



Designation: E1461 – 11

## Standard Test Method for Thermal Diffusivity by the Flash Method<sup>1</sup>

This standard is issued under the fixed designation E1461; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 This test method covers the determination of the thermal diffusivity of primarily homogeneous isotropic solid materials. Thermal diffusivity values ranging from  $10^{-7}$  to  $10^{-3}$   $\text{m}^2\cdot\text{s}^{-1}$  are measurable by this test method from about 75 to 2800 K.

1.2 Practice E2585 is adjunct to this Test Method and contains detailed information regarding the use of the flash method. The two documents are complementing each other.

1.3 This test method is a more detailed form of Test Method C714, having applicability to much wider ranges of materials, applications, and temperatures, with improved accuracy of measurements.

1.4 This test method is intended to allow a wide variety of apparatus designs. It is not practical in a test method of this type to establish details of construction and procedures to cover all contingencies that might offer difficulties to a person without pertinent technical knowledge, or to stop or restrict research and development for improvements in the basic technique.

1.5 This test method is applicable to the measurements performed on essentially fully dense (preferably, but low porosity would be acceptable), homogeneous, and isotropic solid materials that are opaque to the applied energy pulse. Experience has shown, however, that some deviation from these strict guidelines can be accommodated with care and proper experimental design, substantially broadening the usefulness of the method.

1.6 This test method can be considered an absolute (or primary) method of measurement, since no reference standards are required. It is advisable to use reference materials to verify the performance of the instrument used.

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

1.8 For systems employing lasers as power sources, it is imperative that the safety requirement be fully met.

1.9 *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>

C714 Test Method for Thermal Diffusivity of Carbon and Graphite by Thermal Pulse Method

E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

E2585 Practice for Thermal Diffusivity by the Flash Method

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *thermal conductivity,  $\lambda$ , of a solid material*—the time rate of steady heat flow through unit thickness of an infinite slab of a homogeneous material in a direction perpendicular to the surface, induced by unit temperature difference. The property must be identified with a specific mean temperature, since it varies with temperature.

3.1.2 *thermal diffusivity,  $\alpha$ , of a solid material*—the property given by the thermal conductivity divided by the product of the density and heat capacity per unit mass.

3.2 *Description of Symbols and Units Specific to This Standard:*

3.2.1  $D$ —diameter, m.

3.2.2  $C_p$ —specific heat capacity,  $\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ ).

3.2.3  $k$ —constant depending on percent rise.

3.2.4  $K$ —correction factors.

3.2.5  $K_1, K_2$ —constants depending on  $\beta$ .

3.2.6  $L$ —specimen thickness, m.

3.2.7  $t$ —response time, s.

<sup>1</sup> This test method 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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<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.

3.2.8  $t_{1/2}$ —half-rise time or time required for the rear face temperature rise to reach one half of its maximum value, s.

3.2.9  $t^*$ —dimensionless time ( $t^* = 4\alpha_s t/D_T^2$ ).

3.2.10  $T$ —temperature, K.

3.2.11  $\alpha$ —thermal diffusivity,  $m^2 \cdot s^{-1}$ .

3.2.12  $\beta$ —fraction of pulse duration required to reach maximum intensity.

3.2.13  $\rho$ —density,  $kg \cdot m^{-3}$ .

3.2.14  $\lambda$ —thermal conductivity,  $W \cdot m^{-1} \cdot K^{-1}$ .

3.2.15  $\Delta t_5 - T(5t_{1/2}) / T(t_{1/2})$ .

3.2.16  $\Delta t_{10} - T(10t_{1/2}) / T(t_{1/2})$ .

3.2.17  $\Delta T_{max}$ —temperature difference between baseline and maximum rise, K.

3.3 *Description of Subscripts Specific to This Standard:*

3.3.1  $o$ —ambient.

3.3.2  $s$ —specimen.

3.3.3  $T$ —thermocouple.

3.3.4  $x$ —percent rise.

3.3.5  $C$ —Cowan.

3.3.6  $R$ —ratio.

3.3.7  $m$ —maximum.

3.3.8  $t$ —time.

