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Thermal conductivity of samples from three limestone cores.

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REPORT

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Title:
Thermal conductivity of samples from three limestone cores.

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Summary:

Thermal conductivity were measured on three limestone core samples. The measurements were carried out using a transient method. The measured thermal conductivity on oil-saturated samples are as follows:
core I : 3.1 W/m·K
core II : 3.1 W/m·K.
core III : 1.9 W/m·K.

Keywords: thermal conductivity	core samples	limestone

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1. DATA OF MEASUREMENTS.

2. DESCRIPTION OF THE THERMAL CONDUCTIVITY APPARATUS.

SAMPLES.

Thermal conductivity were measured on samples cut from three cores with diameter 37.5 mm. The cores were saturated with oil.

Core I is a fine grained limestone with a well developed lamination and visible dark and light striations. The lamination is perpendicular to the core axis but some of the striations are parallel to the core axis. The core was cut in four samples with length 24, 18, 14 and 9 mm

Core II is taken from the same limestone as Core I. The core axis of Core II is parallel with the lamination. The material has also visible striations in different direction. Three thermal conductivity samples were cut with length 29, 26 and 21 mm

Core III is a porous limestone, rich in fragments and fossils. The core was cut in three samples of length 24, 23 and 20 mm. The sample of length 23 mm was broken during the first measurements and a new sample of length 9 mm was prepared from the broken one.

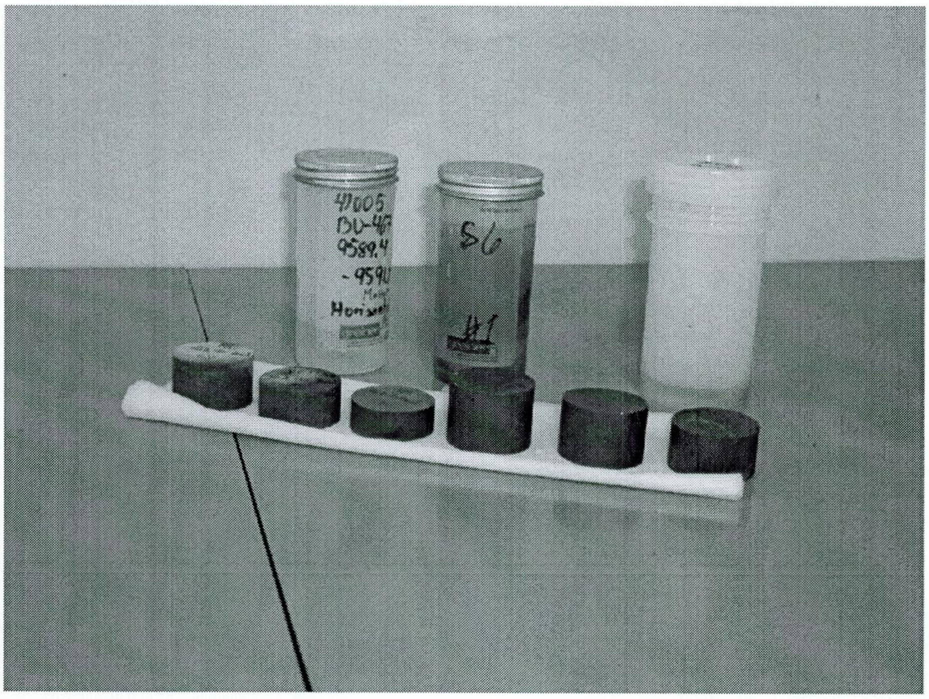


Figure 1. Some samples prepared from cores I and II.

