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Standard Practice for Thermal Conductivity of Materials Using a Thermal Capacitance (Slug) Calorimeter¹

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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 *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 Apparatus³

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

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³ Withdrawn.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

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, m²

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/m³

3.3 Subscripts/Superscripts:

$SPEC$ = material specimen being evaluated

SSS = stainless steel slug (thermal capacitance transducer)

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 and/or local dimensional changes 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 (e.g., 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 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

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

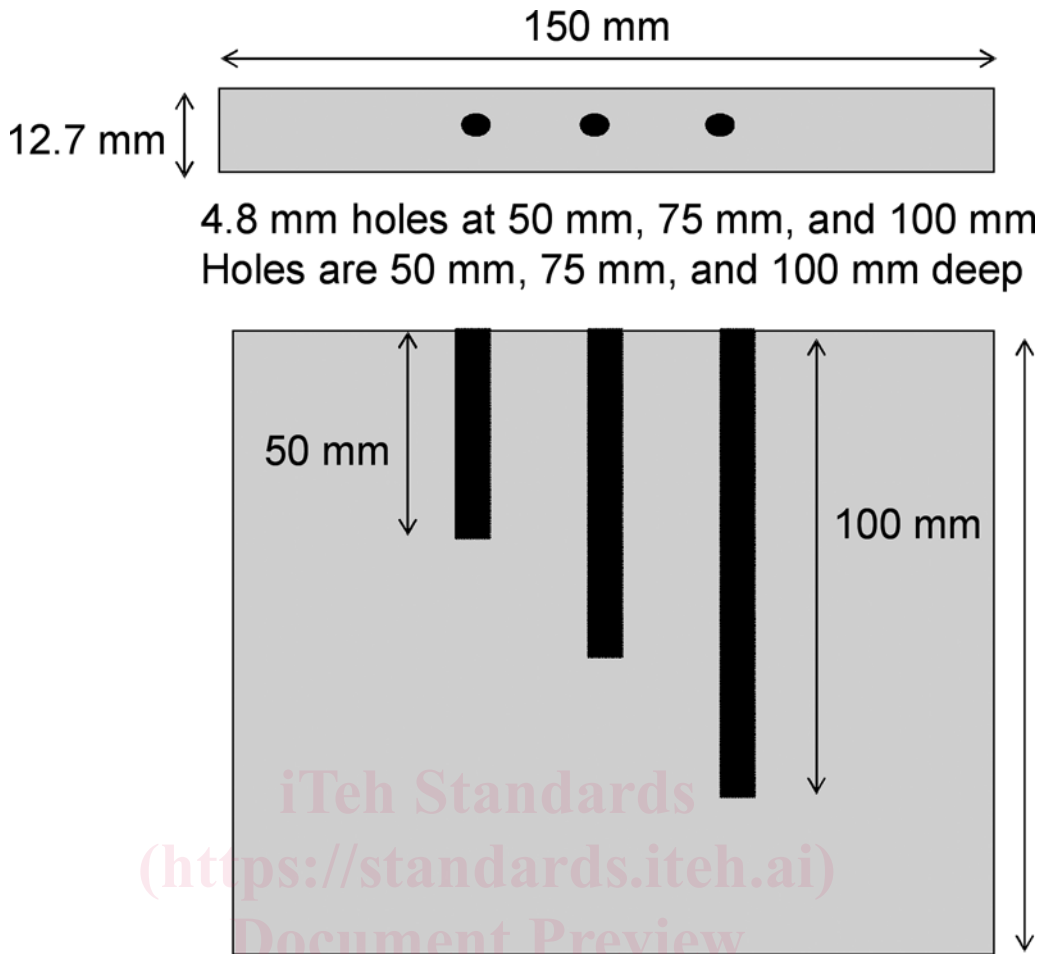


FIG. 1 Schematic of AISI 304 Stainless Steel Slug Calorimeter

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 Specification 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 minute 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 (e.g., gloves, safety glasses, 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 and/or vented.

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 mm by 150 mm and the thickness selected for the particular test (e.g., 25 mm) shall be prepared (e.g., 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 replanned 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 mm by 163 mm and a centered 150 mm 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 is 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