % Si).

[C] 82/14/4 %, Cu/Zn/Sn.

6.6 Pouring height shall be 305 mm [12 in.] (~~s~~See Fig. ~~3).~~ 2).

6.7 ~~*Recorder*—Any two-pen strip chart recorder with full scale deflection~~Data Acquisition System—The system shall be capable of ~~150~~ recording the calorimeter outputs as required by the test.

6.7.1 The temperature data (calorimeter outputs) shall be acquired at a minimum sampling rate of four samples per second per calorimeter. The acquisition system shall be able to ~~300°F (66~~ record temperatures to ~~149°C) or 5 to 10 mV~~ 150°C [300°F], have at least a resolution of 0.1°C [0.2°F], and ~~sufficient sensitivity and scale divisions to read sensor response to ±1°F (0.5°C) or ±0.25 mV. A sufficiently fast response to read exposure time to ±0.2 s is required (0.5 in./s, 13 mm/s is satisfactory).~~ measuring accuracy of ±0.75°C [±1.4°F].

7. Materials

7.1 Description of standard substances for test purposes is included in Table 1.

8. Hazards

8.1 Perform the test in a ventilated area to carry away combustion products, smoke, and fumes. Care shall be exercised in handling the hot substance. A full fire extinguisher, preferably of the carbon dioxide type, ~~should~~ shall be readily available. ~~N~~ Use normal safety practices for the handling of the molten substance and potentially flammable ~~materials should be used. A~~ materials. Place a catch pan filled with dry sand ~~should be placed~~ under the assembly. *Extreme care shall be taken to prevent contact between the molten substance and water because explosive quantities of steam can result.*

8.2 The sensor board and calorimeter assembly become heated during prolonged testing. Use protective gloves when handling these hot objects and during the pour.

9. Sampling and Specimen Preparation

9.1 *Lot Size*—For acceptance sampling purposes, a lot is defined as 4572 m [5000 yd] or a single shipment of a single style of material, whichever is smaller. A lot may constitute all or part of a single customer order.

9.2 *Lot Sample*—As a lot sample, take a 2- m [2.2 yd] full-width piece of material from both the beginning and end of the lot.

9.3 *Test Specimens*—Cut and identify three test specimens from each sample. Make each test specimen at least 12 305 ± 2 mm by 18± 460 ± 2 mm [12 ± 1⁄16 in. (300 by 460 18 ± 2 mm). 1⁄16 in.]. Do not cut samples closer than 10 % of the material width from the edge and arrange the specimens across the sample swatch so as to get as representative a sampling as possible.

9.4 *Preparation of Human Tissue Burn Tolerance Overlay*—The end point criterion is a plot of energy versus time to cause a second-degree burn in human skin. Plot the calorimeter equivalent from Table 2, which corresponds to the recorder scale (T°F, T°C, or mV) on the vertical axis and the corresponding time on the horizontal axis. Use chart units based on the recorder full scale deflection and the chart speed to give a plot that compares directly with the recorder sensor trace. If pen deflection is from left to right, and paper movement down, prepare criterion plot from right to left—origin at lower right. If recorder trace differs, adjust the plot accordingly. Make exact transparent duplicate of the tolerance criterion to produce the overlay. Compare the overlay with original to insure duplication did not change the overlay size.

9.5 *Sensor Care*:

9.54.1 *Initial Temperature*—Cool the sensor after a pour exposure with a jet of air to approximately 70°F (21°C) 21°C [70°F] just prior to positioning the test specimen. Do not adjust the zero setting of the recorder.

9.5.2 specimen.

9.4.2 *Surface Reconditioning*—Wipe the sensor face with a nonabrasive material immediately after each run, while it is hot, to remove any decomposition products which condense, since these could be a source of error. If there is a deposit on the surface of the sensor that appears to be thicker than a thin layer of paint, or is irregular, the sensor surface requires reconditioning. Carefully clean the cooled sensor with acetone or petroleum solvent, making certain there is no ignition source nearby. Repaint the surface, if bare copper is showing, with a thin layer of flat black spray paint. (Air dry thoroughly before use.)

9.6 *Alignment of Equipment and Specimen*—Position the specimen or multiple layers with the face out, on the sensor board. Align the assembly so the stream of molten substance will strike the material directly over the top calorimeter. paint as identified in 6.2.2 above.

