<span id="page-0-0"></span>

# **Standard Test Method for Measuring Heat-Transfer Rate Using a Thermal Capacitance (Slug) Calorimeter<sup>1</sup>**

This standard is issued under the fixed designation E457; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

#### **1. Scope**

1.1 This test method describes the measurement of heat transfer rate using a thermal capacitance-type calorimeter which assumes one-dimensional heat conduction into a cylindrical piece of material (slug) with known physical properties.

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

NOTE 1—For information see Test Methods E285, E422, E458, E459, and E511.

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:*<sup>2</sup>

- E285 [Test Method for Oxyacetylene Ablation Testing of](http://dx.doi.org/10.1520/E0285) [Thermal Insulation Materials](http://dx.doi.org/10.1520/E0285)
- E422 [Test Method for Measuring Heat Flux Using a Water-](http://dx.doi.org/10.1520/E0422)[Cooled Calorimeter](http://dx.doi.org/10.1520/E0422)
- E458 [Test Method for Heat of Ablation](http://dx.doi.org/10.1520/E0458)
- E459 [Test Method for Measuring Heat Transfer Rate Using](http://dx.doi.org/10.1520/E0459) [a Thin-Skin Calorimeter](http://dx.doi.org/10.1520/E0459)
- E511 [Test Method for Measuring Heat Flux Using a Copper-](http://dx.doi.org/10.1520/E0511)[Constantan Circular Foil, Heat-Flux Transducer](http://dx.doi.org/10.1520/E0511)

# **3. Summary of Test Method**

3.1 The measurement of heat transfer rate to a slug or thermal capacitance type calorimeter may be determined from the following data:

3.1.1 Density and specific heat of the slug material,

3.1.2 Length or axial distance from the front face of the cylindrical slug to the back-face thermocouple,

3.1.3 Slope of the temperature—time curve generated by the back-face thermocouple, and

3.1.4 Calorimeter temperature history.

3.2 The heat transfer rate is thus determined numerically by multiplying the density, specific heat, and length of the slug by the slope of the temperature–time curve obtained by the data acquisition system (see Eq 1).

3.3 The technique for measuring heat transfer rate by the thermal capacitance method is illustrated schematically in [Fig.](#page-1-0) [1.](#page-1-0) The apparatus shown is a typical slug calorimeter which, for example, can be used to determine both stagnation region heat transfer rate and side-wall or afterbody heat transfer rate values. The annular insulator serves the purpose of minimizing heat transfer to or from the body of the calorimeter, thus approximating one-dimensional heat flow. The body of the calorimeter is configured to establish flow and should have the same size and shape as that used for ablation models or test specimens.

3.3.1 For the control volume specified in this test method, a thermal energy balance during the period of initial linear temperature response where heat losses are assumed negligible can be stated as follows:

> Energy Received by the Calorimeter (front face)  $=$  Energy Conducted Axially Into the Slug

$$
q_c = \rho C_p l \left( \Delta T / \Delta \tau \right) = \left( M C_p / A \right) \left( \Delta T / \Delta \tau \right) \tag{1}
$$

where:

 $\dot{q}_c$  = calorimeter heat transfer rate,  $\frac{W}{m^2}$ ,

- $p^2$  = density of slug material, kg/m<sup>3</sup>,
- $\rho$  = density of slug material, kg/m<sup>3</sup>,<br>  $C_p$  = average specific heat of slug material during the temperature rise (∆*T*), J/kg·K,
- $l =$  length or axial distance from front face of slug to the thermocouple location (back-face), m,
- $\Delta T$  =  $(T_f T_i)$  = calorimeter slug temperature rise during exposure to heat source (linear part of curve), K,
- $\Delta \tau$  = ( $\tau_f \tau_i$ ) = time period corresponding to  $\Delta T$  temperature rise, s,
- $M$  = mass of the cylindrical slug, kg,
- $A = \csc{c}$  is extended area of slug, m<sup>2</sup>.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee [E21](http://www.astm.org/COMMIT/COMMITTEE/E21.htm) on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee [E21.08](http://www.astm.org/COMMIT/SUBCOMMIT/E2108.htm) on Thermal Protection.

