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Comparison of Thermal Properties Measured by Different Methods

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This report concerns a study which was conducted for SKB. The conclusions and viewpoints presented in the report are those of the authors and do not necessarily coincide with those of the client.

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Foreword

This report presents a comparison between different measurement methods for determining thermal properties of rock samples in the laboratory. The measured rock samples come from the Aspö HRL. The project was conducted by Geo Innova AB, on a commission by the Swedish Nuclear Fuel and Waste Management Co.

For the comparison, results were used from previous investigations where TPS measurements had been performed by two different Swedish laboratories; CIT Thermoflow AB and Hot Disk AB. In this investigation, measurements of the rock samples were performed using the divided bar method and the calorimetric method of the Geological Survey of Finland.

Summary

A strategy for a thermal site descriptive model of bedrock is under development at SKB. In the model different kinds of uncertainties exist. Some of these uncertainties are related to the potential errors in the methods used for determining thermal properties of rock.

In two earlier investigations /Sundberg and Gabrielsson, 1999; Sundberg, 2002/ thermal properties of rock samples were analysed according to the TPS method (transient plane source). Thermal conductivity and thermal diffusivity were determined using the TPS method. For a comparison, the same samples have been measured at the Geological Survey of Finland (GSF), using different laboratory methods. In this later investigation, the thermal conductivity was determined using the divided-bar method and the specific heat capacity using a calorimetric method.

The mean differences between the results of different methods are relatively low but the results of individual samples show large variations.

The thermal conductivity measured by the divided bar method gives for most samples slightly higher values, in average about 3%, than the TPS method. The specific heat capacity measured by the calorimetric method gives lower values, in average about 2%, than the TPS method. Consequently, the thermal diffusivity calculated from thermal conductivity and specific heat capacity gives higher values, in average about 6%, than the TPS method.

Reasons for the differences are estimated mainly to be dependent on differences between the samples, errors in the temperature dependence of specific heat and in the transformation from volumetric to specific heat. The TPS measurements are performed using two pieces (subsamples) of rock. Only one of these two subsamples was measured using the divided bar method and the calorimetric method. Further, sample preparation involved changes in the size of some of the samples. The mean differences between the results of different methods are within the margins of error reported by the measuring laboratories. However, systematic errors in one or both methods cannot be excluded.

For future investigations a set of thermal conductivity standard materials should be selected for testing using the different methods of the laboratories. The material should have thermal properties in the range of typical rocks, be fine-grained and suitable for making samples of different shapes and volumes adjusted to different measurement techniques.

Because of large obtained individual variations in the results, comparisons of different methods should continue and include measurements of temperature dependence of thermal properties, especially the specific heat. This should cover the relevant temperature range of about 0–90°C. Further comparisons would add to previous studies of temperature dependence of the present rocks /Sundberg, 2002; Sundberg and Gabrielsson, 1999/.

Sammanfattning

En strategi för en platsbeskrivande termisk modell för berg håller på att utvecklas vid SKB. Modellen innehåller flera osäkerheter. En del av dessa osäkerheter är kopplade till potentiella felkällor i de metoder, som används för bestämning av bergs termiska egenskaper.

I tidigare undersökningar /Sundberg och Gabrielsson, 1999; Sundberg, 2002/ analyserades bergprovers termiska egenskaper enligt TPS-metoden (transient plane source). Med TPS metoden bestäms provets värmekonduktivitet och värmediffusivitet. För en jämförelse har samma prover mätts vid Finlands geologiska forskningscentral (Geological Survey of Finland, GSF), användandes olika typer av laboriemetoder. I denna senare undersökning bestämdes värmekonduktiviteten med $s k$ divided bar och den specifika värmekapaciteten bestämdes med en kalorimetrisk metod. Slutligen beräknades värmediffusiviteten genom dela värmekonduktiviteten med produkten av densitet och värmekapacitet.

Medelvärde för skillnaden mellan de olika metodernas resultat är relativt liten men resultaten för individuella prover visar på stor spridning.

Värmeledningsförmågan uppmätt med divided bar är för de flesta prover något högre, i medeltal ca 3 %, än motsvarande för TPS-metoden. Värmekapaciteten uppmätt med den kalorimetriska metoden är i de flesta lägre, i medeltal ca 2 %, än motsvarande för TPS-metoden. Följdaktligen, blir värmediffusiviteten beräknad från värmekonduktivitet och specifik värmekapacitet högre, i medeltal ca 6 %, än motsvarande för TPS-metoden.

Orsaker till skillnaderna bedöms främst bero på skillnader mellan de aktuella proverna, fel i temperaturberoendet för omräkningen av värmekapaciteten och i omvandlingen från volumetrisk värmekapacitet till specifik. TPS mätning utförs på ett bergprov delat i två delar (delprov). Endast ett av dessa delprov användes vid mätning med divided bar och den kalorimetriska metoden. Dessutom innebar provberedning en förändring av provstorleken för en del av proverna. Medelskillnaderna mellan de olika metodernas resultat är inom de felmarginaler som angetts av de olika mätlaboratorierna. Emellertid går det inte att utesluta systematiska fel i en av metoderna eller i båda metoderna.

För fortsatta undersökningar rekommenderas att välja ut lämpliga standardmaterial med känd värmekonduktivitet, som kan testas med de olika laboratoriernas mätmetoder. Materialens termiska egenskaper bör ligga i intervallet för typiska bergarter och de bör vara finkorniga samt lämpliga för tillverkning av prover med form och volym anpassad till de olika mätteknikerna.

På grund av stora enskilda variationer i resultaten bör jämförelser av olika metoder fortsätta och inkludera mätningar av temperaturberoendet hos de termiska egenskaperna, särskilt värmekapaciteten. Dessa mätningar bör omfatta temperaturintervallet ca 0–90°C. Fortsatta jämförelser kompletterar tidigare studier av temperaturberoende för de aktuella bergarterna /Sundberg, 2002; Sundberg och Gabrielsson, 1999/.

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1 Introduction

1.1 Background

A strategy for a thermal site descriptive model of bedrock is under development at SKB. In the model different kinds of uncertainties exist. Some of these uncertainties are related to possible errors in the applied measurement methods for determining thermal properties of rock. However, comparisons between different methods have rarely been made and systematically errors may occur in the methods.

