A COMPARISON OF THE DIVIDED-BAR AND QTM METHODS OF MEASURING THERMAL CONDUCTIVITY

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Abstract—Reliable determinations of thermal conductivity (K) are essential for any evaluation of terrestrial heat flow. In an extension and confirmation of an earlier study (Sass *et al.*, 1984a), operational requirements of the relatively new and easy-to-use and maintain QTM (Quick Thermal Conductivity Meter) technique are compared and contrasted with those of the well established divided-bar method. Analysis of QTM experimental data leads to several recommendations: most importantly, (a) at least 5–6 measurements per sample for isotropic rocks (twice as many for anisotropic rocks)—limiting sample processing to no more than 2 per hour—and (b) slab dimensions of at least $20 \times 50 \times 70$ mm (if K > 5 W/mK). For saturated specimens, experimental uncertainty ($\pm 5\%$) and reproducibility ($\pm 5\%$) are greater than for the divided-bar apparatus (± 4 and $\pm 2\%$, respectively).

Conductivity measurements obtained with the two techniques for 15 dry and water-saturated samples ranging in conductivity from 0.6 to 5.4 W/mK have been compared. For saturated samples, agreement is excellent, with divided-bar values on average several percent greater than QTM values; this discrepancy is most probably related to the different conductivity standards used with the two types of apparatus. Systematic differences of 10-20% arising at conductivities greater than 5 W/mK require further study. For dry samples, divided-bar values are 10% greater on average than QTM values; this difference is attributable to the application in the divided-bar method of an axial load, the principal effect of which is to close low-conductivity air-filled cracks. The need for establishing a uniform international conductivity reference standard is emphasized.

INTRODUCTION

Major uncertainty in the determination of terrestrial heat flow frequently arises from the difficulty in measuring thermal conductivity. This difficulty is attributable to (1) the variation of conductivity with degree of saturation, (2) the marked conductivity anisotropy of certain rock types, and (3) the necessity of estimating conductivity from measurements on only a few rock samples, supplemented by a general understanding of the relationship between conductivity and lithology. It is therefore essential to have a clear understanding of the various measurement techniques available, and of their limitations.

The thermal conductivity of rocks is usually measured by one of two methods: the steady-state "divided bar" (Birch, 1950; Beck, 1957) or the transient line-source "needle probe" (DeVries and Peck, 1958; Von Herzen and Maxwell, 1959). The needle probe is more suited to unconsolidated sediments and the divided bar to low porosity well-consolidated rocks. A modification of the needle probe to a half-space configuration (Sass *et al.*, 1984b) is being used increasingly in both commercially available (the Shotherm QTM = Quick Thermal Conductivity Meter) (Ito *et al.*, 1977; Čermák *et al.*, 1984) and similar (in principle), "home-made" apparatuses (e.g. Cull, 1976; Carvalho *et al.*, 1980; Vacquier, 1984; Sass *et al.*, 1984b).

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Sass et al. (1984a) presented a comparison of OTM and divided-bar thermal conductivity results for 17 isotropic rock samples, and concluded that the two techniques yield, within experimental error, the same value for isotropic rocks. The notable advantages of the OTM are however, the relative ease of sample preparation and measurement, its size $(15 \times 37 \times 40 \text{ cm})$ and portability, and, not least, its off-the-shelf availability and moderate or negligible in-house construction, maintenance, and calibration requirements. Nevertheless, there are several potential problems with the OTM that must be carefully considered in view of its increasing use by the geothermal community. First, in common with other line-source techniques, anisotropy can be characterized only through an interpretative procedure (as e.g. that outlined by Grubbe et al., 1983). Second, as noted by Sass et al. (1984a), sample size requirements are greater than for the divided bar; indeed, the OTM, in contrast to both the divided bar and the needle probe. cannot be used to obtain conductivities on drill cuttings. Third, in the divided-bar technique. conductivities are measured under an axial load typically of about 100 bars (10 MPa). corresponding to an overburden of several hundred metres or more. The application of an axial load has two aims: it closes cracks, the presence of which can have an important effect on conductivity (Walsh and Decker, 1966; Simmons and Nur, 1968), and it reduces contact resistance at the ends of the sample (Birch, 1950). On the other hand, conductivity values obtained using the OTM do appear to be relatively insensitive to minor surface roughness (Sass et al., 1984a).

