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Standard Test Method for Specific Heat of Rock and Soil¹

This standard is issued under the fixed designation D 4611; 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 determination of instantaneous and mean specific heat of rock and soil.

1.2 This test method employs the classical method of mixtures. This provides procedures and apparatus simpler than those generally used in scientific calorimetry, an accuracy that is adequate for most rocks and soils, and a degree of precision that is reproducible by laboratory technicians of average skill. While this test method was developed for testing rock and soil, it is easily adaptable to measuring the specific heat of other materials.

1.3 The testing procedure provides an instantaneous specific heat over the temperature 25 to 300°C or a mean specific heat in that temperature range.

1.4 The test procedure is limited to dry samples.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

C 303 Test Method for Dimensions and Density of Preformed Block and Board-Type Thermal Insulation

C 351 Test Method for Mean Specific Heat of Thermal Insulation

D 618 Practice for Conditioning Plastics for Testing

D 2766 Test Method for Specific Heat of Liquids and Solids

E 230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

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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.

E 344 Terminology Relating to Thermometry and Hydrometry

3. Terminology

3.1 Definitions:

3.1.1 *instantaneous specific heat*—the rate of change of sample enthalpy, h , per unit mass with respect to temperature, T , at constant pressure, p ,

$$c_p = (\delta h / \delta T)_p \quad (1)$$

3.1.2 *mean specific heat*—the quantity of heat required to change the temperature of a unit mass of a substance one degree, measured as the average quantity over the temperature range specified. (It is distinguished from true specific heat by being an average rather than a point value. The unit of measurement is joule per kilogram kelvin, J/kgK).

3.1.3 *thermal capacity*—the amount of heat necessary to change the temperature of the body one degree. For a homogeneous body, it is the product of mass and specific heat. For a nonhomogeneous body, it is the sum of the products of mass and specific heat of the individual constituents. Thermal capacity has the units of joule per kelvin, J/K.

3.1.4 *thermal diffusivity*—the ratio of thermal conductivity of a substance to the product of its density and specific heat. Common unit for this property is m^2/s .

3.2 Symbols:

3.2.1 ΔH —enthalpy change (J/kg).

3.2.2 mc_p —thermal capacity (J/K).

3.2.3 T_m —final temperature of the mixture obtained by extrapolation (K).

3.2.4 T_c —temperature of the calorimeter immediately prior to drop obtained by extrapolation.

3.2.5 T_h —temperature of capsule and sample, capsule or standard in the heater prior to drop (K).

3.2.6 ΔT —temperature difference.

3.2.7 \bar{c}_p —mean specific heat (J/kgK).

3.2.8 c_p —instantaneous specific heat (J/kgK).

4. Summary of Test Method

4.1 The method of mixtures consists essentially of adding a known mass of material at a known temperature to a known mass of calorimetric fluid at a known lower temperature and determining the equilibrium temperature that results. The heat

absorbed by the fluid and containing vessel can be calculated from calibrations and this value equated to the expression for the heat given up by the hot material. From this equation, the unknown specific heat can be calculated. If only one drop from a single temperature is performed, then only the mean specific heat can be calculated. If several drops are performed, the instantaneous specific heat can be calculated.

5. Significance and Use

5.1 Specific heat is a basic thermodynamic property of all substances. The value of specific heat depends upon chemical composition and temperature. The rate of temperature diffusion through a material, thermal diffusivity, is a function of specific heat; therefore, specific heat is an essential property of rock and soil when these materials are used under conditions of unsteady or transient heat flow.

6. Apparatus

6.1 *Calorimeter and Accessories*—The calorimeter shall be an unlagged Dewar flask. The capacity of the Dewar flask shall be such as to yield a 1 to 5 K temperature rise of the receiver fluid with average sample size used during testing (Note 1). The flask shall have an insulated cover or stopper. Other accessories shall include a magnetic stirrer equipped with a speed regulating device.

NOTE 1—Typical volumes are approximately 500 to 750 mL with rock or soil samples of 50 g in thin wall stainless steel containers.

6.2 *Calorimeter Temperature-Sensing Device*—A temperature-sensing device capable of 0.0025 K resolution and covering a minimum of 5 K range shall be used.

NOTE 2—A suitable temperature sensor is a multijunction thermopile typically referenced to an ice bath.

6.3 *Calorimeter Fluid*—The calorimeter fluid should be a high specific heat fluid, stable to 250 to 300°C and having a low vapor pressure. Silicone based fluids are frequently used.

