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Forsmark site investigation

Borehole KFM04A

Thermal properties – Anisotropic thermal conductivity and thermal diffusivity determined using the Hot Disk thermal constants analyser (the TPS technique)

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December 2006

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Abstract

Drill core samples from borehole KFM04A, Forsmark, Sweden, have been measured with the Hot Disk Thermal Constants Analyser (TPS technique) with respect to anisotropic thermal properties.

The axial thermal conductivity is increasing versus depth, from 2.07 W/m·K to 3.28 W/m·K (58%) between samples 6, 7 and 8. Sample 8 is also where the thermal conductivity peaks. Sample 9 has a lower thermal conductivity in the axial direction than sample 8: 3.19 W/m·K, whereas sample 10 displays the lowest axial thermal conductivity of all samples, 1.05 W/m·K.

The axial diffusivity is following the same pattern as the axial thermal conductivity. Here the increase is almost as high as for the conductivity, 56% (1.07 to 1.67 mm^2/s). Sample 9 shows an axial diffusivity of 1.59 mm^2/s. Also here sample 10 demonstrates the lowest value: 0.53 mm^2/s.

The radial thermal conductivity values are fluctuating between 3.50 and 5.64 W/m·K. No trend depending on depth is visual.

The radial diffusivity also fluctuates without a trend. The values all lie in the range 1.77–2.87 mm^2/s.

The results in this report indicate that the TPS technique is capable of measuring anisotropic thermal properties of rock samples.

Sammanfattning

Borrkärneprover från borrhål KFM04A i Forsmark har testats med Hot Disks ”Thermal Constants Analyser” (s k TPS-teknik) avseende anisotropa termiska egenskaper.

Den axiella termiska konduktiviteten karaktäriseras först av en ökning med djupet från 2,07 W/m·K till 3,28 W/m·K (58 %) mellan proverna 6, 7 och 8. Prov 8 har därmed det högsta värdet i den aktuella provserien. Prov 9, däremot, har lägre termisk konduktivitet i axiell riktning än prov 8, 3, 19 W/m·K, medan prov 10 uppvisar den lägsta termiska konduktiviteten av samtliga prov, 1,05 W/m·K.

Den axiella diffusiviteten följer samma mönster som den axiella konduktiviteten. I detta fall uppgår ökningen från prov 6 över prov 7 till prov 8 till 56 % (från 1,07 till 1,67 mm^2/s). Prov 9 har en axiell diffusivitet på 1,59 mm^2/s, medan prov 10 resulterat i det lägsta värdet, 0,53 mm^2/s, även för denna parameter.

Den radiella termiska konduktiviteten fluktuerar mellan 3,50 och 5,64 W/m·K. Någon djuptrend har i detta fall inte observerats.

Även den radiella diffusiviteten varierar, men utan någon speciell trend. Samtliga värden ligger i intervallet 1,77–2,87 mm^2/s.

Resultaten i denna rapport indikerar att TPS-tekniken är väl lämpad att identifiera anisotropa termiska egenskaper hos bergprover.

Contents

1 Introduction

The objective of this investigation is to measure anisotropic conductivity and diffusivity of drill core samples from borehole KFM04A, Forsmark, Sweden, by using the anisotropic TPS method. The measurements were carried out on water saturated specimens cut from rock core samples. The samples were selected based on the preliminary core logging and with the strategy to primarily investigate the properties of the dominant rock types.

The principle of the TPS instrument is to place a circular temperature probe and heater between 2 pieces of the sample /1/. This probe consists of a Ni-spiral covered by an insulating material (Mica or Kapton). For measurements below the temperature 230°C, generally the Kapton covered sensor is used. During the measurement a constant power is emitted by the sensor and the heating of the specimen is recorded simultaneously. The data are then treated in the Hot Disk software and the anisotropic thermal conductivity and thermal diffusivity are determined provided that the specific heat capacity, ρC_p, is known prior to the anisotropic measurements. A requirement for the anisotropic technique is that the unique axis of the sample is perpendicular to the plane of the sensor. Therefore the samples tested here were cut parallel to the foliation.

The test programme follows the activity plan AP PF 400-04-61, version 1.0 (SKB internal controlling document).

Prior to the measurements the samples were water saturated for at least 7 days.

The rock cores arrived at Hot Disk AB in May 2004, and the measurements were carried out in June 2004.

The samples were sent to Hot Disk AB directly from SKB.

The specific heat capacity values necessary for the calculations were provided by SP, Borås, in January 2006. These values can be found in Appendix 2.

This report is a third revision based on the measured C_p- and density values provided by SP. Revision information and comments have been added in the end of the results section.