In view of the potential problems and of the likelihood that the QTM will become more commonly used, further comparison of the QTM with the divided-bar apparatus was thought desirable. This study represents an extension of that conducted by Sass *et al.* (1984a). This earlier study considered only isotropic, saturated specimens, and, moreover, only two specimens of conductivity that fell outside the range 2–3 W/mK. In this study, 15 samples covering a wide range of rock types (plus a sample of polyethylene) and ranging in conductivity from 0.6 to 5.4 W/mK were measured in both dry and water-saturated states (Table 1). In addition, several samples of the same lithology were compared to test for internal consistency, and an attempt was made to determine better the operational requirements of the QTM.

DIVIDED-BAR APPARATUS

The divided-bar apparatus used was designed and built at Oxford University, and later moved to Cambridge University (see Richardson and Oxburgh, 1978). The apparatus is modified from the one described by Birch (1950) and is similar to the USGS apparatus used by Sass *et al.* (1984a). The most significant difference from the latter is that the Cambridge instrument is operated in the one-sample mode whereas USGS practice is to include two samples in each "stack" (see Sass *et al.*, 1971). In addition, the Cambridge instrument contains 10 stacks capable of measuring cored disks with diameters ranging between 25 and 38 mm. Samples are usually cut to thicknesses of about 28 mm (USGS: 10 mm), although much thinner or thicker disks are also acceptable. Sample ends are ground (either manually or on an automatic lap using a 9 μ m grinding powder) parallel to within 70 μ m and flat to within 20 μ m. Contact resistances are minimized by the use of a film of heat-transfer compound.

When account is taken of the time required to assemble the stacks, attain thermal equilibrium (45–60 min is usually allowed), make the measurements (usually repeated 3–4 times), and disassemble the stacks, the 10-track configuration enables the measurement of 5 samples/hr on average (USGS: 8 samples/hr, due mainly to thinner samples used, requiring smaller times to achieve a state of thermal equilibration). Sample preparation is relatively difficult, generally involving coring, precise cutting, and fine grinding, and slow: typically, only 3 samples/hr can be prepared.

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Table 1

				Dry				S	Saturated			$K(DB)_{sat.}$
Sample	Lithology	$K(QTM)^*$	N^{+}	K(DB)	N	E^{\ddagger}	K(QTM)	N	K(DB)	N	E	$K(DB)_{dry}$
ZI	dolomite		10	2.67 (3)	4	- -	3.51 (4)	10	3.53 (3)	ŝ	+	1.32
Z2	peridotite		10	2.08 (<0.5)	ŝ	+4	2.37 (9)	10	2.30 (2)	9	÷.	1.11
Z3	gypsum		10	1.07 (2)	ę	+9	1.15 (9)	10	1.23	6	+7	1.15
Z4	marble	2.77 (3)	10	2.99 (1)	ŝ	+7	2.99 (9)	6	3.12 (2)	ŝ	+	1.04
Z5	limestone		10	2.30 (1)	4	- 1	2.56 (2)	10	2.54 (2)	ŝ	-1	1.10
Z6	sandstone	1.23 (3)	10	1.48(3)	4	+17	2.32 (7)	٢	2.31 (2)	e	0	1.56
LZ	claystone	2.40 (4)	Ś	-	4	+1	2.61 (3)	6	2.53 (2)	4	ς Γ	1.04
Z8	metagabbro	3.37 (6)	10	3.77 (2)	4	+11	3.51 (3)	10	3.78 (2)	ŝ	+7	1.00
6Z	polyethylene	0.49 (2)	9	0.57	7	+14	0.49(2)	9	0.57	0	+14	1.00
Z11	anhydrite	3.53 (7)	10	4.54 (1)	ŝ	+22	4.43 (1)	2	5.38	7	+18	1.19
SB1§	granite	2.69 (5)	20		ŝ	+11	3.52 (5)	20		ę	0	1.17
SB2	granite	2.60 (4)	12		e	+14	3.49 (5)	16		ę	+2	1.17
SB3	granite	2.64 (4)	20	3.04 (3)	2	+13	3.42 (6)	20	3.54 (1)	9	+3	1.16
SB5	granite	2.45 (5)	32		×	+19	3.29 (5)	28		×	6+	1.20
SB6	ğranite	2.43 (5)	24	2.72 (<0.5)	3	+11	3.10 (5)	16	3.26 (2)	Э	+5	1.20
Average SB1–SB5 Average all sample	Average SB1–SB5 Average all samples	2.60 (4) (4)		3.03 (<0.5) (2)		+14 +10	3.43 (3) (5)		3.56 (1) (2)		+3.5	

