

Designation: D5334 – 22a

Standard Test Method for Determination of Thermal Conductivity of Soil and Rock by Thermal Needle Probe Procedure¹

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 rock using a transient heat method. This test method is applicable for both intact specimens of soil and rock and reconstituted soil specimens, and is effective in the lab and in the field. This test method is most suitable for homogeneous materials, but can also give a representative average value for non-homogeneous materials.

1.2 This test method is applicable to dry, unsaturated or saturated materials that can sustain a hole for the sensor. It is valid 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 near 0°C or 100°C.

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. Reporting of test results in units other than SI shall not be regarded as nonconformance with 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 procedures 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.

Note 1—This test method is also applicable and commonly used for determining thermal conductivity of a variety of engineered porous materials of geologic origin including concrete, Fluidized Thermal Backfill (FTB), and thermal grout.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.6 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: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
- 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 and Data Records in Geotechnical Data

3. Terminology

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

3.2 Definitions of Terms Specific to This Standard:

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.

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*—heat conductive resin material having a value of $\lambda > 0.5$ W/(m·K).

3.2.3 *thermal grease*, *n*—heat conductive lubricating material having a value of $\lambda > 1.5$ W/(m·K).

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 heating element over a period of time and the temperature rise is recorded. The temperature decay with time after the cessation of heating can also be included in the analysis. Thermal conductivity is obtained from an analysis of the temperature time series data during the heating cycle and (optionally) the cooling cycle, by comparing it to a theoretical curve using non-linear least-squares inversion technique.

5. Significance and Use

5.1 The thermal conductivity of intact soil specimens, reconstituted soil specimens, and rock specimens is used to analyze and design systems involving underground transmission lines, oil and gas pipelines, radioactive waste disposal, geothermal applications, and solar thermal storage facilities, among others. Measurements can be made on site (in situ), or samples can be tested in a lab environment.

NOTE 2—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 Appendix X1.

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 *Digital Multimeter (DMM)*—A device to read voltage and current to the nearest 0.01 V and 0.001 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—(for rock specimens)* A drill capable of making a straight axial hole having a diameter equivalent 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 General Specimen Preparation Guidelines:

7.1.1 The main factors affecting the accuracy of a thermal conductivity reading include the density and water content of the sample, the size of the specimen, the sensitivity and accuracy of temperature measurements, the amount of heat applied, and the relative conductivities of the needle and the sample. Annex A1 contains more information for configuring the test.

7.1.2 Because the density and water content of the sample are major factors in its thermal conductivity, take care to make the specimen the same density and water content as the material it represents, whether that is the undisturbed soil or the installed state of a backfill. As a general reference, a density of more than 2000 kg/m³ is necessary for resistivity to be under 50 °C·cm/W.

7.1.3 The specimen radius needs to be large enough that a heat pulse is not reflected off the outside boundary, and so that the surroundings do not factor into the reading. The diffusivity of the sample determines how fast heat can travel through it, independent of its conductivity or the temperature difference at the source. By assuming that a 99 % heat reduction at distance r is sufficiently small to have a negligible effect on the reading, curves delineating the minimum size of the specimen (that is, the radius, and also the approximate length beyond the end of the needle) can be derived empirically from Eq 3 parameterized by the diffusivity (D) of the specimen, time duration (t) of the reading including heating and cooling if included, and the radius of the needle. Fig. 1 plots three such curves generated for probe sizes selected to span common needle radii. Given the product of the sample diffusivity (D) and reading time duration (t) on the x-axis, the minimum specimen radius can be read off the y-axis. In addition, a power law equation approximates the results for each of the curves. For other needle radii, interpolation or generating a new curve may be appropriate.

$$r = 3.971(D_{t})^{0.4382} \quad a = 2mm$$
(1)

$$r = 3.5453(D_{t})^{0.4526} \quad a = 1.2mm$$

$$r = 3.2392(D_{t})^{0.4623} \quad a = 0.64mm$$

where:

- r = distance from the heated needle (mm) (minimum radius of the specimen),
- D = thermal diffusivity of the specimen (mm²/s),
- t = time from the beginning of heating to the end of the test (s), and
- a =radius of the needle.