1.2 Objectives

The work has been conducted in order to reach the following objectives;

- Compare the result of methods for determination of thermal properties of rock in order to quantify the uncertainty.
- If discrepancies occur, analyse the methods with the focus on possible systematic errors.

The TPS method (transient plane source) is suggested for the determination of thermal conductivity and heat capacity on drill core samples during the site investigations. In earlier investigations /Sundberg and Gabrielsson, 1999; Sundberg, 2002/, the thermal properties were analysed according to this method. In the present study these results has been compared with measurements of thermal conductivity, using the divided bar method, and of specific heat, using the calorimetric method. The Geological Survey of Finland (GSF) performed these measurements.

2 Methods and Samples

2.1 Methods to determine thermal properties

Two different methods have been used to determine the thermal properties on rock samples from Äspö HRL:

- TPS (transient plane source) method.
- Divided bar method and calorimetric method.

The TPS method is a transient method that gives values of thermal conductivity and thermal diffusivity. TPS is a selected investigation method for the site investigations planned by SKB /SKB, 2001/.

The divided bar method is a steady-state method that only gives values of the thermal conductivity. The specific heat capacity is complementary determined using a calorimetric method. The methods used at the GSF have been described by /Kukkonen and Lindberg, 1995, 1998/ and /Kukkonen, 2000/.

The measured samples come from two different investigations at Äspö HRL. In the first investigations, the TPS measurements were performed by CIT Thermoflow and in the second investigation by Hot Disk, Table 2-1. In both these investigations, modelling of thermal properties from the mineralogical composition were also made.

Table 2-1. Compared methods and performing laboratories.

Method	Laboratory
TPS	Hot disk ¹ CIT Thermoflow ²
Divided bar	GSF
Calorimetric method	GSF

1) /Sundberg, 2002/

2) /Sundberg and Gabriellsson, 1999/.

2.1.1 TPS method

Description

Measurements of thermal properties in the laboratory were performed using the TPS method /Gustafsson, 1991/. The TPS (transient plane source) method is used for measurements of thermal diffusivity and thermal conductivity of both fluids and solids, from cryogenic temperatures to about 250°C (if the sensor insulation is made of Kapton). The thermal conductivity that can be measured ranges from 0.005 W/mK to 500 W/mK.

The method uses a sensor element with an engraved pattern of a thin double spiral. The spiral is made of Ni metal and has specific electrical resistivity properties. The spiral is embedded between two layers of Kapton, to give it mechanical strength and electrical insulation. Thus measurements can also be performed in electrically conductive materials. The total thickness of the sensor is 0.65 mm and for this specific application the diameter was 20 mm. The probing depth in a transient experiment should be of the same order as the diameter of the hot disk. To achieve this for different materials and sample-sizes, measurement-times and sensor size can be varied.

Measurements are performed by placing the sensor between two samples of the same material. The surfaces of the samples have to be fairly smooth and reasonably flat in order to limit the contact resistance between the sensor and the sample surfaces. During the measurement, the sensor acts both as a heat generator of a heat pulse and as sensor for the temperature response. The temperature vs. time response is measured in 200 data points.

The evaluation uses the fact that the electrical resistance for a thin Ni spiral at any time is a function of its initial resistance, the temperature increase and the temperature coefficient of the resistivity. A model of heat propagation through the sample, assuming a plane source (sensor) and an infinite sample in perfect contact with the sensor surfaces is stored in the software. By fitting measured temperatures to this model, through a number of iterations, the thermal diffusivity and thermal conductivity are determined.

The accuracy of the thermal conductivity measurements is better than $\pm 3\%$ for the interval 0.01–400 W/mK, and the repeatability is better than 0.5% according to the manufacturer. The accuracy of the thermal diffusivity is probably somewhat lower than that of the thermal conductivity.

The volumetric heat capacity is calculated from obtained values of thermal conductivity and thermal diffusivity (equation 7-1) and recalculated to a specific heat capacity using the determined densities of the samples.

Sample preparation and test procedures

The results from the thermal conductivity measurements come from two different investigations and were made at two different laboratories; CIT Thermoflow AB and Hot Disk AB, hereafter named as the first /Sundberg and Gabriellson, 1999/ and the second investigation /Sundberg, 2002/.

In the *second* investigation rock samples from core drillings had a diameter of about 60 mm. The selected samples were cut in two halves, each with a thickness of about 40–45 mm. The two intersection surfaces were then carefully polished.

In order to water saturate the samples, all samples were placed under vacuum in water for 4–12 h, and dried in air for >3 h.

For each sample five measurements were performed with 30 minutes interval at room temperature. Thermal properties are presented as mean values of these measurements. For the thermal conductivity the standard deviations within the groups were better than $\pm 0.5\%$. For two samples, one and two measurements, respectively, were excluded due to deviant results. This may have been caused by a non-equilibrium in the moisture content at the time of the measurement.

In the *first* investigation the same procedure was used with some exceptions. The rock samples from core drillings had a diameter of about 45 mm. The selected samples were cut in two halves, each with a thickness of about 15 mm.

For each sample, at least three measurements were performed at room temperature. The sensor was dismounted after each measurement, in order to regain thermal balance before the next measurement. Measurements were performed after water saturation of the samples in a water bath. In order to achieve water saturation, the samples were placed in boiled water, first semi-immersed for 24 hours followed by 24 hours completely immersed.

2.1.2 Divided bar and calorimetric methods

Divided bar method

The divided bar method is a steady-state method for determination of thermal conductivity of rock samples. The rock disk is placed in series with one disk of thermal conductivity standard and three copper disks with temperature sensors (each side, and between). A constant temperature difference is applied over the disks. The temperature difference over the sample and the reference standard, respectively, is measured carefully. The heat flow is calculated from the measured temperature drop (difference) across the standard and the known thermal conductivity of the standard. With known heat flow, the thermal conductivity is calculated from the temperature difference across the rock sample and thickness of the rock disk.

