TK04 Thermal Conductivity: User Guide

Author:	M. Hastedt, Y.-G. Kim, M.A. Kominz
Reviewer(s):	T. Gorgas, M. Vasilyev, R. Wilkens, K. Milliken, H. Barnes, S. Hermann
Management Approval (Name, Title, Date):	D.J. Houpt (Supervisor of Analytical Systems)
Audience:	Scientists, Technicians
Origination date:	2/14/2009; 11/14/2009
Current version:	V1.0 | 08/15/2011 V1.0
Revised:	V362 | 09/20/2016; V1.1 | 1/6/2014 (IODP-II)
Domain:	Physical Properties
System:	Thermal Conductivity
Keywords:	Full-space needle probe, half-space needle probe, TK04

Introduction

Thermal conductivity is the coefficient of proportionality relating conductive heat flow to a thermal gradient. The Teka Berlin TK04 system determines thermal conductivity based on a transient heat flow method. A line source is heated with constant power while recording source temperature. Thermal conductivity is calculated from the resulting heating curve.

The TK04 uses two types of probes: the full-space (VLQ) needle probe for soft sediments and the half-space (HLQ) probe for hard rock samples. Measuring a single point in a section takes ~10 min per sample. A self-test including a drift study is conducted at the beginning of each cycle. To measure thermal conductivity the heater circuit is closed and the temperature rise in the probe is recorded. Thermal conductivity is calculated from the rate of temperature rise while the heater current is flowing. The thermal conductivity of each sample is the average of three repeated measurements for the full-space method and three to six repeated measurements for the half-space method. For basalt samples, drift calculations are based on a Macor standard (1.637 ± 0.033 W/[m·K]) because its properties are closest to basalt cores.

Precision of the method is better than 2%, based on extended evaluation of the method; accuracy is about 5% because of random variations of thermal conductivity in natural materials.

Theory of method

Thermal conductivity is measured by transient heating of an isotropic material with a known heating power generated from a source of known geometry and measuring the temperature change with time. The needle probe contains a heater wire and calibrated thermistor. It is assumed to be a perfect conductor because it is much more conductive than unconsolidated sediments. With this assumption, the temperature of the probe has a linear relationship with the natural logarithm of the time after initiation of heating:

$$T(t) = (q/4\pi k) \ln(t) + C,$$

where:

T = temperature,

q = heat input per unit length per unit time (W),

k = thermal conductivity (W/[m·K]),

t = time after initiation of the heat, and

C = a constant.

A simple way of calculating the thermal conductivity coefficient k is picking temperatures T_1 and T_2 at times t_1 and t_2, respectively, from the temperature vs. times measurement curve:

$$ka(t) = q/4\pi[\ln(t_2) - \ln(t_1)]/(T_2 - T_1).$$

$ka(t)$ is the apparent thermal conductivity because the true conductivity (k) is approached only by a sufficiently large heating duration. The method assumes that the measurement curve is linear and ignores the imperfections of the experiment expressed in the constant C.

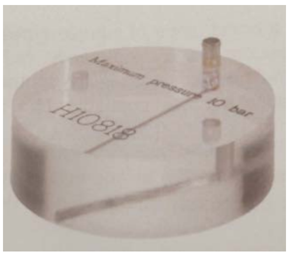
Thermal conductivity is an intrinsic material property for which the values depend on chemical composition, porosity, density, structure, and fabric of the material. Thermal conductivity profiles are used along with in situ temperature measurements to determine heat flow, which is an indicator of age of ocean crust and fluid circulation processes.

Available Probe Types

All probes consist of a source (i.e., a metal needle with an embedded heating wire and a temperature sensor), a handle or body (depending on the probe type), and a connection cable.

Full-space probes (VLQ) are needle probes equipped with a handle at one end of the source. They are completely inserted into the sample.

Half-space probes (HLQ) are placed on top of the sample. The source is embedded into the bottom side of a puck-like probe body and has on-site contact with the material/sample.

