Standard Practice for
Thermal Conductivity of Materials Using a Thermal Capacitance (Slug) Calorimeter[1]

This standard is issued under the fixed designation E2584; 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 practice describes a technique for the determination of the apparent thermal conductivity, λ_a, of materials. It is for solid materials with apparent thermal conductivities in the approximate range $0.02 < \lambda_a < 2$ W/(m·K) over the approximate temperature range between 300 K and 1100 K.

NOTE 1—While the practice should also be applicable to determining the thermal conductivity of non-reactive materials, it has been found specifically useful in testing fire resistive materials that are both reactive and undergo significant dimensional changes during a high temperature exposure.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1113 Test Method for Thermal Conductivity of Refractories by Hot Wire (Platinum Resistance Thermometer Technique)
D2214 Test Method for Estimating the Thermal Conductivity of Leather with the Cenco-Fitch Apparatus (Withdrawn 2008)[3]
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples
E457 Test Method for Measuring Heat-Transfer Rate Using a Thermal Capacitance (Slug) Calorimeter
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *thermal conductivity,* λ—the time rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area.

3.1.2 *apparent thermal conductivity,* λ_a—when other modes of heat transfer (and mass transfer) through a material are present in addition to thermal conduction, the results of the measurements performed according to this practice will represent the apparent or effective thermal conductivity for the material tested.

3.2 *Symbols:*

A = specimen area normal to heat flux direction, m^2
C_p = specific heat capacity, J/(kg·K)
F = heating or cooling rate, (K/s)
L = thickness of a specimen (slab) in heat transfer direction, m
M = mass, kg
Q = heat flow, W
T = absolute temperature, K
T_{inner}^{SSS} = mean temperature of the stainless steel slug, K
T_{outer}^{SPEC} = mean temperature of outer (exposed) specimen surfaces, K
T_{mean}^{SPEC} = mean temperature of specimen, K
ΔT = temperature difference across the specimen, given by $(T_{outer}^{SPEC} - T_{inner}^{SSS})$, K
λ = thermal conductivity, W/(m·K)
λ_a = apparent thermal conductivity, W/(m·K)
ρ^{SPEC} = bulk density of specimen being tested, kg/m^3

3.3 *Subscripts/Superscripts:*

$SPEC$ = material specimen being evaluated
SSS = stainless steel slug (thermal capacitance transducer)

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[1] This practice is under the jurisdiction of ASTM Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.05 on Thermophysical Properties.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

4. Summary of Practice

4.1 *Principle of Operation*—In principle, a slug of thermally conductive metal, capable of withstanding elevated temperatures, is surrounded with another material of a uniform thickness (the specimen) whose thermal conductivity is substantially lower than that of the slug. When the outer surface of this assembly is exposed to a temperature above that of the slug, heat will pass through the outer layer, causing a temperature rise in the slug itself. The temperature rise of the slug is controlled by the amount and rate of heat conducted to its surface (flux), its mass, and its specific heat capacity. With the knowledge of these properties, the rate of temperature rise of the slug is in direct proportion to the heat flux entering it. Thus, under these conditions, the slug becomes a flux-gauging device. From this measured flux, along with the measured thermal gradient across the outer (specimen) layer, the apparent thermal conductivity of the specimen can be calculated. When the heat source is removed, during natural cooling, the direction of the heat flow will be reversed. Still, from the measured flux and thermal gradient, the apparent thermal conductivity can be calculated.

4.2 *Boundary Conditions*—The ideal model described above is based on heat flow toward the slug, perpendicularly to the specimen, and always through the specimen. Deviating from ideality can be due to:

4.2.1 Thickness non-uniformity of the outer layer.

4.2.2 Inhomogeneity (chemical or microstructural) of the outer layer.

4.2.3 Parasitic paths through cracks, gaps or other mechanically induced paths.

4.2.4 Parasitic paths through wires, sheaths (thermocouples), etc., that are unavoidable parts of a practical embodiment.

4.2.5 Delamination of the specimen from the slug's surface (gap formation).

NOTE 2—The user of this method should be very aware of the fact that the contact resistance between the specimen(s) and the slug may not always be neglected, and in some cases may be even significant, becoming probably the most important source of uncertainty in the measurement. For low-density porous materials, however, it was found that, generally, the contact resistance between the specimen(s) and the slug may be neglected.