The method is commonly used in laboratories all over the world. The actual measurements were made using a divided bar apparatus constructed at the Laboratory of Geothermics of the Geological Survey of Finland, Espoo. The method is described in Appendix 1 and by /Kukkonen and Lindberg, 1995, 1998/. The accuracy in the thermal conductivity is $\pm 5\%$ or better.

Calorimetric method

Specific heat capacity was measured using the calorimetric method. The sample is heated to the temperature of boiling water in a vessel, then transferred to a calorimeter, and the initial and final temperatures of the calorimeter are measured. The specific heat is calculated from the masses of the sample and the fluid in the calorimeter, temperatures measured (boiling temperature, initial and final calorimeter temperatures), and the heat capacity of the calorimeter. The calorimetric method used at GSF is described by /Kukkonen and Lindberg, 1998/ and /Kukkonen, 2000/. The relative accuracy of the specific heat capacity measurements is better than $\pm 5\%$.

The sample dimensions were determined using an electronic ruler with an accuracy of 0.01 mm. The sample shapes, however, are usually much coarser than this limit.

Thermal diffusivity is calculated from obtained values by dividing the conductivity value with the product of density and specific heat capacity (equation 7-1). The accuracy in the thermal diffusivity is $\pm 10\%$ or better.

Sample preparation

The large samples from the second investigation (No. 1–11) were reduced in length (from about 60 mm to 10–11 mm) and in diameter (from diameter 63 mm diameter to square shaped samples of about 28x28 mm). The smaller samples from the first investigation (No. 12–17) could be used without changing the size. All samples were slightly polished in order to get a plane surface and reduce the contact resistance. All samples were soaked in water for two days before the measurements.

2.2 Method to determine density

In the earlier investigation using the TPS method, selected rock samples were examined in the laboratory with respect to density and water absorption. Water absorption is a measure of the amount of water that can be accumulated in pores. The measured water absorption can be approximated with the porosity of the samples (pore volume in relation to total volume).

The density was determined according to standards, DIN 52102-RE VA. The water absorption was determined according to standards, DIN 52103-A. The precision was increased compared to the standard, to make it possible to detect differences between materials with very low water absorption properties.

The determinations of density, in the earlier investigations, were performed by the Swedish National Testing and Research Institute (SP).

For the divided bar and calorimetric methods the density was measured again by GSF. The bulk density was measured using the Archimedean principle by weighing the sample in air and water. The applied scale has a resolution of 0.001 g.

The accuracy of the density determinations is $\pm 0.2\%$ or better.

2.3 Samples

Samples from different drill cores at the prototype repository and from borehole KA2599G01 were used for the investigations. The samples were selected to represent both different depths and rock types. In Table 2-2 the samples are identified by the core number, depth, sample size and the investigation (reference) where the samples were originally analysed using the TPS method. In addition, rock type and identification of subsamples for measurements of thermal properties are given.

One of the two subsamples from the TPS measurements was sent to the GSF for measurements using the divided bar and the calorimetric method. For No. 1–11, those subsamples are indicated in italics in the core section column of the table, e.g. *A*.

Table 2-2. Identification of samples used for comparison of different measurement methods.

Sample	Core section (m)	Rock type	Reference	
1	KA 2599 G01	5.90–5.94 (A) + 6.0–6.04 (B)	Äspö diorite	Sundberg, 2002
2	KA 2599 G01	14.58–14.63 (A) + 14.63–14.67 (B)	Äspö diorite	Sundberg, 2002
3	KA 2599 G01	25.32–25.36 (A)+ 25.42–25.46 (B)	Äspö diorite	Sundberg, 2002
4	KA 2599 G01	44.28–44.32 (A) + 44.32–44.36 (B)	Ävrö granite	Sundberg, 2002
5	KA 2599 G01	50.10–50.14 (B) + 50.20–50.24 (A)	Fine-grained granite	Sundberg, 2002
6	KA 2599 G01	61.89–61.93 (B) + 61.99–62.03 (A)	Fine-grained granite	Sundberg, 2002
7	KA 2599 G01	70.60–70.64 (A)+ 70.64–70.68 (B)	Äspö diorite	Sundberg, 2002
8	KA 2599 G01	85.10–85.50 (B) + 85.52– 85.56 (A)	Äspö diorite	Sundberg, 2002
9	KA 2599 G01	101.85–101.89 (B) + 101.95–101.99 (A)	Altered Äspö diorite	Sundberg, 2002
10	KA 2599 G01	120.05–120.09 (A) + 120.15–120.19 (B)	Ävrö granite	Sundberg, 2002
11	KA 2599 G01	126.35–126.39 (B) + 126.45–126.49 (A)	Ävrö granite	Sundberg, 2002
12	KA 3539-2	5.50–5.68	Altered Äspö diorite	Sundberg & Gabrielsson, 1999
13	KA 3545	0.83–1.11	Altered Äspö diorite	Sundberg & Gabrielsson, 1999
14	KA 3551	0.95–1.15	Altered Äspö diorite	Sundberg & Gabrielsson, 1999
15	KA 3563	0.88–1.12	Äspö diorite	Sundberg & Gabrielsson, 1999
16	KA 3581	1.10–1.33	Äspö diorite	Sundberg & Gabrielsson, 1999
17	KA 3587	0.97–1.14	Äspö diorite	Sundberg & Gabrielsson, 1999

3 Thermal Conductivity

The thermal conductivity measured by two different methods is shown in Figure 3-1 and Table 3-1. The later results, from the divided bar method, are in most cases slightly higher, about 3.4% on average, than the results from the TPS method. Differences of individual samples range from -8.9% to +10.6%. The standard deviation for TPS measurements performed on the same sample, was for most samples less than 0.1% (0.05–0.5%).