6.4 *Heater*—The heater shall be designed to provide a uniform heating zone. A maximum variation of $\pm 1\%$ of the mean heater temperature along the heater length corresponding to the sample is permitted.

NOTE 3—Typically, open-end radiation type heaters similar to the cylindrical device shown in Fig. 1 are used. Such heaters are usually heated by electricity; however, other means of heating are acceptable as long as the requirements for the heater can be met. The relative dimensions of the heater and capsule shall be such that the specimen will be heated to a uniform and constant temperature as required. The heater should be provided with an insulated removable cover designed to permit passage of sample capsule temperature sensing devices and suspension wire. The bottom should be closed with a removable insulated cover to permit free dropping of the capsule. Typically, the heater assembly is mounted so it can be swung quickly into place over the calorimeter immediately prior to drop and swung away after the sample has been dropped.

6.5 *Capsule*—The capsule shall be of the hermetically sealed type. The capsule heat capacity should be minimized and in no instance should be greater than the heat capacity of the sample. The capsule should be made of high conductivity material. Typically, capsules are thin wall copper or stainless steel containers.

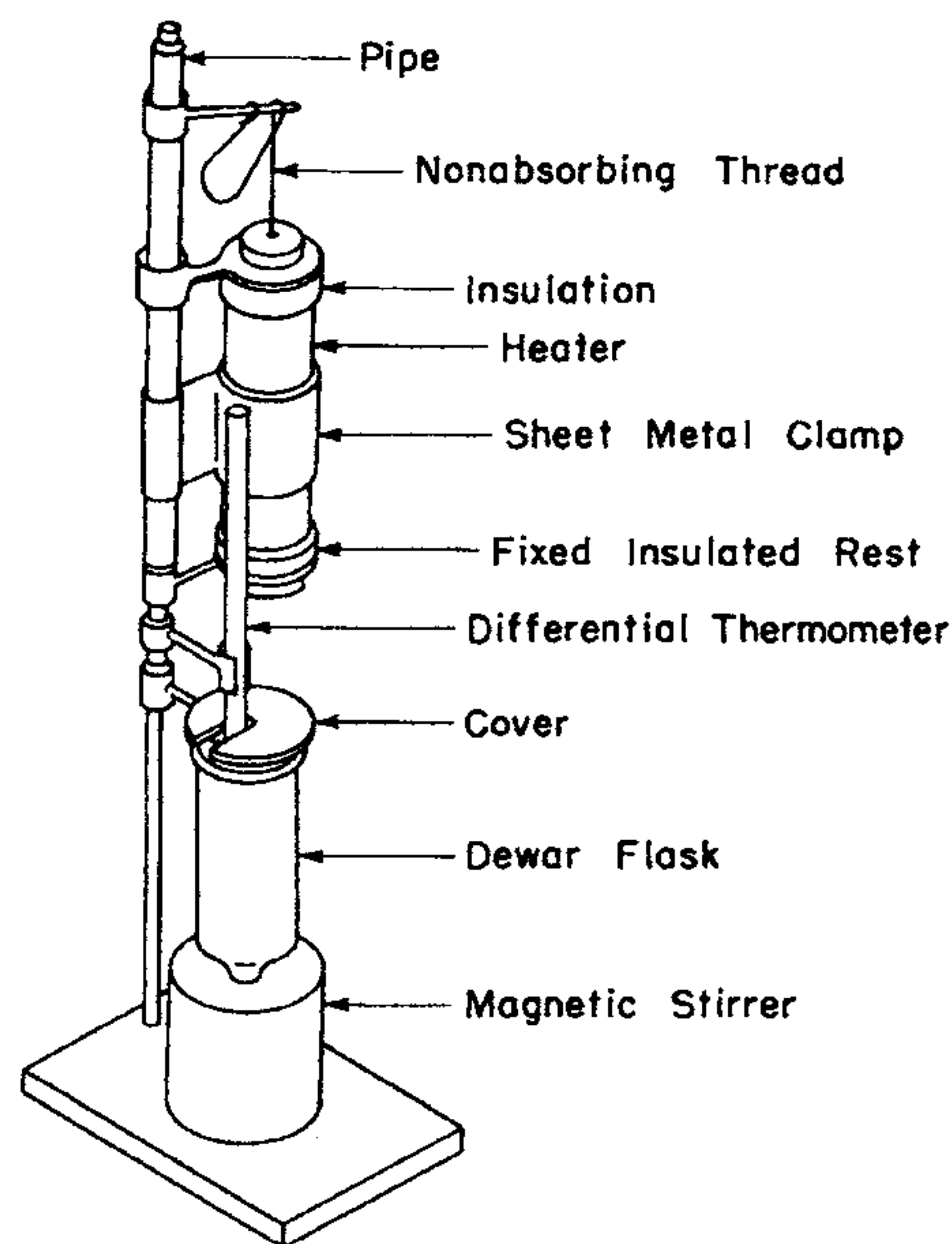


FIG. 1 Specific Heat Calorimeter

6.6 *Specimen Temperature Readout Device*—A convenient method of measuring the temperature of the sample in the heater unit shall be provided. It is desirable to measure the sample temperature inside the container; however, measuring of the outside of the container is permitted. Typically, a thermocouple calibrated to the special limits of error specified in EMF Tables E 230 is used for sample temperature readout. The temperature shall be measured to $\pm 1\%$ of the test temperature.

6.7 *Test Room*—The room temperature in which the tests are conducted shall be maintained at $23 \pm 2^\circ\text{C}$.

6.8 *Calibration Standards*—A minimum of three calibration standards are required. The standards must be traceable to the National Institute of Standards and Technology (NIST) or other National Metrology Institute.

7. Test Specimen

7.1 *Form*—In order to increase the accuracy of this test method, the sample mass should be maximized for a given capsule volume. This usually means, for dense rocks, that the sample should be machined to fit the container tightly. However, crushed rocks in powder form or soils can be tested with a decrease in accuracy due to the lower contribution of the sample to the total measured heat capacity of the sample/container combination. Porous rocks are usually tested in powder form.