Name	**Standard VLQ**	**Standard HLQ**	**Mini HLQ**
Probe type:	Full-space	Half-space	Half-space
Dimension (source, mm):	L: 70 × diameter (D): 2	L: 70 × D: 2	L: 45 × D: 1.5
Dimension (handle/body, mm):	L: 90 × D: 16	L: 30 × D: 88	L: 30 × D: 50
Evaluation parameter set:	Standard VLQ (VLQ Source 70x2)	Standard HLQ (HLQ D88 Source 70x2)	Mini HLQ (HLQ D50 Source 45x1.5)
Measuring range (W/m·K):	0.1–10	0.3–10	0.3–3
Accuracy (%):	±2	±2	±5
Duration of 1 measurement (s):	80	80	60
Min. sample size (mm):	L: 75 × D: 30	L: 15 × D: 80	L: ~15 × D: 50
Picture:			

Analytical process

The approximate amount of time needed per sample is as follows:

Process	Time (min)	Comments
1. Obtain a whole-round core section from the core rack	0.3	See ***Preparing Samples for Analysis***
2. Locate the appropriate needle probe for the sample type	0.5	
3. Verify sample identification in software	0.5	See ***Configuring Measurement Program***
4. Configure measurement program	0.3	
5. Perform drift control	5	See ***Measuring Samples***
6. Heat and measure sample	2	
7. Upload results to LIMS	0.2	See ***Uploading Data to LIMS*** and ***Verifying Data in LIMS***
8. Check results in LIMS	1	
9. Remove the section and deliver to cutting room	0.2	
Total Time per sample:	10 (max)	

Preparing Samples for Analysis

Soft-Sediment Samples

1. Equilibrate core sections to room temperature for at least 4 hours in the core rack before bringing a target section to the thermal conductivity workstation.
2. Select measuring points in the core
   A. Intact core: middle of section
   B. Cracked core: just above/below the middle; record offset in cm
3. Use the cordless drill to drill a ~2 mm hole into the core liner at the borderline between working and archive halves. If the sediment is semiconsolidated, drill a small hole in the sediment for the probe as well.
4. Optional - Apply thermal joint compound to the probe unless the sample is very soft and/or moist.
5. Carefully insert a clean full-space needle into the sediment. Avoid twisting the needle into the core. The needle must be completely inserted into the sample up to the handle.

Figure 1. Thermal conductivity measurement on a soft-sediment section using full-space probe.

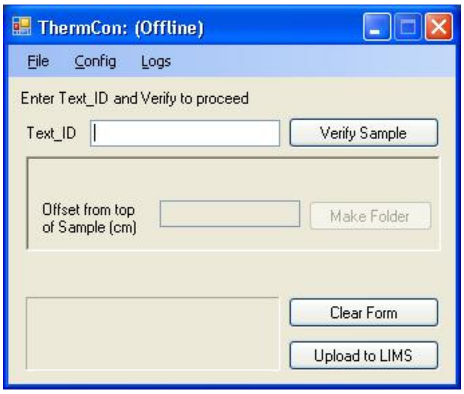
Hard Rock Samples

1. Place hard rock samples in an ambient temperature seawater bath to equilibrate and saturate (4–12 hr). Keep the sample saturated until measurement. A bell jar and vacuum pump available in the Physical Properties lab can also be used to aid in saturation.
2. If the surface of the split core is excessively rough, use a lap plate and grit from the Thin Section Lab to prepare a smooth surface on a split-core piece. Pieces must be at least 10 cm long. The sample diameter has to be at least equal to the probe diameter and the sample should be at least 2–3 cm thick. The needle on the pucks must be in contact with the sample material on its whole length.
3. Equilibrate the sample and sensor needle in an insulated seawater bath for at least 15 min prior to measurement. Do not submerge the puck—let the water level rise to half the depth of the puck or less.
4. Recommendation: Apply thermal joint compound to the side of the probe where the line source is located.
5. We do not at this time recommend using the Teka press to ensure good contact between the puck and the specimen due to overtightening possibly causing puck damage. Use rubber bands to secure the puck to the specimen.

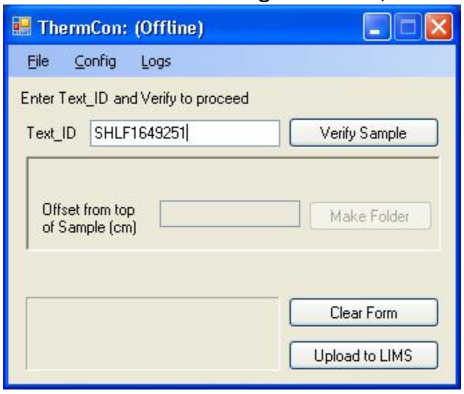
Figure 2. Thermal conductivity measurement on hard rock sample using half-space puck.

