



Designation: E2584 – 20

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

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 , and thermal diffusivity, α_a , of materials. It is for solid materials with apparent thermal conductivities in the approximate range $0.02 < \lambda_a < 20$ W/(m·K) over the approximate temperature range between 300 K and 1100 K, if used stainless steel 304 slag calorimeter and K thermocouples, and up to 1600 K, if used Alumina slag calorimeter and PtRh thermocouples.

NOTE 1—While the practice should also be applicable to determining the thermal conductivity and thermal diffusivity 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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D2214 Test Method for Estimating the Thermal Conductivity of Leather with the Cenco-Fitch Apparatus (Withdrawn 2008)³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 for 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 *apparent thermal conductivity, λ_a , n* —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.

3.1.2 *apparent thermal diffusivity, n* —the apparent thermal conductivity divided by bulk density and apparent specific heat capacity:

$$\alpha_a = \lambda_a / C_p \rho^{SPEC}$$

3.1.3 *specific heat capacity, C_p , n* —the amount of heat required to change a unit mass (or unit quantity, such as mole) of a substance by one degree in temperature.

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.

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

3.2 Symbols:

A	= specimen area normal to heat flux direction, m^2
C_p	= specific heat capacity, $J/(kg \cdot K)$
F	= heating or cooling rate, (K/s)
L	= thickness of a specimen (slab) or distance between central and surface thermocouples, 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, $W/(m \cdot K)$
λ_a	= apparent thermal conductivity, $W/(m \cdot K)$
ρ^{SPEC}	= bulk density of specimen being tested, kg/m^3
α_a	= $\lambda_a / C_p / \rho^{SPEC}$ = apparent thermal diffusivity, m^2/sec

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*—Calculations are performed in the range of monotonous heating, closed to a quasi-steady state conditions, when rates of heating/cooling at the surface and the center of the sample and slug calorimeter are the same (that means that initial stage of heating and cooling should be excluded during treatment of the experimental data).

4.1.1 *Thermal Conductivity Testing*—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.

4.1.2 *Thermal Diffusivity Testing*—Apparent thermal diffusivity is measured by using the two similar samples assembly as for thermal conductivity testing, but without the slug

calorimeter between the samples. The thermal diffusivity is calculated based on measuring of temperature difference between surface and center of the sample and heating rate. Heating rate should provide temperature difference in the samples about 5 K – 50 K.

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—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 practice 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.

4.4.1.1 The specimens for measurement of thermal diffusivity can be the same shape and sizes as specimens for thermal conductivity.

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.

4.5 If the specimens for measurement of thermal conductivity and diffusivity are transparent for heat radiation, the

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

radiation can be decreased by screens between heaters and specimens, or by not transparent coating on the surface of the specimens.

5. Significance and Use

5.1 This practice is useful for testing materials in general, including composites and multi-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 practice 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

