Designation: C1784 − 20

Standard Test Method for
Using a Heat Flow Meter Apparatus for Measuring Thermal Storage Properties of Phase Change Materials and Products[1]

This standard is issued under the fixed designation C1784; 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 test method covers the measurement of non-steady-state heat flow into or out of a flat slab specimen to determine the stored energy (that is, enthalpy) change as a function of temperature using a heat flow meter apparatus (HFMA).

1.2 In particular, this test method is intended to measure the sensible and latent heat storage capacity for products incorporating phase-change materials (PCM).

1.2.1 The storage capacity of a PCM is well defined via four parameters: specific heats of both solid and liquid phases, phase change temperature(s) and phase change enthalpy (**1**).[2]

1.3 To more accurately predict thermal performance, information about the PCM products' performance under dynamic conditions is needed to supplement the properties (thermal conductivity) measured under steady-state conditions.

NOTE 1—This test method defines a dynamic test protocol for products or composites containing PCMs. Due to the macroscopic structure of these products or composites, small specimen sizes used in conventional Differential Scanning Calorimeter (DSC) measurements, as specified in E793 and E967, are not necessarily representative of the relationship between temperature and enthalpy of full-scale PCM products.

1.4 This test method is based upon the HFMA technology used for Test Method C518 but includes modifications for specific heat and enthalpy change measurements for PCM products as outlined in this test method.

1.5 Heat flow measurements are required at both the top and bottom HFMA plates for this test method. Therefore, this test method applies only to HFMAs that are equipped with at least one heat flux transducer on each of the two plates and that have the capability for computerized data acquisition and temperature control systems. Further, the amount of energy flowing through the transducers must be measureable at all points in time. Therefore, the transducer output shall never be saturated during a test.

1.6 This test method makes a series of measurements to determine the thermal energy storage of a test specimen over a temperature range. First, both HFMA plates are held at the same constant temperature until steady state is achieved. Steady state is defined by the reduction in the amount of energy entering the specimen from both plates to a very small and nearly constant value. Next, both plate temperatures are changed by identical amounts and held at the new temperature until steady state is again achieved. The energy absorbed or released by the specimen from the time of the temperature change until steady state is again achieved will be recorded. Using a series of temperature step changes, the cumulative enthalpy stored or released over a certain temperature range is determined.

1.6.1 The specific heats of the solid and liquid phases are determined from the slope of the temperature-dependant enthalpy function during sensible heating/cooling, before and after the phase change process.

1.7 Calibration of the HFMA to determine the 'correction factors' for the energy stored within the plate heat flux transducers and any material placed between the test specimen and the HFMA plates must be performed following Annex A1. These correction factors are functions of the beginning and ending temperatures for each step, as described in Annex A1.

1.8 This test method applies to PCMs and composites, products and systems incorporating PCMs, including those with PCM dispersed in or combined with a thermal insulation material, boards or membranes containing concentrated or dispersed PCM, etc. Specific examples include solid PCM composites and products, loose blended materials incorporating PCMs, and discretely contained PCM.

1.9 This test method may be used to characterize material properties, which may or may not be representative of actual conditions of use.

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

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[1] This test method is under the jurisdiction of ASTM Committee C16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

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

1.11 *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.*

1.12 *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:*[3]

C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus

C168 Terminology Relating to Thermal Insulation

E793 Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry

E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

2.2 *Other Standard:*

RAL-GZ 896 Phase Change Material, Quality Association PCM e.V.

3. Terminology

3.1 *Definitions*—Terminology C168 applies to terms used in this specification.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *phase change material (PCM), n*—a material that changes it physical state (solid to liquid or vice-versa) over a certain temperature range, used in engineering applications specifically to take advantage of its latent heat storage properties.

3.2.2 *PCM Active Range, n*—a broad temperature range in which a PCM changes phase from solid to liquid (melting) or liquid to solid (freezing), with associated enthalpy changes.