Configuring Measurement Program

1. Load **ThermCon** software in offline mode. Ensure that the *Text_ID* field is blank.

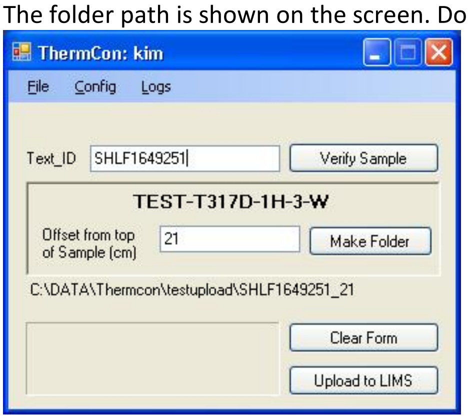


2. Scan the core label using a scanner, then click **Verify Sample**.

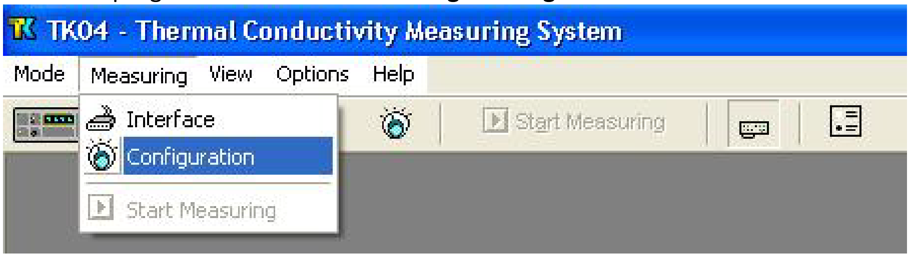


3. If login is requested, enter *UserName* and *Password* and then click **OK**.

4. The folder path is shown on the screen. Do not close this window during measurement.



5. Run **TK04** program and choose **Measuring > Configuration**.



6. Set configuration parameters as follows:

   A. *Probe Number:* serial number of probe to be used in the measurement (Note: results may be wrong by several percent if the wrong serial number is entered or by a factor of ~2 if the wrong type of probe is entered),

   B. *Root Name:* six characters or less; suggest Core-Type-Section (no special characters in the root name).

   C. *Serial Number*: number of repeat measurements at each measurement point (1–99 single measurements).

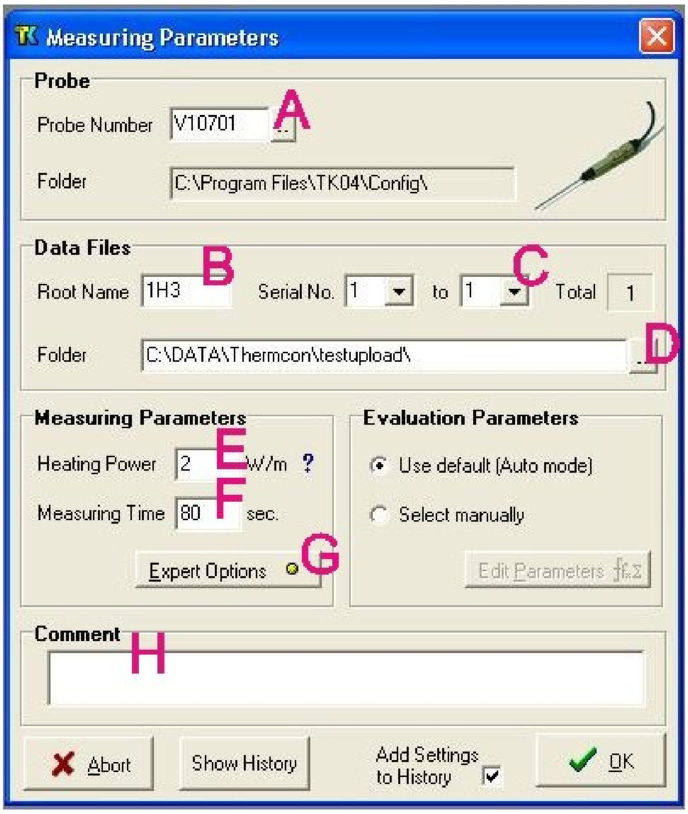
   D. *Folder:* path for saving data results.

   E. *Heating Power:* for the VLQ (needle probe), set to twice the estimated thermal conductivity value of measured sediment. For example, 2–3 is good for sediment. (See the APPENDIX: TK04 RECOMMENDED HEATING POWER for power guidance.)