7.2 *Sample Size and Number of Samples*—The sample shall be representative of the type of rock or soil tested. In cases where inhomogeneity is a problem, multiple samples of the same rock or soil shall be tested. In case of doubt, multiple samples shall be used.

7.3 *Sample Machining*—Samples shall be machined in such a manner that the machining process does not affect the specific heat properties of the sample. Any fluids used in the process shall be compatible with the sample and removed from the sample prior to tests.

7.4 The sample shall dry to constant weight in accordance with Method D 618, Procedure B, prior to testing.

8. System Calibration

8.1 *Calorimeter Fluid Calibration*—Perform a total of 15 drops in order to calibrate the receiver fluid. Drop three standards from five temperatures each approximately evenly spaced from 100 to 300°C.

8.2 *Calculation of the Heat Capacity of the Receiver*—After the 15 drops have been performed as described in Section 9, calculate the mc_p of the receiver for each drop (see 3.2). Plot the results as a function of drop temperature. The results (at each drop temperature) should be within $\pm 1.5\%$. Plot a straight line through the averaged results at each temperature. This is the calibration curve to be used in data reduction. To allow for minor loss in calibration fluid, the mc_p can be adjusted each time for weight loss. Maximum total weight loss shall be less than 5%.

8.3 *System Verification*—Every 10 drops or every 24 h, check the calibration of the receiver by dropping one standard from 200°C. The result must be within $\pm 1.5\%$ of the calibration curve determined in 8.2. If this is not the case, then the receiver fluid must be recalibrated or changed and the new fluid calibrated.

8.4 *Capsule Calibration*—The capsule shall be calibrated by testing it in accordance with the sample testing procedure. The capsule shall be recalibrated every time a change is made (that is, a new gasket), and its calibration shall be verified with a single drop, once a week.

9. Procedure

9.1 Dry the sample to constant weight in accordance with Method D 618, Procedure B. Record the weight to 0.1%.

9.2 Measure the mass of the calorimeter fluid in the receiver (Note 4) to 0.1%, and cover the Dewar.

9.3 Install the sample in the capsule and seal the capsule. Suspend the sealed capsule in the heating system and monitor the temperature of the sample. Record sample temperature at least once every five minutes until thermal equilibrium is achieved (Note 5). It shall be assumed that thermal equilibrium is achieved when the sample temperature is within 0.5% of the furnace temperature and the sample temperature does not change by more than 0.02°C/min over a 10-min period.

9.4 During the entire time that the sample temperature is equilibrating in the furnace, the temperature of the receiver should be monitored and recorded to 0.0025°C at least once every 5 min. If there is a drift in the receiver temperature, it should be constant and less than 0.05°C/min.

