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

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 (e) 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 of soil and soft rock using a transient heat method. This test method is applicable for both undisturbed and remolded soil specimens and soft rock specimens. This test method is suitable only for isotropic materials.

1.2 This test method is applicable to dry materials over a wide temperature range from <0 to >100°C, depending on the suitability of the thermal needle probe construction to temperature extremes. This method may also be used for specimens containing moisture. 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, and (2) phase change (melting) of ice in specimens with temperatures <0°C. Both of these errors can be minimized by adding less total heat to the specimen either through minimizing power applied to the needle probe and/or minimizing the heating duration of the measurement.

1.3 For satisfactory results in conformance with this test method, the principles governing the size, construction, and use of the apparatus described in this test method should be followed. If the results are to be reported as having been obtained by this test method, then all pertinent requirements prescribed in this test method shall be met.

1.4 It is not practicable in a test method of this type to aim to establish details of construction and procedure to cover all contingencies that might offer difficulties to a person without technical knowledge concerning the theory of heat flow, temperature measurement, and general testing practices. Standardization of this test method does not reduce the need for such technical knowledge. It is recognized also that it would be unwise, because of the standardization of this test method, to resist in any way the further development of improved or new methods or procedures by research workers.

1.5 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

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

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:

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
D6026 Practice for Using Significant Digits in Geotechnical Data

3. Terminology

3.1 For terminology used in this test method, refer to Terminologies D653 and D4439.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 heat input—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—any thermally conductive filled epoxy material having a value of \( \lambda > 4 \text{ W/(m-k)} \).

3.2.3 thermal grease—any thermally conductivity grease having a value of \( \lambda > 4 \text{ W/(m-k)} \).

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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 The 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. The thermal conductivity is obtained from an analysis of the time series temperature data during the heating cycle and cooling cycle if applicable.

5. Significance and Use

5.1 The thermal conductivity of both undisturbed and remolded 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, and solar thermal storage facilities.

NOTE 1—Notwithstanding the statements on precision and bias contained in this test method, the precision of this test method 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 test method are cautioned that compliance with Practice D3740 does not in itself assure reliable testing. Reliable testing depends 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 temperature measuring element (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 or produce a digital readout of temperature in degrees Celsius with enough resolution to resolve changes in temperature induced by heating of the needle (typically 0.1 to 0.01 K).

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

6.5 Timer, stopwatch or integrated electronic timer capable of measuring to the nearest 0.1 s for the duration of the measurement.

6.6 Equipment, capable of drilling a straight vertical hole having a diameter as close as possible to that of the needle and to a depth at least equal to the length of the needle.

7. Specimen Preparation

7.1 Undisturbed Soil Specimens:

7.1.1 Thin-Walled Tube or Drive Specimens —Cut a 200 ± 30-mm (8.0 ± 1-in.) long section of a sampling tube containing an undisturbed soil specimen. The tube section should have a minimum diameter of 51 mm (2 in.).

7.1.2 Weigh the specimen in a sampling tube or brass rings.

7.1.3 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). Care should be taken to ensure that 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 equal to the diameter of the needle probe to ensure a tight fit. A device, such as a drill press, may be used to insert the probe to ensure that the probe is inserted vertically and that no void spaces are formed between the specimen and the probe.

7.2 Remolded Soil Specimens:

7.2.1 Compact the specimen to the desired density and 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). The tube should have a minimum diameter of 51 mm (0.2 in.) and a length of 200 ± 30 mm (8.0 ± 1 in.).

7.2.2 Perform 7.1.2 and 7.1.3.

7.3 Soft Rock Specimens:

7.3.1 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 equal to the length of the probe. Care should be taken to ensure that 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 should 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, $C$, should be calculated where:

$$C = \frac{\lambda_{\text{material}}}{\lambda_{\text{measured}}}$$

where $\lambda_{\text{material}}$ and $\lambda_{\text{measured}}$ are the thermal conductivities of the standard material and the specimen, respectively.

Note 3 The boldface numbers given in parentheses refer to the list of references at the end of this standard.
where:

\[ \lambda_{\text{material}} = \text{the known thermal conductivity of the calibration material,} \]

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

8.1.1 All subsequent measurements with the thermal needle probe apparatus should be multiplied by \( C \) before being reported. This is especially important with large diameter needle probes (that is, \( d > 2.5 \text{ mm} \)) 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 (typically \( 0.2 < \lambda < 5 \text{ W/m·K} \)). Suitable materials include dry Ottawa sand, Pyrex 7740, Fused Silica, Pyroceram 9606 (4), glycerine (glycerol) with a known thermal conductivity of 0.292 W m\(^{-1}\) K\(^{-1}\) at 25°C (4), or water stabilized with 5 g agar per liter (to prevent free convection) with a known thermal conductivity of 0.607 W m\(^{-1}\) K\(^{-1}\) at 25°C (4). (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 equal 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 should 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 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.)