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<sup>&</sup>lt;sup>2</sup> 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.

<span id="page-1-0"></span>

**FIG. 1 Schematic of a Thermal Capacitance (Slug) Calorimeter**

In order to determine the steady-state heat transfer rate with a thermal capacitance-type calorimeter, [Eq 1](#page-0-0) must be solved by using the known properties of the slug material<sup>3</sup> (for example, density and specific heat)—the length of the slug, and the slope (linear portion) of the temperature–time curve obtained during the exposure to a heat source. The initial and final temperature transient effects must be eliminated by using the initial linear portion of the curve (see [Fig. 2\)](#page-2-0).

3.3.2 In order to calculate the initial response time for a given slug, Eq 2 may be used. $4$  This equation is based on the idealization of zero heat losses from slug to its holder.

$$
\tau_R = \frac{l^2 \rho C_p}{k \pi^2} ln \left( \frac{2}{1 - \frac{q \text{ indicated}}{q \text{ input}}} \right)
$$
 (2)

where:

 $k =$  thermal conductivity of slug material, W/m·K  $q_{indicated}$  = *q* that would be measured at the back-face of the slug by Eq 1,  $W/m^2$ 

 $q_{\text{input}}$  = constant  $q_{\text{input}}$  at the front-face of the slug beginning at  $\tau = 0$ , W/m<sup>2</sup>

3.3.3 Although the goal of good slug calorimeter design is to minimize heat losses, there can be heating environments, such as very high heat fluxes, where even a good slug calorimeter design cannot meet the recommended 5 % maxi-mum heat loss criterion of [6.1.](#page-3-0) Also, this criterion only deals with heat losses measured during the cooling phase, not losses during the heating phase, which can be greater than the cooling losses. Under these circumstances, significant heat losses from slug to holder during the heating phase, as well as other possible decaying processes such as a drop in surface catalycity, can cause the Temperature-Time slope to decrease significantly more than can be accounted for by the increasing heat capacity with temperature of the Copper slug alone, making it important that the slope be taken early in the process before the losses lower the slope too much, introducing more error to the downside on the heat flux calculated (see [Fig. 3\)](#page-2-0). The degree of losses affect the exact position where the best slope begins to occur, but typically it should be expected at about time  $\tau = \tau_R$  calculated by Eq 2 for  $q_{indicated}/q_{input} = 0.99$ , which value of  $\tau_R$  is abbreviated as  $\tau_{R0.99}$ . [Fig. 2](#page-2-0) and [Fig. 3](#page-2-0) assume that "heat source on" is a step function. This is an idealization, but the reality can be significantly different. For example, in some cases a calorimeter may experience a higher heat flux prior to reaching its final position in the heat source, which can cause the initial maximum slope to be higher than what is wanted for the calculation of the heat flux at the final position. Therefore, it is important to note that "zero" time, to which  $\tau_{R0.99}$  is added to determine where to start looking for the desired slope, is when the calorimeter has reached its final position where it is desired to measure the heat flux. Therefore, choosing the best place to take the slope can be very important. Should more accurate results be required, the losses form the slug should be modeled and accounted for by a correction term in the energy balance equation. $5$ 

3.3.4 For maximum linear test time (temperature–time curve) within an allowed surface temperature limit, the relation shown as Eq 3 may be used for a calorimeter which is insulated by a gap at the back face.<sup>6</sup>

$$
\tau_{\text{max,opt.}} = 0.48 \text{ p}l C_p \left(\Delta T_{\text{frontface}}/q\right) \tag{3}
$$

<sup>&</sup>lt;sup>3</sup> "Thermophysical Properties of High Temperature Solid Materials," TPRC, Purdue University, or "Handbook of Thermophysical Properties," Tolukian and Goldsmith, MacMillan Press, 1961.