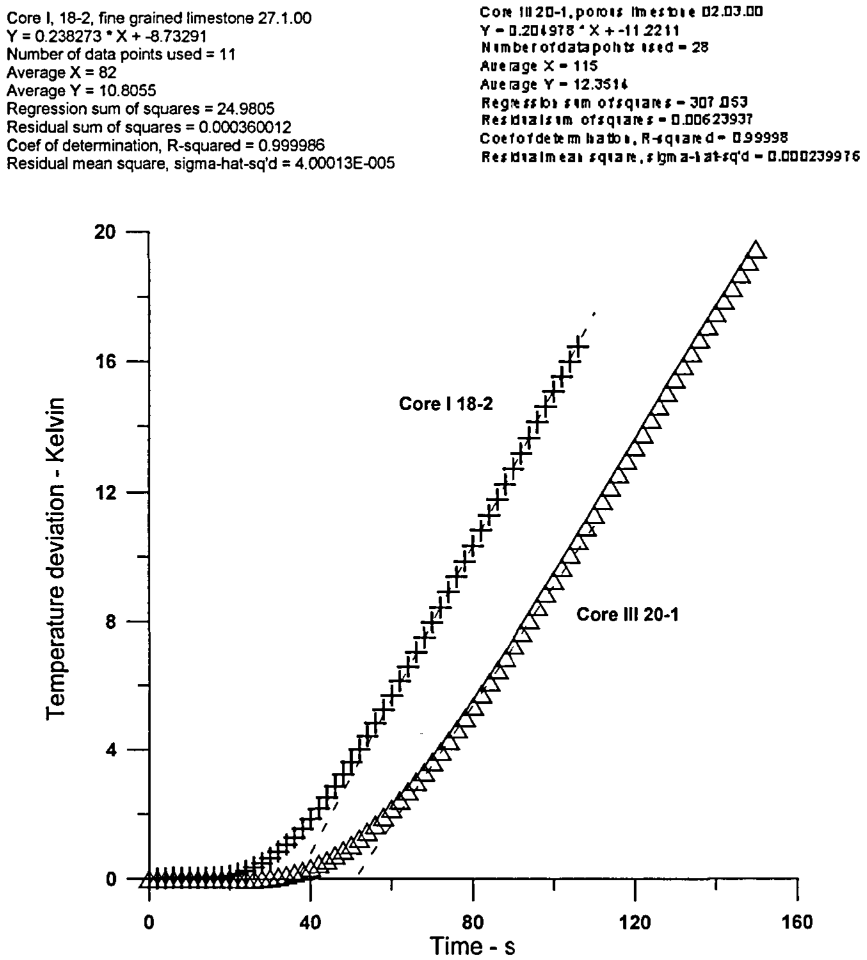
METHOD

The thermal conductivity of the samples was measured using a transient method. A constant heat flow is induced from the top of the sample. The sample is insulated on all other surfaces. The temperature is measured at the base of the sample. Thermal diffusivity (α) is estimated from the temperature – time plot, and the thermal conductivity is calculated from thermal diffusivity, density (ρ) and specific heat (c_p) of the sample (see Table in Appendix 1). The theory of the method is described by Middleton (1993). The apparatus at NGU is shown in Figure 2, while a description of the thermal conductivity apparatus is given in Appendix 2.

Quality control of the thermal conductivity measurements is carried out by measurments on Pyroceram 9606 a well known standard material, and by inter-laboratory comparison with University of Aarhus, Norwegian University of Technology and Science, and Sintef Energy Research. Comparison measurements with this method are also published in Midttømme et al. (1998) and Midtømme & Roaldset (1999) .

The test measurements show that the thickness of a sample has an effect on the thermal conductivity measurement. The measured thermal conductivity increases with increasing thickness of the samples. Final results from measurements are corrected for this effect.

Figure 2. Thermal conductivity apparatus at NGU.



```
Core I, 18-2, fine grained limestone 27.1.00
Y = 0.238273 * X + -8.73291
Number of data points used = 11
Average X = 82
Average Y = 10.8055
Regression sum of squares = 24.9805
Residual sum of squares = 0.000360012
Coef of determination, R-squared = 0.999986
Residual mean square, sigma-hat-sq'd = 4.00013E-005
```

```
Core III 20-1, porous limestone 02.03.00
Y = 0.204978 * X + -11.2211
Number of data points used = 28
Average X = 115
Average Y = 12.3514
Regression sum of squares = 307.053
Residual sum of squares = 0.00623937
Coef of determination, R-squared = 0.99998
Residual mean square, sigma-hat-sq'd = 0.000239976
```

Figure 3 Time – temperature plot of sample I-18-2 and III-20-1.