* K is the mean thermal conductivity in W/mK; the standard deviation expressed as a percent of the mean value is given in parentheses.

 \dagger N is the number of determinations per sample. \ddagger E is the percent deviation of QTM from divided-bar results: {[K(DB) – K(QTM)]/[K(DB)]}. \$ Samples SB1–SB6 have been described previously by Schaerli and Rybach (1984); very slight differences in K(QTM) between this and the 1984 study are due mainly to different averaging methods used.



Fig. 1. The cumulative mean conductivity with standard deviation (thick lines) of 20 replicate QTM measurements (thin line) on sample SB1 saturated. The first 10 measurements were made with the probe face parallel to a presumed direction of principal conductivity and the next 10 with the probe face in a perpendicular alignment. The sample can be seen to be thermally isotropic.

The primary standard used in Cambridge is gem-quality (single-crystal) quartz cored perpendicular to the c (optic) axis, for which the value of 6.19 W/mK at 300 K (27°C) given by Ratcliffe (1959) was taken. This standard is used to calibrate the secondary standards—1 mm thick polycarbonate discs—actually used for the conductivity measurements. The variation in replicate measurements on different Lexan discs implies an uncertainty in conductivity of about 4% (Richardson and Oxburgh, 1978).

In this study, the samples were measured in both dry (after heating at 60° C for 12 hr) and water-saturated (under vacuum for 12 hr) conditions. These conditions were somewhat different from those used for the QTM (dried at 90°C for 24 hr and saturated under vacuum for 1–2 hr) but are routine at Cambridge.

QUICK THERMAL CONDUCTIVITY METER

The QTM used is located at the ETH in Zurich and is almost identical to the one described by Sass *et al.* (1984a). One minor modification is that the face of the QTM "probe" in Zurich is covered by a plastic film, so that saturated specimens can be more easily measured (as opposed to covering the specimens with plastic food-wrap). The experiments of Ito *et al.* (1977) and Sass *et al.* (1984a) indicate that any additional contact resistance thereby introduced ought to be negligible. Several tests were made to determine the minimum number of measurements required, the minimum sample size required, and the reproducibility of results.

The cumulative mean conducitivity for twenty measurements—measuring the sample in different locations and orientations in an attempt to account for heterogeneity and thermal anisotropy—of sample SB1 (saturated), a medium-grained (ca. 2–4 mm) granite (described by Schaerli and Rybach, 1984) is shown in Fig. 1. The first 10 measurements were made with the heating element aligned parallel to a presumed direction of principal conductivity, and the second 10 measurements with a perpendicular alignment. It can be seen that the rock is isotropic and, that after about six measurements, the calculated mean conductivity varies insignificantly,



Fig. 2. Histogram of conductivity values obtained for QTM measurements on sample SB5 in dry (32 separate determinations) and water-saturated (28 separate determinations) conditions. The different shadings show results for different pieces of the same sample. The value above each bar is the total number of determinations that lie within each 0.1 W/mK conductivity interval.

so that no further improvement in accuracy is achieved. Nonetheless, more measurements were generally made in order to determine if the rock was significantly anisotropic before measuring with the divided bar. Moreover, samples of larger grain size may require more than six measurements before a sufficiently accurate mean is obtained. The distribution of individual values obtained for several pieces of the same specimen (SB5) is shown in Fig. 2. Although individual conductivity measurements may differ by up to 20% from the mean (see also Fig. 1), the different pieces yield similar distributions and identical conductivities within experimental error.