<sup>4</sup> Ledford, R. L., Smotherman, W. E., and Kidd, C. T., "Recent Developments in Heat-Transfer Rate, Pressure, and Force Measurements for Hotshot Tunnels," AEDC-TR-66-228 (AD645764), January 1967.

<sup>5</sup> Childs, P. R. N., Greenwood, J. R., and Long, C. A., "Heat flux measurement techniques," *Proceedings of the Institution of Mechanical Engineers*, Vol 213, Part C, 1999, pp. 664–665.

<sup>6</sup> Kirchhoff, R. H., "Calorimetric Heating-Rate Probe for Maximum-Response-Time Interval," *American Institute of Aeronautics and Astronautics Journal*, AIAJA, Vol 2, No. 5, May 1964, pp. 966–67.



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 $\tau$ , TIME  $\rightarrow$ **FIG. 2 Typical Temperature–Time Curve for Slug Calorimeter**



**FIG. 3 Temperature–Time Curve when Heat and Other Items are Significant During Heating Phase**

<span id="page-3-0"></span>where:

 $\Delta T$ <sub>front face = the calorimeter final front face temperature</sub> minus the initial front face (ambient) temperature,  $T_{\alpha}$ .

3.3.5 [Eq 3](#page-1-0) is based on the optimum length of the slug which can be obtained by applying Eq 4 as follows:

$$
l_{\text{opt.}} = 3 k \Delta T_{\text{front face}} / 5 \dot{q}_c \tag{4}
$$

3.4 To minimize side heating or side heat losses, the body is separated physically from the calorimeter slug by means of an insulating gap or a low thermal diffusivity material, or both. The insulating gap that is employed should be small, and recommended to be no more than 0.05 mm on the radius. Thus, if severe pressure variations exist across the face of the calorimeter, side heating caused by flow into or out of the insulation gap would be minimized. Depending on the size of the calorimeter surface, variations in heat transfer rate may exist across the face of the calorimeter; therefore, the measured heat transfer rate represents an average heat transfer rate over the surface of the slug.

3.5 Since interpretation of the data obtained by this test method is not within the scope of this discussion, such effects as surface recombination and thermo-chemical boundary layer reactions are not considered in this test method.

3.6 If the thermal capacitance calorimeter is used to measure only radiative heat transfer rate or combined convective/ radiative heat transfer rate values, the surface reflectivity of the calorimeter should be measured over the wavelength region of interest (depending on the source of radiant energy).

## **4. Significance and Use**

4.1 The purpose of this test method is to measure the rate of thermal energy per unit area transferred into a known piece of material (slug) for purposes of calibrating the thermal environment into which test specimens are placed for evaluation. The calorimeter and holder size and shape should be identical to that of the test specimen. In this manner, the measured heat transfer rate to the calorimeter can be related to that experienced by the test specimen.

4.2 The slug calorimeter is one of many calorimeter concepts used to measure heat transfer rate. This type of calorimeter is simple to fabricate, inexpensive, and readily installed since it is not water-cooled. The primary disadvantages are its short lifetime and relatively long cool-down time after exposure to the thermal environment. In measuring the heat transfer rate to the calorimeter, accurate measurement of the rate of rise in back-face temperature is imperative.

4.3 In the evaluation of high-temperature materials, slug calorimeters are used to measure the heat transfer rate on various parts of the instrumented models, since heat transfer rate is one of the important parameters in evaluating the performance of ablative materials.

