P-07-19

Forsmark site investigation

Drill holes KFM01A, KFM07A, and KFM08A

Specific heat capacity of rocks using calorimetric measurements

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January 2007

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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 author and do not necessarily coincide with those of the client.

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Abstract

The specific heat capacity on eleven specimens from drill holes KFM01A, KFM07A, and KFM08A, Forsmark, Sweden, was measured by a calorimeter. The samples were taken from the dominant rock type granite (101057) in the mentioned drill holes.

The specific heat capacity was in the range of 0.74–0.83 [J/g,K].

Sammanfattning

Specifika värmekapaciteten hos elva provkroppar från borrhålen KFM01A, KFM07A och KFM08A, Forsmark, bestämdes med en kalorimeter. Proverna hade tagits från den dominerande bergarten granit (101057) i de nämnda borrhålen.

Den specifika värmekapaciteten uppgick till 0,74–0,83 [J/g,K].

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1 Introduction

SKB is planning to build a final repository for nuclear waste in bedrock, which demands knowledge about thermal properties of the rock. Forsmark, Sweden, is one of the areas selected for site investigations. The activity presented in this report is part of the site investigation program at Forsmark /1/.

Several boreholes at Forsmark have previously been investigated with respect to the thermal properties. This report presents investigations of specific heat capacity of rock samples from boreholes KFM01A /2/, KFM07A /3/, and KFM08A /4/. The location of the three telescopic boreholes is shown in Figure 1-1. The specific heat capacity of the samples was measured by a calorimetric method.

Rock samples were selected at Forsmark based on the Boremap core logging with the strategy to investigate the properties of the dominant rock type.

The specimens to be tested were cut from the rock samples in the shape of circular discs. The rock samples from KFM01A, KFM07A, and KFM08A arrived at SP in February 2003, April 2005, and August 2005 respectively. The specific heat capacity was determined according to SP-Method 4221 /5/ on water-saturated specimens. Testing was performed during December 2006.

The controlling documents for the activity are listed in Table 1-1. The Activity Plan is SKB's (The Swedish Nuclear Fuel and waste Management Company) internal controlling document, whereas the Method Description and the Quality Plan (SP-QD 13.1) referred to in the table are SP's (SP Technical Research Institute of Sweden) internal controlling documents.

Activity plan	Number	Version
Kompletterande termiska laboratorieanalyser	AP PF 400-06-087	1.0
Method Description	Number	Title
SP-Method	4221	Determination of specific heat capacity using calorimetric measurements
Quality Plan		
SP-QD 13.1		

Table 1-1. Controlling documents for performance of the activity.





2 Objective and scope

The purpose of this activity is to determine specific heat capacity in order to complete the previously measured thermal properties of rock specimens /2/, /3/, and /4/. The obtained thermal properties will be used as input data for mechanical and thermal analysis in a site descriptive model that will be established for the candidate area selected for site investigation at Forsmark.

3 Equipment

The measurement equipment used for the calorimetric determination of the specific heat capacity is shown in Figure 3-1 and consisted of:

- · Calorimeter, made of Macrolon with low heat capacity and very low heat conductivity.
- Magnetic stirrer, IKA type BigSquid.
- Temperature logger, Keithley 2000 multimeter with scanner Keithley 7700 (temperature resolution 0.01 mK, accuracy 5 mK).
- Temperature controlled bath, Heto Thermostat 13 DT-1 (resolution 0.1°C).
- Three temperature sensors, Pt-100 Pentronic (2 for calorimeter, 1 for temperature controlled bath).
- Thermometer for Air, Pentronic CRL 206, s/n 270210 (resolution 0.01°C).
- Balance Mettler PM 2000 (resolution 0.01 g, accuracy 0.02 g).
- Air conditioning equipment, µAC Carel, Essén Company.
- Laptop computer Toshiba programmed on Visual Basic 6 for the temperature monitoring of three channels per three seconds.
- Pure and de-aerated water, crushed ice for fast preparation of a "steady state" condition.
- Various accessories (stand, holder, clamps, hoses, dewar, syringe, timer, etc).

All measurement instruments are traceable via in-house calibration to national and international standards. The three temperature sensors connected to respective logger channel were calibrated immediately before the measurements. The balance was several times checked using relevant weight pieces.