Finally, the variations in conductivity for specimens of high (anhydrite) and medium (two-mica gneiss with grain size *ca*. 1 mm) conductivity as functions of sample thickness and surface area are shown in Fig. 3 (after Schaerli, 1980). These samples were used only for determining minimum acceptable sample sizes, and are not listed in Table 1. Such data indicate that slabs with dimensions of as little as *ca*. $15 \times 30 \times 50$ mm should yield reliable results for thermally isotropic rocks with conductivities of 2–3 W/mK or less, the minimum slab dimensions increasing to *ca*. $20 \times 50 \times 50$ mm for rocks of high conductivity (>5 W/mK). Conservative recommendations for minimum sample sizes are, therefore, $20 \times 50 \times 70$ mm for weakly heterogeneous rocks (such as dense limestone, sandstone and claystone), rising to $30 \times 70 \times 70$ mm for strongly layered and heterogeneous, or highly conductive rocks.

These dimensions should be compared to the values given by Sass *et al.* (1984a): $30 \times 60 \times 100$ mm as a minimum for rocks of conductivity less than 3.5 W/mK, increasing to $50 \times 100 \times 100$ mm for rocks of higher conductivity.

Thus, although it can still be said that the constraints on sample size are a notable drawback to the QTM method, being more limiting than those imposed by the divided-bar method, the constraints are probably not as restrictive as stated in the previous study. More serious is the inability with the QTM to obtain conductivities using rock chips or drill cuttings, as noted in the Introduction.



Fig. 3. Thermal conductivity of selected gneiss and anhydrite samples as a function of sample surface area (a) and sample thickness (b). The dashed lines show error limits of the mean conductivities. After Schaerli (1980).

If each sample is measured 5-6 times (measuring the sample in different locations in an attempt to account for heterogeneity), about 3 samples/hr can be processed. Yet, because of the difficulty in knowing *a priori* that a sample is thermally isotropic, generally at least 10 measurements per sample must be made, with the QTM probe aligned both parallel and perpendicular to the principal mineral alignment, bedding, schistosity or compositional banding. Therefore, it is normally not possible to measure more than 2 samples/hr, particularly if the samples are known to be thermally anisotropic. The great savings in time and energy with the QTM arises from the relative ease (frequently, only cuts required) and rapidity of sample preparation: preparation times for one sample range from 20 min for hard granites to 6 min for limestone or dolomites to as little as 4 min tor sandstones. Considering preparation and measurement time together, however, it cannot be said that the QTM method is substantially quicker—although it is simpler—than the divided-bar method.

The reproducibility of an individual conductivity value as expressed by the percent standard deviation of N measurements ranged, for the samples studied, from 1 to 9%, and, on average, was about 5% (Table 1), in agreement with the results of Sass *et al.* (1984a). Further uncertainties may be related to difficulties in fixing the thermal conductivity of the quartz glass standard (Sass *et al.*, 1984; see below). On the other hand, reproducibility with the divided-bar apparatus was considerably better: standard deviations ranged from less than 0.5-5% of the mean value, with an average deviation of 2% (Table 1). The greater precision of the divided-bar apparatus is also indicated by the comparison of the values obtained for four lithologically similar granitic samples (SB1, SB2, SB3 and SB5): the standard deviation expressed as a percent of the mean conductivity for these four samples ranged from 4% and 3% for dry and saturated samples, as measured with the QTM, to less than 0.5% and 1% for dry and saturated samples, as measured with the divided bar (Table 1).

The primary standards used for calibrating the QTM in Zurich are fused quartz and Plexiglass, for which the manufacturer's recommended values of 1.320 and 0.229 W/mK, respectively, were taken.

COMPARISON OF THE TWO APPARATUSES

Saturated samples

The results of the comparison for dry and water-saturated samples are summarized in Table 1 and in Figs 4–6. The agreement for silica-bearing saturated specimens is excellent (coefficient of correlation r = 1.00), the divided-bar apparatus yielding values on average only 2% higher than the QTM values. When specimens Z9 (polyethylene) and Z11 (anhydrite) are included in the regression analysis, the agreement is somewhat poorer (r = 0.98), divided-bar values being on average 4% greater than QTM values. The agreement is better than 10% for 13 of 15 samples and better than 5% for 10 of 15 samples (Table 1, Fig. 5).