(f) D5334 - 22aMinimum Specimen Radius $(f) \int y = 3.971 \times 0.4382$ $(f) \int y = 3.971 \times 0.4382$

FIG. 1 Minimum Radius of a Specimen

(2)

7.1.4 There are many ways to get an estimate of the specimen's diffusivity. It can be measured directly with an instrument designed for that purpose. Alternately, it can be calculated from a previous measurement of the thermal Conductivity and the specimen's volumetric heat capacity ($\rho_s c_s$) in MJ/(m³·K) according to the equation:

 $D = \frac{\lambda}{\rho_{c}c_{s}}$

where:

- $D = diffusivity (m^2/s), catalog$
- λ = conductivity (W/(m·K)),
- $\rho_s = \text{density (kg/m^3), and}$
- c_s = specific heat (J/(kg·K)).

Another option is to estimate it from a graph of diffusivity values, such as the one in Fig. 2(1).³

7.1.5 The specimen length needs to be greater than or equal to that of the sensor needle. If the specimen and needle are close to the same length, then the nature of the material contacting the end of the specimen may adversely affect the reading; highly conductive materials affect the reading more than insulating materials. An addition to the sample length equal to its minimum radius would provide a sufficient security measure.

Note 3—The specimen dimensions are specified as if the specimen was in the shape of a cylinder, with the needle to be inserted (and a hole to be drilled if necessary) along the axis of the cylinder. In actuality, as long as the specimen can circumscribe a cylinder of the specified radius and length, the shape of the specimen is immaterial.

7.2 Intact Soil Specimens (Thin-Walled Tube or Drive Specimens):



7.2.1 Cut a section of a sampling tube containing an intact soil specimen diameter compliant to 7.1. Consider cutting the section in a way that facilitates determining the volume of the specimen and preserves the integrity of the sample.

7.2.2 Seal the specimen to prevent water loss and redistribution during storage or measurement.

7.3 Reconstituted Soil Specimens:

7.3.1 Compact the specimen to the desired dry density and gravimetric water content in a thin-walled metal or plastic tube that complies with the size guidelines in 7.1 using an appropriate compaction technique (compaction and water content both affect the thermal conductivity). For further guidance on

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

the effect of the various compaction techniques on thermal conductivity, refer to Mitchell et al. (2).

7.4 In Situ Soil Specimens:

7.4.1 Dig a pit or trench to the depth of the desired measurement. Prepare a flat, even soil face for sensor insertion.

7.4.2 Make sure to make the thermal measurements soon after excavating or exposing the soil face to avoid non-homogenous moisture conditions due to evaporation from the exposed face.

7.5 Rock Specimens:

7.5.1 Select the specimen to comply with the size guidelines in 7.1, to avoid fractures and inconsistencies, and follow provide a good locale to drill a hole for the needle that will give a representative reading.

8. Verification of Apparatus

8.1 Every month or after 50 readings check that the apparatus is working correctly. Evaluate the integrity and condition of the sensor/needle. Observe a reading and confirm that the temperature values are reasonable, that they increase in a smooth non-decreasing curve during the heating cycle, and that the expected amount of heating is taking place (using a known sample would be helpful).

8.2 Yearly, conduct the test specified in Section 9 using one of the verification standards specified in 8.2.1.

8.2.1 Verification 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), with size and shape compliant to 7.1. Suitable materials include dry Ottawa sand, Pyrex 7740, fused silica, Pyroceram 9606 (3), glycerine (glycerol) with a known thermal conductivity of 0.285 W/(m·K) at 25°C (3), or water stabilized with 5 g agar per liter (to prevent free convection) with a known thermal conductivity of 0.606 W/(m·K) at 25°C (3). (See Appendix X2 for details on preparation of verification standards.)

8.2.2 The measured thermal conductivity of the verification specimen should agree within 5.0 % of the published value of thermal conductivity, or within ± 5.0 % of the value of thermal conductivity determined by an independent method.

8.2.3 For purposes of comparing a measured value with specified limits, round the measured value to the nearest decimal given in the specification limits in accordance with the provisions of Practice D6026.

9. Procedure

9.1 Determine and record the mass of the specimen to the nearest 0.01 gram (may not be needed for verification).

9.2 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 (may not be needed for verification).

