UNITED STATES DEPARTMENT OF THE INTERIOR

GEOLOGICAL SURVEY

Laboratory line-source methods for the measurement of thermal conductivity of rocks near room temperature

by

J. H. Sass, J. P. Kennelly, Jr., E. P. Smith, and W. E. Wendt

Open-File Report 84-91

1984

Abstract

For the conventional needle probe, the standard mode (line source in an infinite medium) is achieved simply by inserting the probe into unconsolidated material or into a hole drilled in harder material. A halfspace mode is achieved by embedding the needle probe in an epoxy material of low thermal conductivity (<0.2 Wm^{-1} K^{-1}) and grinding the material away until the needle is flush with a flat surface. The sample to be measured is placed on the halfspace surface, and the conductivity is measured in the usual way. Either configuration is capable of yielding values of thermal conductivity of consolidated rocks comparable in accuracy to values obtained with the steady-state, divided-bar technique most commonly used for these rocks. The line-source techniques are of particular advantage for materials that prove difficult to machine into the cylindrical disk specimens required for the steady-state apparatus. The halfspace probe is of additional value for rocks that are too friable to machine, but too hard to allow the drilling of the long, small diameter (38 × 1 mm) holes required for the standard application of the needle probe.

INTRODUCTION

The thermal conductivity of most earth materials is within the range 1 to 5 $Wm^{-1} K^{-1}$. Measurement of this quantity to within a few percent is essential to obtaining reliable estimates of terrestrial heat flow and is important in certain engineering applications. In modern practice, the primary methods for the measurement of rock conductivities have been the steady-state guarded hot-plate or divided-bar techniques (see e.g., Birch and Clark, 1940; Birch, 1950; Ratcliffe, 1959) and the transient line-source "needle probe" (Von Herzen and Maxwell, 1959). These techniques originally were used only for measurements on intact cores of solid or unconsolidated rock, but both methods have been adapted to allow measurement of conductivity on chips or drill cuttings (Horai, 1971; Sass and others, 1971a). Many other novel and ingenious experimental configurations have been proposed and developed, but most have had quite limited application and have not been deployed as routine, production techniques.

Among the various experimental situations envisaged by Jaeger (1959) for which analytical mathematical solutions can be obtained, are those in which a line-source heater and a point temperature sensor are placed on or near the surface of a semi-infinite solid. A commercial version of this configuration, the Shotherm "Quick Thermal Meter" (QTM) has been developed and tested (Ito and others, 1977) and has been shown to give results quite comparable to conventional methods (Sass and others, 1984) provided samples of sufficient size are available. The requirement for large samples (50 mm × 100 mm × 100 mm for rocks with conductivity higher than 3.5 $Wm^{-1} K^{-1}$) seems to be one of the major drawbacks of this instrument in obtaining conductivities from small diameter (~25-50 mm) diamond drill core.

The standard needle-probe has recently been adapted to the "halfspace" mode by Victor Vacquier (Carvalho and others, 1980; Vacquier, 1984) for the measurement of thermal conductivity of large cores from oil wells. We have produced our own version of Vacquier's probe and have found it quite useful in obtaining thermal conductivities from isotropic material that is too friable to be machined into disks for the divided bar but too hard to allow readily, the drilling of holes for insertion of the standard needle probe.

Inasmuch as our needle-probe system has undergone many changes since it was last described (Lachenbruch and Marshall, 1966), and because many colleagues have expressed interest in the details of our halfspace probe, we present here a fairly detailed description of both the probes and the digital data system, which is essentially identical to that deployed in our logging truck.

Acknowledgments. We are grateful to Arthur Lachenbruch and Victor Vacquier for their constructive comments and suggestions.

PHYSICAL DESCRIPTION OF NEEDLE-PROBE SYSTEM

The needle probe (plate 1) is basically a segment of 0.55 mm id 0.91 mm od stainless steel tubing either 38 mm or 64 mm long, having a loop of heater wire of specific resistance 0.7Ω mm^{-1} and a bead thermistor with resistance at 20°C typically in the range 3-5KΩ located in the center of the probe (Figure 1). Probe wall, thermistor and leads, and heater all are mutually insulated with leakage resistance among them greater than 5 GΩ. In the production mode, samples are placed in individual compartments in a large, thermally lagged cabinet. The cabinet is wired so that up to 20 needle probes can be monitored in succession.

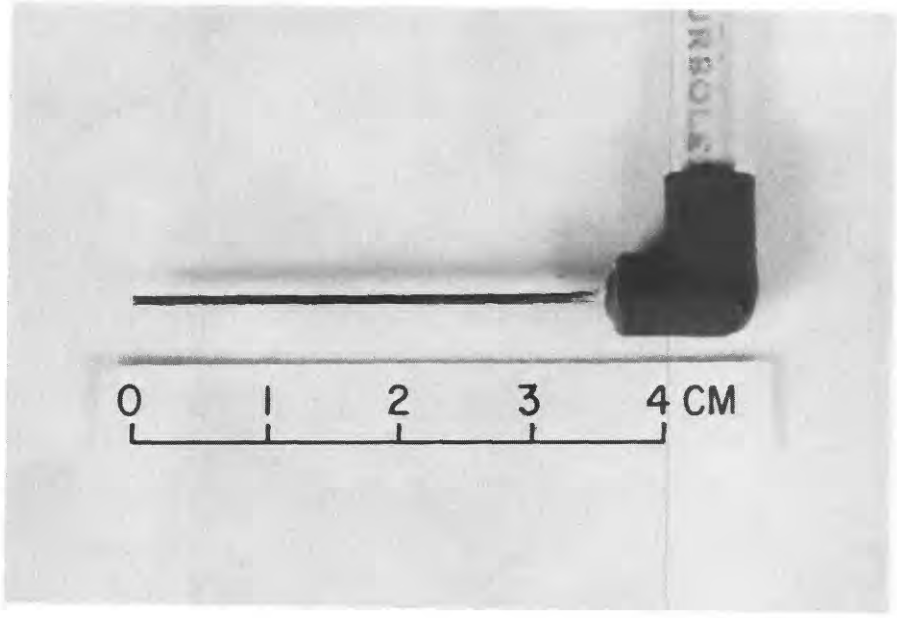
With unconsolidated material, the needle is simply pushed into the sample at a sufficient distance (>20 mm) from the end so that the sample can be considered as an infinite medium for the duration of the experiment (1 to 2 minutes). For consolidated material, a hole of sufficient length is drilled using a twist drill (500 RPM) in a variable speed drill press, or for hard materials, a diamond-tipped drill (8500 RPM). Drill diameters are such that the needle probe is a very tight fit in the hole. Porous materials are measured in a saturated state, insuring a good thermal contact between probe and material. With low porosity materials, the needle is coated with a metallic oxide-silicone paste before being inserted into the drilled hole.