4.3 *Configurations*—This method lends itself to many possible geometrical configurations, a few of which are listed below:

4.3.1 For pipe (tubular) insulations, a cylindrical slug is to be used. End faces are to be blocked with insulation.

4.3.2 For flat plate stock (insulating boards, bulk materials, etc.), a rectangular shaped slug is considered most practical, with the specimen material covering:

4.3.2.1 Both large faces of the slab, with the edges heavily insulated.

4.3.2.2 One large face of the slab, with the other face and the edges heavily insulated.

4.4 *Operation*—For simplicity, only the rectangular embodiment is described below:

4.4.1 *Twin Specimens (Double-Sided)*—A sandwich test specimen is prepared consisting of twin specimens of the material, of known mass and known and nominally identical thickness, between which is sandwiched a stainless steel thermal capacitance transducer (slug) of known mass. The entire sandwich is placed between two (high temperature) metal retaining plates, and the bolts holding the configuration together are tightened with a torque not to exceed 1 kg·m, to maintain a slight compressive load on the specimen. The assembled specimen is placed in a temperature-controlled environment and the temperatures of the steel slug and exposed surfaces of the specimens versus time are measured during the course of multiple heating and cooling cycles. Under steady-state (constant rate) heating or cooling conditions, the apparent thermal conductivity is derived from the measured temperature gradients across the two specimens, the measured rate of temperature increase/decrease of the steel slug, and the known masses and specific heat capacities of the specimens and the stainless steel slug. In principle, the test apparatus is similar to the Cenco-Fitch apparatus (**1**)[4] that is employed in Test Method D2214 for determining the thermal conductivity of leather. Measuring the heat transfer through a material by using a thermal capacitance transducer is similar to the approach that is employed for measuring heat-transfer rates in Test Method E457.

4.4.2 *Single Specimen (One-Sided)*—Similarly to the above, one unknown specimen is placed on one side of the slug and another known specimen (buffer) of extremely high thermal resistance is placed on the other side. In this instance, the outer surface of the buffer may be heated at the same time as the other side, just like in case of a twin specimen, or may be left unheated if it can be established that heat losses from the slug through this face are negligible.

5. Significance and Use

5.1 This practice is useful for testing materials in general, including composites and layered types.

5.2 The practice is especially useful for materials which undergo significant reactions or local dimensional changes, or both, during exposure to elevated temperatures and thus are difficult to evaluate using existing standard test methods such as Test Method C1113.

5.3 Performing the test over multiple heating/cooling cycles allows an assessment of the influence of reactions, phase changes, and mass transfer of reactions gases (for example, steam) on the thermal performance.

NOTE 3—This method has been found to be especially applicable to testing fire resistive materials.

6. Apparatus

6.1 *Thermal Capacitance (Slug) Calorimeter:*

6.1.1 The steel slug shall be manufactured from AISI 304 stainless steel or any other well characterized material of proper temperature service. Dimensions of the steel slug and the holes to be drilled for temperature sensor insertion are provided in Fig. 1. These dimensions and configuration are used for expediency in further discussion, without the intent of

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[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