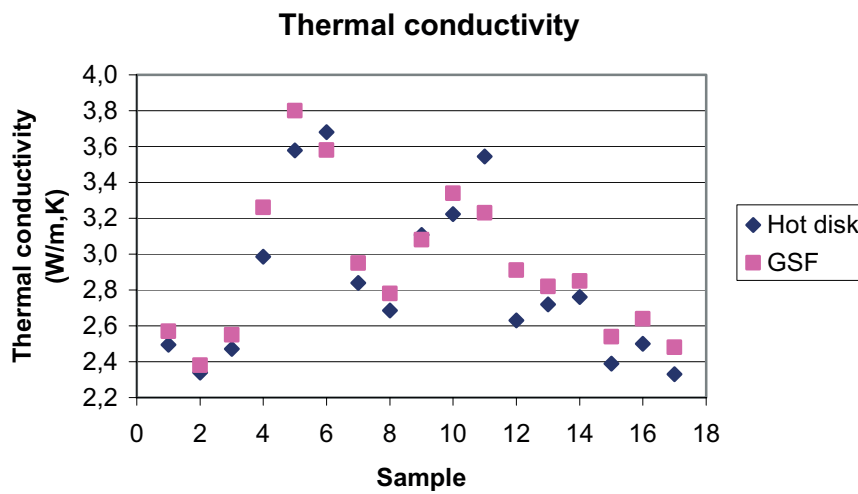


Figure 3-1. Thermal conductivity determined according to the TPS method (Hot Disk) and the divided bar method (GSF).

Table 3-1. Thermal conductivity determined using the TPS method and the divided bar method (GSF).

Thermal conductivity(W/m,K)			
Sample	TPS method	Divided bar method	Difference (%)
1	2.49	2.57	3.0
2	2.34	2.38	1.7
3	2.47	2.55	3.2
4	2.99	3.26	9.2
5	3.58	3.8	6.2
6	3.68	3.58	-2.7
7	2.84	2.95	3.9
8	2.69	2.78	3.5
9	3.11	3.08	-0.9
10	3.22	3.34	3.6
11	3.55	3.23	-8.9
12	2.63	2.91	10.6
13	2.72	2.82	3.7
14	2.76	2.85	3.3
15	2.39	2.54	6.3
16	2.50	2.64	5.6
17	2.33	2.48	6.4
Mean difference			3.4
Standard deviation			4.54

4 Heat Capacity

In the earlier investigations the volumetric heat capacity was calculated from measured thermal conductivity and thermal diffusivity of the TPS method, and the results were given in MJ/(m³ K). At GSF, the specific heat capacity was measured directly in units J/(kg, K). To compare the results, the volumetric heat capacity from the TPS measurements was divided by the bulk density of each sample. For this calculation the density values measured by SP was used (Table 6-1).

Furthermore, the GSF results were corrected to room temperature. The calorimetric measurements at GSF give specific heat capacities at an estimated temperature of about 60°C, which is the mean temperature of the hot bath (app. boiling temperature of water) and the final temperature of the sample in the calorimeter. The TPS results were obtained at room temperature, about 25°C.

For sample No. 3, 4, 8 and 9, a mean temperature dependence in the heat capacity has previously been found at $-0.24\%/K$. This was for a decreasing temperature in the interval 80–25°C /Sundberg, 2002/. For sample No. 13, 15 and 17, plus one additional sample of Äspö diorite, a mean temperature dependence was found at $-0.18\%/K$, in the interval 75–0°C /Sundberg and Gabrielsson, 1999/. A temperature dependence estimated as the mean value of these investigations, $-0.21\%/K$, means that the obtained calorimeter results should be reduced by 7.5% in order to correspond to room temperature. The comparable results are shown in Figure 4-1 and Table 4-1. The two investigations give an indication of the level of the temperature dependence for the present rock. However, the temperature dependence in the specific heat capacity differs between different samples and it may not be linear.

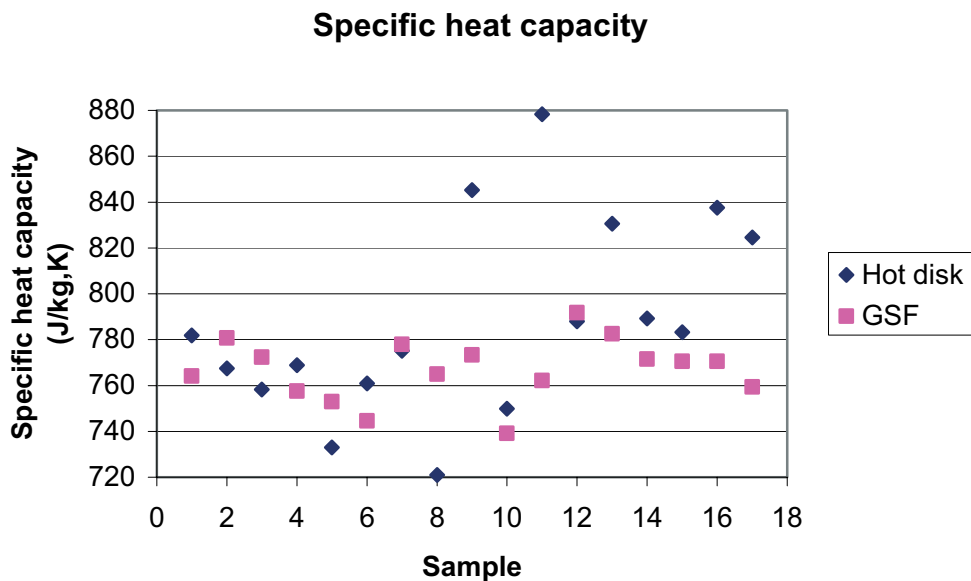


Figure 4-1. Specific heat capacity determined according to the TPS method and measured density (Hot Disk) and the calorimetric method (GSF). The calorimeter results have been corrected to room temperature, 25°C.

Table 4-1. Heat capacity determined by different methods. The calorimeter results have been corrected to room temperature, 25°C. Volumetric heat capacity from TPS measurements has been recalculated to a specific heat capacity.

Heat capacity				
Sample	Volumetric (MJ/m ³ K) TPS	Specific (J/kg K) TPS	Specific (J/kg K) Calorimeter (GSF)	Difference (%)
1	2.16	782	764	-2.3
2	2.13	768	781	1.7
3	2.09	758	772	1.9
4	2.06	769	758	-1.5
5	1.93	733	753	2.7
6	2.02	761	745	-2.2
7	2.10	775	778	0.4
8	1.96	721	765	6.1
9	2.30	845	773	-8.5
10	1.999	750	739	-1.4
11	2.34	878	762	-13.2
12	2.14	788	792	0.5
13	2.27	831	783	-5.8
14	2.15	789	771	-2.3
15	2.15	783	771	-1.6
16	2.32	838	771	-8.0
17	2.29	825	759	-7.9
Mean difference				-2.4
Standard deviation				4.9

In Table 4-2, original calorimeter values uncorrected for the temperature are shown.

