Standard Test Method for Specific Heat of Rock and Soil[1]

This standard is issued under the fixed designation D4611; 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 values of the specific heat of rock and soil.

1.2 This test method employs the classical method of mixtures, which involve procedures and an apparatus that are simpler than those generally used in scientific calorimetry, but with an accuracy that is adequate for most rocks and soils. While this test method was developed for testing rock and soil, it can be adapted to measure the specific heat of other materials.

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

1.4 The test procedure is limited to dry specimens of soil and rock.

1.5 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurements are included in this standard.

1.6 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.6.1 The procedure used to specify how data are collected/recorded or calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analytical methods for engineering design

1.7 *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]

D618 Practice for Conditioning Plastics for Testing

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D6026 Practice for Using Significant Digits in Geotechnical Data

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

3. Terminology

3.1 For definitions of common technical terms used in this standard, refer to Terminology D653.

3.2 *Definitions:*

3.2.1 *instantaneous specific heat*—the rate of change of sample enthalpy, h, per unit mass with respect to temperature, T, at constant pressure, p, (J/kgK).

3.2.2 *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 (J/kgK).

3.2.3 *thermal capacity*—the amount of heat necessary to change the temperature of the body one degree, equal to the product of the mass of the body and its specific heat (J/K).

3.2.4 *thermal diffusivity*—the ratio of thermal conductivity of a substance to the product of its density and specific heat (m^2/s).

3.3 *Symbols:*

3.3.1 ΔH—enthalpy change (J/kg).

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.12 on Rock Mechanics.

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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.

***A Summary of Changes section appears at the end of this standard**

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3.3.2 $(\Delta H)_{cal}$—enthalpy change of the calorimeter (J/kg).

3.3.3 m—mass (kg).

3.3.4 m_{cal}—mass of calorimeter (kg).

3.3.5 m_{cap}—mass of capsule (kg).

3.3.6 m_{spec}—mass of the specimen (kg).

3.3.7 mc_p—thermal capacity (J/K).

3.3.8 T_m—final temperature of the mixture (K).

3.3.9 T_c—temperature of the calorimeter immediately prior to drop (K).

3.3.10 T_h—temperature of capsule and specimen in the heater prior to drop (K).

3.3.11 ΔT—temperature difference (K).

3.3.12 $(\Delta T)_{cal}$—temperature change of the calorimeter (K).

3.3.13 $(\bar{c}_p)_{spec}$—mean specific heat of the specimen (J/kgK).

3.3.14 $(c_p)_{spec}$—instantaneous specific heat of the specimen (J/kgK).

3.3.15 $(c_p)_{cal}$—instantaneous specific heat of the calorimeter (J/kgK).

3.3.16 $(c_p)_{cap}$—instantaneous specific heat of the capsule (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 or mineralogical 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.

NOTE 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etctesting. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Calorimeter*—The calorimeter shall be an unlagged vacuum flask. The capacity of the vacuum 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 2). The flask shall have an insulated cover or stopper.

NOTE 2—Typical volumes of vacuum flasks used for this type of measurement are approximately 500 to 750 mL for rock or soil samples of 50 g in thin-wall copper or stainless steel capsules.

6.2 Magnetic stirrer equipped with a speed regulating device.

6.3 *Calorimeter Temperature-Sensing Device*—A temperature-sensing device with a minimum resolution of 0.0025 K and an accuracy of ±0.5 % and capable of measuring a change in temperature of at least 5 K shall be used.

NOTE 3—A suitable temperature sensor is a multijunction thermopile typically referenced to an ice bath.

6.4 *Calorimeter Fluid*—The calorimeter fluid shall be a high specific heat fluid, stable to 520 to 570 K and having a low vapor pressure.

NOTE 4—Silicone based fluids have been found to meet this requirement.

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

NOTE 5—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.