THERMAL CONDUCTIVITY MEASUREMENTS.

Thermal conductivity was measured on oil-saturated, dried and water-saturated samples. The oil-saturated samples were covered with thin plastic to protect the isolation. The dry measurements were carried out on samples dried at room temperature for at least two days. The samples were then water saturated under vacuum and measured The measurements are shown in Table 1 and Figure 4.

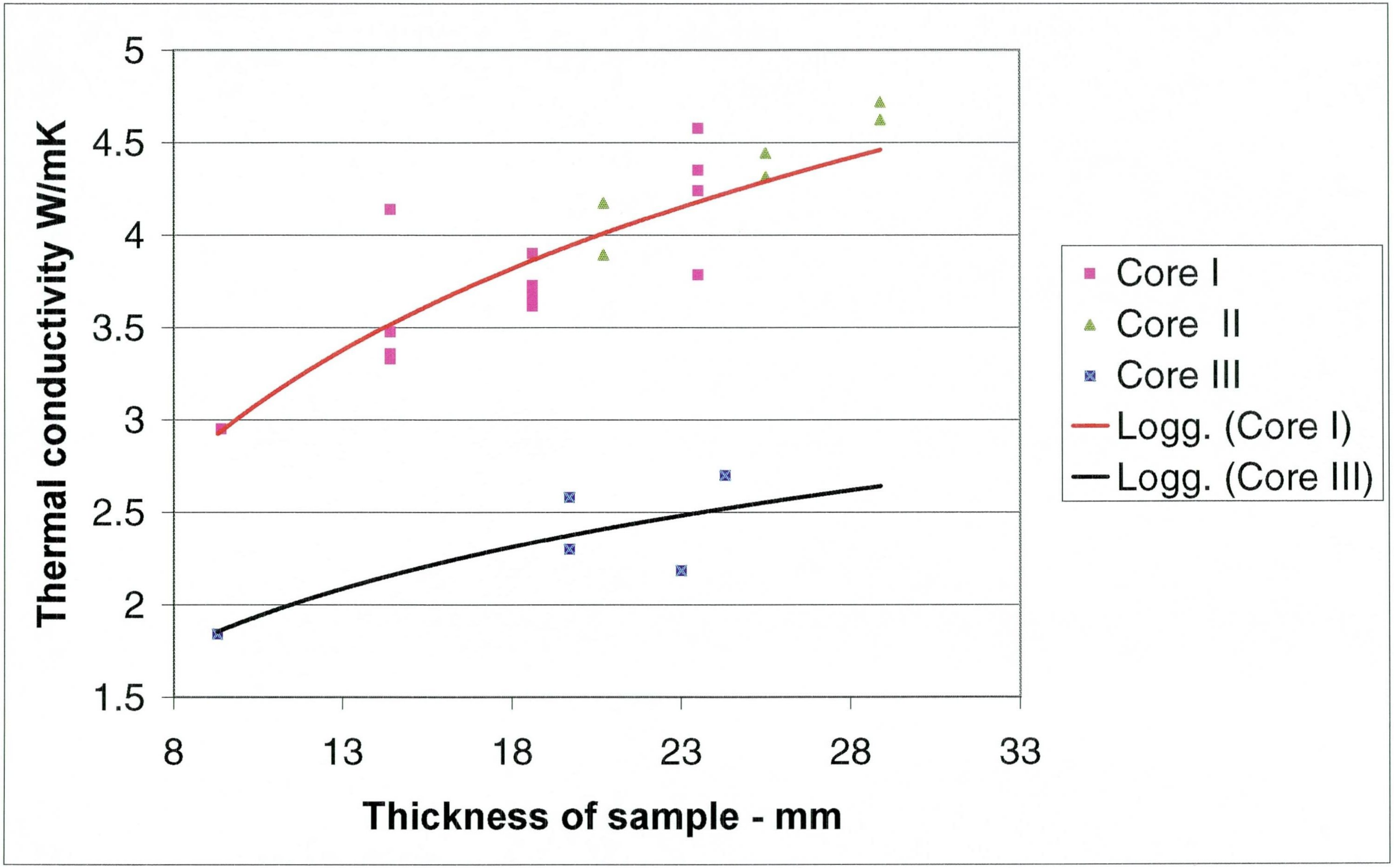
Specific heat was set as 880 W·s/kg·K for all samples (a mean value from Cermak & Ryback, 1982).