After the needle has been inserted, the sample is placed in one of the insulated compartments, the probe is plugged into the switching circuit and all samples (up to 20) to be tested are allowed to come to thermal equilibrium with their surroundings. The temperature of the sample cabinet is not thermostatically controlled so that usually there is a slow temperature drift on the order of 0.1°C hr^{-1}. To take account of this, a five-minute "drift run" is made prior to the conductivity test under control of the timer (Figure 2), the digital voltmeter (DVM) samples the thermistor resistance at one-minute intervals and temperatures are calculated and stored within the memory of the programmable calculator.

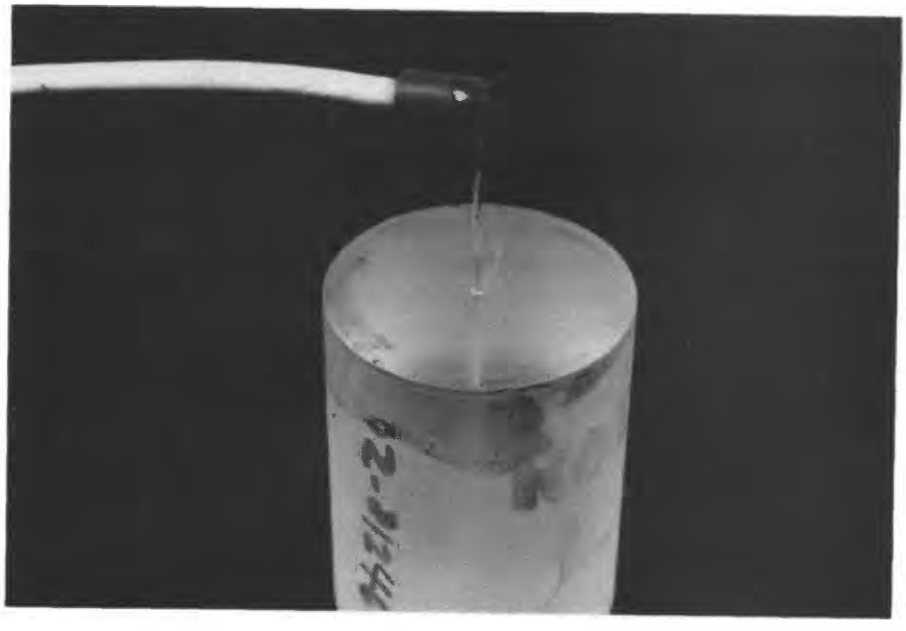
Upon completion of the drift run, the timer activates the constant current source. The current supplied is typically between 50 and 100 mA, for a power input of a few watts per meter resulting in a temperature rise of between 1 and 4 °C during the course of an experiment. The timer also directs the DVM to sample thermistor resistances at one-second intervals for 148 seconds. The calculator converts these resistances to temperatures and stores the data, while at the same time, plotting a temperature versus log time curve in real time and (at the option of the operator) recording the data on the model 4923 digital tape recorder (Figure 2). At the conclusion of the conductivity test, the calculator computes and prints out the drift rate. Drift rates of 1°C hr^{-1} or greater are considered excessive and result in the rejection of the test and a re-test after the drift rate has settled down to an acceptable level. If the drift rate is acceptable and the real-time graph shows no aberrant effects, the operator reduces the data over pre-selected time intervals to obtain values of thermal conductivity. The theory of the line source is abundantly documented (see Carslaw and Jaeger, 1959; Von Herzen and Maxwell, 1959) and temperature-log time data are reduced

PLATE 1

a) Short needle probe.



b) Short needle probe and fused silica standard.