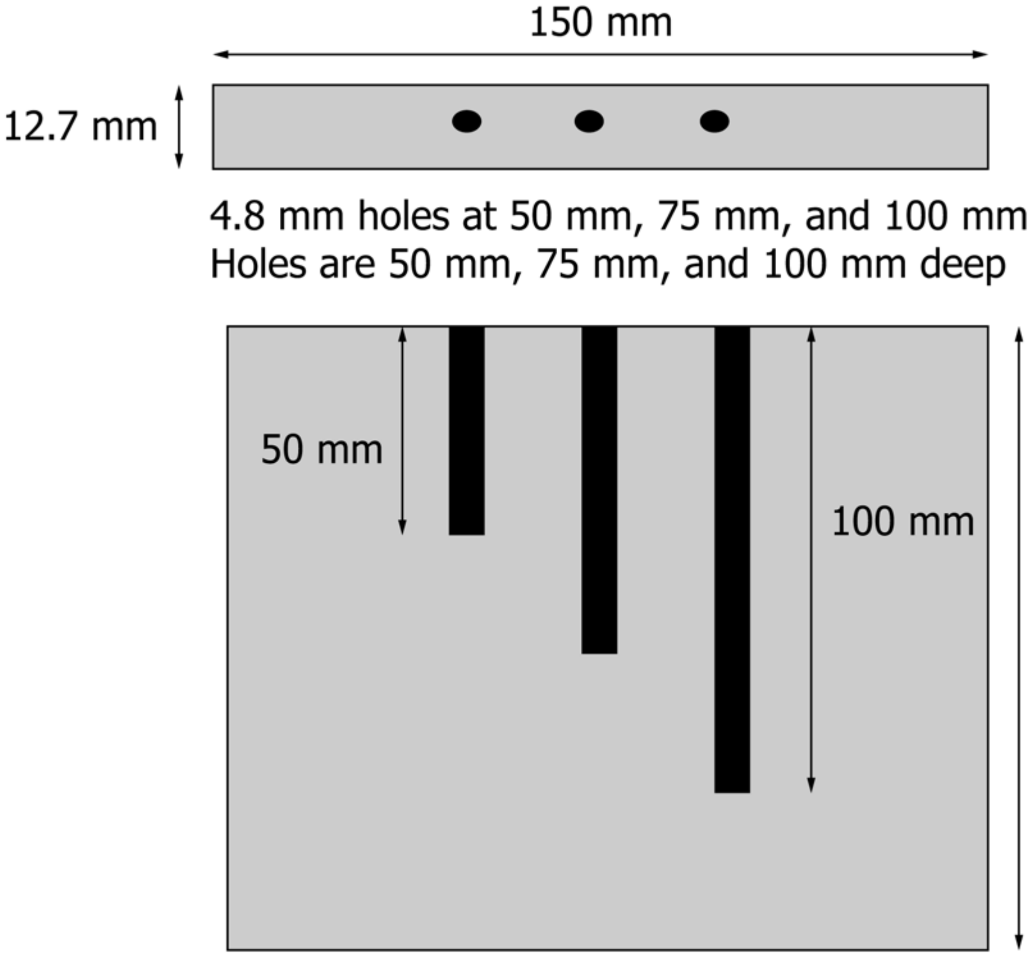

FIG. 1 Schematic of AISI 304 Stainless Steel Slug Calorimeter

posing restrictions on other sizes or configurations, or hindering the adaptation of other engineering solutions.

6.1.2 Two high temperature metal retaining plates, nominally of size 200 by 200 mm, shall be employed to hold the twin specimens, steel slug, and surrounding guard insulation in place and under a slight compression.

6.1.3 The steel slug and twin specimens shall be surrounded on all sides by an appropriate high temperature insulation, nominally of 25 mm thickness. Three holes shall be drilled through the section of insulation that covers the top of the slug in direct line with the corresponding milled holes in the steel slug to allow for insertion of the temperature sensors.

6.2 *Insulation Materials:*

6.2.1 A large variety of materials exists for providing the guard insulation that surrounds the stainless steel slug and specimens. Several factors must be considered during selection of the most appropriate insulation. The insulation must be stable over the anticipated temperature range, have a very low λ, and be easy to handle, cut, and insert holes. In addition, the insulation should not contaminate system components, it must have a low toxicity, and it should not conduct electricity. In general, microporous insulation boards are employed. Typically, these materials exhibit a room temperature thermal conductivity as low as 0.02 W/(m·K) and a thermal conductivity of less than 0.04 W/(m·K) at 1073 K. These values are much lower than those of typical materials that can be tested using this method.

6.3 *Temperature-Controlled Environment:*

6.3.1 The temperature controlled environment shall consist of an enclosed volume in which the temperature can be controlled during heating and monitored during (natural) cooling. The heating units shall be capable of supplying sufficient energy to achieve the temperatures required for the evaluation of the materials under test. Typically, the heated environment ranges in temperature between room temperature and 1000°C during the course of a single heating/cooling cycle.

6.3.2 One example would be a temperature-controlled furnace with an electronic control system that allows the programming of one or more temperature ramps. For example, the following temperature setpoints (versus time) have been successfully employed in the past: 538°C after 45 min, 704°C after 70 min, 843°C after 90 min, 927°C after 105 min, and 1010°C after 2 h.

6.4 *Temperature Sensors:*

6.4.1 There shall be a minimum of three temperature sensors to be inserted into the pre-drilled holes in the stainless steel slug. The multiple sensors are useful to indicate the validity of one-dimensional heat transfer through the specimen(s) to the steel slug. Temperature sensors may also be mounted in milled grooves on the exterior surface of the specimens, one sensor per specimen. When this is not possible, the exposed face of the specimen may be assumed to have a temperature equal to the measured temperature of the temperature-controlled environment.

6.4.2 For the purposes of this test, it is reasonable to postulate that the surface temperature of the slug is identical to its mean temperature. When comparatively high thermal conductivity specimens are used, it is practical to embed a temperature sensor near to or on the slug's surface.

6.4.3 Any sensor possessing adequate accuracy may be used for temperature measurement. Type K or Type N thermocouples are normally employed. Their small size and ease of manufacturing are distinct advantages. The sensors simply must fit into the holes present in the thermal capacitance calorimeter, where they can be easily inserted during the assembly of the configuration within the temperature-controlled environment.

6.4.4 When thermocouples are employed, a constant temperature reference shall always be provided for all cold junctions. This reference can be an ice-cold slurry, a constant temperature zone box, or an integrated cold junction compensation (CJC) sensor. All thermocouples shall be fabricated from either calibrated thermocouple wire or from wire that has been certified by the supplier to be within the limits of error specified in Table 1 of Specification E230. Thermocouples can be calibrated as described in Test Method E220.

6.5 *Data Acquisition System*—While manual acquisition of the data is possible, for convenience, increased reliability, and avoidance of transcription errors, it is recommended that an appropriate data acquisition system be employed to automatically monitor all of the temperature sensors at regular (1 min for example) intervals. As examples, data may be acquired using a thermocouple input module or a voltmeter/multimeter system. In the latter case, the measured signals shall be converted to temperatures using the appropriate tables from Specification E230.

7. Hazards

7.1 It shall be verified that specimens or the test assembly have cooled adequately before attempting to remove them from the temperature-controlled environment.

7.2 The recommendations of each material manufacturer shall be followed when handling their materials (for example, gloves, safety glasses, or respirators).