#### 4. Summary of Test Method

4.1 A small, thin disc specimen is subjected to a high-intensity short duration radiant energy pulse (Fig. 1). The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear face temperature rise (thermogram) is recorded. The thermal diffusivity value is calculated from the specimen thickness and the time required for the rear face temperature rise to reach certain percentages of its maximum value (Fig. 2). When the thermal diffusivity of the specimen is to be determined over a temperature range, the measurement must be repeated at each temperature of interest. This test method is described in detail in a number of publications (1, 2)<sup>3</sup> and review articles (3, 4, 5). A summary of the theory can be found in Appendix X1.

<sup>3</sup> The boldface numbers given in parentheses refer to a list of references at the end of the text.

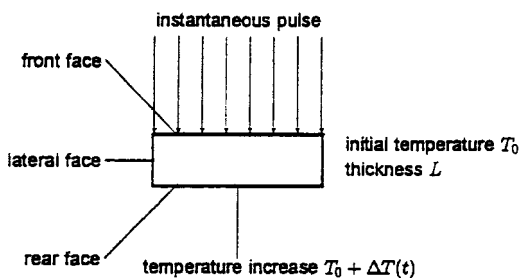


FIG. 1 Schematic of the Flash Method

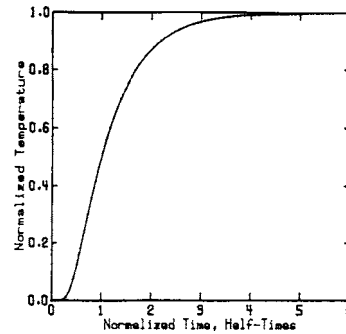


FIG. 2 Characteristic Thermogram for the Flash Method

#### 5. Significance and Use

5.1 Thermal diffusivity is an important property, required for such purposes under transient heat flow conditions, such as design applications, determination of safe operating temperature, process control, and quality assurance.

5.2 The flash method is used to measure values of thermal diffusivity,  $\alpha$ , of a wide range of solid materials. It is particularly advantageous because of simple specimen geometry, small specimen size requirements, rapidity of measurement and ease of handling..

5.3 Under certain strict conditions, specific heat capacity of a homogeneous isotropic opaque solid specimen can be determined when the method is used in a quantitative fashion (see Appendix X2).

5.4 Thermal diffusivity results, together with related values of specific heat capacity ( $C_p$ ) and density ( $\rho$ ) values, can be used in many cases to derive thermal conductivity ( $\lambda$ ), according to the relationship:

$$\lambda = \alpha C_p \rho. \quad (1)$$

#### 6. Interferences

6.1 In principle, the thermal diffusivity is obtained from the thickness of the specimen and from a characteristic time function describing the propagation of heat from the front surface of the specimen to its back surface. The sources of uncertainties in the measurement are associated with the specimen itself, the temperature measurements, the performance of the detector and of the data acquisition system, the data analysis and more specifically the finite pulse time effect, the nonuniform heating of the specimen and the heat losses (radiative and conductive). These sources of uncertainty can be considered systematic, and should be carefully considered for each experiment. Errors random in nature (noise, for example) can be best estimated by performing a large number of repeat experiments. The relative standard deviation of the obtained results is a good representation of the random component of the uncertainty associated with the measurement. Guidelines in performing a rigorous evaluation of these factors are given in (31).

**7. Apparatus**

The essential components of the apparatus are shown in Fig. 3. These are the flash source, specimen holder, environmental enclosure (optional), temperature response detector and recording device.

7.1 The flash source may be a pulse laser, a flash lamp, or other device capable to generate a short duration pulse of substantial energy. The duration of the pulse should be less than 2 % of the time required for the rear face temperature rise to reach one half of its maximum value (see Fig. 2), to keep the error due to finite pulse width less than 0.5 %, if pulse width correction (17, 18, 19) is not applied.

7.1.1 The energy of the pulse hitting the specimen’s surface must be spatially uniform in intensity.

7.2 An environmental control chamber is required for measurements above and below room temperature.

7.3 The detector can be a thermocouple, infrared detector, optical pyrometer, or any other sensor that can provide a linear electrical output proportional to a small temperature rise. It shall be capable of detecting 0.05 K change above the specimen’s initial temperature. The detector and its associated amplifier must have a response time not more than 2 % of the half-rise time value.