10. Procedure

10.1 *Specimen Mounting*—A piece of the test material, at least 12 by 18 in., should be laid prepared in accordance with 9.3, against the sensor board with the sample material hung over the sensor board, centered horizontally over the calorimeters with the top of the material about 130 mm [5 in.] above the top of the top upper sensor. The Attach the test material shall be held to the sensor board by an appropriate means at the top. means.

10.1.1 *Multiple Layer Samples*—For the standard tests to measure the protective property of multiple layer samples, place the surface of the material to be used as the outside of the garment is placed facing out. The Place the subsequent layer(s) is placed underneath in the order used in wearing with the surface to be worn toward the skin facing the calorimeters.

10.2 *Pour Method*—Molten substance of sufficient quantity for the test series is first heated in a furnace. When the sample is mounted and all is in readiness, the test substance shall be brought to about 100°F (56°C) 56°C [100°F] above the pour temperature. T Make molten substance temperature measurements may be taken with an appropriate device such as an optical pyrometer or other heat measuring device with an accuracy of at least ±25°F (±14°C). A ±14°C [±25°F]. Use a preheated crucible appropriate to the substance being used, as shown in Table 1, shall be used. A 1. Put a quantity of substance, ±10 % of the weight shown in Table 1 is then put in the test crucible, and then is transferred transfer it into the pouring rig. The Align the assembly holding the pouring crucible shall be aligned so that the main part of the molten substance stream impacts on the material specimen directly above the center of the top calorimeter.

10.2.1 Slightly

10.2.1 The data acquisition system is started several seconds before the test temperature actual molten material pour is reached, initiated. This establishes the recording device is started. When average starting copper slug calorimeter temperatures for the heat energy determination.

10.2.2 When the temperature of the molten substance drops to the appropriate pour temperature, tilt the crucible is tilted and pour the substance is poured on onto the material specimen set on the inclined sensor panel board for the test. (ETake extreme care should be taken to avoid being splashed by the hot substance.) Continue the exposure for 45 s then stop data acquisition.

10.2.3 Observe the recorder. Observe the specimen during and immediately after the pour.

10.2.4 Expose two specimens for each sample tested.

10.2.5 If the temperature rise of the two tests differ by more than 10 %, test a third specimen and average the results of the three tests. Note any explainable reason for the variation.

10.2.26 *Post Test Inspection*—After the pour, inspect the ladle should be inspected to be sure that all of the test substance has been poured out of the crucible and onto the test material. If visible substance remains in the pouring crucible after the test, this shall be removed remove and weigh thed material to be sure assure that the amount poured is within the tolerances.

10.3 *Direct Pour Method*—As an alternative to the procedure in 10.2, place a quantity of substance ±10 % of the weight shown in Table 1 is placed in a crucible in an induction furnace. T Mount this assembly is mounted in the pouring assembly. The Bring the substance is brought to the pouring temperature as indicated in 10.2 and poured pour directly from the furnace and crucible onto the test material.

10.3.1 PFollow post test inspection is followed as in 10.2.26.

11. Calculation of Results

11.1 *Sensor Response*—The response of each calorimeter is determined shortly before, during, and for 45 seconds after a pour has been initiated.

11.1.1 Once a pour initiation point is determined, the temperature data collected from the calorimeters before and up to the initiation point are averaged to obtain a starting calorimeter temperature, $T_{initial}$ (°C) for each respective sensor.

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

$$C_p = \frac{4.1868 \times (A + B \times t + C \times t^2 + D \times t^3 + E / t^2)}{63.546\ g/mol} \quad (1)$$

where:

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

11.1.3 *Discussion*—The heat capacity of copper in J/g°C at any temperature between 289 K and 1358 K is determined by means of Eq. 1 (Shomate Equation with coefficients from NIST).

11.1.4 The total incident energy versus time is determined and plotted for both panel heat energy sensors.

11.1.4.1 The copper slug heat capacity is determined at each time step. This is done by calculating an average heat capacity for each sensor from the initial heat capacity, determined in 11.1.2, and the time step measured temperature,

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

11.1.4.2 The total incident energy at each time step is determined in J/cm^2 by using the relationship,

$$Total\ Heat\ Energy,\ Q = \frac{mass \times \overline{C}_p \times (Temp_{final} - Temp_{initial})}{area} \quad (3)$$

where:

Q = Heat energy in J/cm^2
$mass$ = mass of the copper disk/slug (g)
$\overline{C}_p$ = Average heat capacity of copper during the temperature rise (J/g°C)
$temp_{final}$ = Final temperature of copper disk/slug at $time_{final}$(°C)
$temp_{initial}$ = Initial temperature of copper disk/slug at $time_{initial}$(°C)
$area$ = Area of the exposed copper disk/slug (cm^2)

