Designation: ~~D5334 – 14~~ D5334 – 22

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~~ specimens of soil and rock and reconstituted soil ~~specimens and soft rock specimens.~~ specimens, and is effective in the lab and in the field. This test method is most suitable ~~only for homogeneous~~ for homogeneous materials, but can also give a representative average value for non-homogeneous materials.

1.2 This test method is applicable to ~~dry~~ dry, unsaturated or ~~unsaturated materials~~ 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 ~~<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.~~ 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 ~~procedure~~ 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~~ safety, health, and ~~health~~ environmental practices and determine the applicability of regulatory limitations prior to use.*

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

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
~~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 and Data Records in Geotechnical Data

3. Terminology

3.1 *Definitions*—For definitions of common technical ~~terms,~~ terms used in this standard, 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 λ > ~~4~~40.5 W/(m·K).

3.2.3 *thermal grease, n*—~~any~~ heat conductive lubricating material having a value of λ > ~~4~~41.5 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 ~~and the temperature rise with time is recorded over a period of time.~~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 to minimize effects of temperature drift during measurement.~~ analysis. Thermal conductivity is obtained from an analysis of the temperature time series data during the heating cycle and ~~cooling cycle if applicable.~~(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 ~~both~~ intact ~~and~~ soil specimens, reconstituted soil ~~specimens as well as soft~~ specimens, and rock specimens is used to analyze and design systems ~~used, for example, in~~ involving underground transmission lines, oil and gas pipelines, radioactive waste disposal, geothermal applications, and solar thermal storage ~~facilities.~~ 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 ~~Annex A1~~Appendix X1.

[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.

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)—*~~*Digital Multimeter (DMM)—*A device to read voltage and current to the nearest 0.01 V and ~~0.01~~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 ~~vertical~~axial hole having a diameter ~~as close as possible~~ 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 ~~*Intact Soil Specimens:*~~ *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 ~~*Thin-Walled Tube or Drive Specimens*—Cut a 200 ± 30-mm long section of a sampling tube containing an intact soil specimen. The tube~~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/m^3 ~~section shall have a minimum diameter of 50 mm.~~is necessary for resistivity to be under 50 °C·cm/W.

~~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~~ 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~~and diameter of the specimen~~) of the reading including heating and cooling if included, and the radius of the needle. Fig. 1~~to 0.1 mm. Take a~~ 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~~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.~~) 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.

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

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

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

where:

r = distance from the heated needle (mm) (minimum radius of the specimen),
D = thermal diffusivity of the specimen (mm^2/s),

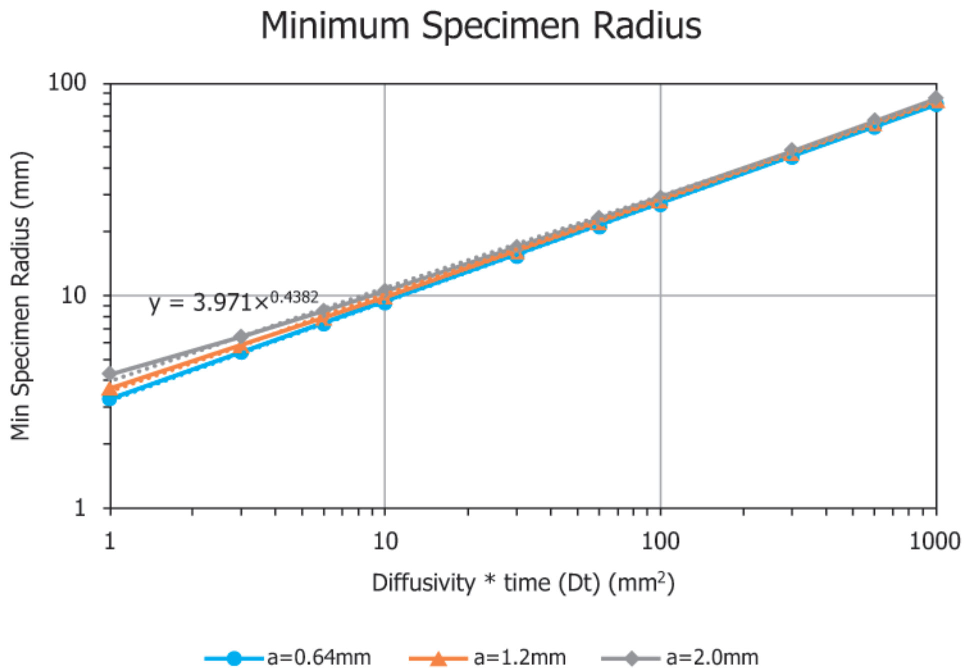

FIG. 1 Minimum Radius of a Specimen

t = time from the beginning of heating to the end of the test (s), and
a = radius of the needle.