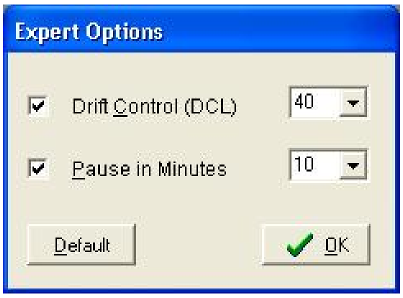
   F. *Measuring Time:* set to at least 80 s, or for mini HLQ 60 s.

   G. Click **Expert Options** to configure *Drift Control* and *Pause in Minutes* (see Step 7).

   H. Enter comments.

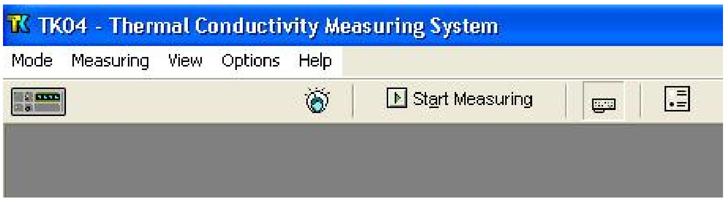


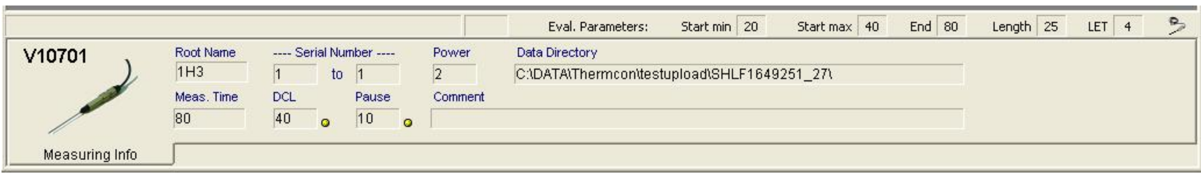
7. To configure *Drift Control* and *Pause in Minutes* in **Expert Options:**
   A. *Drift Control (DCL)*: limit for the range of temperature drift allowed prior to heating and measuring. A larger number allows quicker but less accurate measurement. Default (unchecked) DCL = 10; Recommended DCL = 40.
   B. *Pause in Minutes*: insert a pause between single measurements; recommended = 10 min. This parameter does not apply when conducting a single measurement on each core or each core section.



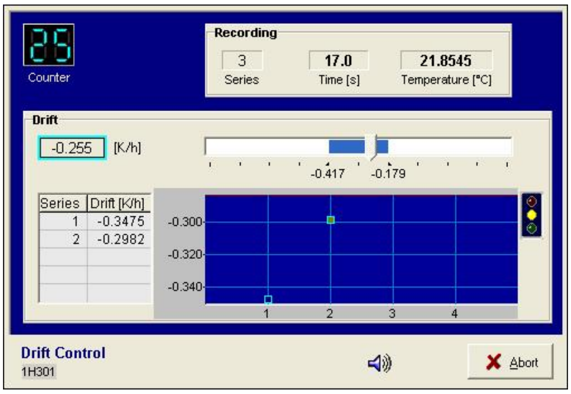
Measuring Samples

1. Confirm configuration settings shown in the lower part of the **TK04** screen, insert the probe into the hole drilled into the sample, and click **Start Measuring.**

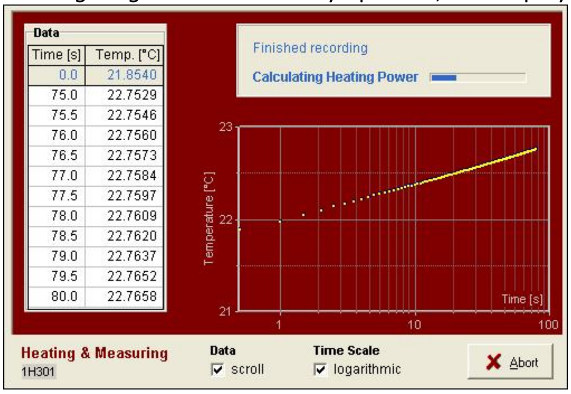




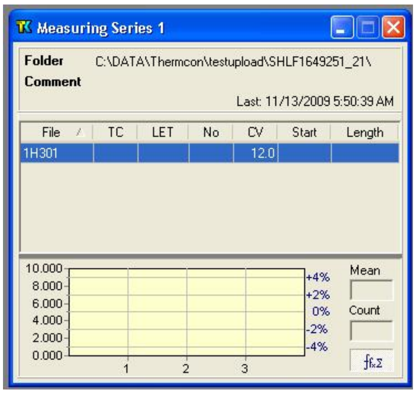
2. Drift control (DCL) repeats until the criterion for drift (set in the **Expert Options** window) is met. Each drift control measurement takes 0.5 min. For DCL = 40, drift control is <10 series (~5 min). For DCL = 10, drift control is <30 (~15 min).
   A. *Counter*: remaining number of measurements in the current drift series
   B. *Recording*: currently recorded time and temperature and drift series being recorded
   C. *Drift*: drift value from the last drift series; indicated in blue on the slider scale when acceptable
   D. *Start signal light*:
      i. Yellow: drift has reached the threshold; approximately half the drift time has passed
      ii. Green: drift limit has been reached and measurement can begin