2 Scope and objective

In order to better understand anisotropic properties of thermal transport (conductivity and diffusivity), anisotropic properties of five rock samples from borehole KFM04A were measured with the Hot Disk Constants Analyser (TPS technique).

3 Equipment

The equipment used for the measurements was a Hot Disk thermal constants analyser.

The following items are included in the Hot Disk instrument (Figure 3-2):

1. Hot Disk bridge unit.
2. Keithley 2400 source/meter.
3. Keithley 2000 volt meter.
4. PC.
5. Computation Device.
6. Hot Disk SW, version 5.7.

The sensor used for the measurements was Kapton insulated, had a radius of 6.401mm (S/N C5501) (for a picture of a typical Hot Disk sensor, see Figure 3-1). Sample set-up is shown in Section 4.2.

Additional equipment were rubber bands, plastic bags, table cloth and plastic straps, see Section 4.2 and Figure 3-3.

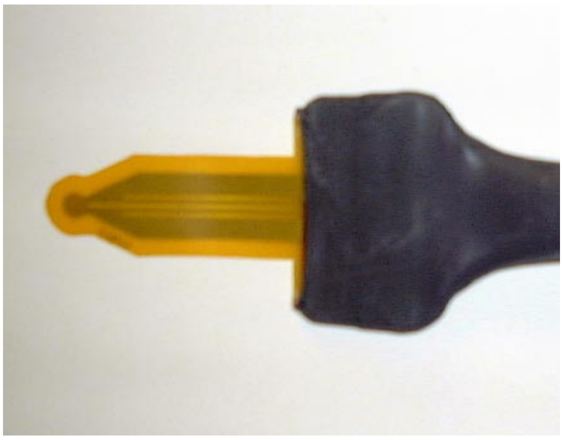

Figure 3-1. *A typical Hot Disk sensor (S/N 7577). For the measurements in this report, the sensor used had larger radius.*

Figure 3-2. *Hot Disk instrument.*

Figure 3-3. *Plastic bag, rubber band sample and sensor used for the measurements in this report.*

4 Execution

4.1 Samples

A total of 10 drill core specimens were sampled from borehole KFM04A, Forsmark. Borehole KFM04A is inclined c 60° from the horizontal plane. The samples were labelled 1–10, where 1st comes from the most shallow part of the drill hole and 10 from the deepest. Each sample was divided into two sub-samples, denoted A and B respectively. The sample diameter was c 50 mm. The samples were cut parallel or perpendicular to the foliation. Due to the method requirements, measurements have been carried out only on the samples cut parallel to foliation (nos. 6–10), see Table 4-1. The sampling levels ranged between 530.89 m and 531.31 m borehole length for these five samples, see Table 4-1. Detailed geological description of the rock is given in SKB's Boremap mapping of KFM04A in SKB´s database SICADA. No further information on the rock type was available except that the samples are granite. Table 4-1 also provides the specific heat capacity values used for the Hot Disk measurements. These values are calculated based on the *average densities, ρ, of the individual specimens* and the measured C_p measurements from SP (see Appendix 2). This makes the values different to the ones displayed in Appendix 2.

4.2 Measurements

All measurements were carried out with the Hot Disk Bridge system equipped with software for anisotropy measurements.

Prior to the measurement all samples were soaked in water for a period of at least 7 days. With a rough frequency of 2 samples per day the samples were taken out from the water and dried with a wet cloth.

The samples were then prepared for the measurement in the following way:

The sensor was sandwiched between the samples and the sample pieces forced together with a dead weight on top of the set-up, see Figure 4-1. The plastic bag was sealed off with a rubber band.

Five measurements were carried out at room temperature (22 ± 2°C) for each sample. The sensor chosen for the measurements were C5501 (radius 6.401 mm) and power and measurement time were 0.7 W and 20 s respectively. These measurement parameters proved to give a rough temperature increase of 0.5–1.5 K in the interval of calculation, a total to characteristic time well within the interval 0.3 and 1.0 and an experimental probing depth less than the maximum allowed probing depth. The specific heat capacities and the densities of each body measured by SP, Borås, were used for each sample.

Table 4-1. Sample data with specific heat capacity. The samples were labelled at Hot Disk.

Sample ID	Cut	Unique axis perpendicular to plane of sensor	Sampling depth (Secup–seclow)	Specific heat cap. [MJ/m³K]
6	Parallel Foliation	Yes	530.89–530.97	1.937
7	Parallel Foliation	Yes	530.97–531.07	1.989
8	Parallel Foliation	Yes	531.07–531.15	1.962
9	Parallel Foliation	Yes	531.15–531.23	2.013
10	Parallel Foliation	Yes	531.23–531.31	1.964

Figure 4-1. *Sensor sandwiched between two sample pieces. The plastic bag was sealed off with a rubber band and a dead weight was mounted on top of the sample set-up.*

Calculations of the measurement files were carried out according to the Hot Disk manual, meaning that the selected points of calculation were chosen so that the points as well as possible fitted the thermal conductivity equation. Data were then transported into Microsoft® Excel for further treatment.