9.3 Insert the thermal needle probe down the axis of the specimen by pushing the probe into the specimen. If the specimen is dense, insert the needle into a hole predrilled to a

depth equal 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 4.

Note 4—If it is necessary to drill a hole in the specimen, drill the hole after measuring the mass. The diameter of the hole should be equivalent to the diameter of the needle probe to make sure there is a tight fit. To provide better thermal contact between the specimen and the probe, the probe may be coated with a thin layer of thermal grease. A device such as a drill press may be used to drill the hole and to insert the probe in a straight line, thus increasing contact between the specimen and probe, and reducing void spaces.

9.4 Allow the specimen to stabilize at the selected testing temperature and allow the probe to come to equilibrium inside the specimen. Stability and equilibrium can be estimated by observing the temperature over a period of time.

9.5 Connect the heater wire of the thermal probe to the constant current source. (See Fig. 3.)

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

9.7 Apply a known constant current to the heater wire.

9.8 Record the Current (nearest 0.001 A) and/or Voltage (nearest 0.01 V) across the heater as needed to compute the power.

9.9 Record time and temperature readings for at least 20-30 steps throughout the heating period.

9.10 Turn off the constant current source.

9.11 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.

9.12 Use a suitable method to determine thermal conductivity. (See Section 10, Calculations and Data Analysis.)

9.13 Determine and record the initial gravimetric water content in accordance with Test Method D2216 and calculate the dry density (or unit weight) 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:

$$\Delta T = -\frac{Q}{4\pi\lambda} Ei\left(\frac{-r^2}{4Dt}\right) \quad 0 < t \le t_1 \tag{3}$$



FIG. 3 Thermal Probe Experimental Setup

where:

- t = time from the beginning of heating (s),
- ΔT = change in temperature from time zero (K),
- Q = heat input (heat per unit length of heater, W/m),
- r = distance from the center of the heater (m),
- D = thermal diffusivity (m²/s),
- λ = thermal conductivity (W/(m·K)),
- Ei = exponential integral, and
- t_1 = heating time (s).

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

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

10.1.3 The behavior of finite diameter and finite length probes can be approximated using these same equations, but D and λ may not represent the actual diffusivity and thermal conductivity of the specimen.

10.1.4 The most direct and precise method to calculate thermal conductivity is to use Eq 3 and Eq 4 directly with the time series data collected as described in Section 9. Eq 3 and Eq 4 cannot be solved for λ and *D* explicitly, so a non-linear least-squares inversion technique is used. A simplified analysis, which gives adequate results, approximates the exponential integral in Eq 3 and Eq 4 by the most significant term of its series expansion from De Vries (4):

$$T = \frac{Q}{4\pi\lambda} \ln(t+t_0) + C \quad 0 < t \le t_1$$

$$T = \frac{Q}{4\pi\lambda} \ln\left(\frac{t+t_0}{t-t_1+t_0}\right) + C \quad t > t_1$$
(6)

where:

- t = time from the beginning of heating (s),
- T = temperature at time t (K),
- Q = heat input (heat per unit length of heater, W/m), 76
- λ = thermal conductivity (W/(m·K)),
- t_1 = heating time (s),
- t_0 = time offset (s), and
- C = temperature axis intercept (a constant) (K).

Because these equations (generally with $t_0 = 0$) are easily inverted to obtain conductivity, they are almost universally used to obtain thermal conductivity from probe temperature data.

10.1.5 Blackwell (5) produced an exact solution in the Laplace domain for the finite probe sizes actually used for measurements. Knight et al. (6) transformed that solution to the time domain using the Stefest algorithm. Using the Blackwell/Knight model in inverse mode to find thermal conductivity is difficult and opaque, but their model allows us to compare Eq 5 and Eq 6 to actual probe performance. Conclusions from those comparisons are:

(1) Eq 5 and Eq 6 model finite probes better than Eq 3 and Eq 4, so no advantage is gained by using exponential integrals.

(2) Eq 5 and Eq 6 model probe performance accurately at long times (5 - 10 min), even when t_0 is set to zero; however, the signal to noise ratio is much worse than for short time data because temperature changes are so small.