3.2.3 *PCM composite, n*—material embedded with PCM to enhance its thermal performance.

3.2.4 *PCM product, n*—material amended to include energy storage capabilities via inclusion of PCM or PCM composites.

3.2.5 *PCM system, n*—array or assembly of PCM products.

3.3 *Symbols and Units*—The symbols used in this test method have the following significance:

3.3.1 A—HFMA metering area, m^2.

3.3.2 $C_{hfi}(T_{begin},T_{end})$—correction factor for heat storage in the heat flux transducers, J/(m^2-°C).

3.3.3 $C_{other}(T_{begin},T_{end})$—correction factor for heat storage in other materials used to surround the test specimen, J/(m^2-°C).

3.3.4 $c_p(T)$—specific heat as a function of temperature, J/kg-°C.

3.3.5 c_{pM}—specific heat of a melted PCM product, defined at a temperature greater than the upper limit of the PCM Active Range, J/kg-°C.

3.3.6 $c_{pM,A}$—areal specific heat of a melted PCM product, defined at a temperature greater than the upper limit of the PCM Active Range, J/m^2-°C.

3.3.7 $c_{pM,V}$—volumetric specific heat of a melted PCM product, defined at a temperature greater than the upper limit of the PCM Active Range, J/m^3-°C.

3.3.8 c_{pF}—specific heat of a frozen PCM product, defined at a temperature less than the lower limit of the PCM Active Range, J/kg-°C.

3.3.9 $c_{pF,A}$—areal specific heat of a frozen PCM product, defined at a temperature less than the lower limit of the PCM Active Range, J/m^2-°C.

3.3.10 $c_{pF,V}$—volumetric specific heat of a frozen PCM product, defined at a temperature less than the lower limit of the PCM Active Range, J/m^3-°C.

3.3.11 E—heat flux transducer output, µV.

3.3.12 f—fraction of total PCM mass in the sample that has undergone phase change, dimensionless.

3.3.13 h—enthalpy, J/kg.

3.3.14 h_A—areal enthalpy, J/m^2.

3.3.15 h_{fs}—latent heat per unit mass, J/kg.

3.3.16 $h_{fs,A}$—latent heat per unit area, J/m^2.

3.3.17 h_V—latent heat per unit area, J/m^2.

3.3.18 k—thermal conductivity, W/m-K.

3.3.19 L—thickness of the test specimen, usually equal to the separation between the hot and cold plate assemblies during testing, m.

3.3.20 N—number of heat flux readings at a specific temperature step.

3.3.21 q—heat flux (heat flow rate, Q, through area A), W/m^2.

3.3.22 $q_{equilibrium}$—average heat flux at the end of a specific temperature step, W/m^2.

3.3.23 Q—heat flow rate in the metered area, W.

3.3.24 R—thermal resistance, (m^2·K)/W.

3.3.25 S—calibration factor of the heat flux transducer, (W/m^2)/V.

3.3.26 T—temperature, °C.

3.3.27 T_{begin}—beginning temperature for each temperature step, °C.

3.3.28 T_{end}—ending temperature for each temperature step, °C.

3.3.29 T_L—lower temperature limit of the PCM Active Range, °C.

3.3.30 T_U—upper temperature limit of the PCM Active Range, °C.

3.3.31 ΔT—temperature difference during a temperature step ($T_{end} - T_{begin}$), °C.

3.3.32 α—thermal diffusivity, m^2/s.

3.3.33 ρ—(bulk) density of the material tested, kg/m^3.

3.3.34 λ—thermal conductivity, W/(m·K).

3.3.35 τ—time interval, s.

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[3] 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.

3.3.36 $\Delta\tau$—time interval corresponding to each individual flux reading (data value), s.

3.4 *Subscripts and Superscripts:*

3.4.1 *A*—areal, per m^2.

3.4.2 *F*—frozen, solid.