7.4 The signal conditioner includes the electronic circuit to bias out the ambient temperature reading, spike filters, amplifiers, and analog-to-digital converters.

**7.5 Data Recording:**

7.5.1 The data acquisition system must be of an adequate speed to ensure that time resolution in determining half of the maximum temperature rise on the thermogram is at least 1 %, for the fastest thermogram for which the system is qualified.

7.6 Measurement of specimen’s temperature is to be done by accepted means, such as calibrated thermocouple, optical pyrometer, platinum RTD, etc. whichever is appropriate for the temperature range. In all cases, such a device must be in intimate contact with or trained on the sample holder, in close proximity of the specimen. Touching the specimen with thermocouples is not recommended. Embedding thermocouples into the specimen is not acceptable.

7.7 The temperature controller and/or programmer are to bring the specimen to the temperatures of interest.

**8. Test Specimen**

8.1 The usual specimen is a thin circular disc with a front surface area less than that of the energy beam. Typically, specimens are 10 to 12.5 mm in diameter (in special cases, as small as 6 mm diameter and as large as 30 mm diameter have been reported as used successfully). The optimum thickness depends upon the magnitude of the estimated thermal diffusivity, and should be chosen so that the time to reach half of the maximum temperature falls within the 10 to 1000 ms range. Thinner specimens are desired at higher temperatures to minimize heat loss corrections; however, specimens should always be thick enough to be representative of the test material. Typically, thicknesses are in the 1 to 6 mm range.

8.2 Specimens must be prepared with faces flat and parallel within 0.5 % of their thickness, in order to keep the error in thermal diffusivity due to the measurement average thickness, to less than 1 %. Non-uniformity of either surface (craters, scratches, markings) of significant depth compared to the specimen thickness should be avoided

8.3 *Specimen Surface Preparation*—It is a good practice to apply a very thin, uniform graphite or other high emissivity coating on both faces of the specimen to be tested, prior to performing the measurements. The coating may be applied by spraying, painting, sputtering, etc. This will improve the capability of the specimen to absorb the energy applied, especially in case of highly reflective materials. For transparent materials, a layer of gold, silver, or other opaque materials must be deposited first, followed by graphite coating. For some opaque reflective materials, grit blasting of the surface can provide sufficient pulse absorption and emissivity, especially at higher temperatures, where coatings may not be stable or may react with the material.

**9. Calibration and Verification**

9.1 Calibrate the micrometer used to measure the specimen thickness, so that the thickness measurements are accurate to within 0.2 %.

9.2 The Flash Method is an absolute (primary) method by itself, therefore it requires no calibration. However, actual execution of the measurement itself is subject to random and systematic errors. It is therefore important to periodically verify the performance of a device, to establish the extent these errors may affect the data generated. This can be accomplished by testing one or several materials whose thermal diffusivity is well known. While most materials used are not true certified

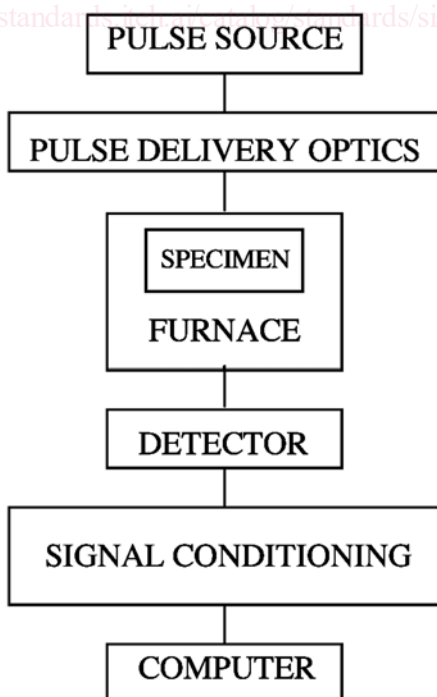


FIG. 3 Block Diagram of a Flash System

standards, they are generally accepted industry-wide with the best available literature data (see [Appendix X3](#)).