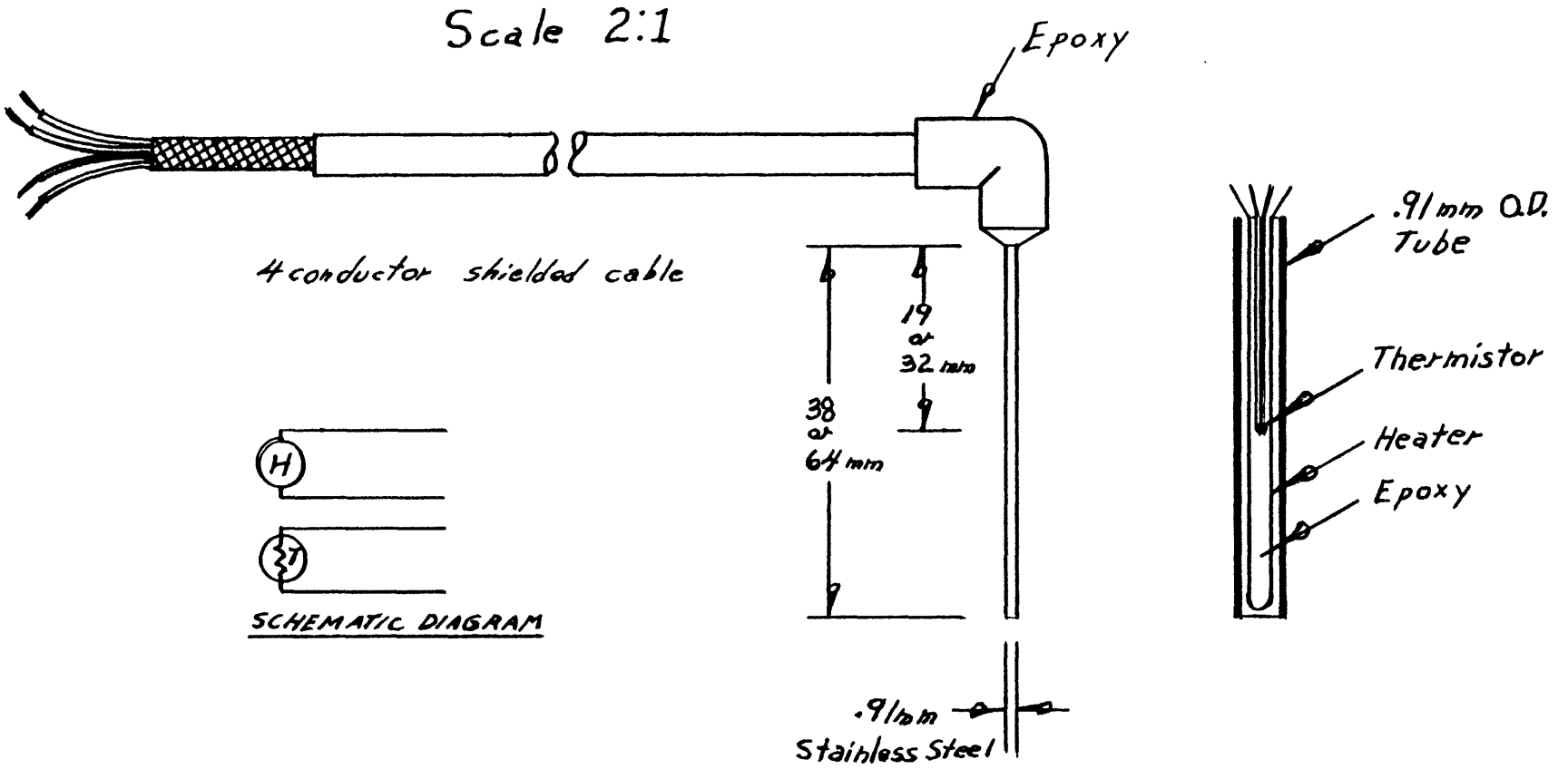


Figure 1. Scale diagram of needle probe.

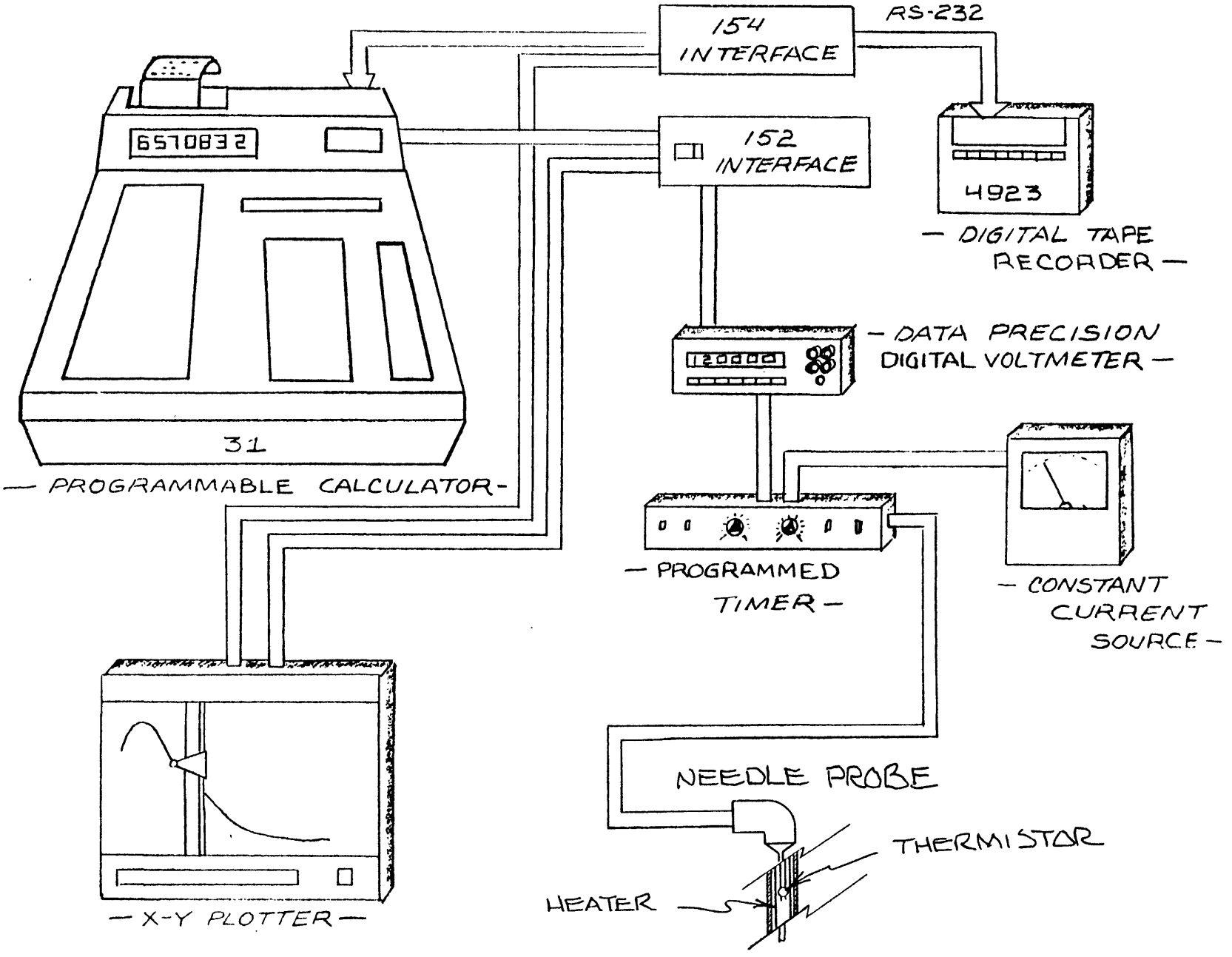


Figure 2. Schematic block diagram illustrating the various components of the USGS needle-probe system.

according to the well-known relation

$$T(\tau) = \frac{I^2R}{4\pi K} \ell n\ \tau \qquad (1)$$

where T and τ are temperature and time, respectively, I is the constant current delivered to the heater, R is the specific resistance of the heater, and K is the thermal conductivity. We define for each probe a constant (C) which is nominally equal to $R/4\pi$ and which is adjusted periodically as part of the calibration procedure (discussed below).

