Designation: D5334 – 14

Standard Test Method for Determination of Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure[1]

This standard is issued under the fixed designation D5334; 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 presents a procedure for determining the thermal conductivity (λ) of soil and soft rock using a transient heat method. This test method is applicable for both intact and reconstituted soil specimens and soft rock specimens. This test method is suitable only for homogeneous materials.

1.2 This test method is applicable to dry or unsaturated materials over temperatures ranging from <0 to >100°C, depending on the suitability of the thermal needle probe construction to temperature extremes. However, care must be taken to prevent significant error from: *(1)* redistribution of water due to thermal gradients resulting from heating of the needle probe; *(2)* redistribution of water due to hydraulic gradients (gravity drainage for high degrees of saturation or surface evaporation); *(3)* phase change of water in specimens with temperatures <0°C or >100°C. These errors can be minimized by adding less total heat to the specimen through either minimizing power applied to the needle probe and/or minimizing the heating duration of the measurement.

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

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

1.4.1 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.

1.5 *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]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

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

D4439 Terminology for Geosynthetics

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

3. Terminology

3.1 *Definitions*—For definitions of common technical terms, refer to Terminology standards D653 and D4439.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *heat input, n*—power consumption of heater wire in watts per unit length that is assumed to be the equivalent of heat output per unit length of wire.

3.2.2 *thermal epoxy, n*—any heat conductive resin material having a value of $\lambda > 4$ W/(m·K).

3.2.3 *thermal grease, n*—any heat conductive lubricating material having a value of $\lambda > 4$ W/(m·K).

[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

4. Summary of Test Method

4.1 Thermal conductivity is determined by a variation of the line source test method using a needle probe having a large length to diameter ratio to simulate conditions for an infinitely long, infinitely thin heating source. The probe consists of a heating element and a temperature measuring element and is inserted into the specimen. A known current and voltage are applied to the probe and the temperature rise with time is recorded over a period of time. The temperature decay with time after the cessation of heating can also be included in the analysis to minimize effects of temperature drift during measurement. Thermal conductivity is obtained from an analysis of the temperature time series data during the heating cycle and cooling cycle if applicable.

5. Significance and Use

5.1 The thermal conductivity of both intact and reconstituted soil specimens as well as soft rock specimens is used to analyze and design systems used, for example, in underground transmission lines, oil and gas pipelines, radioactive waste disposal, geothermal applications, and solar thermal storage facilities.

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. Users of this standard are cautioned that compliance with Practice D3740 does not in itself ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Thermal Needle Probe*—A device that creates a linear heat source and incorporates a thermocouple or thermistor to measure the variation of temperature at a point along the line. The construction of a suitable device is described in Annex A1.

6.2 *Constant Current Source*—A device to produce a constant current.

6.3 *Temperature Readout Unit or Recorder*—A device to record the temperature from the thermocouple or thermistor with a readability of 0.01 K or better.

6.4 *Voltage-Ohm-Meter (VOM)*—A device to read voltage and current to the nearest 0.01 V and 0.01 A.

6.5 *Timer*—A clock, stopwatch, digital timer, or integrated electronic timer capable of measuring to the nearest 0.1 s or better for the duration of the measurement.

6.6 *Drilling Device*—A drill capable of making a straight vertical hole having a diameter as close as possible to that of the needle and to a depth equivalent to the length of the needle.

6.7 *Balance*—A balance that meets the requirements of Guide D4753 and has a readability of 0.01 g for specimens having a mass of up to 200 g and a readability of 0.1 g for specimens with a mass over 200 g. However, the balance used may be controlled by the number of significant digits needed.

7. Specimen Preparation

7.1 *Intact Soil Specimens:*

7.1.1 *Thin-Walled Tube or Drive Specimens*—Cut a 200 ± 30-mm long section of a sampling tube containing an intact soil specimen. The tube section shall have a minimum diameter of 50 mm.

7.1.2 Determine and record the mass of the specimen in a sampling tube or brass ring to the nearest 0.01 gram.

7.1.3 Measure and record the length and diameter of the specimen to 0.1 mm. Take a minimum of three length measurements 120° apart and at least three diameter measurements at the quarter points of the height. Determine the average length and diameter of the specimen.