9.2.1 It must be emphasized that the use of reference materials to establish validity of the data on unknown materials has often led to unwarranted statements on accuracy. The use of references is only valid when the properties of the reference (including half-rise times and thermal diffusivity values) are closely similar to those of the unknown and the temperature-rise curves are determined in an identical manner for the reference and unknown.

9.2.2 One important check of the validity of data (in addition to the comparison of the rise curve with the theoretical model), when corrections have been applied, is to vary the specimen thickness. Since the half times vary as  $L^2$ , decreasing the specimen thickness by one-half should decrease the half time to one-fourth of its original value. Thus, if one obtains the same thermal diffusivity value (appropriate heat loss corrections being applied) with representative specimens from the same material of significantly different thicknesses, the results can be assumed valid.

## 10. Procedure

10.1 For commercially produced systems, follow manufacturer's instructions.

10.2 The testing procedure must contain the following functions:

10.2.1 Determine and record the specimen thickness.

10.2.2 Mount the specimen in its holder.

10.2.3 Establish vacuum or inert gas environment in the chamber if necessary.

10.2.4 Determine specimen temperature unless the system will do it automatically.

10.2.5 Especially at low temperatures, use the lowest level of power for the energy pulse able to generate a measurable temperature rise, in order to ensure that the detector functions within its linear range.

10.2.6 After the pulse delivery, monitor the raw or processed thermogram to establish in-range performance. In case of multiple specimen testing, it is advisable (for time economy) to sequentially test specimens at the same temperature (including replicate tests) before proceeding to the next test temperature.

10.2.7 In all cases, the temperature stability (base line) prior and during a test must be verified either manually or automatically to be less than 4 % of the maximum temperature rise. Testing on a ramp is not recommended.

10.2.8 Determine the specimen ambient temperature and collect the base line, transient-rise and cooling data, and analyze the results according to [Section 11](#).

10.2.9 Change or program the specimen temperature as desired and repeat the data collection process to obtain measurements at each temperature.

10.2.10 If required, repeat the measurements at each temperature on the specimen's cooling or on its re-heating over the same cycle.

## 11. Calculation

11.1 Determine the baseline and maximum rise to give the temperature difference,  $\Delta T_{\max}$ . Determine the time required

from the initiation of the pulse for the rear face temperature to reach  $\Delta T_{1/2}$ . This is the half-rise time,  $t_{1/2}$ . Calculate the thermal diffusivity,  $\alpha$ , from the specimen thickness,  $L$ , in m and the half-rise time  $t_{1/2}$  in s, as follows:

$$\alpha = 0.13879 L^2 / t_{1/2} \quad (2)$$

Check the validity of the experiment by calculating  $\alpha$  at a minimum of two other points on the rise curve. The equation is as follows:

$$\alpha = k_x L^2 / t_x \quad (3)$$

where:

$t_x$  = the time required for the temperature rise to reach  $x$  percent of  $\Delta T_{\max}$ . Values of  $k_x$  are given in [Table 1](#).

11.1.1 Ideally, the calculated values of  $\alpha$  for different values of  $x$  should all be the same. If the values at 25, 50 and 75 %  $\Delta T_{\max}$  lie within  $\pm 2$  %, the overall accuracy is probably within  $\pm 5$  % at the half-rise time. If the  $\alpha$  values lie outside of this range, the response curve should be analyzed further to see if thermal radiation heat loss, finite-pulse time or non-uniform heating effects are present.

11.1.2 Thermal radiation heat loss effects are most readily determined from the temperature of the specimen and the rear-face temperature response after  $4t_{1/2}$ . The recommended procedure is to plot the experimental values of  $\Delta T / \Delta T_{\max}$  versus  $t / t_{1/2}$  along with the values for the theoretical model. Some numbers for the theoretical model are given in [Table 2](#).

11.1.3 A plot of the normalized experimental data and the theoretical model can be prepared readily on line with a computer-based data acquisition system or by preparing graphs using the tabulated values of  $\Delta T / \Delta T_{\max}$  and  $t / t_{1/2}$  and plotting the corresponding experimental data at several percent levels of the rise. All normalized experimental curves must pass through  $\Delta T / \Delta T_{\max} = 0.5$  and  $t / t_{1/2} = 1.0$ . Therefore, calculations including the 25 to 35 % and 65 to 80 % ranges are required to compare the experimental data with the theoretical curve.