4.4 Regardless of the source of thermal energy to the calorimeter (radiative, convective, or a combination thereof) the measurement is averaged over the calorimeter surface. If a significant percentage of the total thermal energy is radiative, consideration should be given to the emissivity of the slug surface. If non-uniformities exist in the input energy, the heat transfer rate calorimeter would tend to average these variations; therefore, the size of the sensing element (that is, the slug) should be limited to small diameters in order to measure local heat transfer rate values. Where large ablative samples are to be tested, it is recommended that a number of calorimeters be incorporated in the body of the test specimen such that a heat transfer rate distribution across the heated surface can be determined. In this manner, more representative heat transfer rate values can be defined for the test specimen and thus enable more meaningful interpretation of the test. The slug selection may be determined using the nomogram as a guide (see [Appendix X1\)](#page-4-0).

## **5. Apparatus**

5.1 *General—*The apparatus shall consist of a thermal capacitance (slug) calorimeter and the necessary instrumentation to measure the thermal energy transferred to the calorimeter. All calculations should use only those data taken after the heat source has achieved steady-state operating conditions. Wherever possible, it is desirable that several measurements be made of the required parameters.

5.2 *Back-Face Temperature Measurement—*The method of temperature measurement must be sufficiently sensitive and reliable to ensure accurate temperature rise data for the back-face thermocouple. Procedures should be adhered to in the calibration and preparation of the thermocouples. Attachment of the thermocouples should be such that the true back-side temperatures are obtained. Although no standardized procedures are available, methods such as resistance welding (small spot) and peening have been successfully used. The error in measurement of temperature difference between the initial and final times should not exceed  $\pm 2$  %. The temperature measurements shall be recorded continuously using a commercially available recorder whose frequency response is at least ten times the expected frequency response of the slug to provide the accuracy required. During the course of operation of the plasma arc or other heat source, care must be taken to minimize deposits on the calorimeter surface.

5.3 *Data Acquisition—*The important parameter, back-face temperature rise, shall be automatically recorded throughout the calibration period. Recording speed will depend on the heat transfer rate level such that the time range shall approach the temperature rise displacement on the recording paper. Timing marks shall be an integral part of the recorder output.

#### **6. Procedure**

6.1 It is essential that the thermal energy source (environment) be at steady-state conditions prior to testing if the thermal capacitance calorimeter is to produce representative heat transfer rate measurements. Make a millivolt scale calibration of the recorder prior to exposure of the calorimeter to the environment. With the recorder operating at the proper speed (see 4.3), expose the calorimeter to the thermal environment as rapidly as possible. After removal from the thermal environment, record the back-face temperature for sufficient time to determine the heat loss rate from the slug. Significant differences between the maximum and post-test values may

<span id="page-4-0"></span>indicate heat conduction losses to the calorimeter body. If feasible, obtain more than one measurement with more than one test method for a given thermal environment. To ensure that energy losses are minimized, the cooling rate slope should compare with the heating rate slope according to the following equation:

$$
(\Delta T/\Delta \tau) \text{ cooling} \le 0.05 \, (\Delta T/\Delta \tau) \text{ heating} \tag{5}
$$

# **7. Heat Transfer Rate Calculation**

7.1 The quantities as defined by [Eq 1](#page-0-0) shall be calculated based on the physical properties of the slug material, dimensions of the slug, and the slope of the temperature–time curve of the calorimeter. The choice of units shall be consistent with the measured quantities.

7.2 An uncertainty analysis shall be performed according to the standard NIST  $TN-1297$ .<sup>7</sup> Both Type A and Type B uncertainties shall be included in the analysis. The heat transfer rate shall be reported with its total uncertainty at a stated confidence level. Values that went into the uncertainty analysis, including those derived from calibration reports and manufacturers' specifications, as well as any assumptions or estimates, shall be documented.