Table 1. Thermal conductivity measurements

	Thermal conductivity W/m·K						Density	Porosity	Specific heat
	Oil-sat 1	Oil-sat.2	Oil-sat 3	Oljem 4	Dry	Water-sat.	g/cm^3	%	Ws/kgK
Core I - 24	4.2	3.8	3.0[1]	4.2	4.4	4.6	2.70	0.1	880
Core I - 18	3.6	3.7			3.9	3.7	2.69	0.2	880
Core I -14	3.5	3.4			3.3	4.1	2.66	1.5	880
Core I - 09	3.0	broken							880
Core II - 29	4.6	4.7					2.68		880
Core II – 26	4.3	4.4					2.68		880
Core II – 21	3.9	4.2					2.68		880
Core III - 24	2.7						2.25		880
Core III – 23	2.2[2]						2.22		880
Core III – 20	2.3	2.6					2.21		880
Core III – 09	1.8						2.23		880

[1] There were air bubbles under the plastic which reduced the contact between sample and heat source and probably lowered the measured thermal conductivity.

[2] The sample was broken during the measurement.


Thermal conductivity W/mK
5
4.5
4
3.5
3
2.5
2
1.5
8
13
18
23
28
33
Thickness of sample - mm
Core I
Core II
Core III
Logg. (Core I)
Logg. (Core III)

DISCUSSION.

Thermal conductivity of samples from **core I** is 3.1W/m·K after correction for the effect of sample thickness by normalizing for a 10 mm thick sample. Recent measurements at NGU on 22 limestone samples give thermal conductivity values between 2.6 and 2.9 W/m·K. The higher thermal conductivity value for this sample may be due to the striations in the samples. Heat is probably transferred easier through these structural elements than through the matrix. The sample seems to have a high content of quartz and pyrite. These minerals have high thermal conductivity. Because the quartz and pyrite grains seems to be fine and isolated from each other the high content of these minerals does probably have a small effect on the thermal conductivity of the sample.
Sample I-24 and I-18 have low porosity values. The thermal conductivity of these samples is not affected by the fluid saturation. The porosity of sample I-14 is estimated to 1.5%. Thermal conductivity of water-saturated samples was measured to be 19 % higher compared with oil-saturated and dry sample. The effect of water saturation might be due to the higher porosity of this sample since water (k_{water} = 0.60 W/m·K) has a considerably higher thermal conductivity than air (k_{air}=0.024 W/m·K) and oil (k_{oil}= 0.24 W/m·K, value from Jensen & Dore (1993)).

The thermal conductivity of **core II** at 3.1 W/m·K is equal to core I. This result shows that there is no effect of anisotropy on the thermal conductivity, even though the material is anisotropic. This is probably due to the striations which is seen in different direction in the material.

The thermal conductivity of **core III** is 1.9 W/m·K. The samples from this core are porous. The weight of sample III-20 was reduced with 0.5 % during measurement indicating that the sample underwent drying. Drying of samples will lower the measured value of thermal conductivity. The measurements were carried out on oil-saturated samples. Thermal conductivity of water-saturated sample is probably higher than those measured on oil because of the higher thermal conductivity of water than of oil

CONCLUSION.

The thermal conductivity values for oil-saturated samples of limestone are determined to be as follows:
core I: 3.1 W/mK
core II: 3.1 W/mK.
core III: 1.9 W/mK..