11.1.4 Examples of the normalized plots for experiments that approximate the ideal case, in which both radiation heat losses and finite pulse time effect exist, are shown in [Figs. 4 and 5](#), and [Fig. 6](#). Various procedures for correcting for these effects are also given in [Refs. \(4, 15, 16, 17, 18, 19, 25, 26\)](#) and specific examples are given in [11.2 and 11.3](#).

11.1.5 The corrections can be minimized by the proper selection of specimen thickness. The finite pulse time effect decreases as the thickness is increased, while heat losses decrease as the thickness is reduced.

**TABLE 1 Values of the Constant  $k_x$  for Various Percent Rises**

$x(\%)$	$k_x$	$x(\%)$	$k_x$
10	0.066108	60	0.162236
20	0.084251	66.67	0.181067
25	0.092725	70	0.191874
30	0.101213	75	0.210493
33.33	0.106976	80	0.233200
40	0.118960	90	0.303520
50	...	...	...

TABLE 2 Values of Normalized Temperature Versus Time for Theoretical Model

$\Delta T/\Delta T_{max}$	$t/t_{1/2}$	$\Delta T/\Delta T_{max}$	$t/t_{1/2}$
0	0	0.7555	1.5331
0.0117	0.2920	0.7787	1.6061
0.1248	0.5110	0.7997	1.6791
0.1814	0.5840	0.8187	1.7521
0.2409	0.6570	0.8359	1.8251
0.3006	0.7300	0.8515	1.8981
0.3587	0.8030	0.8656	1.9711
0.4140	0.8760	0.8900	2.1171
0.4660	0.9490	0.9099	2.2631
0.5000	1.0000	0.9262	2.4091
0.5587	1.0951	0.9454	2.6281
0.5995	1.1681	0.9669	2.9931
0.6369	1.2411	0.9865	3.6502
0.6709	1.3141	0.9950	4.3802
0.7019	1.3871	0.9982	5.1102
0.7300	1.4601	...	...

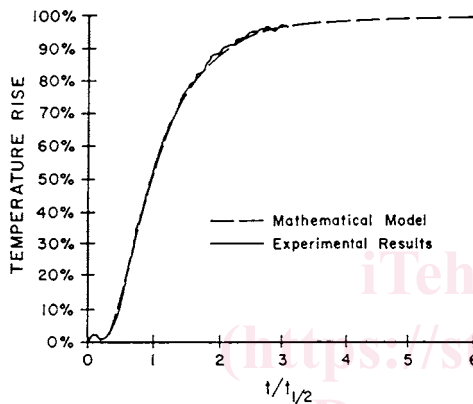


FIG. 4 Comparison of Non-dimensionalized Temperature Response Curve to Mathematical Model

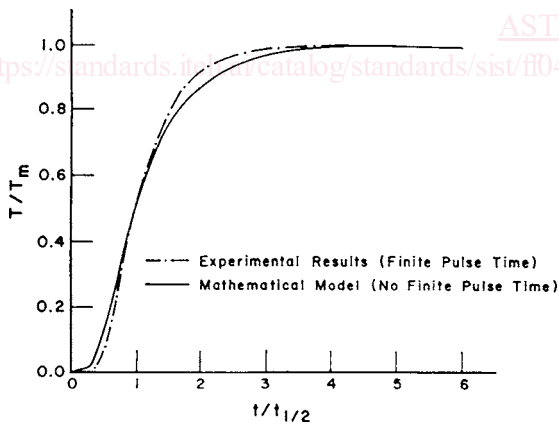


FIG. 5 Normalized Rear Face Temperature Rise: Comparison of Mathematical Model (No Finite Pulse Time Effect) to Experimental Values with Finite Pulse Time