## **8. Report**

8.1 Report the following information:

8.1.1 Physical properties of the slug material,

8.1.2 Configuration of the calorimeter body,

8.1.3 Dimensions of the slug,

8.1.4 Slope of the temperature–time curve (linear portion), both heating and cooling histories,

8.1.5 Calculated (apparent) heat transfer rate,

8.1.6 Corrected (for losses) heat transfer rate for increased accuracy if required, and

8.1.7 Uncertainty of results.

#### **9. Keywords**

9.1 calorimeter; heat transfer rate; slug calorimeter; thermal capacitance

# **APPENDIX**

## **(Nonmandatory Information)**

## **X1. USE OF THE CALORIMETER SELECTION NOMOGRAM**

X1.1 The calorimeter selection nomogram presented in this Appendix may be used to assist instrumentation personnel in choosing the appropriate calorimeter material, exposure time, front-face (surface) temperature rise for a given heat transfer rate, or any other combination of these parameters. This graphical method is intended as a guideline, not as a design criteria, and therefore should be used with an understanding of the basic test method for thermal capacitance (slug) calorimeters.

X1.2 The time from initial heat, τ, determined using the nomogram, will indicate the total exposure time, and not necessarily the optimum value. Average values of specific heat,  $C_p$ , thermal conductivity, *k*, and density, *ρ*, have been used in order to present a simple graphical representation of the basic equation below:

$$
\tau = \pi (\rho k C_p) \left(\frac{\Delta T}{2 \dot{q}c}\right)^2 \tag{X1.1}
$$

X1.3 For the slug to provide accurate results, the slope of the temperature-time curve must be obtained within the linear portion of the curve as defined by the following equation:

$$
l^2/[2(k/\rho C_p)] \le \tau \le 100 l^2/(k/\rho C_p) \tag{X1.2}
$$

NOTE X1.1—The upper limit of the operating range is reduced by a factor of up to 100, if the calorimeter back face is in contact with a solid insulating material.

X1.4 To use the calorimeter selection nomogram (see [Fig.](#page-5-0) [X1.1\)](#page-5-0), the known (or assumed) parameters must be noted on the appropriate scales (A, B, C, or D). A straight line must connect scales A and D, while another straight line connects scales B and C. The crossover line (without numbers) provides the pivot point for the two straight lines, as both must be coincident on the crossover line.

<sup>7</sup> Taylor, B. N., and Kuyatt, C. E., *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, September 1994.

<span id="page-5-0"></span>**E457 − 08 (2015)**  $\overline{B}$  $\mathsf{C}$  $\overline{A}$  $\mathsf{D}$ Fused Silica  $4000 \cdot$ 3  $-200$ 3000  $\pm 100$ E 2500  $-50$ 2000  $10<sup>6</sup>$  $\cdot 10^6$ 20 უ 1500  $-\text{deg}$ crossover line  $10$  $1.5$ 1000 Time from initial heating, pkCp), Slug material parameter, W<sup>2</sup> - s/m<sup>4</sup> 5  $\overline{\phantom{a}}$ 3  $\cup$ AT, Surface temperature rise, deg  $\overline{2}$ Heat-transfer rate, W/m<sup>2</sup>  $-1.0$  $500$ 400  $-0.5$ St.Stl 300  $-0.2$ – Ta<br>1020 Stl  $-0.10$  $1.5$  $8C$ <br>  $W$ 200  $\overline{2}$  $-0.05$  $10<sup>3</sup>$ Pt 150 3  $0.02$ Ni  $1.5$ 100  $0.01$ Al  $\cdot$  2 Au Example: Copper 50 Ag If  $\dot{q}_c = 5 \times 10^7$  W/m<sup>2</sup> 40 Cu  $1.5$ and  $\Delta T = 500$  deg C 30 and  $pkC_p = 1.45 \times 10^9$  w<sup>2</sup> – s/m<sup>4</sup> – deg C<sup>2</sup>  $(\rho kC_P)$   $\pi$ then 20 thus  $\tau = 0.11$  s **FIG. X1.1 Slug Selection Nomogram**

#### **NON-CITED REFERENCES**

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- **(2)** Coleman, H. W., and Steele, W. G., *Experimentation and Uncertainty Analysis for Engineers*, Second Edition, John Wiley & Sons, Inc., New York, NY, 1999.

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