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APPENDIX

Table 2.

Limestone -fine grained					*Thickness*	**Beregnet**			**Diffusivity**		16	V=16cm3	From referenc	Estimated
					mm	**t(0) s**	-T(i) K	m K/s	**@ m2/s**	Weight	Volum	Density	Spesif. Heat	**Thermal cond**
Sample	Number	Date	Saturation	Information					**10(-6)**	g	h*A (cm3)	g/cm3	Ws/kg K	**W/mK**
RL-24-1	1	27-Jan	oil	plastic	23.5	**51.603586**	8.87592	0.172002	**1.7836E-06**	69.47	26.085	2.70	880	**4.24**
RL-18-1	2	27-Jan	oil	plastic	18.6	**37.7556459**	8.72684	0.23114	**1.5272E-06**	55.78	20.646	2.69	880	**3.62**
RL-14-1	3	27-Jan	oil	plastic	14.4	**23.2933145**	9.37309	0.402394	**1.4837E-06**	42.95	15.984	2.66	880	**3.47**
RL09-1	4	27-Jan	oil	plastic	9.4	**11.6884535**	7.54755	0.645727	**1.2599E-06**	27.76	10.434	2.66	880	**2.95**
RL24-2	5	27-Jan	oil	plastic	23.5	**57.7890883**	8.12948	0.140675	**1.5927E-06**	69.38	26.085	2.70	880	**3.78**
RL18-2	6	27-Jan	oil	plastic	18.6	**36.6482606**	8.732291	0.238273	**1.5733E-06**	55.74	20.646	2.69	880	**3.72**
RL14-1	7	27-Jan	oil	plastic	14.4	**24.1063139**	10.0485	0.416841	**1.4336E-06**	42.95	15.984	2.66	880	**3.36**
RL24-3	8	28-Jan	oil	plastic	23.5	**72.7975971**	8.47051	0.116357	**1.2644E-06**	69.38	26.085	2.70	880	**3.00**
RL24-4	9	31-Jan	oil	plastic	23.5	**51.5886431**	9.00325	0.17452	**1.7841E-06**	69.38	26.085	2.70	880	**4.24**
RL18-3	10	31-Jan	dried	heat compoun	18.6	**35.0161763**	8.52875	0.243566	**1.6467E-06**	55.65	20.646	2.69	880	**3.90**
RL14-3	11	31-Jan	dried	heat compoun	14.4	**24.311564**	9.68845	0.398512	**1.4215E-06**	42.95	15.984	2.66	880	**3.33**
RL18-4	12	1-Feb	water		18.6	**37.1652726**	8.66434	0.23313	**1.5514E-06**	55.65	20.646	2.69	880	**3.67**
RL14-4	13	1-Feb	water	heat compoun	14.4	**19.5526763**	10.0324	0.513096	**1.7675E-06**	42.67	15.984	2.66	880	**4.14**
RL24-5	14	2-Feb	dried		23.5	**50.2983349**	9.61508	0.191161	**1.8299E-06**	69.29	26.085	2.70	880	**4.35**
RL24-6	15	2-Feb	water	heat compoun	23.5	**47.8032431**	9.86984	0.206468	**1.9254E-06**	69.32	26.085	2.70	880	**4.57**
Limestone -fine grained. Parallel with layering														
RL2-29-1	16	14-Feb	oil	plastic not top	28.9	**71.1927871**	8.87532	0.124666	**1.9553E-06**	86.15	32.079	2.69	880	**4.62**
RL2-26-1	17	14-Feb	oil	plastic not top	25.5	**59.4061512**	9.54942	0.160748	**1.8243E-06**	76.04	28.305	2.69	880	**4.31**
RL2-21-1	18	14-Feb	oil	plastic not top	20.7	**43.3385031**	9.19344	0.212131	**1.6478E-06**	61.66	22.977	2.68	880	**3.89**
RL2-29-2	19	14-Feb	oil	plastic not top	28.9	**69.7220857**	9.36019	0.13425	**1.9965E-06**	86.13	32.079	2.68	880	**4.72**
RL2-26-2	20	14-Feb	oil	plastic not top	25.5	**57.656295**	9.4151	0.163297	**1.8797E-06**	76.01	28.305	2.69	880	**4.44**
RL2-21-2	21	14-Feb	oil	plastic not top	20.7	**40.3848591**	10.8334	0.268254	**1.7684E-06**	61.63	22.977	2.68	880	**4.17**
Limestone,porous with fossils and fragments						**#DIV/0!**			**#DIV/0!**		0	#DIV/0!	880	**#DIV/0!**
RL3-23-1	22	2-Mar	oil	plastic not top	23	**79.0001391**	8.5174	0.107815	**1.116E-06**	56.74	25.53	2.22	880	**2.18**
RL3-20-1	23	2-Mar	oil	plastic not top	19.7	**54.742948**	11.2211	0.204978	**1.1816E-06**	48.36	21.867	2.21	880	**2.30**
RL3-24-1	24	2-Mar	oil	plastic not top	24.3	**72.1007266**	10.3391	0.143398	**1.365E-06**	60.63	26.973	2.25	880	**2.70**
RL3-09-1	25	2-Mar	oil	plastic not top	9.3	**15.3610509**	11.4128	0.74297	**9.3841E-07**	23.04	10.323	2.23	880	**1.84**
RL3-20-2	26	2-Mar	oil	plastic not top	19.7	**48.6851006**	10.2913	0.211385	**1.3286E-06**	48.28	21.867	2.21	880	**2.58**