7.1.4 Insert the thermal needle probe down the axis of the specimen by either pushing the probe into a predrilled hole (dense specimen) to a depth equal to the length of the probe or pushing the probe into the specimen (loose specimen). Make sure the thermal probe shaft is fully embedded in the specimen and not left partially exposed. See Note 2.

NOTE 2—To provide better thermal contact between the specimen and the probe, the probe may be coated with a thin layer of thermal grease. If a hole is predrilled for the needle probe, the diameter of the hole should be equivalent to the diameter of the needle probe to make sure there is a tight fit. A device, such as a drill press, may be used to insert the probe to make sure the probe is inserted vertically and that no void spaces are formed between the specimen and the probe.

7.2 *Reconstituted Soil Specimens:*

7.2.1 Compact the specimen to the desired dry density and gravimetric water content in a thin-walled metal or plastic tube using an appropriate compaction technique. For further guidance on the effect of the various compaction techniques on thermal conductivity, refer to Mitchell et al. (**1**).[3] The tube shall have a minimum diameter of 50 mm and a length of 200 ± 30 mm.

7.2.2 Follow the procedure given 7.1.2, 7.1.3, and 7.1.4.

7.3 *Soft Rock Specimens:*

7.3.1 Determine and record the mass of the specimen to the nearest 0.01 g and follow the procedure given in 7.1.4 to determine the specimen diameter and length. The specimen dimensions shall be no less than those of the calibration standard (8.3).

7.3.2 Insert the thermal needle probe into the specimen by predrilling a hole to a depth equivalent to the length of the probe. Make sure the thermal probe shaft is fully embedded in the specimen and not left partially exposed. See Note 2.

8. Calibration

8.1 The thermal needle probe apparatus shall be calibrated before its use. Perform calibration by comparing the experimental determination of the thermal conductivity of a standard material to its known value. A calibration factor, C, is calculated as follows:

$$C = \frac{\lambda_{material}}{\lambda_{measured}} \quad (1)$$

where:

$\lambda_{material}$ = the known thermal conductivity of the calibration material, and

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

$\lambda_{measured}$ = the thermal conductivity of that material measured with the thermal needle probe apparatus.

8.1.1 All subsequent measurements with the thermal needle probe apparatus shall be multiplied by *C* before being reported. Although calibration is mandatory, it is especially important with large diameter needle probes (that is, $d > 2.54$ mm) where departures from the assumption of an infinitely thin probe cause potentially significant differences in estimation of the thermal conductivity due to non-negligible heat storage and transmission in the needle probe itself.

8.1.2 The calibration factor, *C*, has been shown to be a function of thermal conductivity when using a large diameter needle probe (see Hanson et al., 2004) (**2**). For users of large diameter probes, it may be necessary to determine *C* at several thermal conductivities in the range of measurement and construct a calibration function which is then applied to subsequent data collected with the thermal needle probe.

8.2 Conduct the test specified in Section 9 using a calibration standard as specified in 8.3.

8.3 *Calibration Standard*—One or more materials with known values of thermal conductivity in the range of the materials being measured, which is typically $0.2 < \lambda < 5$ W/m·K. Suitable materials include dry Ottawa sand, Pyrex 7740, fused silica, Pyroceram 9606 (**3**), glycerine (glycerol) with a known thermal conductivity of 0.286 W/(m·K at 25°C (**3**), or water stabilized with 5 g agar per litre (to prevent free convection) with a known thermal conductivity of 0.607 W/m·K at 25°C (**3**). (See Annex A2 for details on preparation of calibration standards.) The calibration standard shall be in the shape of a cylinder. The diameter of the cylinder shall be at least 40 mm or 10 times the diameter of the thermal needle probe, whichever is larger, and the length shall be at least 20 % longer than the needle probe. On solid specimens, a hole is drilled along the axis of the cylinder to a depth equivalent to the length of the probe. The diameter of the hole shall be equal to the diameter of the probe so that the probe fits tightly into the hole. For drilled specimens the probe shall be coated with thermal grease to minimize contact resistance.

8.4 The measured thermal conductivity of the calibration specimen must agree within one standard deviation of the published value of thermal conductivity, or with the value of thermal conductivity determined by an independent method.

8.5 For purposes of comparing a measured value with specified limits, the measured value shall be rounded to the nearest decimal given in the specification limits in accordance with the provisions of Practice D6026.