3.4.3 *fs*—latent, associated with the transition from solid to liquid or liquid to solid.

3.4.4 *i,k*—index denoting ith, kth member of a series.

3.4.5 *L*—lower.

3.4.6 *M*—melted, liquid.

3.4.7 *U*—upper.

3.4.8 *V*—volumetric, per m^3.

4. Summary of Test Method

4.1 This test method describes a method of using a heat flow meter apparatus (HFMA) to perform heat flux measurements on samples exposed to dynamic, that is non-steady-state, temperature conditions. The HFMA plates are allowed to stabilize at a certain identical temperature, above or below the PCM Active Range, and then their temperatures are incrementally decreased or increased. The plates are allowed to stabilize after each temperature step and the enthalpy change of the test specimen is determined for each step change in temperature, hence the dynamic nature of the test.

NOTE 2—Since the 'dynamic' portion of the test method does not involve measurements made under steady-state conditions, nor lead to determination of steady-state thermal transmission properties, the Test Method C518 cannot be used.

4.1.1 The test method is specifically designed to address materials and products that undergo physical changes with latent heat absorption or release during the course of the test. In particular, a phase transition will occur within PCM products, when the test temperatures span the PCM Active Range.

4.2 The object of the test, especially for a PCM product, is generally to determine the temperature dependence of the enthalpy of the specimen.

5. Significance and Use

5.1 Materials used in building envelopes to enhance energy efficiency, including PCM products used for thermal insulation, thermal control, and thermal storage, are subjected to transient thermal environments, including transient or cyclic boundary temperature conditions. This test method is intended to enable meaningful PCM product classification, as steady-state thermal conductivity alone is not sufficient to characterize PCMs.

NOTE 3—This test method defines a dynamic test protocol for complex products or composites containing PCMs. Due to the macroscopic structure of these products or composites, conventional measurements using a Differential Scanning Calorimeter (DSC) as specified in E793 and E967, which use very small specimens, are not necessarily representative of the relationship between temperature and enthalpy of full-scale PCM products due to the specimen size limitation.

5.2 Dynamic measurements of the thermal performance of PCM products shall only be performed by qualified personnel with understanding of heat transfer and error propagation. Familiarity with the configuration of both the apparatus and the product is necessary.

5.3 This test method focuses on testing PCM products used in engineering applications, including in building envelopes to enhance the thermal performance of insulation systems.

5.3.1 Applications of PCM in building envelopes take multiple forms, including: dispersed in, or otherwise combined with, a thermal insulation material; a separate object implemented in the building envelope as boards or membranes containing concentrated PCM that operates in conjunction with a thermal insulation material. Both of these forms enhance the performance of the structure when exposed to dynamic, that is, fluctuating, boundary temperature conditions.

5.3.2 PCMs can be studied in a variety of forms: as the original "pure" *PCM*; as a *composite* containing PCM and other embedded materials to enhance thermal performance; as a *product* containing PCM or composite (such as micro- or macro-encapsulated PCM); or as a *system*, comprising arrays or assemblies of PCM products.

5.4 This test method describes a method of using a heat flow meter apparatus to determine key properties of PCM products, which are listed below. Engineers, architects, modelers, and others require these properties to accurately predict the in-situ performance of the products (**2**).

5.5 The objective is generally to conduct a test under temperature conditions that will induce a phase transition (for example, melting or freezing) in the PCM product during the course of the test.

5.6 Determination of thermal storage properties is the objective of this test method, and key properties of interest include the following:

5.6.1 PCM Active Range, that is the temperature interval over which the phase transitions occur, for both melting and freezing of the PCM product or composites containing PCMs.

5.6.2 Specific heat of the fully melted and fully frozen product, defined outside the PCM Active Range.

5.6.3 Enthalpy as a function of temperature, *h(T)*.

5.6.4 *Enthalpy plot*—a histogram or table that indicates the change in enthalpy associated with incremental temperature changes that span the tested temperature range.