Figure 3-1. Calorimeter with two temperature sensors on magnetic stirrer.

4 Execution

Specific heat capacity was determined according to SP-Method 4221 at SP Measurement Technology. Peter Lau at SP Measurement Technology conducted the specific heat capacity measurements, whereas Ingrid Wetterlund at SP Fire Technology prepared the report.

4.1 Description of the samples

Eleven pairs of cores (designated A and B) were sampled from boreholes KFM01A, KFM07A, and KFM08A, Forsmark, Sweden. The drill cores in question had previously been investigated with respect to e.g. rock type identification (so called Boremap mapping). The rock samples had also been used for earlier performed laboratory tests at SP. The twenty-two specimens with a thickness of 25 mm each had been cut from the rock samples at SP. The diameter of the specimens was about 50 mm. The identification marks, rock type and sampling levels of the specimens are presented in Table 4-1. Detailed geological description of the entire cores of KFM01A, KFM07A and KFM08A is given in SKB's database SICADA (Boremap data).

4.2 Test procedure

The present activity was to determine specific heat capacity by calorimetry at SP Measurement Technology on specimens used in earlier activities at SP Building Technology and Mechanics and at SP Fire Technology. The earlier activities are reported in SKB P-04-159 /2/, SKB P-05-214 /3/, and SKB P-05-219 /4/.

Table 4-1. Rock type and identification marks (Rock-type classification according to Boremap). Sampling level refers to borehole length.

Identification	Sample idenfication in earlier reports	Rock type/ occurence	Sampling level (Sec low)
KFM01A-90D-1	KFM01A-90V-10	(101057)	389.68
KFM01A-90D-2	KFM01A-90V-12	(101057)	493.85
KFM01A-90D-3	KFM01A-90V-14	(101057)	494.09
KFM07A-90D-4	KFM07A-90V-02	(101057)	319.86
KFM07A-90D-5	KFM07A-90V-04	(101057)	358.85
KFM07A-90D-6	KFM07A-90V-05	(101057)	379.92
KFM08A-90D-7	KFM08A-90V-01	(101057)	200.89
KFM08A-90D-8	KFM08A-90V-04	(101057)	350.19
KFM08A-90D-9	KFM08A-90V-09	(101057)	503.61
KFM08A-90D-10	KFM08A-90V-11	(101057)	650.05
KFM08A-90D-11	KFM08A-90V-05	(101057)	424.29

The following logistic sequence was applied for the present activities:

- 1. Specimens were picked up from the storage by SP Building Technology and Mechanics.
- 2. Specimens were water saturated by SP Building Technology and Mechanics.
- 3. Specimens were sent from SP Building Technology and Mechanics to SP Measurement Technology.
- 4. Specific heat was determined by SP Measurement Technology.
- 5. Specimens were sent from SP Measurement Technology to SP Building Technology and Mechanics.

The rock samples were water saturated and stored under this condition for 7 days. This yielded complete water saturation, whereupon the specific heat capacity was determined.

Determination of the specific heat capacity was performed during December 2006.

The calorimetric technique involves heating the samples after mass determination to a well defined temperature. The samples are placed in a temperature controlled water bath long enough to stabilize.

The calorimeter is filled with prepared water (pure and de-aerated of 17°C) to a predefined level and stirred to produce nearly steady state conditions. Thereafter it is placed on the balance and excessive water is extracted with a syringe to reach a nominal mass, chosen with respect to the sample volume.

The so prepared calorimeter is stirred and the temperature logging program is started. After 90 to 150 seconds the sample is quickly moved (3 to 5 seconds) from the bath into the calorimeter. The temperature rise of water can be followed graphically during the equalization process, which typically takes 150 seconds and the experiment is terminated after another 300 to 600 seconds.

The calorimeter, water and sample are weighed again to determine the amount of water that unavoidably did follow with the sample into the calorimeter. This amount is typically 0.28 to 0.36% of the water contained in the calorimeter. If accidentally a water splash happens during the sample insertion, those droplets are absorbed with a small piece of prepared tissue that is weighed dry and wet. The corresponding mass is subtracted from the initial water mass. In extreme cases it has amounted to 0.03% of the total water mass.