Appendix 2

DESCRIPTION OF THE THERMAL CONDUCTIVITY APPARATUS.

APPARATUS CONFIGURATION

Figure 1 shows the set up used at NGU for measuring thermal conductivity using a transient method. A constant heat flow is induced from the top of the sample. The sample is insulated on all other surfaces. The temperature is measured at the base of the sample. Thermal diffusivity (α) is estimated from the temperature vs. time plot, and the thermal conductivity is calculated from thermal diffusivity, density (ρ) and specific heat (c_p) of the sample (see Table in Appendix 1). The theory of the method is described by Middleton (1993).

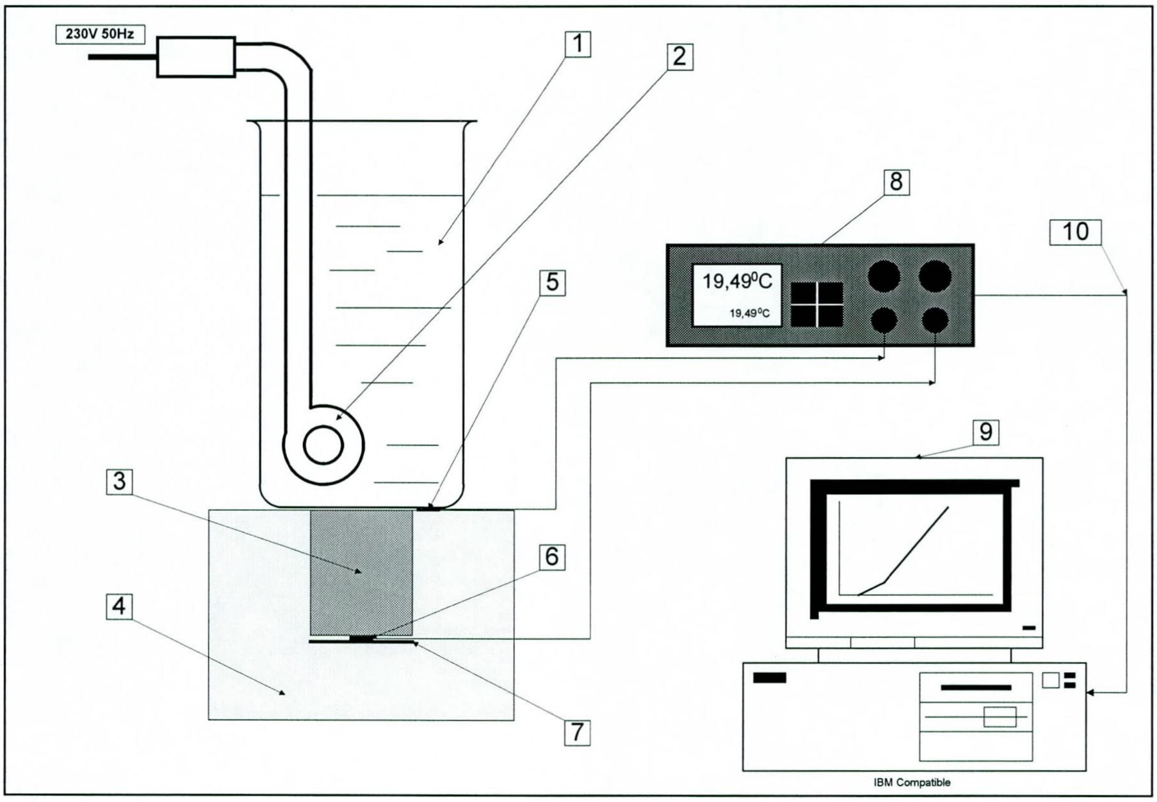

Figure 1. Diagram showing the configuration of NGU apparatus for transient method of measuring the thermal properties of rock.

Explanations to Figure 1:

1 - steel pan with boiling water
2 - 300 W heating element
3 – sample of rock
4 – insulation
5 – Pt100 sensor no 1
6 – Pt100 sensor no 2
7 – aluminium foil
8 – DOSTMANN® T855 thermometer
9 – PC with SmartGraph® recording software
10 – interface between thermometer and PC

ROCK SAMPLE

Rock samples used for the measurements can consist of drillcore sections (20-100 mm ∅; 10-40 thick) or square cut coupons (35 – 100mm on a side; 10 – 40 mm thick).

HEAT SOURCE

The heat source is a stainless steel pan with boiling water. The water is heated continuously by a 300 W heating element installed inside the pan. The diameter of the pan is 14 cm. The temperature under the pan is measured. The temperature has to be constant and above 95°C before the measurements start.

TEMPERATURE SENSORS

a) RTD Sensor – Introduction