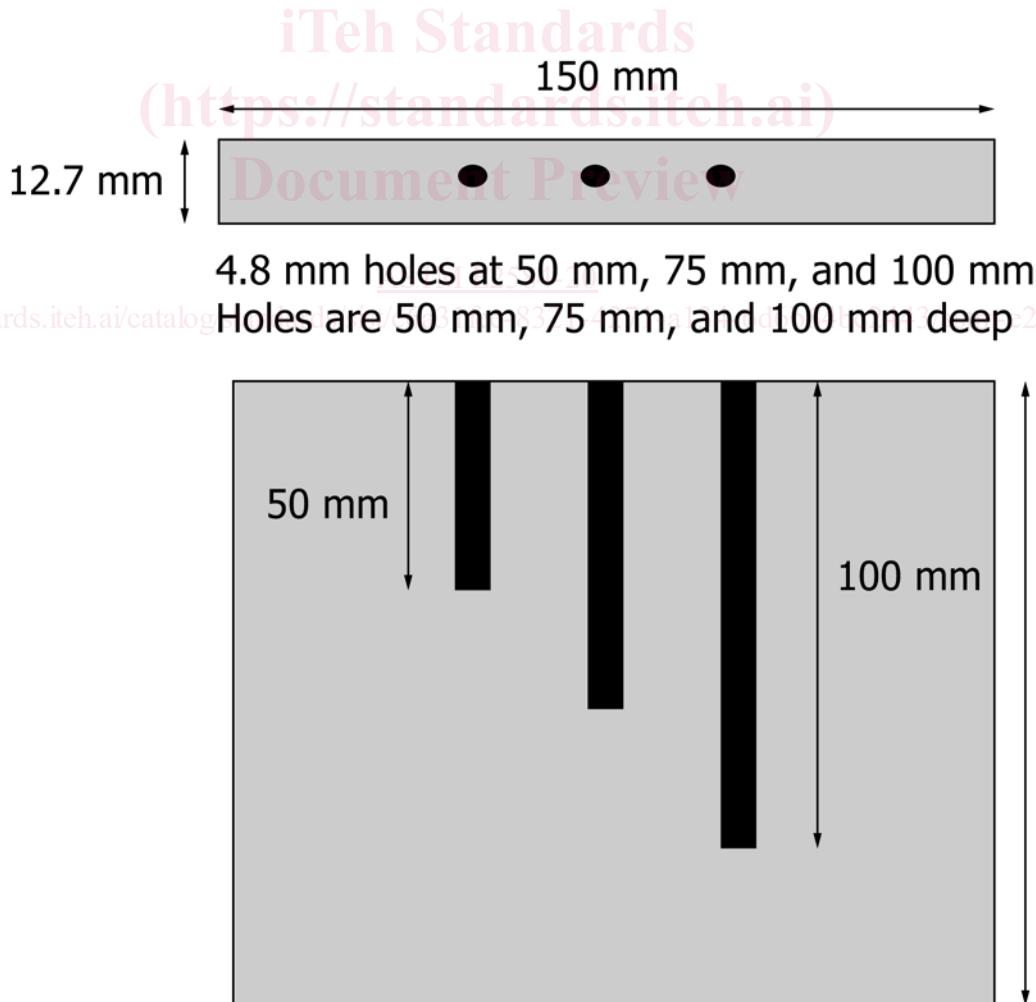


FIG. 1 Schematic of AISI 304 Stainless Steel Slug Calorimeter

proper temperature service. Recommended 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 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 practice.

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 one temperature sensor 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 contact heat resistances are negligible, the exposed face of the specimen may be assumed to have a

temperature equal to the measured temperature of the temperature-controlled retaining plates (see Fig. 2).

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 practice 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 similar shape and sizes of