For the short (38 mm) probe, equation (1) is valid for times greater than ~15-20 seconds and less than ~60-90 seconds depending on the conductivity of the material being tested. The upper limit may be extended to 2 minutes or more for the 64-mm long probe provided the sample is large enough.

Before data reduction, the observed temperatures are corrected for the drift rate in effect prior to the heater-test. For example, if the least-squares linear drift rate is $+0.1°C\ hr^{-1}$ ($\sim 3 \times 10^{-5}\ °C\ sec^{-1}$), then the time elapsed in seconds is multiplied by the drift rate (per second) and the resulting temperature is subtracted from the observed temperature. The corrected temperature-log time pairs are then subjected to a linear least-squares regression analysis over standardized time intervals, typically 20-40 seconds for the 38-mm probe and 30-80 seconds for the 64-mm probe. From equation (1) and our definition of the probe constant ($C = R/4\pi$), it follows that the slope of the least-squares line is I^2C/K from whence

$$K = \frac{I^2C}{\text{slope}} \qquad (2)$$

In the calibration procedure, the probe is inserted into a hole drilled along the axis of a fused silica rod having the same diameter as the probe length. The conductivity test is then carried out as outlined above and the constant C is adjusted to produce the value of K established by Ratcliffe (1959) at the mean temperature of the test. Calibrations are carried out once or twice a year and usually result in changes to C of 1% or less. Larger changes indicate serious faults in the heater or thermistor circuit and usually result in repairs, recalibration, or disposal of the probe in question. In addition to regular calibrations, frequent calibration checks are made with fused silica when a series of samples is being measured.

COMPONENTS OF THE SYSTEM

Because of the fast-paced development in the electronics industry, we shall not present detailed descriptions and specifications of the individual components. Instead we present a brief summary of the specifications of the major components as a guide in selecting from the large number of components currently available.

Programmed Timer. Designed and fabricated by W. E. Wendt in our electronics shop. Basic time generation is by a crystal-controlled oscillator with base frequency (f) of 100.000 KHZ ±0.0001%. The temperature coefficient Δf% between 0-70°C varies from +0.0075 to -0.032. Variable intervals of between 1 and 60 seconds can be programmed based on division of the crystal-controlled base frequency.

Constant current source. Soltec Model 6141

Programmable
DC voltage/current generator

Accuracy (6 months + 23°C ±5°C)
10 mA/100 mA ranges:
10 mA: ±0.035% setting + ±3μa
100 mA: ±0.035% setting + ±30μa

Temperature coefficient (0°C to + 40°C)
10 mA/100 mA ranges:
10mA: ±0.002% setting + ±200na
100 mA: ±0.002% setting + ±2μa

Range	Span	Resolution
10 mA	0 to ±11.999 mA	1 μa
100 mA	0 to ±119.99 mA	10 μa

Digital multimeter. Data Precision Model 3500 Digital Multimeter

Accuracy: (6 months, =23°C ±5°C)

1KΩ/10KΩ/100KΩ ranges:

± .007% reading
± .001% full scale
± 1 least significant digit

Temperature coefficient (0°C to 50°C):

1KΩ/10KΩ/100KΩ ranges:

(±0.001% reading ± 0.0004% full scale)/°C

Resistance measuring configuration:

True four-wire

Range	Resolution	Max. Test Current
1.00000KΩ	10 mΩ	1 mA
10.0000KΩ	100 mΩ	100 μA
100.000KΩ	1 Ω	10 μA

Data processing. The data acquisition and processing system was assembled from Tektronix components in 1974. The individual components are identified in the sketch (Figure 2) and are described in detail in the appropriate TEK manuals. The Tektronix 31 programmable calculator system is considered obsolete by the manufacturer and is thus no longer sold; however, a variety of suitable substitutes having larger memories and faster response is available commercially (any microcomputer system and the appropriate peripherals will do the job). Even though this system is theoretically obsolete, we intend to leave it in place until the unavailability of spare parts and/or increased down time and maintenance costs dictate a replacement.

HALFSPACE PROBE

There is a class of rock material from which it is difficult to prepare disks for the divided-bar apparatus and to drill holes for the needle probe but for which it is relatively easy, using slabbing saws and coarse laps, to prepare a smooth, flat surface. In fact, even where it is possible to machine or drill rocks, it is usually quicker and simpler to prepare a flat surface on an existing slab. Because of this, a reliable technique using the principle of a line source on the surface of a semi-infinite medium or halfspace represents a considerable saving in the time of sample preparation and consequently, a higher rate of productivity in measuring conductivities than that achievable using conventional methods.

The commercially available QTM showed promise in a comparison with the divided bar (Sass and others, 1984), but it suffers from two drawbacks: 1) As already mentioned, the requirements for minimum sample size render the QTM unsuitable for studying the small core usually associated with diamond core holes; 2) The purchase price (~$15,000 at this time) makes it unfeasible for any but the most richly funded thermal labs.

An alternative that reduces the size requirement and costs only a few percent of the price associated with the QTM (particularly if a needle probe capability already exists) was perfected by Victor Vacquier as a means of dealing with slabbed core from deep oil wells (usually 50 mm or more in diameter). The design consisted of encapsulating a needle probe in material of low conductivity, grinding away the material until the needle was flush with a flat surface of the low-conductivity material, and placing samples of rock directly on to this surface for measurement of conductivity. To a first approximation and for an appropriate time period, the sample can be considered a semi-infinite medium with a line source on the surface with temperature-time relation governed by

$$T(\tau) = \frac{I^2R}{2\pi K} \ell n\ \tau \qquad (3)$$

Thus in theory, all we need do is to double our probe constant (C, equation 2) to obtain the proper value for thermal conductivity.