9. Procedure

9.1 Allow the specimen to come to equilibrium at the selected testing temperature. This equalization is especially important if only the heating data are to be analyzed as temperature drift will cause a significant error in the thermal conductivity measurement. Errors from small temperature drifts are minimized if both heating and cooling data are used in the analysis.

9.2 Connect the heater wire of the thermal probe to the constant current source. (See Fig. 1.)

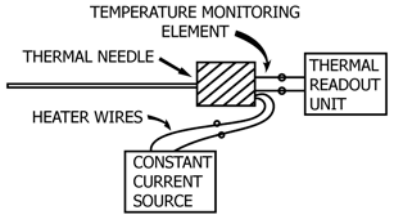


FIG. 1 Thermal Probe Experimental Setup

9.3 Connect the temperature measuring element leads to the readout unit.

9.4 Apply a known constant current, for example, equivalent to 1.0 A, to the heater wire such that the temperature change is less than 10 K in 1000 s.

9.5 Record time and temperature readings for at least 20–30 steps throughout the heating period. The total heating time should be appropriate to the thermal needle probe size. For a small diameter needle (that is, $d < 2.54$ mm), a 30 to 60 second heating duration is sufficient to accurately measure thermal conductivity. With a larger diameter needle, a longer heating duration may be necessary. However, this method is only valid if the thermal pulse does not encounter the boundaries of the specimen, so care must be taken not to choose too long a heating duration. Also note that potential errors from redistribution of water in unsaturated specimens increase with heating time as discussed in 1.2.

9.6 Turn off the constant current source.

9.7 If cooling data are to be included in the analysis, record the time and temperature readings for at least 20–30 steps throughout a cooling period equivalent in duration to the heating cycle.

9.8 Use a suitable inverse method (graphical or statistical) to determine thermal conductivity. (See Section 10, Data Analysis.)

9.9 Determine and record the initial gravimetric water content in accordance with Test Method D2216 and calculate the dry density of a representative sample of the specimen.

10. Calculations and Data Analysis

10.1 *Theory:*

10.1.1 If a constant amount of heat is applied to a zero mass heater over a period of time, the temperature response is:

$$\Delta T = -\frac{Q}{4\pi\lambda} Ei\left(\frac{-r^2}{4Dt}\right) \quad 0 < t \le t_1 \tag{2}$$

where:

t = time from the beginning of heating (s),
ΔT = change in temperature from time zero (K),
Q = heat input per unit length of heater (W/m),
r = distance from the heated needle (m),
D = thermal diffusivity (m^2/s),
λ = thermal conductivity (W/(m·K)),
Ei = exponential integral, and
t_1 = heating time.

10.1.2 The change in temperature after the heat input is turned off is given by:

$$\Delta T = -\frac{Q}{4\pi\lambda}\left[-Ei\left(\frac{-r^2}{4Dt}\right)+Ei\left(\frac{-r^2}{4D(t-t_1)}\right)\right] \quad t>t_1 \qquad (3)$$

10.1.3 The behavior of finite diameter and finite length probes can be approximated using these same equations, but D and λ will not represent the actual diffusivity and thermal conductivity, so calibration factors must be obtained for these probes as outlined in Section 8.

10.1.4 The most direct and precise method to calculate thermal conductivity is to use Eq 2 and 3 directly with the time series data collected as described in Section 9. Unfortunately, Eq 2 and 3 cannot be solved for λ and D explicitly, so a non-linear least-squares inversion technique must be used. A simplified analysis, which gives adequate results, approximates the exponential integral in Eq 2 and 3 by the most significant term of its series expansion:

$$\Delta T \cong \frac{Q}{4\pi\lambda}\ln(t) \quad 0<t\leq t_1 \qquad (4)$$

$$\Delta T \cong \frac{Q}{4\pi\lambda}\ln\left(\frac{t}{t-t_1}\right) \quad t>t_1 \qquad (5)$$

10.2 *Simplified Method:*

10.2.1 For thermal needle probes with diameter of 2.54 mm or less, exclude from the analysis the first 10 to 30 seconds of data from both the heating and, if used, cooling data. For larger diameter thermal needle probes it will be necessary to plot the data on a semi-log plot as described in 10.2.2 and identify the duration of the non-linear portion of initial data that shall be excluded. These data are most strongly affected by terms ignored in Eq 4 and 5, and will result in decreased accuracy if they are included in the subsequent analysis. The total time duration of the data included in the analysis, and duration of initial values excluded from the analysis, shall be fixed for any thermal needle probe configuration and used during calibration and all subsequent thermal conductivity measurements with that probe type to avoid biasing results due to subjective selection of the time range for analysis.