A resistance-temperature detector (RTD) is a temperature sensing device whose resistance increases with temperature. A RTD consists of a wire coil or deposited film of pure metal. RTDs can be made of different metals and have different resistances, but the most popular RTD is platinum and has a nominal resistance of 100 Ω at 0° C – called **Pt100.** Platinum RTDs are known for their excellent accuracy and stability over a wide temperature range.

b) The Relationship of Resistance and Temperature in RTDs

The output of an RTD is relatively linear with respect to temperature. The temperature coefficient, (α) differs between RTD curves. Although various manufacturers may specify alpha differently, alpha is most commonly defined as the change in RTD resistance from 0 to 100°C, divided by the resistance at 0°C, divided by 100°C:

$$\alpha\ [\Omega/\Omega/°C] = (R_{100} - R_0)/(R_0 * 100°C)$$

where R_{100} is the resistance of the RTD at 100°C,
and R_0 is the resistance of the RTD at 0°C.
For example, a 100Ω platinium RTD with $\alpha = 0.003911$ will measure 139.11Ω at 100°C.

Although the resistance-temperature curve is relatively linear, accurately converting the measured resistance to a temperature value requires curve fitting.

The Callendar-Van Dusen equation is commonly used to approximate the RTD curve:

$$R_t = R_0\ [1 + At + Bt^2 + C(t - 100)t^3\ ]$$

where R_t is the resistance of the RTD at temperature = t,
R_0 is the resistance of the RTD at 0°C,
A, B, and C are the Callendar-Van Dusen coefficients, see Table 1,
and t is the temperature in °C.

Most platinum RTD curves follow one of three standardized curves:
- the DIN 43760 standard (α=0.00385),
- the U.S. Industrial or 'American' standard (α=0.003911),
- the International Temperature Scale (ITS-90) for wire-wound RTDs (α=0.003925).