11.1.4.3 For a copper disk/slug that has a mass of 18.0 g and exposed area of 12.57 cm^2, the determination of heat energy reduces to:

$$Total\ Heat\ Energy,\ Q = 1.432 \times \overline{C}_p \times (Temp_{final} - Temp_{initial}) \quad (4)$$

11.1.5 *Discussion*—If a copper disk/slug with a different mass and or exposed area is used, the constant factor in Eq. 4 above must be adjusted correspondingly. If required, the value in cal/cm^2 can be determined by multiplying the total heat energy in Eq. 4 by the conversion factor 1/4.1868 cal/J.

11.2 *Predicted Second-Degree Skin Burn Injury Determination (Stoll Curve Comparison)*—The time dependent measured heat energy for each copper slug calorimeter monitor sensor, determined in 11.1.4.3 above, is compared to an empirical human predicted second-degree skin burn injury model[5], commonly referred to as the “Stoll Curve” or “Stoll Response,”

$$Stoll\ Response,\ J/cm^2 = 5.0204 \times t_j^{0.2901} \quad (5)$$

NOTE 2—The “Stoll Response” can also be expressed in cal/cm^2 by means of:

$$Stoll\ Response,\ cal/cm^2 = 1.1991 \times t_j^{0.2901} \quad (6)$$

12. Report

12.1 The description of the specimen including but not limited to the number of material layers in the specimen, the sequence of the layers as they would be worn starting from the outer-most layer, the type of material in each layer, the area density in

[5] 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.

g/m^2[oz/yd ^{2}] of each layer of the material, the color of each layer of the material, the preconditioning in terms of laundering, dry cleaning and/or ambient conditioning for each specimen prior to testing.

12.2 The length of time it would take, under test conditions, to produce a second-degree burn according to in accordance with the Stoll curve (see Table 2, Footnote A) from the heat transferred through the test material (from 11.2). (No blister is one possible value.) V All values from the calorimetershow during the higher temperature rise entire 45 second acquisition period shall be used (worst case interpretation).

11.2 The degrees Fahrenheit of

12.3 The temperature rise at in Celsius degrees during the 30 s of exposure.

112.34 Physical damage to test material.

112.45 Adhesions, flaming, etc., and so forth, observed during test.

11.5

12.6 *Visual Response of Material Substance to Molten Substance Contact*—After the exposed specimen has cooled, carefully remove it from the sensor board and observe the effect of the exposure. This may be described by one or more of the following terms which are defined in 3.5 to3.1.5-3.1.11 3.11: ignition, charring, embrittlement, 3.1.1-3.1.5 and 3.1.7-3.1.10: break open, melting, charring, dripping, embrittlement, ignition, melting, shrinkage, and substance adhesions to test material, such as, number of adhesions, size of adhesions, and location in relation to sensor disc.

123. Precision and Bias

123.1 *Precision*—The precision of this test method was determined in a series of laboratory tests conducted on referenced fabrics and a detailed summary has been placed on file with ASTM headquarters.

12.2 [6]

13.2 *Bias*—No justifiable statement can be made on the bias of this test method for evaluating heat transfer through materials, since the true values of heat transfer cannot be established by an acceptable referee method.

[6] Supporting data are available from ASTM Headquarters. Request RR:F23-1000.

APPENDIXES

(Nonmandatory Information)

X1. SUGGESTED VISUAL RESPONSE REPORT FORM (see 11.5) 12.6)

Property	Yes	No	Comment
Ignition			
Charring			
Embrittlement			
Break-Open			
Melting			
Dripping			
Shrinkage			
Adhesions			

X2. CRUCIBLE TILT RATE

X2.1 The crucible tilt rate of one revolution in 4.7 ± 0.2 s is relatively slow. The low tilt rate results in a stream of metal that hits the calorimeter area as a moving *line* of metal. The total splash interval is about 1 s, which produces a reasonably severe impact event, but not all of the metal actually hits the calorimeter because the impact point moves as the crucible rotates.

X3. CRUCIBLE PARAMETERS

X3.1 The P-5 crucible has been used successfully for this work. The P-5 is available from American Refractories and Crucible Corp., P.O. Box 338, North Haven, CT 06473. A crucible filled to ⅓ to ½ level contains 1 kg of iron.