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/(m^3·K) according to the equation:

$$D = \frac{\lambda}{\rho_s c_s} \quad (2)$$

where:

D = diffusivity (m^2/s),
λ = conductivity (W/(m·K)),
ρ_s = 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 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. SeeThe 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 Note 2.to the sample length equal to its minimum radius would provide a sufficient security measure.

NOTE 3—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.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.

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

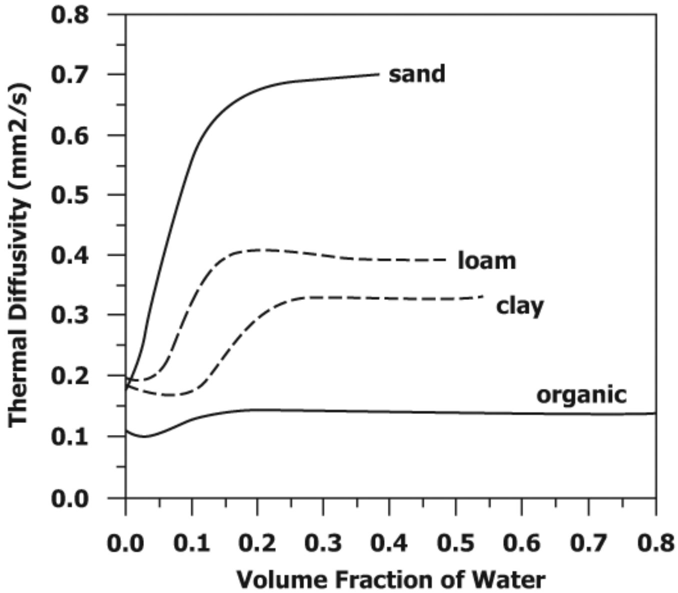

FIG. 2 Diffusivity Values for Select Soil Types

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.~~ technique (compaction and water content both affect the thermal conductivity). For further guidance on the effect of the various compaction techniques on thermal conductivity, refer to Mitchell et al. (~~1~~2). ~~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.~~

7.4 ~~*Soft Rock*~~ *In Situ Soil Specimens:*

7.4.1 ~~Determine and record the mass of the specimen to the nearest 0.01 g and follow the procedure given in~~Dig a pit or trench to the depth of the desired measurement. Prepare a flat, even ~~7.1.4 to determine the specimen diameter and length. The specimen dimensions shall be no less than those of the calibration standard (~~soil face for sensor insertion. ~~8.3).~~

7.4.2 ~~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~~Make sure to make the thermal measurements soon after excavating or exposing the soil face to avoid non-homogenous moisture conditions ~~Note 2.~~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. ~~Calibration~~Verification of Apparatus

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, 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).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
$\lambda_{measured}$ = 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.

8.2 *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 < λ < 5 W/m·K. Suitable materials include dry Ottawa sand, Pyrex 7740, fused silica, Pryoceram 9606 Yearly, conduct the test specified in Section (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 A29 for details on preparation of calibration standards.) The calibration standard shall be in the shape of a cylinder.using one of the verification standards specified in 8.2.1The 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..

8.2.1 *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 < λ < 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.

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 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 ~~come to equilibrium~~ stabilize 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.~~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. ~~1~~3.)

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

9.7 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.~~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~~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.~~

9.10 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~~20-30 steps throughout a cooling period equivalent in duration to the heating cycle.

9.12 Use a suitable ~~inverse method (graphical or statistical)~~ 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.

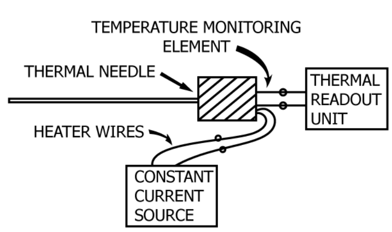

FIG. ~~1~~3 Thermal Probe Experimental Setup