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

9.4 Apply a known constant current (for example, equal 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, \( d < 2.5 \text{ mm} \)), 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 moist 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 equal 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 Perform an initial moisture content test method (see Test Method D2216) and a dry density test method (see Test Method D4439) on a representative sample of the specimen.

10. 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:

\[ \Delta T = -\frac{Q}{4\pi\lambda}\left[\frac{-r^2}{4D}\right] \quad 0 < t \leq t_1 \]

where:

\( t \) = time from the beginning of heating (s),
\( \Delta T \) = temperature rise 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),
\( \lambda \) = thermal conductivity (W m\(^{-1}\) K\(^{-1}\)),
\( Ei \) = exponential integral, and
\( t_1 \) = heating time.

10.1.2 The temperature rise after the heat is turned off is given by:

\[ \Delta T = -\frac{Q}{4\pi\lambda}\left[\frac{-r^2}{4D}\right] + \frac{Ei\left(-\frac{-r^2}{4Dt_1}\right)}{4D(t-t_1)} \]

\( t > t_1 \)

10.1.3 The behavior of finite diameter and finite length probes can be approximated using these same equations, but \( D \) and \( \lambda \) will not represent the actual diffusivity and 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 $\lambda$ 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:

$$D > \frac{Q}{4 \pi \lambda} \ln\left(\frac{t}{t-t_1}\right) \quad t > t_1 \tag{5}$$

10.2 Simplified Method:

10.2.1 For thermal needle probes with diameter of 2.5 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 should 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, should 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 $\ln[1/(t-t_1)]$ for the cooling phase (see Fig. 4). The early and late portions of the test (representing transient conditions and boundary effects, respectively) should 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 should be termed $(S_{h10})$ for the heating phase, and
$(S_{c10})$ for the cooling phase.

10.2.3 The data included in the analysis should 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 analy-
sis too heavily toward the long-term of the testing period. Fig.
4 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 mea-
surement. Averaging the two values minimizes the effects of
the drift, which can cause large errors in determination of $\lambda$.

Note that $C$ is the calibration coefficient determined in Section
8.

$$
\lambda = \frac{CQ}{4\pi S} = \frac{2.3CQ}{4\pi S_{10}}
$$

(6)

where:

$Q = \text{heat input (W/m),}

C = \text{calibration constant from Section 8,}

\lambda = \text{thermal conductivity [W/(m·K)],}

S = \text{slope used to compute thermal conductivity if ln(t) is}

used in analysis,

$S_{10}$ = slope used to compute thermal conductivity if
$\log_{10}(t)$ is used in analysis,

t = time (s),

$I$ = current flowing through heater wire (A),

$R$ = total resistance of heater wire ($\Omega$),

$L = \text{length of heated needle (m), and}$

$E = \text{measured voltage (V).}$

10.3 Derivation of the basis for Eq 2 and 3 are presented by
Carslaw and Jaeger (5), and adapted to soils by VanRooyen and
Winterkorn (6); VanHerzen and Maxwell (7); and Winterkorn
(8).

11. Report

11.1 For each thermal conductivity test, fill out a data sheet
similar to that shown in Fig. 2, reporting the following:

11.1.1 Date of the test and project name or number,

11.1.2 Boring number, sample or tube number, sample
depth, and data recorded in 9.5.

11.1.3 Initial moisture content and dry density,

11.1.4 Time versus temperature plot (see Fig. 3),

11.1.5 Thermal conductivity, and

11.1.6 Physical description of sample including soil or rock
type. If rock, describe location and orientation of apparent
weakness planes, bedding planes, and any large inclusions or
inhomogeneities.

12. Precision and Bias

12.1 An interlaboratory study involving line-source meth-
ods, including needle probes used for rock and soils, was
undertaken by ASTM Committee C16 (9). The materials of
known thermal conductivity that were evaluated included
Ottawa sand and paraffin wax (having a thermal conductivity
similar to certain soil and soft rock types). The results indicated
a measurement precision of between ±10% and ±15%, respec-
tively, with a tendency to a positive bias (higher value) over the
known values for the materials studied. With careful calibra-
tion of thermal needle probes in materials of known thermal
conductivity as outlined in Section 8, this precision should be
improved upon, and the positive bias should be removed.
Subcommittee D18.12 welcomes proposals that would allow
for a more comprehensive precision and bias statement cover-
ing the full range of soil and rock materials.