3. After satisfying drift control, sample heating and measuring begins, and temperature values are corrected automatically for the drift effect predicted from the last drift series. Elapsed measurement time is controlled by the value entered in **Configuring Measurement Program > Step 6F**.
   A. *Recording*: displays currently recorded time and temperature
   B. *Data*: lists recorded time/temperature values from the heating curve
   C. *Heating diagram*: continuously updated; can display as linear or logarithmic scale

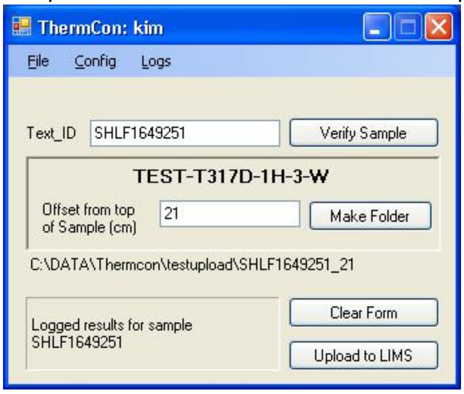


4. Solutions calculated by the algorithm are shown on the screen. Note the result on the handwritten log.
   A. *TC*: thermal conductivity
   B. *LET*: logarithm of the extreme time (lower limit = 4); the measurement with the largest LET is used to calculate thermal conductivity
   C. *CV*: contact value
   D. *PC*: power control is shown on the plot in the lower part of the screen (recommended PC = 2–3. If PC is out of range, adjust the *Heating Power* (HP) in **Configuring Measurement Program > Step 6E** as follows: if PC > 3, decrease HP; if PC < 2, increase HP).
   E. *Mean*: mean thermal conductivity
   F. *Count*: number of measurements used to calculate thermal conductivity



Uploading Data to LIMS

1. To upload results to LIMS, click **Upload to LIMS**. If upload is successful, a message like "Logged results for sample …" is shown. Close the **ThermCon** program.



Verifying Data in LIMS

Verifying Data in Web Tabular Report

Run LIMS Web Tabular Report to check thermal conductivity data:

1. Select **Web Tabular Report > Science Data.**
2. **Select Analysis > TCON** and **Show Report**.

Retrieving Data from LIMS REPORTs

1. Go to *LIMS Reports* at http://webserv.ship.iodp.tamu.edu:8080/UWQ/.
2. Under **Select Report**, choose **Physical Properties** > **Thermal Conductivity (TCON)**.
3. Under **Select Sample Range**, specify *Expedition*, *Site*, *Hole*, and *Section* image(s) to retrieve.
4. Click **View data** or **Download data file** to view results or download a CSV file.

After Verifying Data Upload

1. Once uploaded data are confirmed, clean the needle probe and place it in its styrofoam storage container.
2. Repeat sample measurement process with a new sample.

LIMS Component descriptions

Analysis	Component	Unit	Definition
TCON	Bottom_depth	m	Location of bottom of measurement, measured from the top of the hole
	Comment	None	Comment about the run
	Contact_value	None	Measure of contact quality between probe and sample
	End_time	s	Elapsed time for end of analysis window
	Heating_power	W/m	Power applied to needle during heating
	Length_of_time	s	Elapsed time, start to finish, of analysis
	Log_extreme_time	s	LET, used in calculation algorithm
	Method	None	Data reduction method: SAM or TCON
	Needle_name	None	Full-space or half-space
	Number_of_solutions	None	Number of solutions found by the software
	Offset	cm	Location of measurement from top of section

	Start_time	s	Elapsed time into experiment for start of analysis window
	Therm_con_average	W/(m·K)	Mean thermal conductivity result
	Therm_con_number	None	Number of measurements in the population
	Therm_con_result	W/(m·K)	Individual thermal conductivity result
	Therm_con_stdev	W/(m·K)	Standard deviation (n-1) of measurement population
	Top_depth	m	Location of top of measurement from top of hole