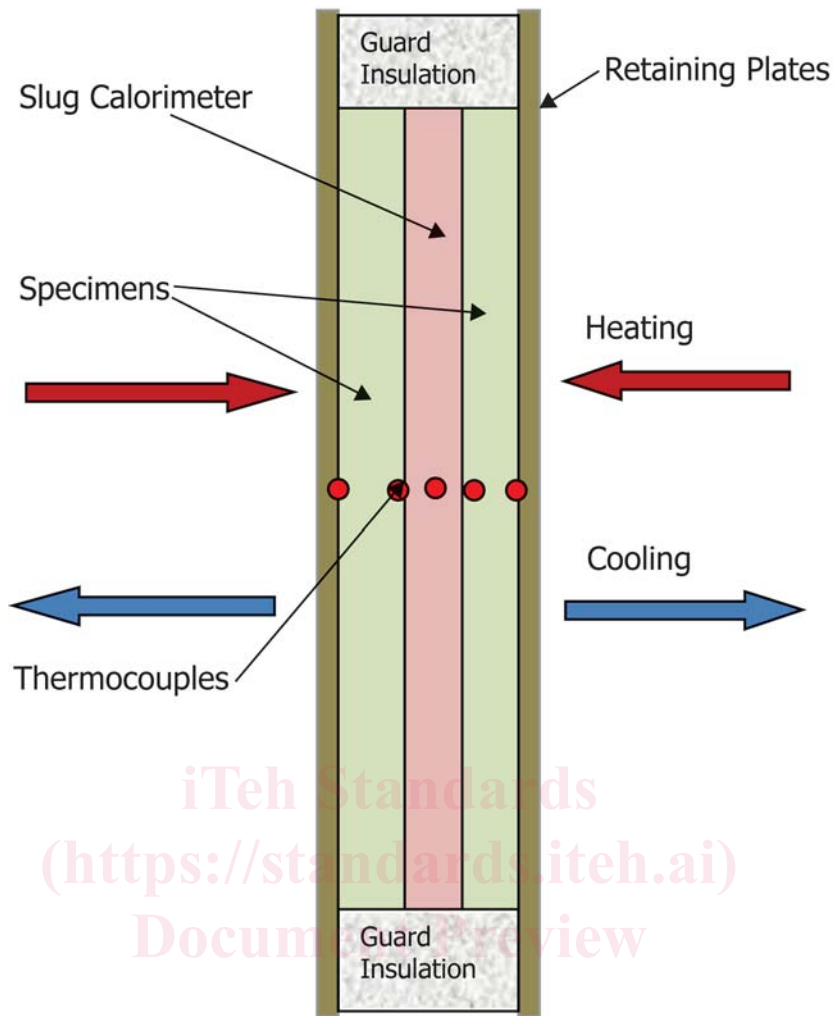


FIG. 2 Schematic of an Assembled Twin Specimen Slug Calorimeter Specimen Ready for Thermal Conductivity Testing

specimens can be used both for thermal conductivity and thermal diffusivity testing. 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 is provided in Fig. 3. 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

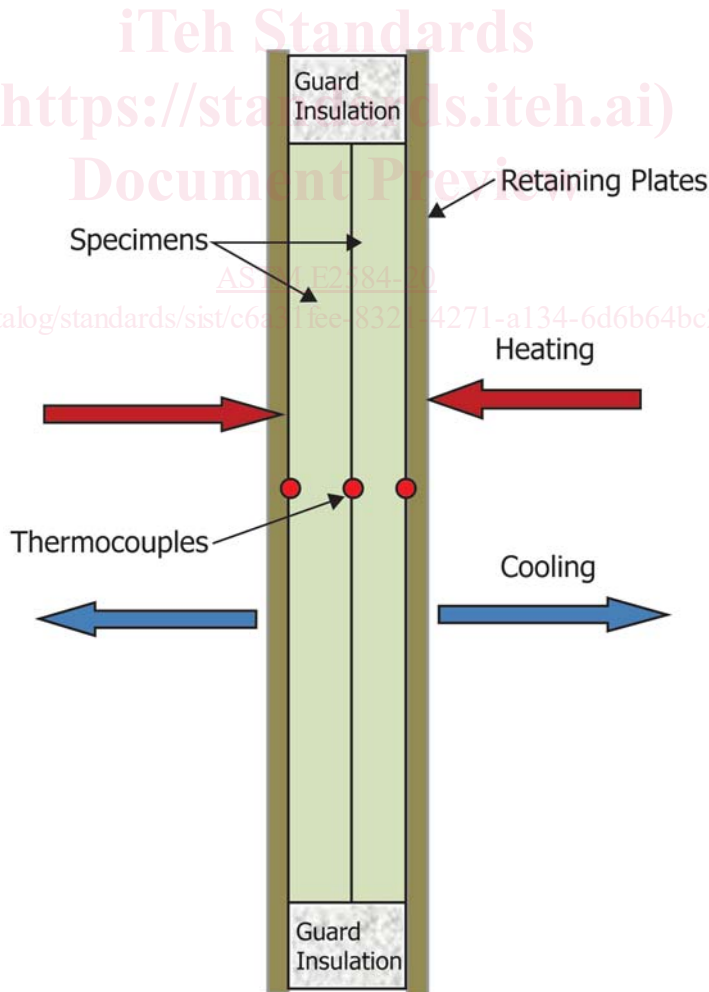


FIG. 3 Schematic of an Assembled Twin Specimen Ready for Thermal Diffusivity Testing

temperature-controlled environment reaches some pre-defined endpoint temperature (1273 K for instance), depending on the purpose of the test being conducted.