Based on a brief visual inspection of one of Vacquier's probes, we converted one each of our short and long needle probes into as close a copy as possible of his halfspace design (plate 2). The encapsulating medium was Emerson and Cummings "Stycast 1090" an epoxy base with hollow sodium borosilicate glass spheres ranging in diameter from 44 to 175 μm dispersed within it. The hardened medium has a density of less than 1 g cm^{-3} and a thermal conductivity of 0.18 $Wm^{-1}\ K^{-1}$, about an order of magnitude lower than most rocks.

In practice, the halfspace probe is immersed in water in a plastic dishpan together with the samples to be measured. The samples are placed, in turn, on the halfspace probe, allowed to come to thermal equilibrium, then the needle probe test as described above is performed. The water ensures a

PLATE 2

Long needle probe embedded in epoxy (halfspace mode).

good contact between sample and probe. Between tests, the probe is "rested" for a minimum of 15 minutes to allow the memory of the previous test to dissipate. This is facilitated by placing a rectangular block of aluminum on the halfspace probe.

The temperature-log time curves for the halfspace probes show slight but distinct curvature relative to the corresponding curves for the needle probe (Figures 3, 4, and 5). For the time intervals used in determining conductivity, however (20-60 seconds for the 38-mm probe; 20-100 seconds for the 64-mm probe), the departures from linearity were not extreme, and the values of the probe constant derived from calibration runs with fused silica were only a few percent different from the theoretical 2C expected if the probe material were a perfect insulator.

We thus handled the problem of curvature in temperature log time curves empirically, deriving different probe constants for three overlapping time intervals for calibration runs with fused silica, and applying these constants over the same time intervals for unknowns as a check on the internal consistency of individual tests. The constants derived from fused silica gave internally consistent values in agreement with divided-bar results over a large conductivity interval with the result that we feel a great deal of confidence in the technique. There were no systematic effects apparent in comparing results from slabbed and rough lapped (~100-mesh lap) surfaces with highly polished surfaces. Asperities or ripples with vertical dimensions greater than ~.05 mm did have an adverse effect on the reproducibility of the technique, however.

A comparison between the halfspace probe and the divided-bar was made on nine specimens ranging in conductivity from 1.4 to 5.8 $Wm^{-1} K^{-1}$ (Table 1, Figure 6) -- a range that includes the vast majority of consolidated geological material. No systematic effects are evident. A regression of the form $y = Ax$ on the points in Figure 6 resulted in a coefficient of correlation of 0.99 and a slope of 1.0 within the errors of the analysis (cf. similar results for the QTM obtained by Sass and others, 1984).

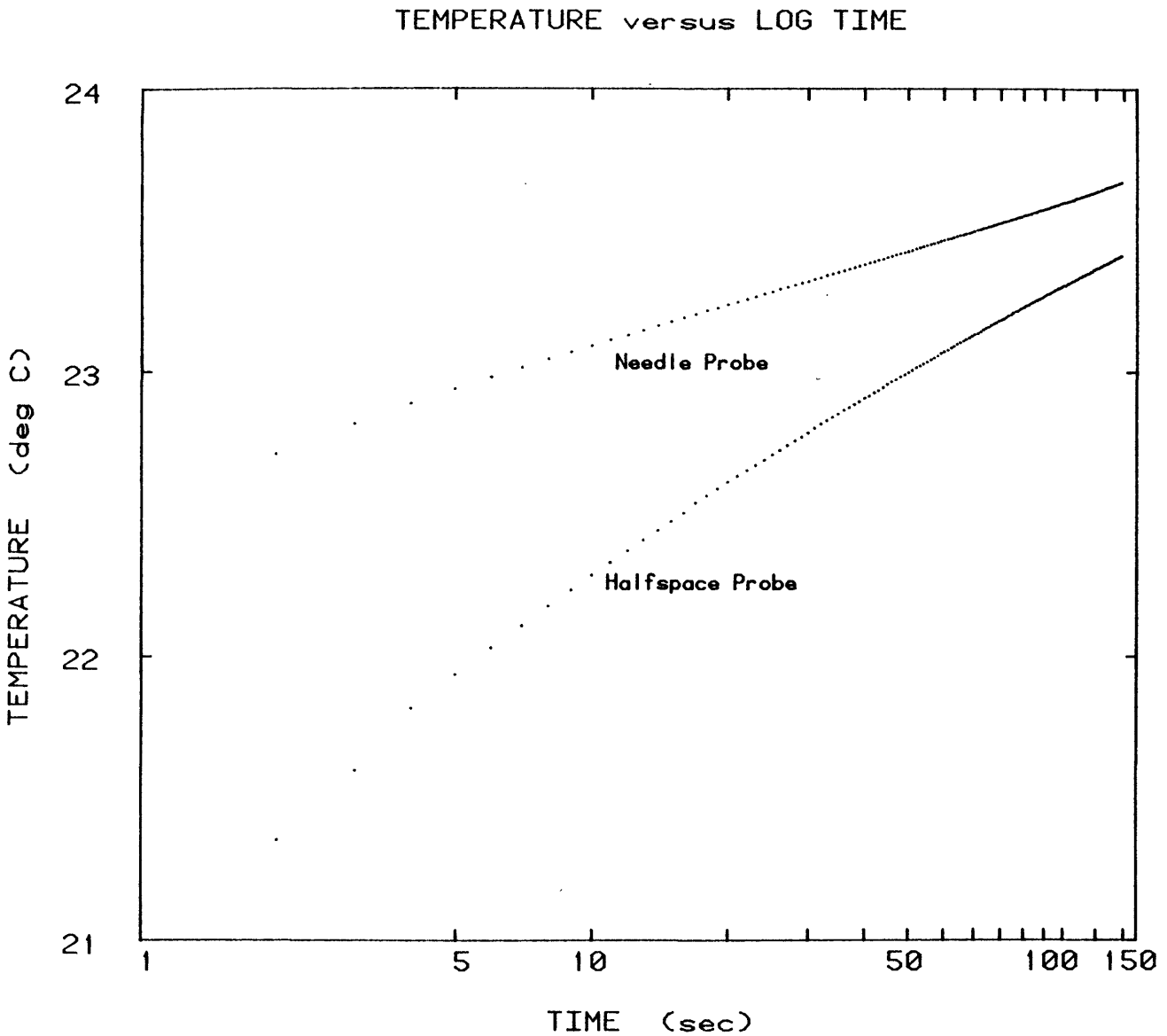


Figure 3. Complete temperature-log time curves for both the needle probe and halfspace probe (38 mm long probes) for calibration runs on fused silica.