Troubleshooting

Drift phase takes too long:

- Ambient temperature is not stable: allow sample more time to equilibrate
- Probe and sample are not in equilibrium with ambient temperature: place sample/probe in insulated case
- If necessary, force measurements by choosing a weaker drift limit

Variation of measurement series is too high:

- Ambient temperature is not stable enough
- Heating power too low to produce sufficient temperature increase: increase heating power
- Start time maximum value is too high: set to 40 s
- Interval length minimum value is too low: set to >25–30 s
- Contact values vary strongly: omit outliers from evaluation

Evaluation returns few or no solutions:

- Wet sample may cause convection effects: reduce heating power
- Poor contact between probe and sample: use contact fluid
- Interval length minimum value too high: set <30 s
- Start time maximum value too low: set to 40 s
- Variations in ambient temperature: insulate sample and/or probe
- Heat transport into the sample is not distributed: smooth sample surface and apply contact fluid

LET values too low:

- Poor contact between probe and sample
- Unstable ambient temperature
- Interval length or start time minimum values too high

Evaluation intervals start later than ~35 s:

- Poor contact between probe and sample
- Heating power too high
- Boundary effects caused by finite probe length

Descending trend in thermal conductivity values:

- Water-saturated samples drying out: keep sample wet during analysis

Appendix: TK04 Recommended Heating Power

Note: for loose sediments, use a lower heating power to avoid convective heat transport of pore fluids.

Material	Thermal Conductivity (W/m·K)		Recommended Heating Power (W/m)	
	Mean	Range	VLQ	HLQ
Wood	0.21	0.1–0.35	0.15–1.3	—
Coal	0.29	0.1–1.5	0.15–5.4	—
Concrete	1.00	0.75–1.4	1.0–5.0	0.5–2.2
Fused silica	1.40	1.33–1.46	1.8–5.2	0.8–2.3
Clay	1.40	1.2–1.7	1.6–6.1	0.7–2.6
Silt	1.60	1.4–2.1	1.9–7.5	0.8–3.2
Basalt	1.95	1.4–5.4	1.9–19.0	0.8–7.6
Siltstone	2.04	0.6–4.0	0.8–14.0	0.4–5.7
Limestone	2.29	0.5–4.4	0.7–16.0	0.4–6.3
Syenite	2.31	1.3–5.3	1.7–19.0	0.8–7.5
Amphibolite	2.46	1.4–3.9	1.9–14.0	0.8–5.6
Claystone	2.46	1.6–3.4	2.1–12.0	0.5–9.3
Lava	2.47	0.2–4.5	0.3–16.0	0.2–6.4
Gabbro	2.50	1.6–4.1	2.1–15.0	0.9–5.9
Dolerite (Diabase)	2.64	1.6–4.4	2.1–16.0	0.5–6.3
Granodiorite	2.65	1.3–3.5	1.7–13.0	0.8–5.0
Quartz sand (wet)	2.70	2.4–3.1	3.2–11.0	1.3–4.5
Marble	2.80	2.1–3.5	1.8–13.0	1.2–5.0
Porphyrite	2.82		3.8–10.0	1.5–4.2
Boulder clay	2.90	2.5–3.3	3.4–12.0	1.4–4.8
Diorite	2.91	1.7–4.2	2.3–15.0	1.0–6.0
Slate (perpendicular)	2.91	1.5–3.9	2.0–14.0	0.9–5.6
Gneiss	2.95	1.2–4.7	1.6–17.0	0.7–6.7
Granite	3.05	1.2–4.5	1.6–16.0	0.7–6.4
Eclogite	3.10	2.4–3.4	3.2–12.0	1.3–4.9
Andesite	3.20	1.6–4.7	2.1–17.0	1.0–6.7
Dolomite	3.62	1.6–6.6	2.1–20.0	1.0–9.3
Slate (parallel)	3.80	2.2–5.2	3.0–19.0	1.2–7.4
Peridotite	3.81		5.0–14.0	2.0–5.5
Anhydrite	4.05	1.0–6.0	1.3–20.0	0.6–8.5
Pyroxenite	4.27	3.2–5.1	4.3–18.0	1.7–7.2
Dunite	4.41	3.5–5.2	4.7–19.0	1.9–7.4
Quartzite	4.55	3.1–>8	4.2–20.0	1.7–11.0
Quartz	9.50	6.5–12.5	8.7–20.0	3.5–17.0