Between each of the anisotropy measurements a measurement was carried out on a SIS 2343 mildsteel sample with the C5501 sensor used for the tests (for results, see Appendix 1).

4.3 Nonconformities

The following are deviations from the measurement plan (AP PF 400-04-61):

- Only samples cut parallel to foliation (nos. 6–10) have been measured, since the Anisotropy method is not designed to measure samples with the unique axis parallel with the plane of the Hot Disk sensor.
- Sample 8 was remeasured 12 hours after it was taken out from the water bath. The reason for this was that the sample surface facing the sensor had severe surface irregularities causing the measurement data to fluctuate too much.

5 Results

Section 5.1 contains the results calculated out of the mean values of measured data, five repeated measurements.

5.1 Measurement results per sample

The results for the measurements are given in the following tables below, see Tables 5-1 to 5-5. All values are means from 5 individual measurements. For pictures of the samples, please refer to Appendix 2.

Table 5-1. Results for sample 6.

Sample	Points for calc.	λ_{axial} [W/m·K]	κ_{axial} [mm^2/s]	λ_{radial} [W/m·K]	κ_{radial} [mm^2/s]
6	(42–132)	2.143	1.106	4.470	2.308
	(42–132)	2.048	1.057	4.533	2.340
	(42–132)	1.908	0.985	5.051	2.608
	(39–107)	2.052	1.060	4.848	2.503
	42–132)	2.190	1.131	4.674	2.413
Average		**2.07**	**1.07**	**4.72**	**2.43**
Stdev		**0.11**	**0.06**	**0.24**	**0.12**

Table 5-2. Results for sample 7.

Sample	Points for calc.	λ_{axial} [W/m·K]	κ_{axial} [mm^2/s]	λ_{radial} [W/m·K]	κ_{radial} [mm^2/s]
7	(77–200)	3.040	1.528	3.569	1.794
	(77–200)	3.057	1.537	3.527	1.773
	(77–200)	3.049	1.533	3.500	1.760
	(77–200)	3.025	1.521	3.497	1.758
	(77–200)	2.996	1.506	3.502	1.761
Average		**3.03**	**1.53**	**3.52**	**1.77**
Stdev		**0.02**	**0.01**	**0.03**	**0.02**

Table 5-3. Results for sample 8, remeasured data set.

Sample	Points for calc.	λ_{axial} [W/m·K]	κ_{axial} [mm^2/s]	λ_{radial} [W/m·K]	κ_{radial} [mm^2/s]
8	(27–200)	3.229	1.646	3.566	1.817
	(27–200)	3.306	1.685	3.492	1.780
	(27–200)	3.288	1.676	3.488	1.778
	(27–200)	3.293	1.678	3.476	1.772
	(27–200)	3.296	1.680	3.467	1.767
Average		**3.28**	**1.67**	**3.50**	**1.78**
Stdev		**0.03**	**0.02**	**0.04**	**0.02**

Table 5-4. Results for sample 9.

Sample	Points for calc.	λ_{axial} [W/m·K]	κ_{axial} [mm^2/s]	λ_{radial} [W/m·K]	κ_{radial} [mm^2/s]
9	(59–200)	3.197	1.588	3.967	1.971
9	(51–200)	3.163	1.571	4.007	1.991
9	(61–200)	3.205	1.592	3.955	1.965
9	(60–200)	3.206	1.593	3.948	1.961
9	(61–200)	3.201	1.590	3.944	1.959
Average		**3.19**	**1.59**	**3.96**	**1.97**
Stdev		**0.02**	**0.01**	**0.03**	**0.01**

Table 5-5. Results for sample 10.

Sample	Points for calc.	λ_{axial} [W/m·K]	κ_{axial} [mm^2/s]	λ_{radial} [W/m·K]	κ_{radial} [mm^2/s]
10	(30–116)	1.154	0.588	5.272	2.684
10	(34–118)	1.006	0.512	5.728	2.917
10	(25–142)	1.066	0.543	5.603	2.853
10	(28–136)	1.058	0.539	5.625	2.864
10	(35–143)	0.950	0.484	5.971	3.040
Average		**1.05**	**0.53**	**5.64**	**2.87**
Stdev		**0.08**	**0.04**	**0.25**	**0.13**

5.2 Summary of results

In the tables below the results are summarized (Table 5-6 and table 5-7).