7.3 Materials that react at high temperatures may release noxious or toxic products. In such cases, necessary precautions should be taken to assure that any gases generated during the execution of this test method are properly filtered or vented, or both.

8. Sampling, Test Specimens, and Test Units

8.1 *Double-Sided Systems:*

8.1.1 Two (twin) parallelepiped specimens, each with a cross sectional area of 150 by 150 mm and the thickness selected for the particular test (for example, 25 mm) shall be prepared (for example, by spraying, brush application, or simply cutting from a larger specimen). The length and width of the specimens shall be determined by making a set of three measurements across the top, middle, and bottom (left, center, and right) of the specimens. The mean and standard deviation of the measurements shall be reported. If the standard deviation is greater than 2.5 mm, the specimen shall be recut in an attempt to obtain a squarer specimen. If necessary, prior to the measurement of the specimen thickness, the planarity (levelness) of the top and bottom surfaces of the specimen shall be verified and any "high" spots (local thickness 1 mm or greater than local background thickness) removed as needed. For example, these high spots may be removed by careful extraction using a utility knife or hacksaw blade for fibrous and soft materials, or by using a file or an electric sander for "harder" materials. The thickness of the specimen shall be determined by measurement using a digital thickness gauge (digital calipers). A minimum of eight measurements (two from each of the four sides of the specimen slab) shall be performed and the mean and standard deviation shall be reported. If the standard deviation is greater than 1 mm, the specimen shall be either discarded or replaned in an attempt to achieve a more uniform thickness.

8.1.2 As an alternative to testing bulk specimens, thinner specimens may be applied to pre-weighed AISI Type 304 stainless steel panels (1.6 mm thick panels for example) and tested with the substrate panels placed against the central steel slug. If the steel panels are 150 by 163 mm and a centered 150 by 150 mm area specimen is applied, the two metal edges may be conveniently used to "grip" the specimen (under a portion of the guard insulation) in the final specimen sandwich configuration. In this case, the mean specimen thickness can be determined using the digital caliper technique outlined above, but first subtracting the previously measured mean thickness of the steel substrate (panel) from each individual thickness measurement.

8.1.3 For specimens of sufficient thickness, a single centered groove of sufficient size for the insertion of a temperature sensor shall be made into the top (exposed) surface of each specimen. After the groove has been properly sized, the initial mass of each specimen shall be determined and recorded. The initial density of each specimen shall be determined by dividing its measured mass by the product of its measured dimensions (volume). The mass of the steel slug shall also be measured and recorded. For thin specimens applied to steel panels, no such groove shall be made and the measured temperature of the temperature-controlled environment shall be used as being representative of the temperature of the exterior (exposed) surfaces of the specimens.

8.2 *Single-Sided Systems:*

8.2.1 For a single specimen device, the preparation procedure is identical, except as to the need for only a single one.

9. Preparation of Apparatus

9.1 *Double-Sided Systems:*

9.1.1 The sandwich specimen shall be assembled by placing one of the outer retaining plates on a flat surface and centering the first specimen on the plate. The stainless steel slug shall be added next, followed by the second specimen. The specimens and slug shall then be surrounded on all four sides by a 25 mm thickness (minimum) of high temperature insulation to serve as a guard insulation material. The insulation material on the top surface shall contain three small holes located directly over the three holes in the steel slug, for insertion of the temperature sensors. The top retaining plate shall be applied and the retaining bolts tightened manually with a wrench, to maintain a slight compression on the entire sandwich specimen construction. A schematic of a final assembled specimen in provided in Fig. 2. The mass of the final assembled specimen shall be measured and recorded.

9.1.2 The assembled specimen shall be centrally located in the temperature-controlled environment and the temperature sensors attached, three into the central steel slug and one each into the grooves (when present) on the exterior surface of the two twin specimens. At this point, the specimen is ready for testing.

9.2 *Single-Sided Systems:*

9.2.1 For single sided application, the slug and the permanent (buffer) specimen are maintained as a unit, including all edge insulation. The unknown is fixed to the slug on the open side, and the rest of the process is essentially the same. In a horizontal configuration, the retaining plates may be omitted.

10. Procedure

10.1 The specimen shall be exposed to a measured time/temperature exposure appropriate for the evaluation of the material being examined. An example would be using a heating rate of 1 to 5 K/min to the desired maximum temperature. Whatever time/temperature exposure is utilized, it should be reported as part of the test results. When the maximum testing temperature is achieved, the heat source shall be turned off and the temperature sensors shall continue to be monitored during the natural cooling of the specimens in the temperature-controlled environment. The maximum testing temperature may be set by when the steel slug reaches a pre-defined endpoint temperature (810 K for instance) or when the temperature-controlled environment reaches some pre-defined endpoint temperature (1273 K for instance), depending on the purpose of the test being conducted.