The results from calorimeter (direct) measurements are in average 2.4% lower than the results from the TPS measurements. The difference ranges from -13.2% to +6.1%.

The largest differences were obtained for sample 9 and 11. During the TPS measurements of sample 11, two anomalous results were received (two out of five), possibly due to non-equilibrium in the moisture content. The anomalous results were excluded.

Table 4-2. Heat capacity determined by different methods. The calorimeter results refer to a measurement temperature of about 60°C and the TPS results to about 25°C. The volumetric heat capacity from TPS measurements has been recalculated to a specific heat capacity.

Heat capacity				
Sample	Volumetric (MJ/m³ K) TPS	Specific (J/kg K) TPS	Specific (J/kg K) Calorimeter (GSF)	Difference (%)
1	2.16	782	826	5.6
2	2.13	768	844	10.0
3	2.09	758	835	10.1
4	2.06	769	819	6.5
5	1.93	733	814	11.1
6	2.02	761	805	5.8
7	2.10	775	841	8.5
8	1.96	721	827	14.7
9	2.30	845	836	-1.1
10	1.999	750	799	6.5
11	2.34	878	824	-6.2
12	2.14	788	856	8.6
13	2.27	831	846	1.9
14	2.15	789	834	5.7
15	2.15	783	833	6.4
16	2.32	838	833	-0.5
17	2.29	825	821	-0.4
Mean difference				5.5
Standard deviation				5.35

5 Thermal Diffusivity

Thermal diffusivity was measured by Hot Disk according to the TPS method. The standard deviation for TPS measurements performed on the same sample was for most samples lower than 0.9%. GSF determined the thermal diffusivity by dividing the thermal conductivity with the product of density and specific heat capacity. The resulting error in the thermal diffusivity, calculated in this way, is $\pm 10\%$ or better. The specific heat capacity values from calorimeter measurements refer to a temperature of 60°C . These values were corrected with respect to an estimated mean temperature dependence of $-0.21\%/K$ in the specific heat capacity of the present rock. This means that the resulting thermal diffusivity uncorrected for temperature dependence was increased by 8% to correspond to room temperature (25°C). The comparable results are shown in Figure 5-1 and Table 5-1.

In Table 5-2, thermal diffusivity values based on original calorimeter values uncorrected for the temperature are shown.

The results from the GSF are in most cases higher, about 5.7% on average, than the results from the TPS method. The difference for individual samples ranges from -2.9% to $+15.1\%$.

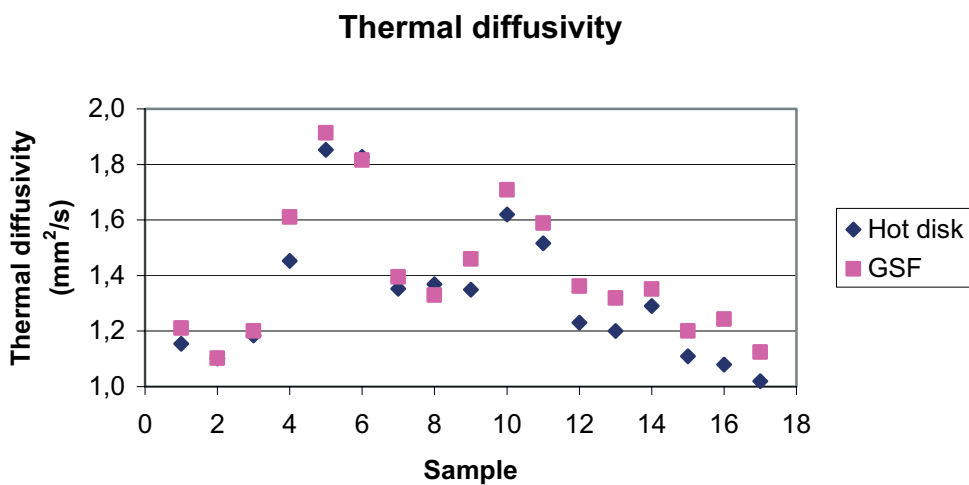


Figure 5-1. Thermal diffusivity determined according to the TPS method (Hot Disk) and calculated from measured data (GSF). The GSF results have been corrected to room temperature, 25°C .

Table 5-1. Thermal diffusivity determined by different methods. Analysing laboratory is specified in the table. The GSF results have been corrected to room temperature, 25°C.

Thermal diffusivity (mm ² /s)			
Sample	Hot Disk	GSF	Difference (%)
1	1.15	1.21	4.9
2	1.10	1.10	0.2
3	1.18	1.20	1.4
4	1.45	1.61	10.9
5	1.85	1.91	3.3
6	1.83	1.82	-0.6
7	1.35	1.40	3.1
8	1.37	1.33	-2.9
9	1.35	1.46	8.2
10	1.62	1.71	5.4
11	1.52	1.59	4.8
12	1.23	1.36	10.7
13	1.20	1.32	9.9
14	1.29	1.35	4.8
15	1.11	1.20	8.1
16	1.08	1.24	15.1
17	1.02	1.12	10.2
Mean difference			5.7
Standard deviation			4.8

Table 5-2. Thermal diffusivity determined by different methods. Analysing laboratory is specified in the table. The GSF results refer to a measurement temperature of about 60°C and the Hot Disk results to about 25°C.

Thermal diffusivity (mm ² /s)			
Sample	Hot Disk	GSF	Difference (%)
1	1.15	1.12	-2.6
2	1.10	1.02	-7.3
3	1.18	1.11	-5.9
4	1.45	1.49	2.8
5	1.85	1.77	-4.3
6	1.83	1.68	-8.2
7	1.35	1.29	-4.4
8	1.37	1.23	-10.2
9	1.35	1.35	0.0
10	1.62	1.58	-2.5
11	1.52	1.47	-3.3
12	1.23	1.26	2.4
13	1.20	1.22	1.7
14	1.29	1.25	-3.1
15	1.11	1.11	0.0
16	1.08	1.15	6.5
17	1.02	1.04	2.0
Mean difference			-2.2
Standard deviation			4.46

6 Density

The results from density measurements are shown in Figure 6-1 and Table 6-1. In the earlier investigations the density was measured at SP. When comparing the results, the differences were less than one percent between the two laboratories, except for the last sample (No. 17) where it was about 4%.