9.5 After the sample has reached thermal equilibrium, position the furnace over the Dewar (Note 6). Momentarily remove the cover from the Dewar and drop the sample into the calorimeter fluid. Replace the cover immediately after the drop. If during the drop, the sample hits anything prior to reaching the calorimeter fluid, the drop shall be disregarded and repeated.

9.6 Continuously monitor the temperature of the calorimeter fluid after the drop until the temperature drift is less than or equal to the drift just prior to the drop.

NOTE 4—A common method for measuring the mass of the fluid is to measure the total mass of the Dewar and fluid and mass of the empty Dewar. The mass of the fluid is the difference in the two masses.

NOTE 5—The time required for the sample to reach thermal equilibrium depends on such factors as furnace design, capsule design, sample size, and thermal diffusivity of the capsule and sample.

NOTE 6—For swinging furnaces, the time that the furnace is over the Dewar should be minimized.

10. Calculation

10.1 Plot the temperature of the calorimeter fluid versus time. An example of a temperature-time plot is shown in Fig. 2.

10.2 From the temperature-time plot, determine the value of T_m and T_c by extrapolation as illustrated in Fig. 2. Calculate the temperature change of the bath as follows:

$$\Delta T_{cal} = T_m - T_c \quad (2)$$

10.3 From the calibration graphs, find the $(mc_p)_{cal}$ of the calorimeter for the given drop temperature T_h . Calculate the enthalpy change of the calorimeter as follows:

$$\Delta H_{cal} = (mc_p)_{cal} \times \Delta T_{cal} \quad (3)$$

10.4 The heat released to the calorimeter is equal to the sum of the enthalpy changes of the sample and capsule, as follows:

$$\Delta H_{cal} = [(mc_p)_{cap} + (mc_p)_{samp}](T_h - T_m) \quad (4)$$

10.5 Therefore, the enthalpy change of the sample can be calculated from the following equation:

$$(mc_p)_{samp}(T_h - T_m) = (mc_p)_{cal} \times \Delta T_{cal} - (mc_p)_{cap}(T_h - T_m) \quad (5)$$

10.6 If only one drop is performed, then the mean specific heat of the sample can be calculated as follows:

$$\bar{c}_p \text{ samp.} = \frac{(mc_p)_{cal} \times \Delta T_{cal} - (mc_p)_{cap}(T_h - T_m)}{m_{\text{samp}}(T_h - T_m)} \quad (6)$$

10.7 If the instantaneous specific heat is to be determined, perform drops from a minimum of three temperatures. Calculate the enthalpy change of the sample for each drop and plot as a function of drop temperature. The enthalpy change should