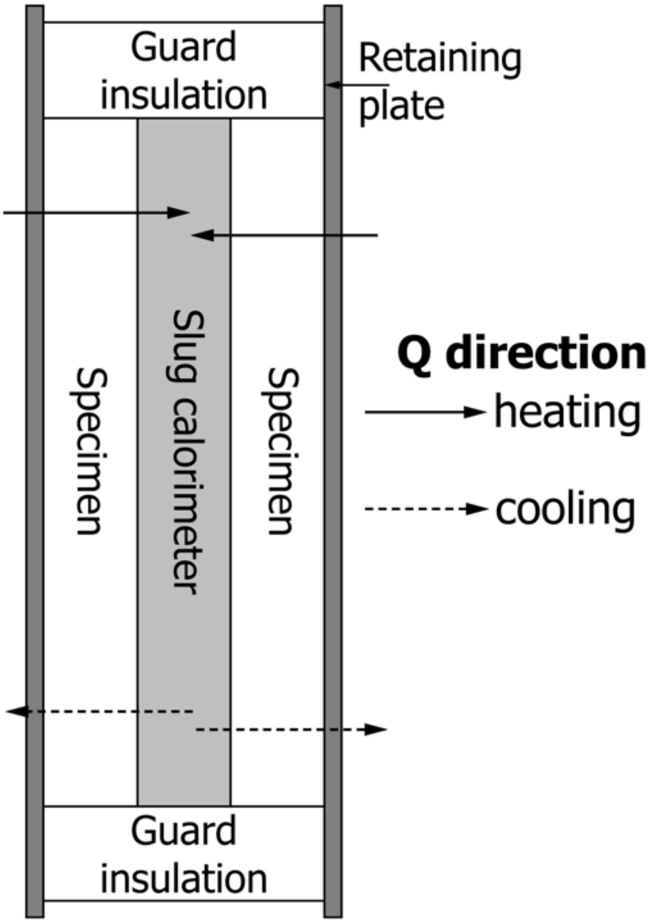

FIG. 2 Schematic of an Assembled Twin Specimen Slug Calorimeter Specimen Ready for Testing

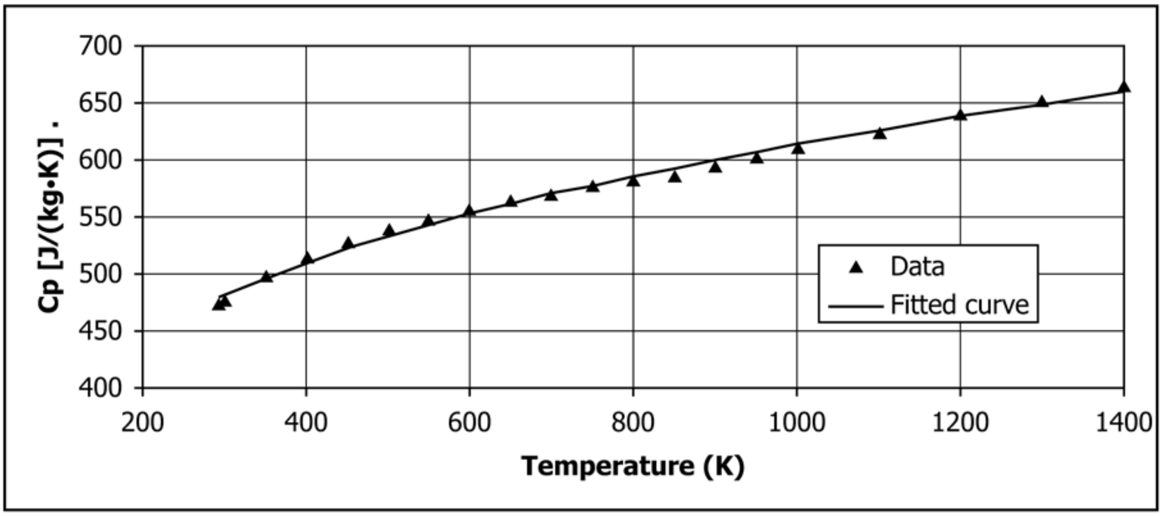

FIG. 3 Literature Values (2) and Fitted Curve for Specific Heat Capacity of 304 Stainless Steel Fitted Curve is of the Form $C_p^{SSS} = 6.683 + 0.04906*T + 80.74*\ln(T)$ with T in K

10.2 When the natural cooling has approached near room temperature (300 ± 40 K) and the measured temperature gradient across the specimen is less than 20 K, the test is completed.

10.3 During the course of the test, the temperature sensors are typically read once per minute during the heating cycles. During the slower natural cooling, the sampling frequency is often decreased to either once every 3 min or once every 5 min.