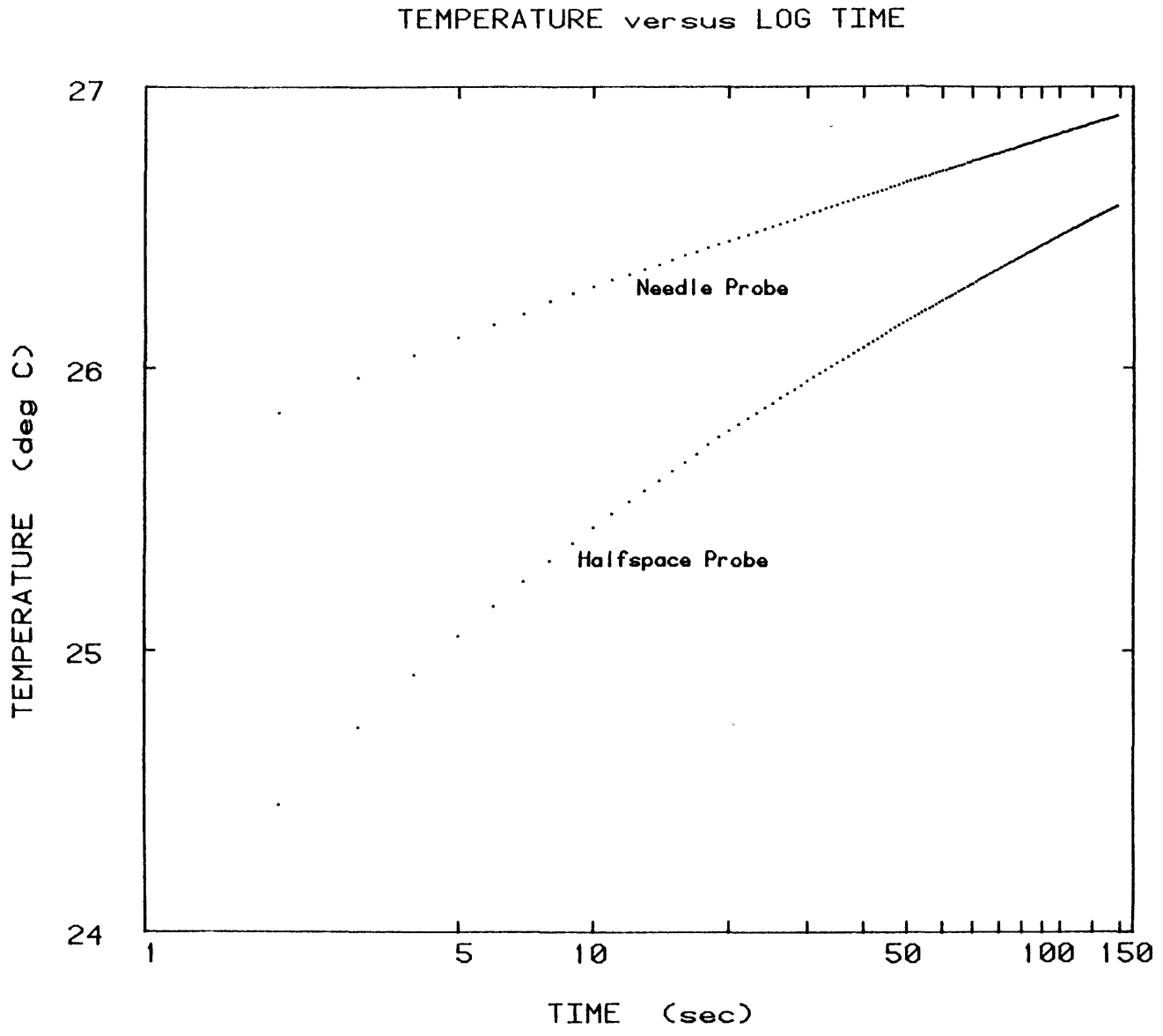


Figure 4. Complete temperature-log time curves for both needle probe and halfspace probe (64 mm long) for calibration runs on fused silica.