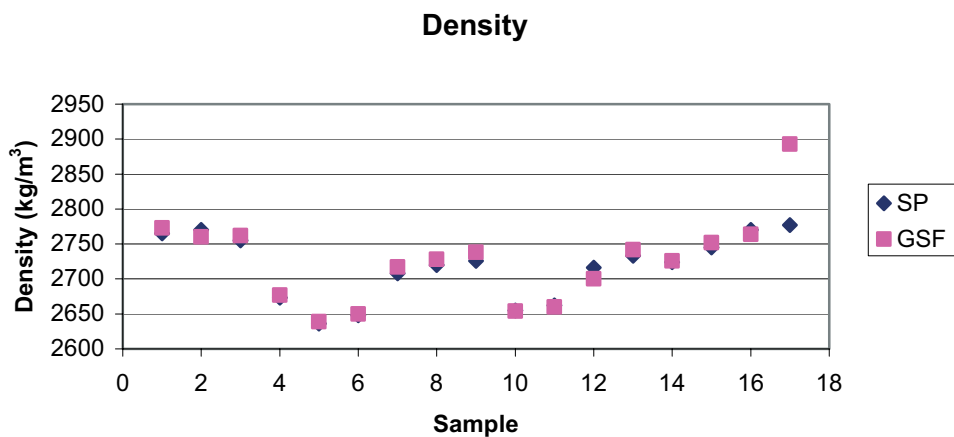


Figure 6-1. Density determined by SP and by GSF.

Table 6-1. Density determined by different methods. Analysing laboratory is specified in the table.

Density(kg/m ³)			
Sample	SP	GSF	Difference (%)
1	2765	2773	0.3
2	2770	2760	-0.4
3	2755	2762	0.3
4	2673	2677	0.1
5	2636	2639	0.1
6	2648	2650	0.1
7	2708	2717	0.3
8	2720	2728	0.3
9	2726	2738	0.4
10	2655	2654	0.0
11	2662	2660	-0.1
12	2716	2700	-0.6
13	2733	2742	0.3
14	2724	2726	0.1
15	2745	2752	0.3
16	2770	2764	-0.2
17	2777	2893	4.2
Mean difference			0.3
Standard deviation			1.07

7 Evaluation

7.1 Comparison between different methods

The thermal conductivity obtained with the divided bar method (GSF) differ -8.9% to $+10.6\%$ from the results of the TPS method. The mean difference is $+3.4\%$ for the divided bar compared with TPS. For most samples the difference is within the expected margin of error reported by the laboratories. For a statistically large number of samples the mean difference in the results should be equal to zero.

The specific heat capacity obtained with the calorimetric method is lower than the results from the TPS method. The mean difference is -2.4% . The difference for individual samples varies between -13.2% to $+6.1\%$.

The calorimetric measurements at GSF give specific heat capacities at approximately 60°C , whereas the TPS results were obtained at room temperature (25°C). A mean temperature dependence in the specific heat of $-0.21\%/K$ was assumed in order to reduce the calorimetric values to room temperature. The comparable thermal diffusivity is consequently higher for the measurements made by GSF than for the TPS method, in average 5.7% . The difference is in the interval -2.9% to $+15.1\%$.

The density measurements show only very small differences between the laboratories (SP and GSF). In general, the difference is smaller than 0.6% . If one extreme value of 4% is excluded the mean difference is smaller than 0.1% .

The results of the thermal properties divided into four different rock types are shown in Table 7-1. The differences between the methods show no obvious trend due to rock type or level of thermal conductivity.

Table 7-1. Mean values of thermal properties for different rock types, determined using different methods. The thermal properties refer to a temperature of about 25°C . Mean values of differences between results of different methods for different rock types.

Rock type	No. of samples	Thermal conductivity W/m,K			Specific heat capacity J/kg,K			Thermal diffusivity mm ² /s		
		TPS	Divided bar	Diff. (%)	Calc. ¹ (TPS)	Calori-metric	Diff. %	TPS	Calc. ² (GSF)	Diff. %
Äspö diorite	8	2.51	2.61	4.2	781	770	-1.2	1.17	1.23	5.0
Altered Äspö diorite	4	2.81	2.92	4.2	813	780	-4.0	1.27	1.37	8.4
Ävrö granite	3	3.25	3.28	1.3	799	753	-5.4	1.53	1.64	7.0
Fine-grained granite	2	3.63	3.69	1.7	747	749	0.3	1.84	1.86	1.4

¹ Measured thermal conductivity divided by the product of measured diffusivity and bulk density.

² Measured thermal conductivity divided by the product of measured specific heat capacity and bulk density.

Only thermal conductivity is measured directly at both laboratories. Properties that were not measured directly were calculated from measured properties. The relation between thermal properties is shown in equation 7-1:

$$\hat{e} = \ddot{e}/(\tilde{n}\cdot c) \text{ or } \hat{e} = \ddot{e}/C \quad 7-1$$

\ddot{e} = thermal conductivity, W/(m·K)

\hat{e} = thermal diffusivity, m²/s

\tilde{n} = density, kg/m³

c = specific heat capacity, J/(kg·K)

C = volumetric heat capacity, J/(m³·K)

The specific heat capacity is measured directly by GSF using the calorimetric method. For the TPS method the volumetric heat capacity is determined from measured thermal conductivity and diffusivity, and the specific heat is calculated using the measured density.

The thermal diffusivity is measured directly with the TPS method. At GSF the thermal diffusivity is calculated from measured thermal conductivity, specific heat capacity and bulk density.

7.2 Possible reasons for differences in the results

The differences in measured thermal conductivity are relatively small between the two laboratories. Thermal conductivity values measured by the divided bar method are about 3% higher than those measured by the TPS method. Specific heat capacity values measured by the calorimetric method are about 2% lower than those measured by the TPS method.