11. Calculation

11.1 *Double-Sided Configuration:*

11.1.1 At each sampling time, the mean specimen temperature (T_{mean}^{SPEC}) is calculated as the average of the mean slug temperature (T_{inner}^{SSS}) and the mean exterior specimen temperature (T_{outer}^{SPEC}). The apparent thermal conductivity, λ_a, at each mean specimen temperature is then calculated as:

$$\lambda_a = \frac{FL(M_{SSS}C_p^{SSS} + M_{SPEC}C_p^{SPEC})}{2A\Delta T} \tag{1}$$

where the symbols are as defined in 3.2, (with F representing the actual temperature increase of the slug over a time interval, not the programmed heating rate for the system), and M_{SPEC} refers to one half of the mass of the two (twin) specimens and may be time (temperature)-dependent. Heat capacity data for AISI 304 stainless steel taken from the literature (**2**) is provided in graphical form in Fig. 3, along with an equation that has been fitted to these literature values.

11.2 *Single-Sided Configuration:*

11.2.1 Eq 1 can be easily modified for the single-sided configuration. For example, the single-sided setup can be conveniently thought of as a double-sided configuration in which the thickness of the steel slug has been doubled. This is only true, however, if the contribution of the buffer specimen has been determined to be less than 5 % of the heat conduction, when the specimen apparent thermal conductivity is equal to or higher than 0.1 W/(m·K) for a 25 mm thick specimen. Then, the apparent thermal conductivity, λ_a, at each mean specimen temperature is calculated as:

$$\lambda_a = \frac{FL\left(M_{SSS}C_p^{SSS} + \frac{M_{SPEC}C_p^{SPEC}}{2}\right)}{A\Delta T} \tag{2}$$

12. Report

12.1 The report of the test results shall include the following:

12.1.1 Complete specimen identification including size and pretest mass and density for each specimen(s);

12.1.2 Complete identification of high temperature guard insulation and source;

12.1.3 Statement of temperature sensor type and size;

12.1.4 Mass of steel slug;

12.1.5 Masses of steel substrates (panels) when used with specimens;

12.1.6 Masses of assembled slug calorimeter specimen, before and after testing (if twin type);

12.1.7 Tables of time-temperature set points employed for each cycle's heating curve;

12.1.8 Table of measured temperature sensor results, including that measured for the temperature-controlled environment, during the entire course of the test;

12.1.9 Table or graph of calculated apparent thermal conductivity as a function of mean specimen temperature during all of the applied heating/cooling cycles; and

12.1.10 Configuration of the equipment used (twin, single, etc.).

13. Precision and Bias[5]

13.1 The precision of this test method is based on an interlaboratory study (ILS) of Test Method E2584, conducted in 2009. Each of nine laboratories tested three different materials at twelve temperature levels (ranging from 100 to 650ºC). Every "test result" represents an individual determination. All laboratories were asked to report two replicate test

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1038.

results from a single operator, for every material. Except for the inability of all laboratories to report all requested material, analysis, and replicate combinations, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report RR:E37-1038.

13.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material; "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

13.1.1.1 Repeatability limits are listed in Tables 1-12.

13.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "R" value for that material; "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

13.1.2.1 Reproducibility limits are listed in Tables 1-12.

13.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

13.1.4 Any judgment in accordance with 13.1.1 and 13.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of materials tested and laboratories reporting replicate results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

13.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

13.3 The precision statement was determined through statistical examination of 373 results, from nine laboratories, on three materials. These three materials were described as the following:

Material A	Low density spray-applied fire resistive material 2.54 cm thick
Material B	Medium density spray-applied fire resistive material 2.54 cm thick
Material C	Board fire resistive material 2.54 cm thick

13.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

14. Keywords

14.1 fire resistive material; slug calorimeter; thermal conductivity

TABLE 1 Thermal Conductivity at 100°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.117	0.0104	0.0112	0.029	0.031
B	0.154	0.0035	0.0344	0.010	0.096
C	0.094	0.0013	0.0100	0.004	0.028

[A] The average of the laboratories' calculated averages.

TABLE 2 Thermal Conductivity at 150°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.126	0.0120	0.0183	0.034	0.051
B	0.160	0.0020	0.0323	0.006	0.090
C	0.101	0.0019	0.0108	0.005	0.030

[A] The average of the laboratories' calculated averages.

TABLE 3 Thermal Conductivity at 200°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.139	0.0123	0.0200	0.035	0.056
B	0.168	0.0025	0.0320	0.007	0.090
C	0.108	0.0013	0.0123	0.004	0.035

[A] The average of the laboratories' calculated averages.

TABLE 4 Thermal Conductivity at 250°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.154	0.0141	0.0226	0.039	0.063
B	0.176	0.0030	0.0325	0.008	0.091
C	0.116	0.0012	0.0138	0.003	0.039

[A] The average of the laboratories' calculated averages.

TABLE 5 Thermal Conductivity at 300°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.169	0.0157	0.0274	0.044	0.077
B	0.184	0.0032	0.0333	0.009	0.093
C	0.124	0.0021	0.0119	0.006	0.033

[A] The average of the laboratories' calculated averages.

TABLE 6 Thermal Conductivity at 350°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.186	0.0202	0.0317	0.056	0.089
B	0.191	0.0043	0.0356	0.012	0.100
C	0.135	0.0021	0.0146	0.006	0.041

[A] The average of the laboratories' calculated averages.