The axial thermal conductivity varies between 1.05 W/m·K and 3.28 W/m·K for samples 6 to 10, where sample 10 has the lowest value and sample 8 displays the highest. For samples 6, 7 and 8 the thermal conductivity is increasing from 2.07 W/m·K to 3.28 W/m·K (58%). Sample 9 shows a thermal conductivity in the axial direction of 3.19 W/m·K.

The axial diffusivity is following the same pattern as the axial thermal conductivity. Here the values are within the range 0.53 to 1.67 mm^2/s. Also here sample 10 has the lowest value: 0.53 mm^2/s.

Table 5-6. The averages of each measurement.

Sample	λ_{axial} [W/m·K]	κ_{axial} [mm^2/s]	λ_{radial} [W/m·K]	κ_{radial} [mm^2/s]
6	2.07	1.07	4.72	2.43
7	3.03	1.53	3.52	1.77
8	3.28	1.67	3.50	1.78
9	3.19	1.59	3.96	1.97
10	1.05	0.53	5.64	2.87

Table 5-7. The standard deviations of the results above.

Sample	Std-λ_{axial} [W/m·K]	Std-κ_{axial} [mm^2/s]	Std-λ_{radial} [W/m·K]	Std-κ_{radial} [mm^2/s]
6	0.11	0.06	0.24	0.12
7	0.02	0.01	0.03	0.02
8	0.03	0.02	0.04	0.02
9	0.02	0.01	0.03	0.01
10	0.08	0.04	0.25	0.13

The radial thermal conductivity values are fluctuating between 3.50 and 5.64 W/m·K for all samples.

The radial diffusivity also fluctuates without a trend. The values are within the range 1.77–2.87 mm^2/s.

The standard deviations are all low in comparison with the measured conductivities and diffusivities.

5.3 Discussion

Anisotropic thermal transport properties of samples from borehole KFM04A, Forsmark, have been measured with the Hot Disk instrument at Hot Disk AB, Uppsala.

The results for samples 6–10 seem to vary depending on depth (sample 6 was taken from 530.95–531.03 m borehole length and sample 10 from 531.29–531.37 m borehole length, see Table 4-1). The results show some consistency except for sample 10 which has a lower axial thermal conductivity and also a lower thermal diffusivity in the axial direction.

Some of the samples were unflat and had edges causing problems to mount the sensor properly. If these edges on the samples could be removed, a source of error would be removed.

5.4 Comments on the revision from June 2004

The earlier revision of this report (June 2004) contained thermal conductivity and diffusivity data calculated with an estimated specific heat capacity: 2.271 MJ/m^{3}K. This value was chosen since it is the average of the specific heat values in the report P-04-160, because the samples from borehole KSH01A were similar to the samples produced for the measurements in the present report. Table 5-8 below shows the estimated anisotropical values from the earlier revision together with the new values. For clarity the quotient $\lambda_{radial}/\lambda_{axial}$ and $\lambda_{radial}/\lambda_{axial}$ is given next to each result column.

Table 5-8. The results of the measurements when 2.271 MJ/m^{3}K was used, together with data based on SP's specific heat capacity measurements.

Sample (C$_p$ from SP meas.)	λ_{axial} **[W/m·K]**	κ_{axial} **[mm^2/s]**	λ_{radial} **[W/m·K]**	κ_{radial} **[mm^2/s]**	$\lambda_{radial}/\lambda_{axial}$	$\kappa_{radial}/\kappa_{axial}$	ρC_P **[MJ/m^{3}K]**
6	2.07	1.07	4.72	2.43	2.28	2.28	1.937
7	3.03	1.53	3.52	1.77	1.16	1.16	1.989
8	3.28	1.67	3.50	1.78	1.07	1.07	1.962
9	3.19	1.59	3.96	1.97	1.24	1.24	2.013
10	1.05	0.53	5.64	2.87	5.39	5.39	1.964
Sample (ρC_p = 2.271 MJ/m^{3}K)							
6	2.13	0.94	5.18	2.28	2.43	2.43	2.271
7	2.84	1.25	4.04	1.78	1.42	1.42	2.271
8	2.98	1.31	4.01	1.77	1.35	1.35	2.271
9	3.06	1.35	4.39	1.93	1.43	1.43	2.271
10	0.98	0.43	6.34	2.79	6.47	6.47	2.271

Below it is shown how a 10% variation in the ρC_p value would affect the measured thermal conductivity and diffusivity values. The equations are

$C_p = \lambda_{axial}/\kappa_{axial} = \lambda_{radial}/\kappa_{radial}$

$\lambda_{bulk} = \mathrm{sqrt}\,(\lambda_{axial}\ \lambda_{radial})$

and $\kappa_{bulk} = \kappa_{radial}$

With these equations it can be shown that by increasing the specific heat by 10%

- λ_{axial} is decreased by roughly 10%, λ_{radial} is increased by 10%, κ_{axial} is decreased by roughly10% and κ_{radial} is unchanged.