The small, systematic difference is most likely explained by either of two effects: an incorrect determination of the conductivity of one of the standards or the application of an axial load of ca. 0.1 kb (10 MPa) with the divided-bar apparatus. First, the standard value for fused silica at room temperature given by Ratcliffe (1959) is 1.375 W/mK (this value was taken for the quartz crystal used to calibrate the divided bar), which should be compared to the standard value of 1.320 W/mK used for the QTM (see previous section): this difference in standard values is exactly 4%. A systematic difference of similar but opposite magnitude was observed by Sass *et al.* (1984a), who were able to relate this to a possible incorrect determination of conductivity (by the manufacturer) for the QTM fused-quartz standard. On the other hand, it is also possible that the systematic difference is associated with the variation in conductivity of the Lexan discs, the secondary standard used to calibrate the divided bar.

Second, it is known that the application of an axial stress of the order of hundreds of bars can cause the conductivity of saturated specimens to increase by 1-2% (the effect on dry specimens is, however, considerably greater—see below) (e.g. Walsh and Decker, 1966; Simmons and Nur, 1968). Indeed, that the divided-bar apparatus allows an approach to natural conditions was originally stated by Birch (1950) to be one of its principal advantages. Nonetheless, it should be noted that the effect on conductivity is small for saturated specimens. Both this and the experience of Sass *et al.* (1984a) suggest that uncertainties in the determination of "standard" values can lead to systematic errors in the determination of conductivity, making precise comparisons between different apparatuses difficult. An important conclusion to be drawn is that there is a need for a uniform international reference standard for use by all thermal conductivity laboratories.



Fig. 4. Comparison of thermal conductivity of 15 specimens measured using the QTM and divided-bar (DB) techniques in both dry (a) and water-saturated (b) conditions. The data are from Table 1: for ease of presentation, samples Z1–Z11 are simply numbered 1–11 and samples SB1–SB6 are lettered A–F. Lines of equality K(QTM) = K(DB) (solid) and of various percent deviations (dashed) are shown along with the average uncertainty for individual measurements (black rectangles).



Fig. 5. Histogram of the percent deviation of QTM from divided-bar (DB) measurements on 15 specimens in dry (mean deviation = +10%) and water-saturated (mean deviation = +4%) conditions (data from Table 1). Data are grouped in intervals of 5%. Positive deviations indicate divided-bar values greater than QTM values; negative deviations indicate the opposite.



Fig. 6. Comparison of both dry and water-saturated conductivities for 15 samples (denoted as in Fig. 4) measured using the divided-bar and QTM techniques. For each sample, the line connects the water-saturated (higher) with the dry (lower) value. The relative lengths of the lines give a first-order indication of relative sample porosity, and the slopes, relative to the K(QTM) = K(DB) line, show how the application of an axial load increases the divergence between QTM and divided-bar measurements for differing saturation conditions (see text).

It is still difficult to explain the increased departure between QTM and divided-bar values at higher conductivities (in particular, sample Z11 of this study and USGS2—a dolomite studied by Sass *et al.*, 1984a). Although Sass *et al.* concluded that the condition of the specimen was not crucial with the QTM method, the presence of a contact film could explain the discrepancy. Further comparison of samples with conductivities greater than 3.5 W/mK is required.

Dry samples

As "dry" conditions do not generally occur at levels in the crust at which heat flow is measured, the principal reason for measuring samples in this condition was the conclusion of Schaerli and Rybach (1984) that for low-porosity crystalline rocks, thermal conductivity measurements on dry and water-saturated samples can be used as a rapid method for the determination of *in situ* porosity. Their work was based on QTM measurements. As noted previously, it is to be expected that the application of an axial load will begin to close cracks; thereby, the configuration and amount of pore space will change.

The comparison of QTM with divided-bar measurements on dry samples yields a good correlation (r = 0.97), but the divided-bar values are now on average 10% greater than QTM values; in more than 50% of samples, this difference is between 10 and 20% (Fig. 5). As measured with the divided bar, the dry values are up to 56% lower than the saturated values, depending primarily on the porosity and the pore configuration of the specimens (the conductivity of air is about 23 times lower than that of water at room temperature). The variation between dry and saturated conductivities is even greater for the QTM, as can be clearly seen in Fig. 6, where the relative lengths of the lines give a first-order estimate of variations in porosity and the slopes of the lines relative to the K(QTM) = K(DB) line indicate the effect of changing saturation conditions.