TABLE 7 Thermal Conductivity at 400°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.205	0.0233	0.0405	0.065	0.113
B	0.200	0.0032	0.0360	0.009	0.101
C	0.144	0.0026	0.0166	0.007	0.047

[A] The average of the laboratories' calculated averages.

TABLE 8 Thermal Conductivity at 450°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.224	0.0239	0.0430	0.067	0.120
B	0.217	0.0032	0.0353	0.009	0.099
C	0.153	0.0030	0.0154	0.007	0.043

[A] The average of the laboratories' calculated averages.

TABLE 9 Thermal Conductivity at 500°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.245	0.0252	0.0490	0.071	0.137
B	0.228	0.0020	0.0415	0.006	0.116
C	0.165	0.0030	0.0154	0.008	0.043

[A] The average of the laboratories' calculated averages.

TABLE 10 Thermal Conductivity at 550°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.263	0.0265	0.0593	0.074	0.166
B	0.247	0.0035	0.0411	0.010	0.115
C	0.177	0.0099	0.0184	0.028	0.052

[A] The average of the laboratories' calculated averages.

TABLE 11 Thermal Conductivity at 600°C (W/(m·K))

Material	Average,[A] $(\bar{x})$	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.311	0.0312	0.0611	0.087	0.171
B	0.273	0.0042	0.0456	0.012	0.128
C	0.201	0.0099	0.0177	0.028	0.050

[A] The average of the laboratories' calculated averages.

TABLE 12 Thermal Conductivity at 650°C (W/(m·K))

Material	Average,^{A} ($\bar{x}$)	Repeatability Standard Deviation, (S_r)	Reproducibility Standard Deviation, (S_R)	Repeatability Limit, (r)	Reproducibility Limit, (R)
A	0.342	0.0325	0.0677	0.091	0.189
B	0.294	0.0056	0.0496	0.016	0.139
C	0.220	0.0100	0.0192	0.028	0.054

^{A} The average of the laboratories' calculated averages.

APPENDIX

(Nonmandatory Information)

X1. TESTING FIRE RESISTIVE MATERIALS

X1.1 For fire resistive materials, the measurement is complicated by the changes in the mass and the thickness of the specimens that may occur during testing. A fire resistive material may lose as much as 30 % of its mass upon exposure to a 1250 K temperature. Furthermore, some intumescent fire resistive materials (coatings) may expand up to 40× or more when exposed to temperatures of 450 K and greater. In the case of mass loss, it is recommended that the interferences be handled by simply determining the mass of the assembled specimen before and after testing and distributing the measured mass loss uniformly with the measured mean specimen temperature throughout the course of the first heating cycle. In the case of significant expansion of the fire resistive material, it is recommended that the expansion be visually or otherwise non-destructively assessed during the course of the first heating cycle. If this is not possible in the employed testing configuration, the thickness of the fire resistive material before and after testing should be measured and the expansion assigned as a step function in the thickness, occurring at the known or measured mean temperature of intumescence. In this latter case, if it is not possible to directly measure the temperature of the exposed (expanding) surface of the fire resistive material, it may be assumed to be equal to the temperature measured for the temperature-controlled environment.

X1.2 Fire resistive materials are generally reactive at high temperatures, many releasing physically or chemically bound water or carbon dioxide. Other more toxic materials, such as hydrochloric acid or styrene monomer, may be produced in small quantities during the thermal degradation of one or more components of some of these materials.

X1.3 A second "identical" heating/cooling cycle may be applied. By comparing the results of the second cycle to those of the first, the influences of reactions, phase changes, and mass transfer can be examined. Finally, if desired, a third heating/cooling cycle, typically at a much slower heating rate, may be applied to increase the range of useful (post transient) temperature data that may be obtained during the heating. As an example, the temperature of the temperature-controlled environment could be linearly ramped from room temperature to 870 K over the course of 4 h. After all testing has been completed and the specimen has cooled, it shall be carefully removed from the temperature-controlled environment and its (posttest) mass determined and recorded.

X1.4 The mass loss of the specimens is determined as the difference between the measured posttest and pretest masses of the assembled specimen and then distributed uniformly with the mean specimen temperature during the first heating cycle. Thereafter, the mass is assumed to remain constant throughout the first cooling cycle and any subsequent heating/cooling cycles. By plotting the computed apparent thermal conductivity versus the mean specimen temperature for the various heating/cooling cycles, the transient regions of the test where steady-state conditions do not apply can be readily determined. These will include the initial heating phase of each heating cycle and the transition from heating to cooling, when the direction of heat transfer through the specimens ultimately is reversed. These transient regions are particularly significant during the initial portions of the first two heating cycles due to their high heating rate. More information on this, along with example results, can be found in X1.6 and in Refs (**3**) and (**4**).

X1.5 When the materials are applied to a stainless steel plate (substrate) prior to testing, the mass of both steel plates shall be included as part of the mass of the steel slug, M_{SSS}, in Eq 1.