Also it can be shown that by decreasing the specific heat by 10%

- λ_{axial} is increased by roughly 11%, λ_{radial} is decreased by 10%, κ_{axial} is increased by roughly 11% and κ_{radial} is unchanged.

The early estimation of 2.271 MJ/m^{3}K is approximately 15.1% larger than the average specific heat capacities measured by SP in Borås. Following this means that the axial conductivity and diffusivities should be increased with roughly 15.1% and the radial thermal conductivity should be decreased with approximately 15.1%. The radial thermal diffusivity should remain the same since that value directly is given by the measurement regardless of specific heat capacity.

Looking at Table 5-8 it can be seen that the radial diffusivity is fairly independent of the changes in ρC_p, the radial conductivity is decreased by between 9 and 13%, the axial conductivity is increased by between 4 and 10% (for sample 6 a decrease in conductivity by 2.8%) and the axial diffusivity is increased by between 14 and 27%.

Since the radial conductivity should decrease with decreasing specific heat capacity and the axial conductivity should increase, the quotients between these two should decrease. This can also be viewed in Table 5-8.

There is one main reason for the difference between the experimental values and the theoretical calculations above: The introduction of the new specific heat capacity values was done in the raw data files, meaning a recalculation of the entire set of data files was needed. It is considered a priority to always analyse a set of data points in such a way that the best possible fit to the heat conductivity equation is found. This meant that the same points of calculation were not used.

References

/1/ Instruction Manual, Hot Disk Thermal Constants Analyser, Windows 2000/XP Version 5.6.

Appendix 1

Measurements on stainless steel, SIS 2343 mildsteel, during the same time period as anisotropy measurements

In Table A-1 the results from measurements carried out on a SIS 2343 mildsteel sample are displayed.

Power during measurement: 1W.
Sensor: C5501 (same as used for measurements on rock samples).
Measurement time: 10 s.

The thermal conductivity results are stable, except for the measurement carried out on May 26 where the thermal conductivity value (13.39 W/m·K) is roughly 4.5% lower than the other measurements. Specific heat values and diffusivity values are stable for all measurements. The reason why the May 26 thermal conductivity values are lower than the other values may be a number of factors, but the most significant deviation from the other measurements is the rather high temperature drift in the sample prior to measurement. A temperature drift can be caused by touching the sample for too long, or not allowing the sample to reach an isothermal state.

Table A-1. Results from measurements on SIS 2343 mildsteel.

Date	Temperature Drift [K]	λ [W/m·K]	κ [mm^2/s]	C_p [MJ/m^{3}K]
26 May	0.03	14.11	3.648	3.867
26 May	0.1	13.39	3.597	3.723
01 Jun	0.03	14.12	3.557	3.969
02 Jun	0.03	13.88	3.498	3.969

Appendix 2

A2.1 Determination of specific heat capacity of rock samples

Content of the Commission

The specific heat capacity or, with modern terminology, heat capacitivity of five rock samples, ref. KFM04A, was measured using a calorimetric method. Some other physical quantities of the stone samples in question were determined before, and other thermal quantities are measured after these measurements. For this reason the samples were kept in water all the time. The calorimetric procedure to determine the specific heat was performed very close to room temperature.

The data for these measurements are given with uncertainty estimations based on experimental experience with the calorimetric method and the newly designed equipment. A validation against other methods of determination was not performed.

Purpose of the measurements

The thermal quantities of the stone material (thermal conductivity and specific heat capacity) are important quality measures for estimating the heat transportation and in order to define the compactness how dens nuclear waste can be stored in holes drilled in stone below ground. This information is one of several others concerning the stone quality that is measured and is part of a project that involves several different technical disciplines at SP.

The Measurement Technique

The heat capacitivity is a material quantity specifying the amount of energy needed to increase/lower the temperature of one gram of substance with one degree Kelvin. It is determined by measuring the temperature change when absorbing the energy in a different medium. This material quantity depends on the temperature from which the increase/lowering takes place. In a calorimetric method usually a liquid with known heat capacitivity (water $c_W = 4.185$ J/g·K) is used to measure the amount of delivered or absorbed heat energy.