5.6.5 Enthalpy changes associated with phase transitions during the PCM melting and freezing processes in materials and composites containing PCMs.

5.7 PCM products often possess characteristics that complicate measurement and analysis of phase transitions during a test. Following are some of the known issues with PCMs:

5.7.1 *Imprecise PCM Active Range*—PCMs in general do not have precise melting or freezing temperatures, and the entire active temperature range, from the beginning to the end of phase transitions, must be determined.

NOTE 4—The onset of freezing will not necessarily coincide with the end of melting. Therefore, the freeze and melt enthalpy curves must be independently defined to determine the PCM Active Range.

5.7.2 *Multiple Phase Transitions*—Many PCMs exhibit a solid-solid transition with significant latent heat effects at temperatures near the melting transition.

5.7.3 *Sub-cooling*—Occurs when the specimen cools below its nominal freezing temperature before it actually begins to freeze, thus exhibiting an unusual enthalpy-temperature curve.

Solid-liquid and solid-solid phase changes are often dependent on heating and cooling rate.

5.7.4 *Hysteresis*—Occurs when a specimen heated from one temperature to another, and then returned to the original temperature, absorbs more (or less) heat at any particular temperature during the heating stage than it releases during cooling.

5.8 The properties measured are determined by fundamental thermophysical properties of the constituent materials of the product, and are thus inherent to the PCM product. The desired thermal performance enhancement, however, will depend strongly on the particular environment, climate, and mode of the actual engineering application of the PCM.

6. Apparatus

6.1 Follow the Apparatus section of Test Method C518 with these additional requirements:

6.1.1 A minimum of two heat flux transducers, one mounted on each plate of the apparatus, are required.

6.1.2 The ability to scan temperature and heat flux data at specified intervals and store results in a form that is immediately accessible in real time to the user or other programs running concurrently is required; for example, a text file to which data are written after each scan. The ability to record a time stamp of each scan is required.

6.1.3 The ability to accept a user-defined temperature program for control of both plate temperatures. This test method includes a series of temperature steps, with specified intervals determined by time or equilibrium criteria.

NOTE 5—Independent time or equilibrium criteria control for each setpoint will facilitate the test.

6.1.4 The amount of energy flowing through the transducers must be measureable at all times. To avoid saturating the transducers, either their voltage gain must be variable, or in apparatus without variable transducer gain, the alternative approaches described in Appendix X2 must be followed.

7. Specimen Preparation

7.1 Instructions are given here separately for solid samples, loose blended materials, and discretely contained PCM.

7.2 For solid samples such as gypsum wallboard containing PCM (**3-5**).

7.2.1 Cut the specimen to the same size as the HFMA plate area.

NOTE 6—If the specimen has a conductive facing, for example, foil, place a sheet of craft paper between the facing and the corresponding apparatus plate. If the heat capacity of this sheet is expected to be significant relative to the energy storage of the specimen, independently measure the heat capacity in the same manner as for the HFMA transducers, described in Annex A1. Then correct the measured heat flow into the assembly for this material as described in Section 10.

7.3 For loose material blended with PCM (**6, 7**).

7.3.1 Construct the sides of a frame using thin low mass material between 2.5 to 5 cm in height and sized so the frame will be located at the periphery of the test chamber. Affix a net material to form the frame bottom.

7.3.2 Since the frame is located far from the metering area, it is unlikely that the frame presence will have a significant effect on the thermal measurement. This shall be verified by separate measurement on solid specimens made with and without the sample frame.

7.4 For arrays of PCM pouches or PCM containers (**8**).

7.4.1 Ensure the portion of the product within the metered area is representative of the array pattern.

7.4.2 A sketch or photograph of the test specimen is required for this type of product, due to the spatial non-uniformities and discontinuities that are common with this product type.