11.1.6 Non-uniform heating effects also cause deviations of the reduced experimental curve from the model because of two-dimensional heat flow. Since there are a variety of non-uniform heating cases, there are a variety of deviations. Hot center cases approximate the radiation heat loss example. Cold center cases result in the rear face temperature continuing to rise significantly after  $4t_{1/2}$ . Non-uniform heating may arise from the nature of the energy pulse or by non-uniform

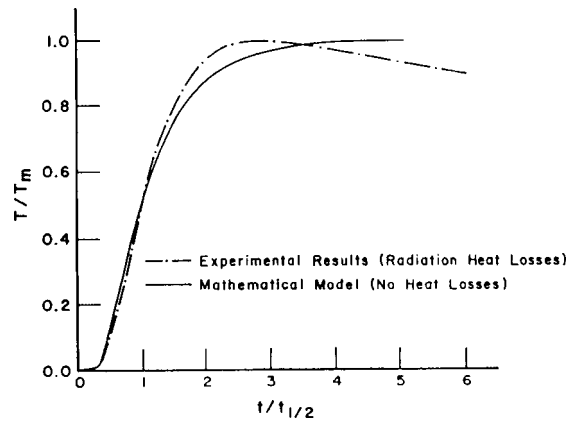


FIG. 6 Normalized Rear Face Temperature Rise: Comparison of Mathematical Model (No Heat Loss) to Experimental Values with Radiation Heat Losses

absorption on the front surface of the specimen. The former case must be eliminated by altering the energy source, while the latter may be eliminated by adding an absorbing layer and using two-layer mathematics (4, 13).

11.2 Finite pulse time effects usually can be corrected for using the equation:

$$\alpha = K_1 L^2 / (K_2 t_x - \tau). \tag{4}$$

For this to be valid, the evolution of the pulse intensity must be representable by a triangle of duration  $\tau$  and time to maximum intensity of  $\beta\tau$  as shown in Fig. 7. The pulse shape for the laser should be determined using an optical detector that can detect the laser pulse as opposed to the flash lamp pulse. From the pulse shape so determined,  $\beta$  and  $\tau$  are obtained. Values of the two constants  $K_1$  and  $K_2$  for various values of  $\beta$  are given in Table 3 for correcting  $\alpha_{0.5}$ .

11.3 Heat loss corrections can be performed using procedures proposed in a (25, 26), for example. Both of these corrections are affected by non-uniform heating effects. Corrections given in (25) by Cowan are affected by conduction heat losses to the holders in addition to the radiation heat losses from the specimen surfaces. Thus, the errors in the correction procedures are affected by different physical phenomena and a comparison of thermal diffusivity values corrected by the two procedures is useful in determining the presence or absence of these phenomena.

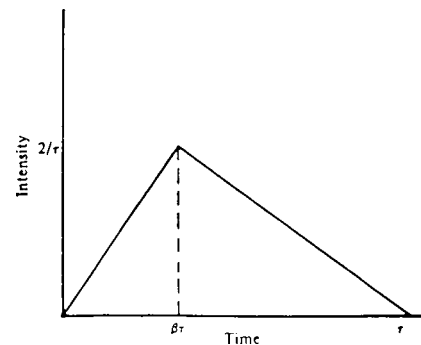


FIG. 7 Laser Pulse Shape

**TABLE 3 Finite Pulse Time Factors**

<i>b</i>	<i>K</i> <sub>1</sub>	<i>K</i> <sub>2</sub>
0.15	0.34844	2.5106
0.28	0.31550	2.2730
0.29	0.31110	2.2454
0.30	0.30648	2.2375
0.50	0.27057	1.9496

11.3.1 To use the Cowan procedure, determine the ratio of the net rise time values at times that are five and ten times the experimental half-rise time value to the net rise at the half-rise time value. These ratios are designated as  $\Delta t_5$  and  $\Delta t_{10}$ . If there are no heat losses  $\Delta t_5 = \Delta t_{10} = 2.0$ . The correction factors ( $K_C$ ) for the five and ten half-rise time cases are calculated from the polynomial fits:

$$K_C = A + B (\Delta t) + C (\Delta t)^2 + D (\Delta t)^3 + E (\Delta t)^4 + F (\Delta t)^5 + G (\Delta t)^6 + H (\Delta t)^7 \quad (5)$$

where:

values for the coefficients *A* through *H* are given in **Table 4**. Corrected values for thermal diffusivity are calculated from the following relation:

$$\alpha_{\text{corrected}} = \alpha_{0.5} K_C / 0.13885 \quad (6)$$

where:

$\alpha_{0.5}$  = the uncorrected thermal diffusivity value calculated using the experimental half-rise time.