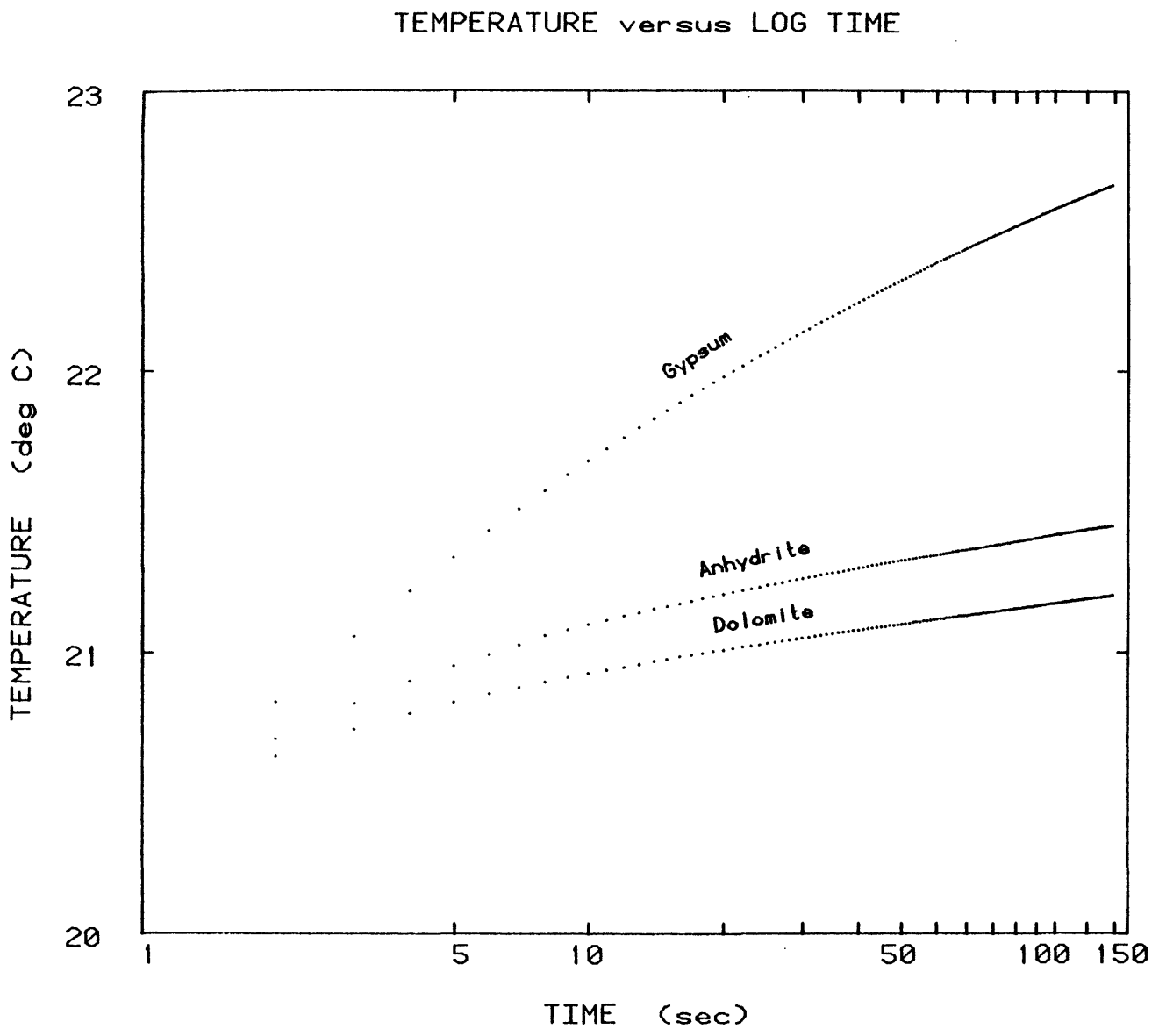


Figure 5. Complete temperature-log time curves for halfspace probe (38 mm) runs on three sedimentary materials. Power input is the same in all cases.

TABLE 1. A comparison between thermal conductivities measured at ~23°C by the USGS needle probe in a "halfspace" configuration and the USGS divided-bar apparatus

Sample designation	Halfspace probe*			Divided bar	Material
	N	$\bar{K}$	SD	K	
BDG1	3	5.83	0.05	5.48	Dolomitic siltstone
BDG2	3	4.73	0.14	5.14	Anhydrite
BDG5	3	5.78	0.11	5.67	Dolomite
USGS2	4	5.22	0.12	5.09	Dolomite
M1S	4	2.96	0.06	2.96	Marble
M2S	4	2.96	0.04	2.78	Marble
M3N	4	2.63	0.06	2.65	Marble
GRW	2	2.27	0.05	2.40	Granite
BDG6	3	1.36	0.02	1.36	Gypsum

*N = number of tests run; $\bar{K}$ is arithmetic mean conductivity, and SD, standard deviation.