7.5 Ensure good contact between the HFMA plates and the product. If necessary, use an elastomeric or soft foam rubber sheet between one or both sides of the product and the corresponding apparatus plate. This sheet will improve contact between the controlled temperature plates and prevent air circulation between the panel and the plates. The energy storage correction for the sheet(s) must be independently measured, in the same manner as for the HFMA transducers, as described in Annex A1. The measured heat flow into the assembly must then be corrected for this material as described in 10.3.

7.6 For PCM products with high lateral thermal conductivity, use an insulating frame to avoid significant edge losses. Ensure the frame is far away from the metered area to maximize the one-dimensional heat flow in the metered area.

8. Calibration

8.1 Prior to using this test method, calibrate the HFMA to determine the temperature-dependent calibration coefficients for both heat flux transducers using the procedure for the multiple temperature and thickness points in the Calibration section of Test Method C518.

8.2 The heat flux levels obtained during an HFMA test run are, in general, determined by heat flowing into or out of the specimen. The heat flux readings are also impacted by the heat that enters or leaves the transducers themselves, as a result of the change of the transducer temperature that corresponds to the change in plate temperature. Such heat flow is incidental to the values used in characterizing the PCM product. Therefore, separately calibrate the heat flux transducers within the HFMA to measure the correction factor for heat storage in the heat flux transducers. This additional apparatus calibration is described in Annex A1.

9. Procedure

9.1 *Personnel Qualifications*—This test method shall only be performed by qualified personnel with experience in heat transfer analysis and experimental error propagation. To ensure accurate measurement, the operator shall be fully proficient in the operation of the equipment and must have detailed familiarity with the configuration of the apparatus, the apparatus control and data reporting software, and the specimen itself.

9.2 *Procedure Overview*—In order to characterize the PCM product, test parameter definitions are required, as are multiple series of measurements at discrete temperature steps. Instructions are given here to first define the general process used during a series of measurements (9.3); describe how to determine the test parameters (9.4); and finally, to apply this process to characterize the PCM product (9.5). Additional instructions are included to describe an optional investigation of the hysteresis within partially melted or frozen specimens (9.6, Appendix X3).

9.3 Define general series of temperature steps for both plates, for example, 11°C and 11°C, 13°C and 13°C, 15°C and 15°C, and so on.

9.3.1 To measure the enthalpy stored in the test specimen in each temperature range, make a series of measurements.

9.3.2 First, both plates shall be held at the same constant temperature until steady state is achieved.

Note 7—Please see Annex A1 for a description of experimental work that has been done with an apparatus with plates at different temperatures to achieve the same goals.

9.3.2.1 Steady state is defined by the reduction in the amount of energy entering the specimen from both plates to a very small and nearly constant value. See 10.2.

9.3.3 After steady state is achieved, both plate temperatures will be changed to the same new temperature and held at that value until steady state is again achieved.

9.3.4 The cumulative amount of energy that enters the specimen from the time of the temperature change until steady state is again achieved will be recorded.

9.3.5 Heat flux readings shall include the proper sign to indicate direction of heat flow; for example, a positive reading may indicate heat entering the test specimen, and negative values indicating heat leaving the specimen.

9.3.6 The initial temperature selection, the temperature difference between setpoints, and the number of temperature steps, will vary according to the purpose of each particular test series.

Note 8—The temperature range available depends on the construction of the HFMA equipment, the heat rejection bath temperature, and the calibration of the equipment.

9.4 *Determine the test parameters:*

9.4.1 An initial test shall be used to estimate the PCM Active Range and determine the time required for each temperature step. This step is not required if the specimen phase change characteristics are already well known, for example from differential thermal analysis (DTA) tests or differential scanning calorimetry (DSC) tests (using the step method or appropriately slow heating and cooling rates, as described by Castellon et al. (**9**)).

9.4.2 Make series of measurements, as described in 9.3, starting at a temperature at least 10°C below the expected melting temperature, or at the lowest temperature available on the HFMA, whichever is higher. Use temperature difference steps of 1.5 ± 0.5°C. Allow a minimum of two hours for each setpoint during the initial specimen characterization.

