

Standard Method of Test for Thermal Conductivity of Rock Using Divided Bar
Submitted to AMTS 1982
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1. Scope

- 1.1 This method presents a laboratory procedure for determining the thermal conductivity of hard rocks in the temperature range 5-95°C (40-203°F) using a steady state divided bar technique. The technique is a relative method and is applicable to rocks from which a coherent cylindrical sample can be cored and saturated.¹
- 1.2 For satisfactory results in conformance with this method, the principles governing the size, construction, and use of the apparatus described in this method should be followed. If the results are to be reported as having been obtained by this method, then all pertinent requirements prescribed in this method shall be met.
- 1.3 It is not practicable in a 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 the 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 method, to resist in any way the further development of improved or new methods or procedures by research workers.

2. Significance and Use

- 2.1 The test method presented here is used to determine the thermal conductivity (K) of cylindrical rock cores in as close to in situ conditions as can be easily reached in the laboratory. The thermal conductivity of a rock determines how fast heat can flow through the rock. Thermal conductivity is used in the thermal analysis of petroleum reservoirs, geothermal sites, electrical transmission lines, oil pipelines, radioactive waste disposal sites, and ground solar thermal storage.

3. Summary of Method

- 3.1 In brief the apparatus shall consist of a thermal stack comprised, in order, of a heat source (usually supplied by a constant temperature circulating bath), an upper heat flow meter, the test sample, a lower heat flow meter, and a heat sink (usually another constant temperature circulating bath). The stack is insulated or supplied with guard heaters to minimize heat loss through the sides. An axial load of 100±25 bars is applied to the sample so that a reproducible contact resistance is obtained and the sample is saturated to approximate the crack closure characteristics of in situ conditions. The average temperature drop across the test sample and the relative thermal conductivity is thus determined. The apparatus is calibrated by use of quartz and silica glass

¹ The upper and lower temperature limits are controlled by the behavior of the saturating fluid, generally water. Under dry conditions, tests can be run at higher temperatures. Tests could also be run under permafrost conditions if an appropriate circulating medium were used for heat sink and source. With minor modifications the apparatus described can be used on dried samples to temperatures of at least 300°C (Somerton, 1973; Sibbitt et al., 1979).

reference standards run in place of the test sample. The technique was originally described by Birch (1950) and has been described in more detail recently by Goss and Combs (1976).

4. Apparatus

4.1 In detail the apparatus shall consist of the following (See Figure 1)

- 4.1.1 Two constant temperature circulating fluid baths capable of maintaining a specified temperature within the temperature range of 5-95°C (40-203°F) to $\pm 0.5^\circ\text{C}$ ($\pm 0.1^\circ\text{F}$).
- 4.1.2 A hydraulic frame, cylinder and pump to apply a desired axial load to the sample.
- 4.1.3 Temperature measuring devices such as thermocouples (Copper-Constantan), thermistors or RTD's with associated measuring instrumentation (a voltmeter, wheatstone bridge, etc.) and a rotary switch to place each of five pairs of thermocouples into the measuring circuit. The temperature measuring instruments should be accurate to $\pm 0.1^\circ\text{C}$ ($\pm 0.2^\circ\text{CF}$).
- 4.1.4 Two heat flow meters consisting of a layer of material of known (relative) thermal conductivity between two copper discs which have been drilled parallel to their faces for insertion of a thermocouple junction or thermistor. The construction of the meter is discussed in an appendix.
- 4.1.5 Heat transfer boxes for top and bottom (hot and cold) isothermal sections (heat sink and source).
- 4.1.6 Standard discs of silica glass (GE 101 or equivalent) and natural quartz (cut with axis of cylinder parallel to optic axis, $\pm 1^\circ$) of same size as unknown core discs. Standards should be prepared with polished faces flat and parallel to ± 0.004 mm (± 0.001 inches). Typical sample dimensions are 2.5-3.7 cm (1-1.5 inches) in length and 2.5-10 cm (1-4 inches) in diameter.
- 4.1.7 Micrometer(s) with an accuracy of ± 0.004 mm (± 0.001 inches) for measuring length and diameter of standards and samples.
- 4.1.8 Digital or mechanical balance for determining weight of standards and samples so that density, porosity, and fluid loss during test can be calculated.

5. Procedure

5.1 Sample preparation

- 5.1.1 Core and face a cylinder of rock of appropriate thickness and diameter (within 10% of standard length and diameter and stack diameter). Each sample length and diameter tolerance should be ± 0.25 mm (± 0.01 inch). The top and bottom should be finished with 400 or finer silicon carbide or diamond powder on a lap to minimize thermal contact resistance, and should be flat within ± 0.025 mm (± 0.001 inch) total.
 - 5.1.1.1 The diameter of the sample shall be at least 10 times larger than the largest mineral grain. The diameter shall be measured across two perpendicular diameters at mid-height of the sample with a dial micrometer capable of measuring to 0.025 mm (0.001 inches). These reading shall be recorded.
 - 5.1.1.2 The thickness of the sample shall be at least 10 times the diameter of the largest grain, but in no case less than 12.7 mm (0.5 inches). For thinner samples thermal contact resistance may become significant. The length shall be measured at three equally spaced points, with a dial micrometer capable of measuring to the nearest 0.025 mm (0.001 inches). These readings shall be recorded as shown on the form.