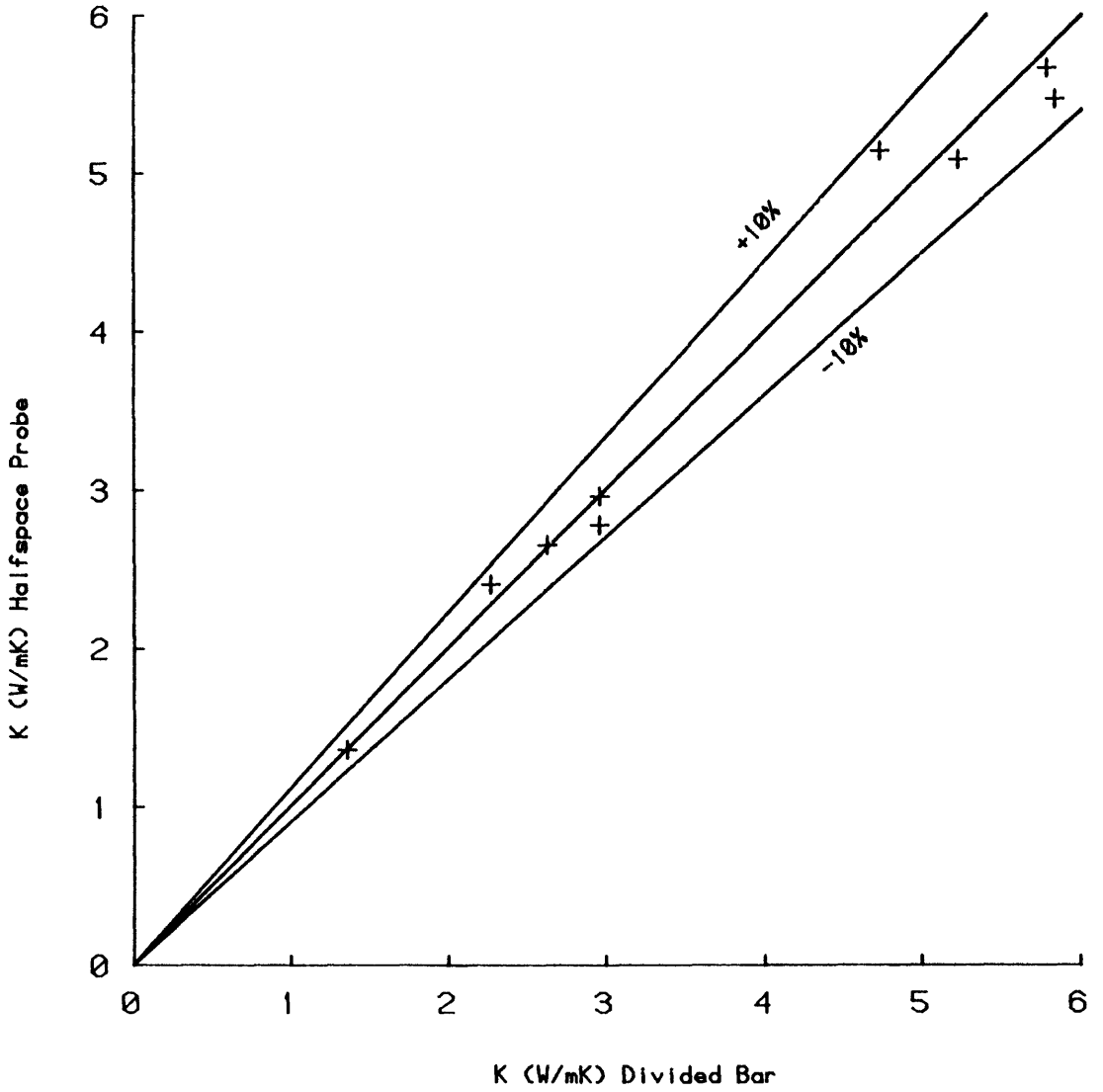


Figure 6. A comparison between the halfspace probe and the USGS divided-bar apparatus for 9 rock specimens (see Table 1).

DISCUSSION

Through careful calibration and intercomparison, we have established that there are three options available in the USGS Geothermal Laboratory for the routine measurement of the thermal conductivity of consolidated rocks near room temperature. Each of the three methods will have advantages and disadvantages in any given situation, but in general, any one of them will give reliable values of thermal conductivity, and all will yield values of comparable accuracy.

For easily machinable crystalline rocks and anisotropic rocks, our preferred technique remains the steady-state divided-bar method described by Sass and others (1971b). We would not hesitate to use the halfspace probe for isotropic crystalline materials however, either as a substitute for or an adjunct to the divided bar, if a bottleneck were to appear in the production of disks for the divided bar and the results were needed quickly.

The divided bar should be used whenever possible with anisotropic material. For heat-flow determinations, the value of conductivity required is that in a vertical direction, and this is a straightforward and unambiguous measurement with the divided bar. For friable materials, either line-source method may be used, but measurements in at least two orientations and a subsequent interpretive procedure are required to specify conductivity in a given direction (see Simmons, 1961; Grubbe and others, 1983).

For measurements on chips, we still prefer the divided bar (Sass and others, 1971a). Reliable conductivities can be obtained on much smaller volumes of sample than with the needle probe (cf. Horai, 1971). It has also been demonstrated by Alan Beck and his associates (see e.g., Frodemisi and Beck, 1983) that for unconsolidated highly permeable material, problems with high temperature gradients and high and variable contact resistances in the vicinity of the probe can lead to serious errors in needle-probe results.

References

Birch, Francis, 1950, Flow of heat in the Front Range, Colorado: Geological Society of America Bulletin, v. 61, p. 567-630.

Birch, Francis, and Clark, Harry, 1940, The thermal conductivity of rocks and its dependence on temperature and composition: American Journal of Science, v. 238, p. 529-558 and 613-635.

Carslaw, H. S., and Jaeger, J. C., 1959, Conduction of Heat in Solids: Oxford University Press, New York.

Carvalho, H. D. S., Purwoko, Siswoyo, Thamrin, M., and Vacquier, V., 1980, Terrestrial heat flow in the Tertiary basin of Central Sumatra: Tectonophysics, v. 69, p. 163-188.

Frodemisi, S. P., and Beck, A. E., 1983, Induced convection during cylindrical probe conductivity measurements on permeable media in thermal conductivity XVII: Proceedings of the 17th International Thermal Conductivity Conference, J. G. Hast, editor, Plenum Press, p. 619-634.

Grubbe, K., Haenel, C. R., and Zoth, G., 1983, Determination of the vertical component of thermal conductivity by line source methods: Zbl. Geol. Palaont., v. 1, p. 49-56.

Horai, K., 1971, Thermal conductivity of rock-forming minerals: Journal of Geophysical Research, v. 76, p. 1278-1308.

Ito, Y., Saito, T., and Nagumeo, M., 1977. Shotherm QTM measurement of rock specimens, Shotherm Sales Information No. 111: abstracted and translated with permission from the original paper in Japanese: Shotherm QTM measurement of rock specimens: Chinetsu, v. 14, p. 21.

Jaeger, J. C., 1959, The use of complete temperature-time curves for determination of thermal conductivity with particular reference to rocks: Australian Journal of Physics, v. 12, p. 203-217.

Lachenbruch, A. H., and Marshall, B. V., 1966, Heat flow through the Arctic Ocean floor: The Canada Basin-Alpha Rise boundary: Journal of Geophysical Research, v. 71, p. 1223-1248.

Ratcliffe, E. H., 1959, Thermal conductivities of fused and crystalline quartz: British Journal of Applied Physics, v. 10, p. 22-28.

Sass, J. H., Lachenbruch, A. H., and Munroe, R. J., 1971a, Thermal conductivity of rocks from measurements on fragments and its application to heat-flow determinations: Journal of Geophysical Research, v. 76, p. 3391-3401.

Sass, J. H., Lachenbruch, A. H., Munroe, R. J., Greene, G. W., and Moses, T. H., Jr., 1971b, Heat flow in the western United States: Journal of Geophysical Research, v. 76, p. 6376-6413.

Sass, J. H., Stone, C., and Munroe, R. J., 1984, Thermal conductivity determinations on solid rock--A comparison between a steady-state divided-bar apparatus and a commercial transient line-source device: Journal of Volcanology and Geothermal Research, in press.

Simmons, G., 1961, Anisotropic thermal conductivity: Journal of Geophysical Research, v. 66, p. 2269-2270.

Vacquier, Victor, 1984, Oilfields, a source of heat-flow data: Tectonophysics, in press.

Von Herzen, R. P., and Maxwell, A. E., 1959, The measurement of thermal conductivity of deep-sea sediments by a needle probe method: Journal of Geophysical Research, v. 64, p. 1557-1563.