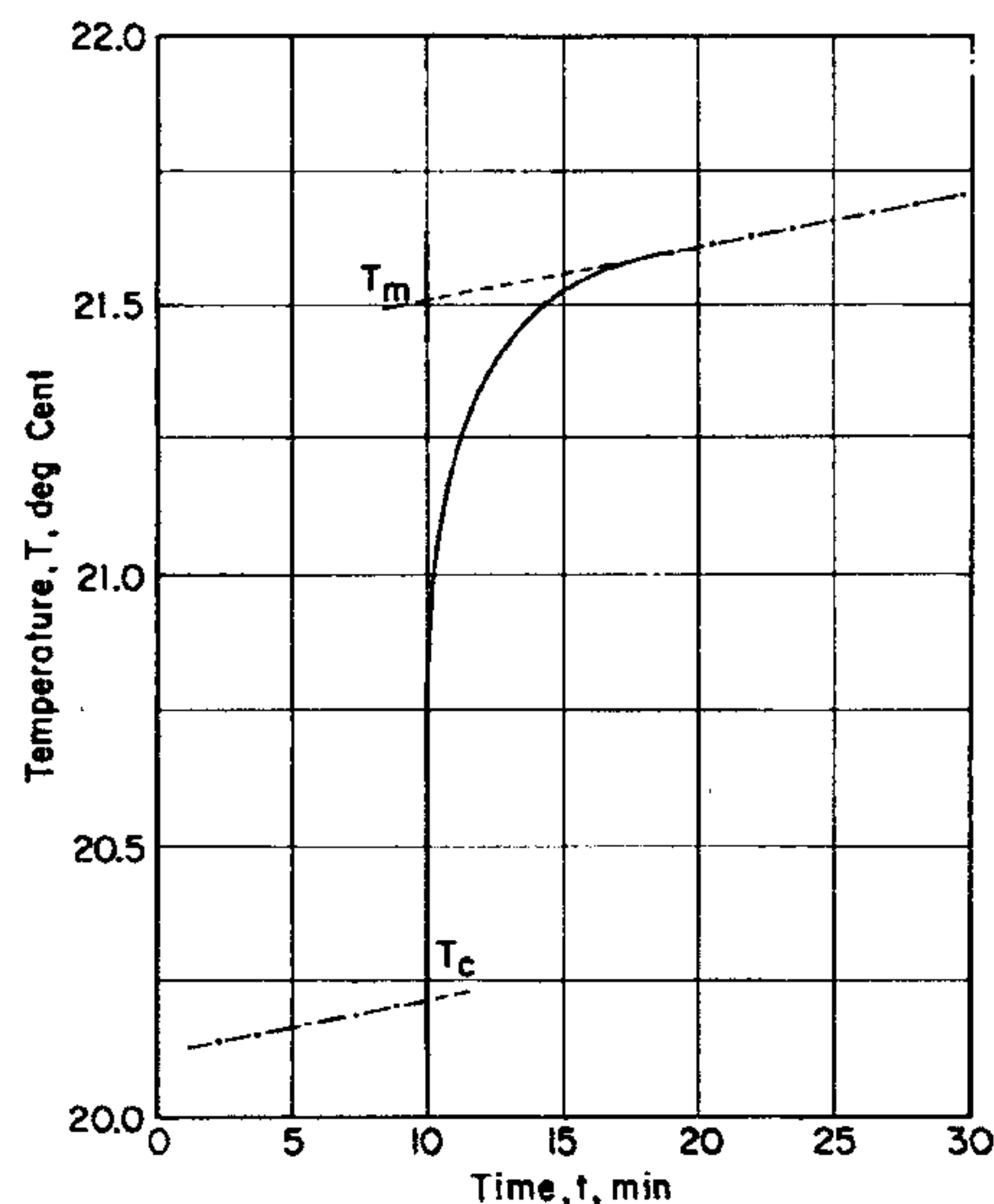


FIG. 2 Curve Showing Relation of Temperature to Time for a Typical Specific Heat Determination

be zero at T_m . Use a curve fitting routine to fit the data and obtain a polynomial equation. Use $\Delta H = 0$ at T_m as one of the points.

10.8 Calculate the instantaneous specific heat by differentiating the enthalpy change versus drop temperature curve as follows:

$$c_{p_{\text{samp}}} = \frac{d(\Delta H_{\text{samp}})}{dT} \quad (7)$$

10.9 *Calculation of Capsule Calibration*— Perform capsule calibration prior to test drops by dropping empty capsules, calculating the enthalpy change, and plotting it versus drop temperature. After an adequate curve has been established over the temperature range of interest, the enthalpy change of the capsule for a given test can be picked off the curve. Usually, a minimum of five drops is required to characterize the capsule.

11. Report

11.1 The report shall contain the following information:

11.1.1 Sample description including size, form, and mass.

11.1.2 The enthalpy change and drop temperatures.

11.1.3 The calculated mean or instantaneous, specific heat, or both.

11.1.4 Type of calorimeter fluid used.

11.1.5 Curve fitting procedure.

11.1.6 Statistical procedures used (if any).

11.1.7 The contribution of the capsule to the total enthalpy change for each drop (%).

11.1.8 Type of temperature readout used (both on the calorimeter and sample).

11.1.9 Any deviation from test procedure.

12. Precision and Bias

12.1 An interlaboratory testing program to determine the precision and bias of this test method for rocks has not been conducted. However, a limited round-robin study on thermal insulation³ and a compilation of adiabatic calorimeter data for Carbon-Carbon composites⁴ indicates that results from two or more tests performed on essentially identical materials should not vary by more than $\pm 10\%$.

13. Keywords

13.1 calibration; calorimeter; enthalpy; equilibrium temperature; heating tests-specific heat; rock; soil; temperature tests; thermal analysis-diffusivity

³ "A Proposed Method of Test for Specific Heat of Thermal Insulating Materials," Normal H. Spear, *ASTM Bulletin*, ASTM, 1950, pp 79–82.

⁴ Desphande, M. S., and Bogaard, R. H., "Evaluation of Specific Heat Data for POCO Graphite and Carbon-Carbon Composites," *Thermal Conductivity* 17, Plenum Press, 1983, pp 45–54.

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