9.4.3 End the series when the amount of energy stored in a temperature step returns to a small value, that is, when the test specimen is fully melted. See 10.2.

Note 9—As described in 10.2, the amount of time required at each temperature step will vary depending on the size of the temperature step, the thermal diffusivity of the specimen, and the amount of energy storage that occurs over that temperature step.

9.4.4 Repeat this procedure starting at the fully melted temperature condition and decreasing the plate temperatures in 1.5 ± 0.5°C steps until the amount of energy stored in a temperature step returns to a small value, that is, when the test specimen is fully frozen. See 10.2.

9.4.5 Examine the data as described in Section 10. Determine the estimated PCM Active Range, the desired temperature step size, and the amount of time required for each step.

9.4.6 An example of such a test series is shown in Annex A2.

9.5 *Characterize the PCM product:*

9.5.1 Make a series of measurements, as described in 9.3, starting at a temperature at least 10°C below estimated PCM Active Range, and heating the plates with temperature difference steps of 1.5 ± 0.5°C. End at a temperature at least 10°C above the estimated PCM Active Range. The amount of time required at each temperature step shall be as determined in 10.2.

Note 10—The minimum and maximum temperature difference step size will be limited by the combined uncertainty of the temperature measurement and heat flux measurement within the HFMA.

Note 11—To enable testing over a sufficient temperature range while considering any HFMA limits on number of allowable temperature steps, variable temperature differences can be utilized. For example, Biswas et al. (**10**) used temperature differences of 1°C close to and within the PCM Active Range and 2°C away from the PCM Active Range.

9.5.2 Make a series of measurements, as described in 9.3, starting at a temperature at least 10°C above the estimated PCM Active Range, and cooling the plates with temperature difference steps of 1.5 ± 0.5°C. End at a temperature at least 10°C below the estimated PCM Active Range.

9.5.3 Examine the data as described in Section 10 to determine:

9.5.3.1 Whether either of the data series shall be repeated using longer equilibrium times at any particular temperature.

9.5.3.2 Whether the temperature range needs to be expanded to capture the full PCM Active Range.

9.5.4 A minimum of three heating series, as described in 9.5.1, and a minimum of three cooling series, as described in 9.5.2, are required.

9.5.4.1 In order to define the enthalpy curve of energy storage vs. temperature with adequate precision, select beginning temperatures for the subsequent heating and cooling series that differ from those used for the initial heating and cooling series.

Note 12—For example: If the initial heating series spanned 10 to 30°C in 2°C steps, retain the 2°C step size, but start the second heating series at 10.6°C and the third heating series at 11.3°C. If the initial cooling series spanned 30 to 10°C in 2°C steps, retain the 2°C step size, but start the second heating series at 29.4°C and the third heating series at 28.7°C.

9.5.4.2 Examine the data as described in 10.2 to determine whether either of the data series shall be repeated using longer equilibrium times at any particular temperature.

9.6 Hysteresis effects when starting from partially frozen or partially melted material may be explored using the method described in Appendix X3.

10. Calculations

10.1 *Calculations Overview*—The calculations require several separate stages. First it is necessary to examine the data to evaluate whether an adequate amount of time was spent at each and every temperature step (10.2). Once this has been established, it is possible to calculate the net energy storage within the test specimen corresponding to each temperature step (10.3). That data form can then be used to express the enthalpy of the product as a function of temperature (10.4); to define the specific heat of the fully melted and fully frozen product (10.5, 10.6); and to define the latent heat of the product (10.7).

10.2 Evaluate adequacy of time intervals at each temperature step.

10.2.1 The amount of time required at each temperature step will vary depending on the size of the temperature step, the thermal diffusivity of the specimen, the material thickness, and the amount of energy storage that occurs over that temperature step. The time interval required to reach steady state during phase change phenomena are much greater than time intervals required when the material is subjected to sensible energy storage phenomena.