Table 1. Callendar-Van Dusen Coefficients Corresponding to Common RTDs

Standard	**Temperature Coefficient (α)**	**A**	**B**	**C***
DIN 43760	0.003850	3.9080 x 10 -3	-5.8019 x 10^{-7}	-4.2735 x 10 -12
American	0.003911	3.9692 x 10 -3	-5.8495 x 10^{-7}	-4.2325 x 10 -12
ITS-90	0.003926	3.9848 x 10 -3	-5.870 x 10^{-7}	-4.0000 x 10 –12

* For temperatures below 0°C only; C=0.0 for temperatures above 0°C.

If a known current, I_{EX}, passes through the RTD and the output voltage (V_0) developed across the RTD, is measured then:

$$\mathbf{t = 2(V_0 - I_{EX} R_0) / I_{EX} R_0 \left[A + \sqrt{A^2 + 4B(V_0 - I_{EX} R_0)/I_{EX} R_0}\,\right]}$$

where V_0 is the measured RTD voltage and I_{EX} is the excitation current.

c) Tolerance Class of RTD Sensors

Sensor manufacturers offer a wide range of sensors that comply with BS1904 class B (DIN 43760): these sensors offer an accuracy of ±0.3 °C at 0 °C. For increased accuracy, BS1904 class A (±0.15 °C), 1/3DIN (±0.1 °C) or tenth-DIN sensors (±0.03 °C). Companies like Isotech can provide standards with 0.001 °C accuracy.

Table 2. Tolerance Class of RTD Sensors

Class	Tolerance (°C)	Resistance Tolerance at 0°C
1/3DIN	± (0.1 + 0.0017t)	± 0.04
A	± (0.15 + 0.002t)	± 0.06
B	± (0.3 + 0.005t)	± 0.12

Other related standards are IEC751 and JISC1604-1989.

d) RTD Measurement Circuits

Because the RTD is a resistive device, a current must drive through the device and the resulting voltage must be monitored. However, any resistance in the lead wires that connect your measurement system to the RTD will add error to your readings. For precision work, sensors have four wires- two to carry the current, and two to measure the voltage across the sensor element. It is also possible to obtain three-wire sensors, but these operate on assumption that the resistance of each of the three wires is the same.

The current through the sensor will cause some self heating. If the sensor element is unable to dissipate this heat, it will report an artificially high temperature. This effect can be reduced by either using a large sensor element, or by making sure that it is in good thermal contact with its environment.

e) RTD Sensor used in NGU Apparatus

A two thick film PT100 sensors type M-FK 422, tolerance class B, privided by ELFA Scandinavia – Sweden are used at the NGU apparatus. The sensors are connected to a digital thermometer. The sensor has a 0.2 s time constant. The sensore's measurement surface has dimensions 2,2 x 4 mm. A sensor is placed in the middle under the sample and above a thin foil of aluminium. The other sensor is located under the pan.

THERMOMETER

A universal bench thermometer DOSTMANN® type T855 is used in the NGU apparatus. Technical data for the thermometer are presented in Table 3.

Table 3. Specifications for the DOSTMANN® type T855 thermometer

Meas. Channel 1	Pt100 4-wire
Meas. Channel 2	Pt100 4-wire
Measuring Range	-200^{0}C ... +200^{0}C
Accuracy for –500C…+1500C	±0.03^{0}C
Accuracy for remainig range	±0.05^{0}C
Resolution	0.01^{0}C
Sampling Rates	1/10/30 sec./1/10/60 min.
Interface	V24 (RS232), 2 anal. outp. 0-5V, printer
Linearisation Standard	DIN 43760

RECORDING SOFTWARE

DOSTMANN® SmartGraph Series P500/T800 recording and graphical presentation software is used in the NGU apparatus. This software offers thermometer control, online documentation, graphic and tabular presentation of the measured temperature values. The data recorded with the NGU apparatus is exported as an Excel spreadsheet file for further data processing and presentation

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