- 5.1.2 Small joints and seams are potentially high-conductivity heat flow routes through the sample. Material that has veins running axially through the test sample should be avoided.²
 - 5.1.3 Measure sample size with a micrometer and weight with a digital or mechanical balance.
 - 5.1.4 Saturate sample with water (or other appropriate fluid). The saturation technique should follow ASTM Standard Number. The sample should be kept in the fluid following saturation until the test is complete. Measurement of the weight of the saturated samples allows determination of the mass of saturating fluid and of the connected porosity if desired. Generally, water is used for hard rock, but other materials may require special fluids, such as saturated brine for salt or glycerin for slaking mudstones.
 - 5.1.5 Weigh sample after test to check for loss of fluid during test. Loss should be less than 10% of fluid present.
6. Procedure for Calibration Measurements
 - 6.1 Allow sufficient turn on time for baths, heat sink and source, and stack to reach an operating temperature equilibrium. A temperature difference of 10-20°C (18-36°F) as required for appropriate accuracy in temperature differences should be maintained across the thermal stack.
 - 6.2 Place first reference standard (quartz or silica glass) into stack and allow to reach equilibrium (typically about 30 minutes for 1.25-4 cm (0.5-1.5 inch) long cylinders). A water soluble grease is applied to the top and bottom of the standard to reduce contact resistance effects. The axial load on the standard should be 100±25 bars (1500 psi).
 - 6.3 Read the temperatures sequentially from top to bottom with a digital voltmeter or other device. Repeat the sequence so that each temperature difference is read at least twice. The readings are then averaged.
 - 6.4 Measurements are recorded as shown on the form.
 - 6.5 Remove standard and replace with second standard or sample.
7. Calibration of the thermal stack
 - 7.1 The apparent thermal conductivity of the reference material used in the heat flow meters is a function of many variables. Since the technique is relative, the effects of most of these variables are removed by calibration using standards of natural (crystalline) quartz (with a higher thermal conductivity than most rocks) and silica glass (fused quartz) (with a lower thermal conductivity than most rocks).
 - 7.2 Calculation of the calibration value is discussed in section 9. The philosophy of the construction of the thermal stack is given in an appendix.
 - 7.3 Agreement within 10% of the calculated apparent thermal conductivity of the material in the upper and lower heat flux meters means that such effects as side heat losses (or gains) are within acceptable error limits. Effects of thermal contact resistance are removed by a similar treatment of standard and sample cores.
 - 7.4 Because of possible environmental effects on the apparent thermal conductivity of the reference material, calibration runs (at least 2 of the silica glass and 2 of the quartz) should be carried out each day the apparatus is operated.
8. Test Procedure for Samples

² Note: The technique is restricted to the determination of thermal conductivity in one direction so that anisotropic materials must be measured in more than one direction.

- 8.1 Following calibration of device, an unknown sample is placed in the thermal conductivity device directly from fluid storage.
 - 8.2 The ends of the sample are coated with water soluble grease to insure good thermal contact with the copper during test.
 - 8.3 An axial load of at least 100 ± 25 Bars (1500 psi) is applied to the stack.
 - 8.4 Sample is allowed to reach temperature equilibrium.
 - 8.5 Temperature differences are measured as was the case with the standard and recorded on the form sheet.
9. Calculations

9.1 The thermal conductivity is calculated from a comparison of the temperature drop across the standards to the temperature drip across the unknown sample. The equation is as follows

$$K_{samp} = \left(\frac{\Delta T_1 + \Delta T_3}{2\Delta T_2} \right) \cdot K_{ref} \cdot Th_{corr} \cdot D_{corr} \quad (1)$$

$$K_{ref} = \frac{1}{2} \left[\frac{\Delta T_1 + \Delta T_3}{2\Delta T_2} K_{QStd} + \frac{\Delta T_1 + \Delta T_3}{2\Delta T_2} K_{SGStd} \right] \quad (2)$$

where

K_{samp} = thermal conductivity of unknown sample at test temperature.

K_{ref} = average apparent (relative) thermal conductivity of reference material at operating temperature in upper and lower heat flux meters.

K_{std} = thermal conductivity of natural quartz or silica glass standard at calibration temperature.

ΔT_1 = temperature drop across a particular portion of apparatus (see Figure 1) during test with sample, natural quartz or silica glass in the bar. The agreement of ΔT_1 and ΔT_3 for each run should be within 10% so that side heat loss (or gain) is within acceptable limits.

Th_{corr} = correction for difference between standard and sample thickness

D_{corr} = correction for difference between standard and sample diameter

9.1.1 The equation for the thickness correction is

$$Th_{corr} = \frac{Th_{samp}}{Th_{std}}$$

9.1.2 The equation for the diameter correction is

$$D_{corr} = \frac{D_{std}^2}{D_{samp}^2}$$

9.2 The thermal conductivity of the silica glass and natural quartz reference materials used varies as a function of temperature. The appropriate reference conductivity must thus be calculated at each sample temperature. The equations based on absolute values determined by Ratcliffe (1959) follow.