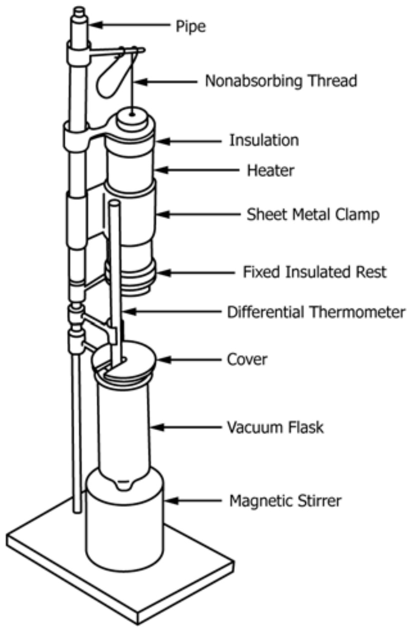

FIG. 1 Specific Ileal Calorimeter

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

6.7 *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 capsule; however, measuring of the outside of the capsule is permitted. Typically, a thermocouple calibrated to the special limits of error specified in EMF Tables E230 is used for sample temperature readout. The temperature shall be measured to ±1 % of the test temperature.

6.8 *Test Room*—The room temperature in which the tests are conducted shall be maintained at 296 ± 2 K.

6.9 *Calibration Specimens*—A minimum of three traceable calibration specimens are required.

NOTE 6—Commonly used calibration specimens are sapphire or platinum.

7. Test Specimen

7.1 *Form*—In order to increase the accuracy of this test method, the specimen mass shall be maximized for a given capsule volume. This usually means, for dense rocks, that the specimen shall be machined to fit the capsule 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 specimen/capsule combination. Porous rocks are usually tested in powder form.

7.2 *Statistical Requirements*—The number of samples and specimens tested shall be sufficient to provide an adequate statistical basis for evaluation of the results. Rock types that are highly variable will require more tests than relatively uniform rocks in order to evaluate the results with equal uncertainty.

7.2.1 The number of samples and specimens required to obtain a specific level of statistically valid results may be determined using Test Method E122. However, it may not be economically practicable to achieve specific confidence levels and professional judgment may be required.

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

7.4 The specimen shall dry to constant mass in accordance with Method D618, 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. Specifically, drop each of the three calibration specimens from five temperatures that are approximately evenly spaced from 373 to 573 K. The procedures described in Section 9 should be followed to perform to perform these calibration drops.

8.2 *Calculation of the Heat Capacity of the Receiver*—After the 15 drops have been performed according to the procedures described in Section 9, calculate the mc_p of the receiver for each drop (see 3.3). Plot the results as a function of drop temperature. The results (at each drop temperature) shall be within ±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 mass loss. Maximum total mass 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 capsule from 473 K. The result must be within ±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 empty capsule shall be calibrated by testing it in accordance with the testing procedure described in Section 9. A minimum of five drops at different temperatures that are approximately spaced evenly from 373 to 573 K is required to calibrate the capsule. The capsule shall be recalibrated every time a change is made (for example, replacing the gasket), and its calibration shall be verified with a single drop, with a frequency of at least once a week during periods that the calorimeter is in regular use.

9. Procedure

9.1 Dry the specimen to a constant mass in accordance with Method D618, Procedure B. Record the mass to an accuracy of ±0.1 %.

9.2 Measure the mass of the calorimeter fluid in the receiver (Note 7) to 0.1 %, and cover the vacuum flask.

9.3 Install the specimen in the capsule and seal the capsule. Suspend the sealed capsule in the heating system and monitor the temperature of the specimen. Record specimen temperature at least once every five minutes until thermal equilibrium is achieved (Note 8). It shall be assumed that thermal equilibrium is achieved when the specimen temperature is within 0.5 % of the furnace temperature and the specimen temperature does not change by more than 0.02 K/min over a 10-min period.

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

9.5 After the specimen has reached thermal equilibrium, position the furnace over the vacuum flask (Note 9). Momentarily remove the cover from the vacuum flask and drop the specimen into the calorimeter fluid. Replace the cover immediately after the drop. If during the drop, the specimen 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 7—A common method for measuring the mass of the fluid is to measure the total mass of the vacuum flask and fluid and mass of the empty vacuum flask. The mass of the fluid is the difference in the two masses.

NOTE 8—The time required for the specimen to reach thermal equilibrium depends on such factors as furnace design, capsule design, specimen size, and thermal diffusivity of the capsule and specimen.

NOTE 9—For swinging furnaces, the time that the furnace is over the vacuum flask should be minimized.