Reasons for obtained differences in the results, summarised in Table 7-1, can be related to different sources of errors:

1. Differences in used rock samples
2. Performance of the tests and applied test procedures.
3. Errors in the methods and limitations of background theories.

The possible reasons for the discrepancy are discussed for each group below.

7.2.1 Differences in used rock samples

The TPS method needs two pieces (subsamples) to perform the measurement, whereas the divided bar method and the calorimetric method only need one piece. Only one of the two subsamples that were used for TPS measurements was used by the GSF. The two subsamples are mostly taken from the core at a distance of a few cm from each other, see Table 2-2. Prior to measurements in the divided bar, the size of the samples was reduced to fit the measurement devices.

It is possible that there are changes in the mineral composition between the subsamples, which can cause differences in the measured thermal properties. However, it is not likely that such variations are systematic in one direction for all 17 samples but it can have an influence on individual samples.

7.2.2 Performance of the tests and applied test procedures

Influence from possible thermal short cuts is possible if the grain size is large. Some core samples contained large grains and may have influenced the divided bar measurements (higher value if the conductivity of the large grain is higher than the true mean value). However, the granitic rocks at Äspö contain mostly potassium feldspar as large grains, and the thermal conductivity of feldspar is usually below the bulk rock values.

No obvious foliation has been observed for the different samples. However, the two methods measure in different directions, TPS in a sphere and divided bar across the sample, and this may have some influence on the results if there is a slight foliation.

The temperature dependence of the thermal conductivity is generally small for the rock types at Äspö HRL. In the present study this effect is negligible since the difference in measurement temperatures at the two laboratories was very small (a few degrees at the most). The divided bar method used a constant temperature difference of about 10°C between the upper and lower ends of the column. The sample temperature was measured at 22–23°C. A significant temperature dependence in the thermal conductivity has only been found for altered Äspö diorite. For this rock type the temperature dependence was found at $-0.07\%/K$, for an increasing temperature in the interval 25–80°C /Sundberg, 2002/.

The temperature dependence of the heat capacity is significant. In the present study, the specific heat capacity of the calorimetric method at GSF refers to a temperature of about 60°C, whereas the heat capacity calculated from TPS (and SP) results refers to room temperature, about 25°C. The GSF results were therefore corrected to room temperature to enable a comparison. For the calculation a mean value of the temperature dependence of two earlier investigations, $-0.21\%/K$, was used, as described in chapter 4. Thus, the original calorimeter results were reduced by 7.5%. The two earlier investigations give an indication of the level of the temperature dependence for the present rock. However, the temperature dependence in the specific heat capacity differs between different samples and it may not be linear.

The porosity is small but has in some cases significant influence on the results, depending on if the porosity is saturated with water or air. The differences in the thermal conductivity are about 0–5%, between saturated and unsaturated conditions, at the actual porosities of about 0.3% /Sundberg, 2002/. This shows the importance of the water saturation procedure of the samples before thermal measurements. In this study, all samples were water saturated before the measurements took place. The TPS results come from two different investigations with slightly different methodology regarding the water saturation procedure. However, no significant differences in the thermal conductivity can be observed because of the different methodologies. Further, the porosity of the samples is low and water saturation differences can not explain the differences between the results of different methods. The experience from the TPS measurements is that for some cases it could be possible that a non-equilibrium in the moisture content has an influence on the measurement.

A contact resistance may occur if the sample surfaces are not completely flat. For the divided bar method a contact resistance normally results in a lower thermal conductivity. In the divided bar apparatus at the GSF, the sample is uniaxially compressed with a mechanism corresponding to a static load of about 180 N. The stress is applied for homogenising the contact resistances. TPS is a transient method and less dependent on effects of contact resistance. It is possible to choose the information used for the evaluation, whereas the initial part can be excluded and the data representing the bulk of the sample can be chosen. However, the thermal diffusivity is somewhat more sensitive to such a procedure than the thermal conductivity.

If the heat flow for the divided bar is not according to the theory an error may also occur. Insufficient insulation of the bar may cause a non 1-dimensional heat flow, which can result in an overestimation of the thermal conductivity. Such an insulation is not normally applied for the measurements at the GSF. Comparisons between measurements with and without an insulation surrounding the divided bar have yielded similar results.

The TPS is an absolute method, meaning that the results are calculated from the measured temperature versus time data. The divided bar method is a steady-state method and the thermal conductivity of the sample is determined in relation to that of a standard material. The result is thus dependent on the accuracy of the standard values.

7.2.3 Errors in the methods and limitations of background theories

Errors in the instrumentation, sensors, temperature measurement devices and standards are also possible and can give systematic errors.

It is not likely that there are errors in the theory, calculations or instrumentation for the TPS method or the divided bar method. The theories for both methods have been published numerous times in scientific papers and are internationally accepted.

The TPS method has been used before in the Äspö prototype repository by /Sundberg and Gabrielsson, 1999/ and compared with a field method (2-probe method) used at the prototype repository. The results differed by 10% (field values higher). No obvious reason for the difference was found but it is possible that unobserved small water movements due to large gradients (large depth) influenced the field measurements.

The measured methods of the GSF have been applied in site investigations in Finland for measurements of thermal properties /Kukkonen and Lindberg, 1995, 1998; Kukkonen, 2000/ as well as for studies of geothermal heat flow /Kukkonen and Peltoniemi, 1998/. A summary of thermal conductivity measurements of about 2,500 samples for geothermal heat flow studies in Finland, along with petrophysical properties, has been reported by /Kukkonen and Peltoniemi, 1998/.

8 Conclusions and Recommendations

Comparable studies of measurements with different methods are rare. In this study, 17 samples were measured using the TPS method and the results were compared with results from the divided bar method and a calorimetric method. The mean differences between the results of the different methods are relatively low but the results of individual samples show large variations.