9.2.1 The equation for thermal conductivity of GE 101 Silica Glass as a function of temperature is

$$K_{SG101} = 3.1628 + 0.004173T$$

where T is temperature in °C.

- 9.2.2 The equation for the thermal conductivity of a natural quartz disk, with axis parallel to the optic axis, as a function of temperature is

$$K_{QTZ} = 16.470 - 0.06479T + 0.0002098T^2$$

where T is temperature in °C.

- 9.2.3 The temperature of the device at the standard and sample position should be constant to within 0.5°C (1°F) during a set of tests.

10. Report

10.1 The report shall include the following.

- 10.1.1 A discussion of the scope of the testing program, which includes the number of specimens, tested, rationale for sample selection and the limitations of the testing program.
- 10.1.2 Description of the specimens. The rock type, structure and fabric, grain size, discontinuities or voids, and weathering of the samples shall be describes as a minimum. Further detail depends on the application of the results.
- 10.1.3 A detailed listing of the equipment actually used for the test shall be included in the report. The name, model number, and basic specifications of each major piece shall be listed.
- 10.1.4 If the actual equipment or procedure has varied from the requirements contained in this procedure, each variation and the reasons for it shall be noted. The effect of the variation upon the test results shall be discussed.
- 10.1.5 A summary table of results including test suite designations, temperature ranges, average coefficients of thermal conductivity, ranges, and uncertainties shall be presented.
- 10.1.6 A table of results for individual samples including, as a minimum, individual specimen number, rock type, temperature range, and thermal conductivity shall be presented.
- 10.1.7 For each suite of rock samples, the mean value of thermal conductivity, range, standard deviation, and 95% confidence limits for the mean shall be calculated as a minimum. The uncertainty of the sample suite shall be compared with the measurement uncertainty to determine whether measurement error f sample variability is the dominant factor in the results.

11. Comments

11.1 Temperature effects on thermal conductivity

- 11.1.1 This standard is designed for measurements between temperature limits obtainable using water as a circulating medium in a constant temperature bath and at temperatures near enough to room temperature that side heat losses (or gains) can be controlled by insulation alone rather than the use of guard heaters. With minor modifications the same apparatus could be used for higher temperature thermal conductivity measurements.
- 11.1.2 Thermal conductivity of rock is a function of temperature. The effect has been described most recently by Roy et al. (1981). Typical effects for granites are 1-2%/10°C. Hence measurement temperature must be specified for each test and use of measured values at other than test temperatures must involve a correction.
- 11.1.3 The standard technique is a one-dimensional measurement of thermal conductivity. Anisotropic rock must be cored in different orientations and enough samples run to determine the tensor components for complete specification of thermal conductivity.

12. Precision and Accuracy

12.1 Precision of individual measurement

12.1.1 Error propagation calculations and repeat measurements indicate a precision of an individual measurement of $\pm 1\%$ if the provisions of the standard are followed. Daily recalibration, constant axial load, controlled side losses, and constant temperature are necessary to achieve this precision.

12.2 Precision of determined thermal conductivity

12.2.1 The standard technique is relative so the intra and interlaboratory precision depends on the precision of the thermal conductivity determination of the standard materials used (see below) and the similarity of material obtained by different laboratories at different times.

12.2.2 In 1975 a comparison was made between 4 laboratories measuring thermal conductivity by this divided bar technique (3 in the U.S. and 1 in Canada). No instructions were given other than to measure the thermal conductivity. The results for 8 samples are shown in Table 1. The results indicate a worst case error of $\leq 4\%$ and error of the mean of several samples of $< 1\%$.

12.3 Accuracy of determined thermal conductivity

12.3.1 The accuracy of the measurement depends on the accuracy of the silica glass and natural quartz thermal conductivity values taken for calibration.

Table 1 *Interlaboratory comparison of divided bar thermal conductivity measurements

Sample Number	Mean Thermal Conductivity (Wm ⁻¹ k-1)	% Deviation from the mean			
		Lab 1	Lab 2	Lab 3	Lab 4
1	1.75	+0.3	+5.4	-6.0	+0.3
2	1.91	+2.5	-3.8	+0.4	+0.9
3	2.36	0.0		+2.8	-2.7
4	2.78	+1.7		-1.2	-0.5
5	2.78	+1.4	-2.2	+1.1	-0.4
6	2.91	+0.3	+4.1	-3.4	-1.0
7	3.44	-4.2		+6.0	-1.8
8	3.75	+0.9	-1.8	+2.5	-1.5

mean deviation from the mean(%) +0.4 +0.3 +0.3 -0.8

rms deviation from the mean (%) 1.9 3.7 3.5 1.4

*Results tabulated by David S. Chapman, Department of Geological Sciences, University of Utah, based on comparisons made in 1975.

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