13. Keywords

13.1 heat flow; temperature; thermal conductivity; thermal
probe; thermal properties
ANNEXES

(Mandatory Information)

A1. COMPONENTS AND ASSEMBLY OF THERMAL NEEDLE

A1.1 The thermal needle consists of a stainless steel hypodermic tubing containing a heater element and a thermocouple as shown in Fig. A1.1. Its components and assembly are similar to the one described by Mitchell et al.\(^1\) and Footnote 5.\(^4\) To construct a thermal needle, hypodermic tubing is cut to 115 mm (4 1/2 in.) in length. The end to be inserted into the bakelite head of a thermocouple jack is roughened for a length of 15 mm (0.5 in.). A copper-constantan thermocouple wire junction previously coated with an insulating varnish is threaded into the hypodermic needle with the junction 50 mm (2 in.) from the end of the needle (see Note A1.1). At the same time, a manganin heater element is inserted with approximately 75-mm (3-in.) pigtails extending from the top of the needle as shown in Fig. A1.2. The uncut end of the needle is then inserted into an evacuating flask through a rubber stopper and the other end is placed in a reservoir of thermal epoxy primer as shown in Fig. A1.2. A vacuum pump connected to the evacuating flask is used to draw the thermal epoxy up through the needle. The needle is removed from the reservoir and flask, and a blob of putty is placed at the end of the needle to hold the thermal epoxy in place for hardening. After the thermal epoxy hardens, the thermocouple wires are soldered to the pins of a polarized thermocouple jack and the roughened end of the needle is placed in the bakelite head of the jack. The heater leads are brought out through two holes in the back of the bakelite head (see Fig. A1.1).

\(^{4}\) Mitchell, J. K., (Personal Communication), 1978 b.

Note 1—4a shows data from the heating portion of the cycle and 4b 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. 4 (a & b) Typical Experimental Test Results

\[ y = 0.0124x + 25.548 \]

\[ y = 0.0143x + 25.495 \]
FIG. A1.1 Typical Probe Components

Heater Element No. 26 Manganin
\* or Nichrome No. 30 Gauge Wire

- Copper No. 30 (0.25mm)
- Constantan No. 30 (0.25mm)

Bakelite Head

Epoxy Filled

Hypodermic Tubing
- 4.8mm (0.187 in.)
- 1.4mm (0.054 in.)

Thermocouple Junction

Epoxy Tip

FIG. A1.2 Drawing Thermal Epoxy Into Hypodermic Tubing
A2. PREPARATION OF CALIBRATION STANDARDS

A2.1 Glycerol:

A2.1.1 Glycerol (Glycerin) has a published thermal conductivity of 0.292 W m\(^{-1}\) K\(^{-1}\) at 25°C (4), and therefore falls within the low end of thermal conductivities expected in soil and rock. The thermal conductivity of glycerol is affected by the amount of water present in the glycerol, so only anhydrous (99% or greater) glycerol should be used for calibration. It should be noted that glycerol will readily take up water in both liquid and vapor phase, so care must be taken to prevent the sorption of water vapor into glycerol stored in improperly sealed containers. It should also be noted that glycerol is a fluid, and can be subject to mixing by free convection. Free convection arises from density gradients in fluids caused by thermal gradients in the fluid. If the thermal needle probe is heated too much, then free convection could occur, so glycerol is only appropriate for thermal needle probes that will heat less than 2°C over the course of the thermal conductivity measurement. 99+% anhydrous glycerol can be obtained from numerous sources, including local drugstores.

A2.2 Water:

A2.2.1 Water (DI or tap) has a published thermal conductivity of 0.607 W m\(^{-1}\) K\(^{-1}\) at 25°C (4), and therefore falls within the range of thermal conductivities expected in soil and rock. However, with most thermal needle probes, the heating of the needle will result in free convection in the water which will cause very large errors in the thermal conductivity measurement. To prevent this error, the water specimen must be physically stabilized. The preferred method for stabilizing the water is with a 5 g Agar/1 L water mixture. The Agar should be added to hot water and stirred well until the mixture is homogenous. The mixture should then be brought to a boil, and then re-homogenized in the container that will be used to hold the specimen during calibration. After cooling back to room temperature, the mixture should be a solid with the consistency of jelly. A readily available source of Agar is Stock #A10752 from Alfa Aesar (CAS#9002-18-0).

REFERENCES

SUMMARY OF CHANGES

Subcommittee D18.12 has identified the location of selected changes to this standard since the last issue (D5334 – 05) that may impact the use of this standard (approved July, 1, 2008).

(1) Edits to Sections 3, 4, 6, 8, 9, 10 and 12.
(2) Revised Fig. 2 and Fig. 4.
(3) Inserted new Annex A2.
(4) Edits to References.

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