Physical Model

$$Q_m = c_m \cdot m_m \cdot \Delta T_m \qquad (1)$$

If the absorbed/delivered heat energy Q_m and the temperature change ΔT_m in the material of known mass m_m is measured, then the heat capacitivity c_m can be calculated according to (1).

In a calorimetric method the energy delivered by the material object Q_m is absorbed in a calorimeter Q_C and dominantly of its working medium (water) Q_W.

$$Q_m = Q_C + Q_W$$

$$c_m \cdot m_m \cdot (T_m - T_{EQ}) = C_C + c_W \cdot m_W \cdot (T_{EQ} - T_C) \qquad (2)$$

Equation (2) gives a simple model for the calorimetric technique. Three temperatures must be measured, T_m the initial temperature of the piece of material, T_C the temperature of the calorimeter and the contained water before introducing the test piece and T_{EQ} the final temperature after an equalization of both temperatures. Further the masses of the test piece m_m and of the energy absorbing water m_W must be measured in advance, to derive the interesting quantity c_m. In standard methods the calorimeter constant C_C depends on the working temperature and the achieved temperature change and is determined in advance by suitable calibration.

The model (2) is mathematically poor and there are a lot of effects that in practice influence the measured temperatures and masses. This leads to a need of corrections and limits the accuracy in the calculated value for c_m.

Limitations and Measurement Concept

The rock samples, prepared from a cylinder of 51 mm diameter by cutting them in slices approximately 45° angle to the axis, with a thickness of approximately 30 mm, were not suitable for commercial standard calorimeters. Further the heat capacitivity data should concern the wet stone. The samples thereforee were kept in water all the time. A second request was to determine the values at room temperature, which means that the equalizing temperature T_{EQ} and the initial temperature T_m should be low, compared to standard conditions.

A calorimeter in the form of a cylindrical container with a lid was produced, giving space for two thermometers, a magnetic stirrer and a rock sample with a minimum of water. The idea is to reach a measurable temperature rise from room temperature with relatively low sample temperature.

The amount of water was adjusted for each sample by weighing the calorimeter filled up to a predetermined level, as just to cover the sample entirely. This was tested in advance.

Selection of test pieces

Of ten rock samples numbered 6A, 6B etc to 10A and 10B supplied by SKB, with the specific cutting mentioned above, the heat capacitivity of five should be measured. Always the A-sample was used except for 6A, which was replaced by 6B as the first one was especially large cut and thus did not fit nicely into the designed calorimeter.

Method of Measurement

The measurement principle used was to keep the rock samples in a temperature controlled water bath at 31.5°C for a time long enough to stabilize.

The calorimeter was filled with thermally prepared water and adjusted to the amount previously decided on a balance (± 0.005 g). The temperature was chosen to be between 20 and 21°C which was about 1.5°C over the controlled room temperature of 19°C. This had proven to quickly reach steady state conditions (loss of energy to the surrounding was compensated by the mechanical energy production of the stirrer).

The temperatures of the calorimeter and the bath were logged during the whole experiment with three temperature sensors (two in the calorimeter and one in the bath) connected to a temperature logger.

Different techniques were tested, but most practical was to fetch a sample by hand out of the bath, get rid of the excess water by a fast hand movement, taking the piece over with the other hand and inserting it, still wet on the surface, into the calorimeter, putting the lid with insulation on top. During this 3 to 5 seconds change over some energy is lost, which was estimated. This temperature is close to the human skin temperature which also minimized the risk to withdraw or add unwanted energy.

The two sensors monitor first an exponential rise of temperature (1.5 to 2°C) up to a maximum and then a very slow exponential decrease. After sufficient measurement points the experiment is terminated, the sensors removed and the calorimeter is weighed again. A non intended transfer of water from the bath into the calorimeter is thus determined by the mass difference, taking into account the mass of the sample. In the same way losses of water by changed test conditions can be evaluated, as well as evaporation losses.

The temperature recorded by the sensors has a resolution of better than 0.01 mK and an uncertainty less than 10 mK. The actual mixing or equalizing temperature is evaluated afterwards by fitting the exponential decay extrapolated to the starting point. This technique is somewhat circumstantial but covers the total overlay of several temperature processes acting together and reduces the need of a calorimeter constant to insignificance.

Considering the relative low temperature changes, energy losses not directly measurable with this method should be kept lower than 2 to 3 percent.