10.2.2 Using the remaining data, determine the slope, S_h of a straight line representing temperature versus ln t for the heating phase, and, if used, the slope, S_c of a straight line representing temperature versus $\ln[t/(t-t_1)]$ for the cooling phase (see Fig. 2). As shown in Fig. 2, the early and late portions of the test (representing transient conditions and boundary effects, respectively) shall not be used for the curve fitting. These slopes can be determined using linear regression with any standard spreadsheet or data analysis software, or manually, by plotting the data and fitting a straight line to the data by eye. If manual methods are used to determine the slope, it may be convenient to use semi-log graph paper with $\log_{10}$ time. If the slope of temperature versus $\log_{10}t$ is used in the analysis, the slopes of the plots are termed S_{h10} for the heating phase, and S_{c10} for the cooling phase.

10.2.3 The data included in the analysis shall be evenly spaced with the logarithm of time (X-axis). If data are collected in even time increments and subsequently plotted on a log time scale, then the distribution becomes uneven biasing the analysis too heavily toward the long-term of the testing period. Fig. 2 shows a data set that has been properly filtered to provide an even data distribution along the log time axis.

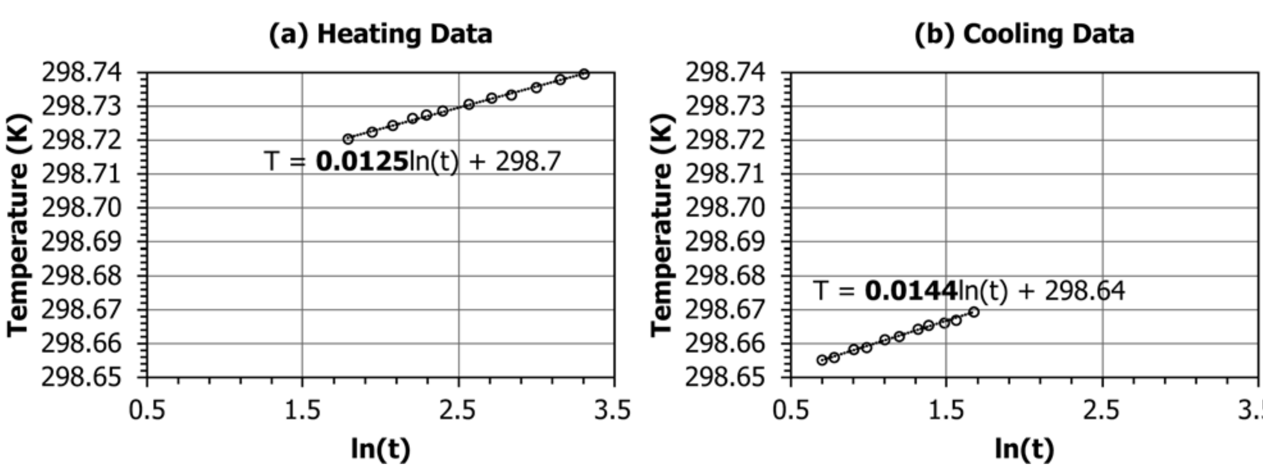
10.2.4 Compute thermal conductivity using Eq 6, where S is the average of S_h and S_c and S_{10} is the average of S_{h10} and S_{c10} if both heating and cooling data are used for the analysis or just S_h (or S_{h10}) if only heating data are used. Typically, S_h and S_c differ because of specimen temperature drift during the measurement. Averaging the two values minimizes the effects of the drift, which can cause large errors in determination of λ. Note that C is the calibration coefficient determined in Section 8.

$$\lambda = \frac{CQ}{4\pi S} = \frac{2.3CQ}{4\pi S_{10}} \qquad (6)$$

where:

Q = $I^2\frac{R}{L} = \frac{EI}{L}$

Q = heat input (W/m),



NOTE 1—2a shows data from the heating portion of the cycle and 2b shows data from the cooling portion of the cycle.

NOTE 2—The slopes (S_h and S_c) are shown in bold.

NOTE 3—The data are approximately evenly spaced on the X-axis to avoid bias as discussed in 10.2.2.

FIG. 2 (a & b) Typical Experimental Test Results