11.3.2 Heat loss corrections based on the procedure given in Clark and Taylor (24) also use ratio techniques. For the  $t_{0.75}/t_{0.25}$  ratio, that is, the time to reach 75 % of the maximum divided by the time to reach 25 % of the maximum, the ideal value is 2.272. Determine this ratio from the experimental data. Then calculate the correction factor ( $K_R$ ) from the following equation:

$$K_R = -0.3461467 + 0.361578 (t_{0.75}/t_{0.25}) - 0.06520543 (t_{0.75}/t_{0.25})^2 \quad (7)$$

The corrected value for the thermal diffusivity at the half-rise time is  $\alpha_{\text{corrected}} = \alpha_{0.5} K_R / 0.13885$ . Corrections based on many other ratios can also be used.

11.4 If the measurements are performed at temperatures different from that where the specimen thickness has been determined, consider the presence of the linear thermal expansion effects. If these effects are not negligible, calculate the specimen thickness at each temperature and apply the usual procedure as described above.

**TABLE 4 Coefficients for Cowan Corrections**

Coefficients	Five Half Times	Ten Half Times
<i>A</i>	-0.1037162	0.054825246
<i>B</i>	1.239040	0.16697761
<i>C</i>	-3.974433	-0.28603437
<i>D</i>	6.888738	0.28356337
<i>E</i>	-6.804883	-0.13403286
<i>F</i>	3.856663	0.024077586
<i>G</i>	-1.167799	0.0
<i>H</i>	0.1465332	0.0

11.5 Other parameter estimation methods may also be used, provided detailed reference to the source is reported with the data.

## 12. Report

12.1 As a minimum, the report shall contain the following information:

12.1.1 Identification of the specimen (material) and previous history;

12.1.2 Specimen thickness (m);

12.1.3 Temperature (°C);

12.1.4 Calculated value of thermal diffusivity at  $x = 50\%$ ,  $\text{m}^2 \cdot \text{s}^{-1}$ , at the reported temperature;

12.1.5 Statements concerning calculated values near  $x = 25\%$  and  $75\%$  as well as  $x = 50\%$ , or a comparison of the reduced experimental curve to the model, at each temperature;

12.1.6 Statements concerning the results of repeat measurements at each temperature;

12.1.7 Statement as to whether or not the data was corrected for thermal expansion. If this correction was made, the thermal expansion values used must be reported;

12.1.8 Discussion of errors and correction procedures that were used for heat losses and finite pulse time effects;

12.1.9 Environmental surroundings of the specimen (gas type, pressure, etc.);

12.1.10 Statements of conformance with requirements of this standard.

12.2 Additionally, it is beneficial to report:

12.2.1 Statement that the response time of the detector, including the associated electronics was adequately checked, and the method used;

12.2.2 Energy pulse source;

12.2.3 Statement of the beam uniformity check, or methods employed to eliminate the need for any;

12.2.4 Type of temperature rise detector.

12.2.5 Manufacturer and model of the instrument used.

## 13. Precision and Bias

13.1 A number of national and international round robin testing programs have shown that a measurement precision of  $\pm 5\%$  can be attained for thermal diffusivity of a variety of materials. The results of several of these programs are detailed in a separate report on file with ASTM. No evidence of bias has been noted for opaque materials. Generally the values were obtained using simple data acquisitions and analysis. It has been shown that the accuracy can be significantly improved using more sophisticated data acquisition and data analysis.

13.2 The above precision levels do not imply that the specific heat capacity and thermal conductivity of the specimen can be derived to the same levels from thermal diffusivity measurements, since such derivations require input of values for other parameters.

13.3 Uncertainty analysis is to be performed for the instrument used for performing the measurements, and the results should be incorporated into the data analysis and reports.