The average difference of 10% between divided-bar and QTM values reflects the importance of axial compression for measurements on dry samples, in contrast to those on water-saturated specimens, where the zero-pressure conductivity is already close to the intrinsic conductivity. An important conclusion to be drawn is that any correlation between dry and saturated conductivities and porosity should be established with the use of a conductivity apparatus—such as the divided-bar—for which it is possible to apply an axial load.

CONCLUSIONS

The principal advantages and disadvantages of the QTM and divided-bar techniques are listed and compared in Table 2. What the QTM technique gains in terms of ease of start-up, use, and sample preparation, it loses in terms of the divided-bar technique's better accuracy, smaller sample size requirements, and better approximation to *in situ* conditions.

The QTM's operational requirements have been elucidated as part of this study:

(1) a minimum of 5-6 measurements at different points of the sample are recommended for fine- to medium-grained isotropic rocks—at least twice as many measurements would be required for anisotropic rocks;

(2) a minimum sample size of $20 \times 50 \times 70$ mm is recommended for weakly heterogeneous rocks of conductivity less than 3 W/mK, this minimum sample size recommendation increasing to $30 \times 70 \times 70$ mm for strongly heterogeneous, anisotropic, or highly conductive (>5 W/mK) rocks;

(3) reproducibility is $\pm 5\%$ (compared to $\pm 2\%$ for the divided-bar technique); and

(4) uncertainty is $\pm 5\%$ for saturated and $\pm 10-20\%$ for dry samples (compared to ± 4 and $\pm 4\%$, respectively, for the divided-bar technique).

Method/property	QTM	Divided bar
Measuring time*	2 samples/hr	5 samples/hr
Preparation	relatively easy (usually, cuts only required) and rapid (3–15 samples/hr)	relatively difficult (coring, cutting, polishing required) and slow (<i>ca.</i> 3 samples/hr)
Sample size	minimum of $15 \times 30 \times 30$ mm (if $K \le 3.0$ W/mK) or $30 \times 50 \times 50$ mm (if $K > 3.0$ W/mK)	core diameter: 25–38 mm; core length: 28 mm typically, but shorter cores (10–15 mm) acceptable; drill cuttings also acceptable
Start up and use	off-the-shelf apparatus; relatively simple to use; easily transportable; requires little space	complex, careful in-house construction required; transportation unfeasible; requires separate room
Reproducibility	±5%	±2%
Uncertainty	saturated: ±5%	±4%
	dry: ±10–20%	±4%
Anisotropic rocks	requires interpretative technique	directly measurable ±4%
Other comments	direct digital read-out of conductivity	better approximation to <i>in situ</i> conditions
Summary of principle advantages	ease of start up, use, and sample preparation	greater accuracy; smaller samples; drill cuttings acceptable; approach to natural conditions

Table 2. Comparison of the advantages and disadvantages of the QTM and divided-bar techniques for measuring the thermal conductivity of rocks

* Sass *et al.* (1984a) gave values of 4 and 8, respectively, for the apparatuses that they used. Depending on the number of stacks and the stack configuration, other divided-bar apparatuses may yield different values.

A comparison of QTM and divided-bar data for 15 isotropic, saturated samples of conductivity varying within the wide range of 0.6 to 5.4 W/mK has confirmed and extended Sass *et al.*'s (1984a) conclusions that the two techniques generally yield the same value, within experimental uncertainty, over a wide range of conductivities. An increasing discrepancy at higher conductivities noted as well by Sass *et al.* (1984a) requires further study. That divided-bar values are systematically greater by several percent than QTM values is most probably related to the use of different calibration standards for the two methods. As a corollary, this result points to *the need for a uniform international calibration standard for use in all thermal conductivity laboratories*.

An extension of the comparison to dry samples has shown that divided-bar values are on average 10% greater than QTM values. This difference is attributable to the application of an axial load, which closes very low-conductivity air-filled cracks, with the divided-bar technique (for water-saturated cracks, the influence on conductivity is only 1-2% or less).

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