Conclusions:

- Thermal conductivity measured by the divided bar method gives for most samples slightly higher values, in average 3.4%, than the TPS method. The difference between individual samples ranges between -8.9% to +10.6%.
- The specific heat capacity measured by the calorimetric method gives for most samples slightly lower values, in average 2.4%, than the calculated specific heat based on the results of the TPS method. The difference between individual samples ranges between -13.2% to +6.1%.
- Consequently, the thermal diffusivity evaluated from the divided bar and the calorimetric methods, is in average 5.7% higher than the thermal diffusivity measured by the TPS method. The difference between individual samples ranges between -2.9% to +15.1%.
- Reasons for the differences are estimated mainly to be dependent on differences between the samples, errors in the temperature dependence of specific heat and in the transformation from volumetric to specific heat. The TPS measurements are performed using two pieces (subsamples) of rock. Only one of these two subsamples were sent to the GSF and measured using the divided bar method and the calorimetric method. Further, sample preparation involved changes in the size of some of the samples. The mean differences between the methods are for most samples within the margins of error reported by the measuring laboratories. However, systematic errors in one or both methods cannot be excluded.

Recommendations:

A set of thermal conductivity standard materials should be selected for testing using the different methods of the laboratories. The material should have thermal properties in the range of typical rocks, be fine-grained and suitable for making samples of different shapes and volumes adjusted to different measurement techniques.

Because of large obtained individual variations in the results, comparisons of different methods should continue and include measurements of temperature dependence of thermal properties, especially specific heat. This should cover the relevant temperature range of about 0–100°C. Further comparisons would add to previous studies of temperature dependence /Sundberg, 2002; Sundberg and Gabrielsson, 1999/.

9 References

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Measurements of thermal properties of rocks from Sweden

Report of laboratory measurements

October 6, 2002

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1 Introduction

The present results of measuring thermal properties of rocks are based on the order of Jan Sundberg, Geoinnova AB, Sweden, of June 11, 2002, to the Laboratory of Geothermics of the Geological Survey of Finland, Espoo. The order consists of measuring thermal properties of 17 drill core samples. The determined parameters are thermal conductivity, specific heat capacity and density. Thermal diffusivity was calculated from the other measured values.

The delivered samples included altogether 17 samples. Six samples have a core diameter of about 45 mm (samples 3539-2 ... 35879), and the rest have a diameter of 61–64 mm (samples 1A ... 11B). Lithologically the samples represent crystalline plutonic rocks with acid to intermediate composition, i.e. granites, granodiorites and diorites (the present author's estimation).

2 Sample preparation

Prior to measurements the sample shapes were prepared to be better compatible with the instruments used at our laboratory. The smaller diameter samples were measured in the condition as they were received, excluding removal of protruding edges left after the original diamond saw cutting and a slight polishing (polishing powder no. 180) for homogenizing the contact surfaces for conductivity measurements. The big diameter samples were originally too long (about 60 mm) for our instruments, and thinner disks (thickness about 10–11 mm) were prepared of them. Even then, thermal conductivity measurements were found to be difficult due to the big core diameter, and smaller size disks with square shape (about 28 mm x 28 mm, thickness 10–11 mm) were prepared of the big diameter cores. Samples were measured after soaking them in water for two days.

3 Measurements

Thermal conductivity, specific heat capacity and density were measured at the Laboratory of Geothermics of the Geological Survey of Finland, Espoo.

Thermal conductivity was measured using a divided bar apparatus constructed at the GSF. The method is a steady-state method in which the sample is placed in the middle of a vertical column consisting of alternating disks of conductivity standards (quartz) and copper (temperature sensors in them). Upper and lower ends of the column are kept in constant temperatures (temperature difference of about 10 degrees), which creates a forced flow of heat through the column. Temperature differences are measured across the standards and the sample, and the conductivity is calculated from the dimensions of the samples, known thermal conductivities of the standards and the temperature differences. The method is described in more detail in references /1–3/. The inaccuracy of the thermal conductivity measurements is smaller than $\pm 5\%$.

Specific heat capacity was measured using the calorimetric method. The sample is heated to the temperature of boiling water in a vessel, then transferred to a calorimeter, and the initial and final temperatures of the calorimeter are measured. The specific heat is calculated from the masses of the sample and fluid in the calorimeter, temperatures measured (boiling temperature, initial and final calorimeter temperatures), and the heat capacity of the calorimeter. The method is described in detail in references /2–3/. The relative inaccuracy of the specific heat capacity measurements is smaller than $\pm 5\%$.

Bulk density was measured using the Archimedean principle by weighing the sample in air and water. The method is described in reference /3/. The applied scale has a resolution of 0.001 g. The inaccuracy of the density determinations is better than $\pm 0.2\%$.

The sample dimensions were determined using an electronic ruler with an accuracy of 0.01 mm. The sample shapes, however, are usually much coarser than this limit.

Thermal diffusivity was calculated by dividing the conductivity value by the product of density and specific heat capacity. The inaccuracy of the thermal diffusivity is better than $\pm 10\%$.

4 Results

Results of the measurements are given in Table A-1. The results are can be considered normal for crystalline acid and intermediate rocks. The biggest variations are measured in conductivity (standard deviation about 14% of the average) and diffusivity (17%), and smallest in specific heat capacity and density (about 2%). Variations in conductivity and diffusivity presumably reflect the variations in mineral content, particularly the quartz content.

Table A-1. Results of measurements

Sample	Thermal conductivity Wm ⁻¹ K ⁻¹	Specific heat capacity J kg ⁻¹ K ⁻¹	Density kg m ⁻³	Diffusivity m ² s ⁻¹
3539-2	2.91	856	2700	1.26E-06
3545	2.82	846	2742	1.22E-06
3551	2.85	834	2726	1.25E-06
3563	2.54	833	2752	1.11E-06
3581	2.64	833	2764	1.15E-06
3587	2.48	821	2893	1.04E-06
1A	2.57	826	2773	1.12E-06
2B	2.38	844	2760	1.02E-06
3A	2.55	835	2762	1.11E-06
4B	3.26	819	2677	1.49E-06
5B	3.8	814	2639	1.77E-06
6B	3.58	805	2650	1.68E-06
7B	2.95	841	2717	1.29E-06
8A	2.78	827	2728	1.23E-06
9B	3.08	836	2738	1.35E-06
10A	3.34	799	2654	1.58E-06
11B	3.23	824	2660	1.47E-06
Average	2.93	829	2726	1.30E-06
St. dev.	0.41	15	62	2.23E-07

5 References

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