X1.6 Example results for a typical fire resistive material specimen are provided in Fig. X1.1. In the two graphs, the measured results as supplied by the thermal capacitance (slug) calorimeter are compared against measured values supplied either by a transient plane source technique (**5**) or Test Method C1113 (**6**). The three methods are seen to provide results that are in sufficient agreement. The transient periods of the slug calorimeter test method, both during the initial phase of each heating curve and during the transition from heating to cooling for each heating/cooling cycle are clearly observed in the top and bottom graphs, respectively. By comparing the heating (*1*) and heating (*2*) curves in the top graph, the complex influence of reactions, phase changes, and mass transfer on the thermal performance of this fire resistive material can be evaluated. This behavior consists of both endothermic events where the

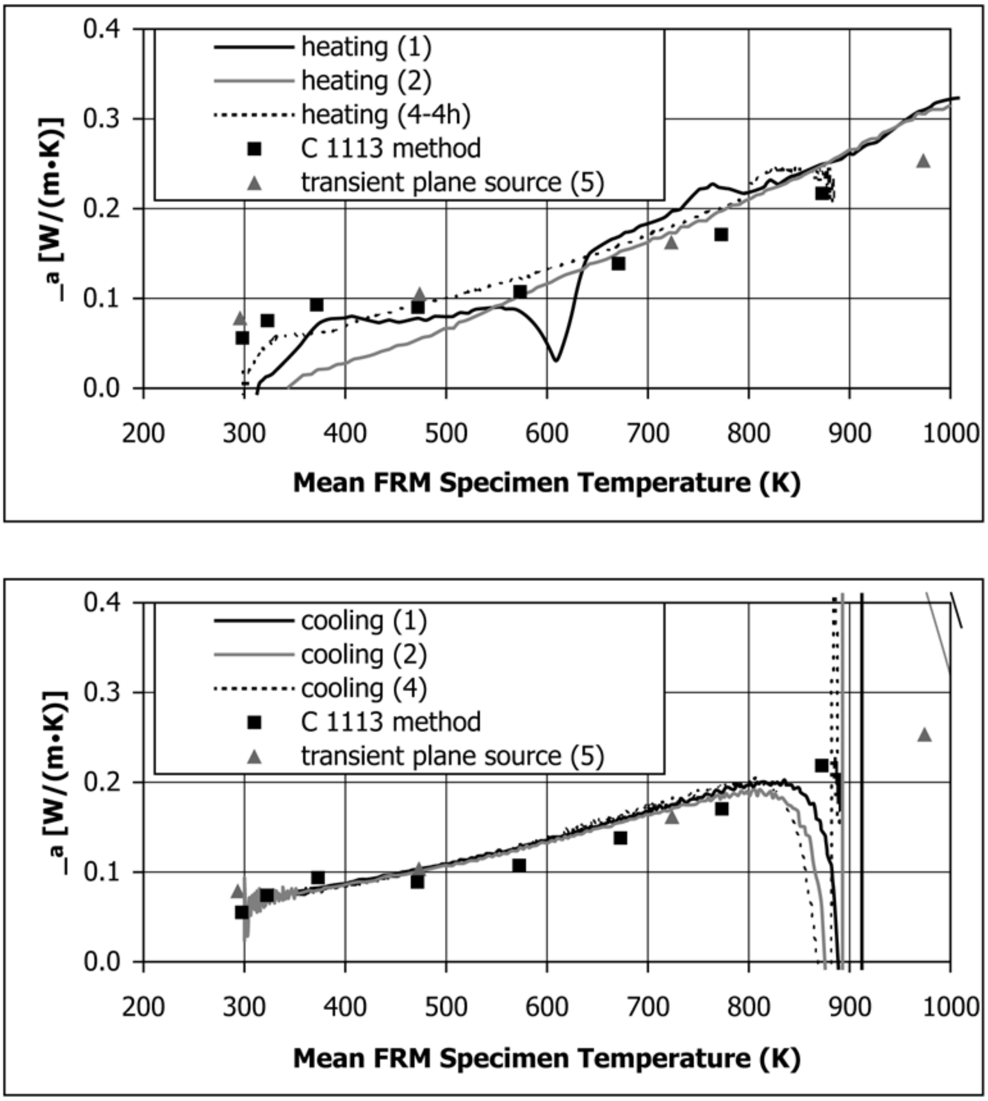

FIG. X1.1 Apparent Thermal Conductivity Results for a Typical Fire Resistive Material Specimen Using the Slug Calorimeter in Comparison to Measured Data from Other Methods for Multiple Heating (Top) and Cooling (Bottom) Cycles

apparent thermal conductivity for the first heating cycle is lower than that in the second heating cycle, and exothermic events where the reverse is true. The endothermic events correspond to endothermic (dehydration) reactions and phase changes, while the exothermic ones are likely due to the subsequent mass transfer of these hot reaction gases through the porous fire resistive materials to the steel slug surface.

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