10. Calculation

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

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

$$\Delta T_{cal} = (T_m - T_c) \tag{1}$$

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

$$\Delta H_{cal} = m_{cal}(c_p)_{cal}\Delta T_{cal} \tag{2}$$

10.4 The enthalpy change of the calorimeter is also equal to the sum of the enthalpy changes of the specimen and capsule, as follows:

$$\Delta H_{\mathrm{cal}} = [(mc_p)_{cap} + (mc_p)_{spec}](T_h - T_m) \tag{3}$$

10.5 Combining Eq 2 and Eq 3:

$$m_{spec}(c_p)_{spec}(T_h - T_m) = m_{cal}(c_p)_{cal}\Delta T_{cal} - m_{cap}(c_p)_{cap}(T_h - T_m) \tag{4}$$

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

$$(\bar{c}_p)_{spec} = \frac{m_{cal}(c_p)_{cal}\Delta T_{\mathrm{cal}} - m_{cap}(c_p)_{cap}(T_h - T_m)}{m_{spec}(T_h - T_m)} \tag{5}$$

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 specimen for each drop and plot as a function of drop temperature as follows:

$$\Delta H_{spec} = m_{cal}(c_p)_{cal}\Delta T_{cal} - m_{cap}(c_p)_{cap}(T_h - T_m) \tag{6}$$

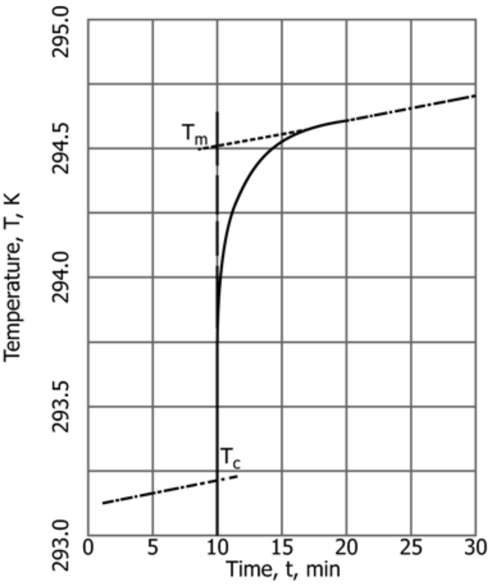

FIG. 2 Plot of Calorimeter Fluid Temperature Versus Time for a Typical Specific Heat Determination

10.8 The enthalpy change should be zero at T_m. Use a curve fitting routine to fit the data and obtain a polynominal equation. Use $\Delta H = 0$ at T_m as one of the points.

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

$$(c_p)_{spec} = \frac{d(\Delta H_{spec})}{dT} \tag{7}$$

10.10 *Calculation of Capsule Calibration*—Calculate the enthalpy change of an empty capsule and plot it versus the drop temperature. After a curve for enthalpy change has been established over the temperature range of interest, the enthalpy change of the capsule for a given test can be obtained from the curve.

11. Report: Test Data Sheet(s)/Form(s)

11.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.6 and Practice D6026.

11.2 Record as a minimum the following general information (data):

11.2.1 Specimen description including size, form, and mass.
11.2.2 The enthalpy change and drop temperatures.
11.2.3 The calculated mean or instantaneous values of specific heat, or both.
11.2.4 Type of calorimeter fluid used.
11.2.5 Details of the calibration specimen used.
11.2.6 Curve fitting procedure, order of the polynomial fitted, and coefficient of determination R^2.
11.2.7 Statistical procedures used (if any).
11.2.8 The contribution of the capsule to the total enthalpy change for each drop (%).
11.2.9 Type of temperature readout used (both on the calorimeter and specimen).
11.2.10 Technician name, and time and date of testing run.

12. Precision and Bias

12.1 *Precision*—Test data on precision is not presented due to the nature of the soil or rock materials tested by this test method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. Also, it is either not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation. However, a limited round-robin study on thermal insulation[3] and a compilation of adiabatic calorimeter data for Carbon-Carbon composites[4] indicates that results from two or

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[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.

more tests performed on essentially identical specimens is not expected to vary by more than ±10 %.

12.1.1 The Subcommittee D18.12 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

12.1.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

13. Keywords

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

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (2008) that may impact the use of this standard. (May 1, 2016)

(1) Updated 1.5 to clarify that the standard is SI-only.
(2) Inserted 1.6.
(3) Added new Note 1.
(4) Added references to other standards.
(5) Updated all temperatures to be in K.
(6) Edited consistency with specimen and sample.
(7) Updated the definitions to meet the style manual.
(8) Updated units in Fig. 2.
(9) Removed references to C303, C351, D2766, and E344.
(10) Updated Precision and Bias statements (Section 12).

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