10.2.1.1 The maximum heat rate into or out of the specimen is limited by apparatus capability and the specimen thermal diffusivity. It is possible to estimate the minimum amount of time ($\tau_{min,est}$) needed for each step by neglecting the apparatus limits and the impact of thermal storage on the thermal transmittance through the specimen, as shown in Eq 1. This approach is only possible when there is some basis for estimating the energy storage needed for that particular temperature step and when an estimate is available for the thermal conductivity of the material. Possible sources for the energy storage estimate include prior heating or cooling series or data from a DSC run.

$$\tau_{min,\,est} = \frac{\text{Estimated enthalpy storage for a particular temperature step}}{\text{Maximum conductance rate through specimen}}$$

or,

$$\tau_{min,\,est} = \frac{c_p(T)\rho L\Delta T}{\left(\frac{k\Delta T}{(L/2)}\right)} = \frac{c_p(T)\rho L^2}{2k} \qquad (1)$$

NOTE 13—If data are available to permit the calculation shown in Eq 1, reasonable rules of thumb for the adequate total time for that temperature step would be: *(1)* for heating away from the expected latent range, use 1.5 times the estimated minimum; *(2)* for heating within the latent range, use 2.5 times the estimated minimum; *(3)* for cooling away from the expected latent range, use 2.5 times the estimated minimum; *(4)* for cooling within the latent range, use 5 times the estimated minimum.

10.2.2 Plot the heat flux signal vs. time for each temperature step for each plate as shown in Fig. 1. This plot is also useful in determining how much time is required at each temperature step. For example, the time spent at temperatures labeled 20.5 and 19.5 is longer than necessary and the time spent at temperature 18.5 is barely sufficient.

NOTE 14—The raw data are evaluated in this step. The raw data, typically in microvolts, will be transformed into the integrated heat flux in a subsequent step as described in 10.3.

NOTE 15—It is useful to examine the equilibrium portions of the curves by either limiting the range of the plot on the y-axis, or plotting the absolute values of the electrical signal on a log axis.

10.2.3 As shown in 10.2.2 and Fig. 1, at steady state conditions at the end of each temperature step, a small non-zero HFMA signal remains, largely due to edge heat losses. For each series of temperature steps, determine whether each temperature step was held for an adequate length of time by examining this residual, or equilibrium, HFMA signal.

10.2.3.1 Calculate the residual heat flux transducer output (E), which is average output over the last 60 minutes of each

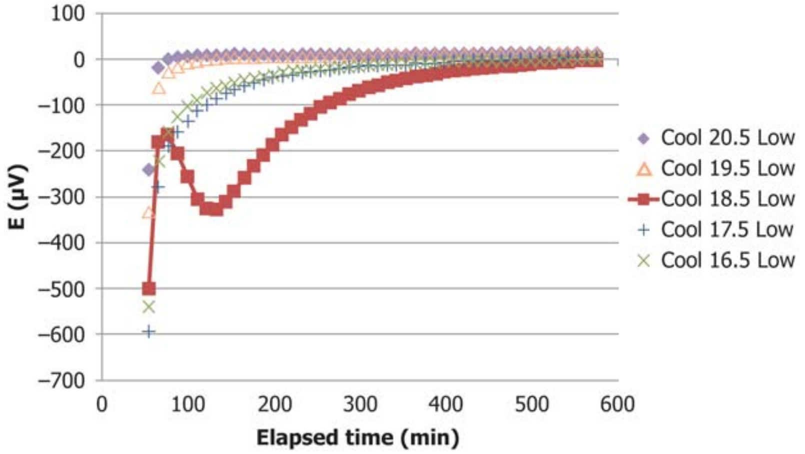

FIG. 1 Example of Transducer Output (E) Data Taken During a Series of Cooling Temperature Steps of the Lower Plate of an HFMA