Equipment

Calorimeter:	Specially made of Macrolon, made for this purpose (low heat capacitivity and very low conductivity).
Magnetic stirrer:	IKA type BigSquid.
Temperature logger:	Keithly 2200 Multimeter with scanner Keithley 7700.
Resolution:	0.01 mK.
Accuracy:	0.005°C.
Temperature sensors:	Pt-100 Pentronic 3 of different shape.
Measurement program:	SP in computer Toshiba with Visual Basic 6.
Balance:	Mettler PM 2000 (resolution 0.01 g).
Air temperature:	Testo 610 (Testoterm).
Air humidity:	Testo 610 (Testoterm).

All measurement instruments are traceable via in-house calibration to national and international standards.

Calculation of c_m

The measurements concern mainly the five quantities in (2) aborting the calorimeter constant:

m_m	the mass of the test piece,
m_W	the mass of the water at start,
T_m	the temperatures of the sample, which is the temperature of the bath reduced by an estimated amount of $\Delta T = 0.4°C$ during transfer,
T_C	the temperature of the water at steady state in the calorimeter,
T_{EQ}	the temperature after total equalisation, which is evaluated experimentally by separating to exponential processes in the mixing phase.

However, due to practical reasons two complementary quantities are introduced into the model:

Δm_W	the mass of the non-intentional transferred water (mass at start – mass at end) is a small amount, typically 0.25 to 0.45 g of water. It adds energy equal to the temperature drop from the bath to the mixing temperature,
ΔQ_E	the estimated amount compensating for the evaporation losses during the insertion in the calorimeter before and during the mixing, which is estimated to between one and two percent. The mass loss connected to this evaporation is barely measurable. The evaporation energy however is large, 540 cal/g (2,259 J/gK).

$$c_m = \frac{c_w \cdot \left[m_W \cdot \left(T_{EQ} - T_C\right) - \Delta m_W \cdot \left(T_m - T_{EQ}\right)\right] + \Delta Q_E}{m_m \cdot \left(T_m - T_{EQ}\right)}$$

Results

The evaluated values are collected in the table below.

Test sample	Secup-seclow	Mass [g]	Heat capacitivity [J/g·K]	Uncertainty [J/g·K]	Uncertainty [%]
6 B	530.89–530.97	204.71	0.73	±0.026	3.6
7 A	530.97–531.07	197.29	0.75	±0.03	4.0
8 A	531.07–531.15	202.81	0.74	±0.029	3.9
9 A	531.15–531.23	223.11	0.76	±0.03	4.0
10 A	531.23–531.31	202.64	0.74	±0.025	3.4

The uncertainty statements are calculated according to EA-4/02 with coverage factor k=2, giving a coverage probability of 95% normal distribution assumed.

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A2.2 P502681 KFM04A: Density and porosity

Table A2-1. Level 1,530–532 m. Specimens KFM04A-6 to KFM04A-10.

KFM04A-6 (530.89–530.97)

The dry density for specimen KFM04A-6A was measured to be 2,655 kg/m^3 and the porosity to 0.30% and the dry density for specimen KFM04A-6B was measured to be 2,653 kg/m^3 and the porosity to 0.27%.

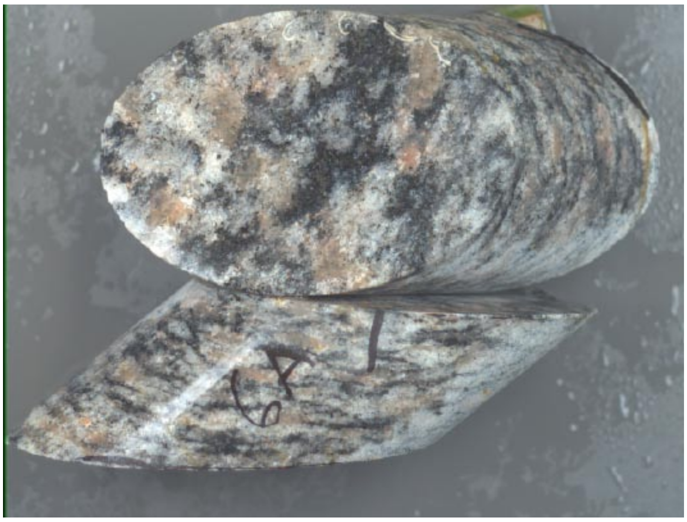

Figure A2-1. Specimen KFM04A-6.

KFM04A-7 (530.97–531.07)

The dry density for specimen KFM04A-7A was measured to be 2,652 kg/m^3 and the porosity to 0.31% and the dry density for specimen KFM04A-7B was measured to be 2,652 kg/m^3 and the porosity to 0.28%.