(3) Eq 5 and Eq 6 accurately model finite size probe performance at relatively short times (1 min) if t_0 is selected to minimize deviations between model and measured values. Allowing t_0 to vary in this way removes effects of probe specific heat and the specimen's diffusivity.

10.2 Simplified Method 1:

10.2.1 Remove the thermal drift (see Annex A1).

10.2.2 Selecting an appropriate data range, 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. 4).

10.2.3 Compute thermal conductivity using Eq 7, where S is either S_h or S_c . If $\log 10(t)$ was used instead of the natural log $(\ln(t))$, then employ the correction factor in the second half of Eq 7, where S_{10} is the heating or the cooling slope.

10.2.4 If using both the heating and cooling curves, average the two values of conductivity.

$$\lambda = \frac{Q}{4\pi S} = \frac{2.3Q}{4\pi S_{10}} \tag{7}$$



FIG. 4 (a & b) Typical Experimental Test Results

where:

Q = heat input (W/m) =

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

- λ = thermal conductivity [W/(m·K)],
- S = 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 (Ω),
- L = length of heated needle (m), and
- E = measured voltage (V).

10.3 Simplified Method 2:

10.3.1 Remove the thermal drift (see Annex A1).

10.3.2 Collect data as in Method 1. Analyze the data by adjusting conductivity and initial time t_0 in Eq 5 and Eq 6 to match the measured temperature data. A non-linear Least Squares method such as Solver in Excel can be used. The t_0 and conductivity combination that gives the best fit identifies the conductivity λ of the medium.

10.4 Derivations forming the basis of Eq 5 and Eq 6 are adapted to soils by Van Rooyen and Winterkorn (7); Von Herzen and Maxwell (8); and Winterkorn (9).

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 1.4.1 and Practice D6026.

11.2 Record as a minimum the following general information (data): <u>ASTM1</u>

11.2.1 Project information, such as project name, number, source of test specimens, including other pertinent data that helps identify the specimen.

11.2.2 Name or initials of the person(s) who prepared and tested the specimens, including the date(s) performed.

11.2.3 Type of material tested and if soil, indicate if the specimen was intact or reconstituted. If sample is concrete, FTB or other engineered material, include the material mix if known and curing time if known for cemented materials.

11.2.4 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 nonhomogeneities.

11.2.5 The size of the needle (diameter to the nearest 0.1mm and length to the nearest 1mm).

11.2.6 Method of needle insertion: pushed or pre-drilled.

11.2.7 Type of thermal grease used (if applicable).

11.3 Record as a minimum the following test specimen data: 11.3.1 Initial gravimetric water content to the nearest 0.01 kg/kg, or 1.0 %.

11.3.2 Dry density (or unit weight) to the nearest 10 Kg/m^2 .

11.3.3 The mass of the specimen to the nearest 0.1 g.

11.3.4 The average diameter and length of the specimen to the nearest 1.0 mm.

11.3.5 Time, nearest 0.1 s, and temperature, nearest 0.01 K, readings for heating, cooling, or both as necessary

11.3.6 (Optional) Time versus temperature plot. See Fig. 5 for an example of an idealized curve of the data.

11.3.7 Slope of the temperature drift measured during equilibration, if it is used in the analysis.

11.3.8 Power per unit length of the heater to the nearest 0.01 W/m.

11.3.9 Thermal conductivity to the nearest 0.01 W/($m \cdot K$).

12. Precision and Bias

12.1 An interlaboratory study involving line-source methods, including needle probes used for rock and soils, was undertaken by ASTM Committee C16 (10). The materials of known thermal conductivity that were evaluated included Ottawa sand and paraffin wax (having a thermal conductivity similar to certain soil and rock types). The results indicated a measurement precision of between ± 10 and ± 15 %, respectively, with a tendency to a positive bias (higher value) over the known values for the materials studied.

12.1.1 Subcommittee D18.12 welcomes proposals that would allow for a more comprehensive precision and bias statement covering the full range of soil and rock materials.

13. Keywords

13.1 heat flow; infinite line heat source; needle probe; temperature; thermal conductivity; thermal probe; thermal properties



(A) IDEALIZED CURVE

FIG. 5 Typical Record of Data (Idealized Curve)