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.

10.4 To provide quasi-steady state temperature field in the assembly sample- slug calorimeter at the requested temperature range, for example, from room temperature, the assembly can be cooled below the room temperature and the initial stage of heating have to be excluded during calculations.

11. Calculation

11.1 Double-Sided Configuration for Thermal Conductivity Testing:

11.1.1 The specific heat is known. 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}). If known specific heat of the sample the apparent thermal conductivity, λ_a , at each mean specimen temperature is then calculated as:

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

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 pro-

vided in graphical form in Fig. 4, 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:

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

11.3 The thermal diffusivity is known. The apparent thermal conductivity, λ_a , is calculated as:

$$\lambda_a = \frac{FLM_{SSS}C_p^{SSS}}{A\left(\Delta T - \frac{FL^2}{2\alpha_a}\right)} \quad (3)$$

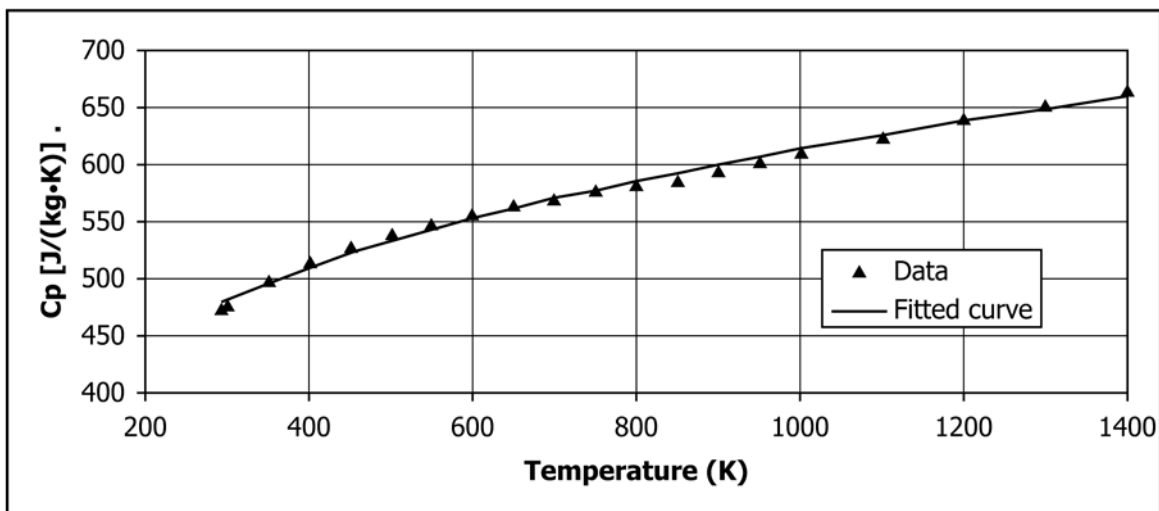
NOTE 4—In the Refs (3, 4) is suggested a method of calculation and software, which allow to decrease initial stage of the process if surface temperature increases with constant rate and no latent heat effects. The method can be applied if no latent heat effects during heating/cooling.

11.4 Thermal Diffusivity Testing:

11.4.1 At each sampling time, the mean specimen temperature (T_{mean}^{SPEC}) is calculated and the mean exterior specimen temperature (T_{outer}^{SPEC}). The apparent thermal diffusivity, α_a , at each mean specimen temperature is then calculated as:

$$\alpha_a = \frac{F_L^2}{2\Delta T} \quad (4)$$

11.5 The correction to the Eq 1-4 or errors can be calculated based on the theory of monotonic heating (see Appendix X1).



NOTE 1—Fitted curve is of the form:

$$C_p^{SSS} = 6.683 + 0.04906 * T + 80.74 * \ln(T)$$

with T in K.

FIG. 4 Literature Values (2) and Fitted Curve for Specific Heat Capacity of 304 Stainless Steel