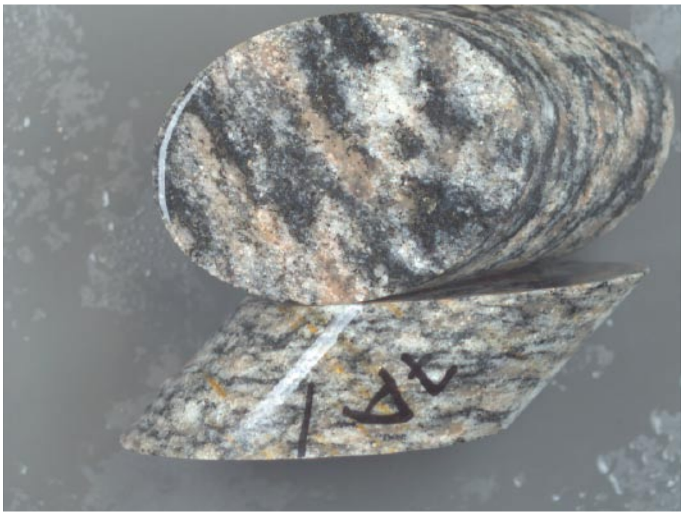

Figure A2-2. *Specimen KFM04A-7.*

KFM04A-8 (531.07–531.15)

The dry density for specimen KFM04A-8A was measured to be 2,651 kg/m^3 and the porosity to 0.26% and the dry density for specimen KFM04A-8B was measured to be 2,653 kg/m^3 and the porosity to 0.27%.

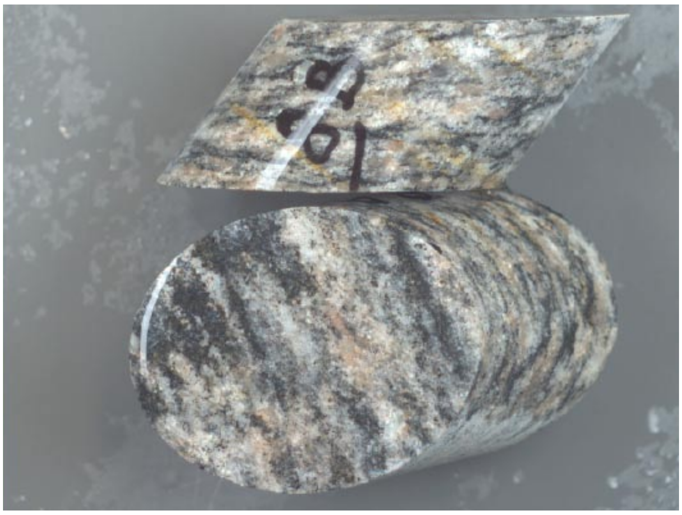

Figure A2-3. *Specimen KFM04A-8.*

KFM04A-9 (531.15–531.23)

The dry density for specimen KFM04A-9A was measured to be 2,649 kg/m^3 and the porosity to 0.27% and the dry density for specimen KFM04A-9B was measured to be 2,654 kg/m^3 and the porosity to 0.26%.

Figure A2-4*.* *Specimen KFM04A-9.*

KFM04A-10 (531.23–531.31)

The dry density for specimen KFM04A-0A was measured to be 2,655 kg/m^3 and the porosity to 0.29% and the dry density for specimen KFM04A-0B was measured to be 2,652 kg/m^3 and the porosity to 0.27%.

***Figure A2-5**. Specimen KFM04A-10.*

Uncertainty of method as expanded uncertainty with covering factor 2 (95% confidence interval):

Density	± 4 kg/m^3
Porosity	± 0.09%
Water absorption	± 0.05%

The tests were performed in accordance with the method descriptions:

- ISRM 1979, Volume 16, Number 2.
- EN 13755, Natural stone test methods – Determination of water absorption at atmospheric pressure.

One exception from the method was the statement of significant numbers. The precision in the method for density gives only three significant digits. The fourth digit given here is thus not significant. The precision in the method for porosity gives only one significant digit the second digit given here is thus not significant. It is important that this is kept in mind when the results are used for further calculation.

A2.3 P502681 KFM04A: Resolved and expanded data table

Test sample	Secup-seclow	Mass [g]	Heat capacitivity [J/kg·K]	Uncertainty [J/kg·K]]	Density [kg/m3]	Heat capacitivity [kJ/m3·K]	Porosity %
6 B	530.89–530.97	204.71	734	±26 (3.6%)	2,653	1.945	0.27
7 A	530.97–531.07	197.29	750	±30 (4.0%)	2,652	1.989	0.31
8 A	531.07–531.15	202.81	739	±29 (3.9%)	2,651	1.959	0.26
9 A	531.15–531.23	223.11	759	±30 (4.0%)	2,649	2.011	0.27
10 A	531.23–531.31	202.64	738	±25 (3.4%)	2,655	1.959	0.29