All mass values for the determination of the specific heat were manually documented in a prepared form that was a printout of the corresponding Excel calculation sheet.

With the termination of the logging program each experiment was saved as raw data in an Excel file on the SP network. The main information was the bath temperature and two calorimeter temperatures as function of time. Finally, the specific heat capacity can be calculated by using the measured parameters.

4.3 Nonconformities

There was one deviation to plan: The reserve specimen KFM08A-90D-11 was tested together with the other specimens.

5 Results

The results of the activity are stored in SKB's database SICADA, where they are traceable by the Activity Plan number.

The specific heat capacity and for each separate sample is reported in 5.1 together with the density and porosity values from the earlier activities /2/, /3/, and /4/. A summary of the specific heat capacity is given in 5.2. The volumetric heat capacity calculated from the measured specific heat capacity is also reported in 5.1 and 5.2.

5.1 Test results of individual specimens

Specimens KFM01A-90D-1A and B



Figure 5-1. Specimens KFM01A-90D-1A and B (KFM01A-90V-10).

Table 5-1.	Porosity, wet and dr	v density of specimens	KFM01A-90D-1A and B,	average values.
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Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM01A-90D-1 Sec low: 389.68	2,660	2,660	0.2

Table 5-2. Heat capacity of specimens KFM01A-90D-1A and B.

Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]	
KFM01A-90D-1 Sec low: 389.68	1.98	0.743	

Specimens KFM01A-90D-2A and B



Figure 5-2. Specimens KFM01A-90D-2A and B (KFM01A-90V-12).

Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM01A-90D-2 Sec low: 493.85	2,660	2,660	0.3

Table 5-3. Porosity, wet and dry density of specimens KFM01A-90D-2A and B, average values.

Table 5-4. Heat capacity of specimens KFM01A-90D-2A and B.

Sample Calculated volumetric heat		Specific heat
capacity [MJ/(m ³ , K)]		capacity [J/(g, K)]
KFM01A-90D-2 Sec low: 493.85	1.98	0.746

Specimens KFM01A-90D-3A and B



Figure 5-3. Specimens KFM01A-90D-3A and B (KFM01A-90V-14).

Table 5-5.	Porosity,	wet and dry	density of	f specimens	KFM01A-90	D-3A and B,	average	values.

Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM01A-90D-3 Sec low: 494.09	2,660	2,660	0.3

Table 5-6. Heat capacity of specimens KFM01A-90D-3A and B.

Sample	Calculated volumetric heat capacity [MJ/(m ³ , K)]	Specific heat capacity [J/(g, K)]
KFM01A-90D-3 Sec low: 494.09	2.02	0.761

Specimens KFM07A-90D-4A and B



Figure 5-4. Specimens KFM07A-90D-4A and B (KFM07A-90V-02).

Table 5-7. Porosi	ty, wet and dry densi	ity of specimens KFM07	A-90D-4A and B, average values
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Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM07A-90D-4 Sec low: 319.86	2,650	2,650	0.4

Table 5-8. Heat capacity of specimens KFM07A-90D-4A and B.

Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM07A-90D-4 Sec low: 319.86	2.20	0.830

Specimens KFM07A-90D-5A and B



Figure 5-5. Specimens KFM07A-90D-5A and B (KFM07A-90V-04).

Table 5-9.	Porosity.	wet and dr	v densitv	of specimens	KFM07A-90D-5A	and B.	average values.
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Sample	Density, wet [kg/m³]	Density, dry [kg/m ³]	Porosity [%]
KFM07A-90D-5 Sec low: 358.85	2,660	2,660	0.3

Table 5-10. Heat capacity of specimens KFM07A-90D-5A and B.

Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM07A-90D-5 Sec low: 358.85	2.09	0.787

Specimens KFM07A-90D-6A and B



Figure 5-6. Specimens KFM07A-90D-6A and B (KFM07A-90V-05).

Table 5-11. Porosity, wet and dry density of specimens KFM07A-90D-6A and B, average values.

Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM07A-90D-6 Sec low: 379.92	2,650	2,650	0.3

Table 5-12. Heat capacity of specimens KFM07A-90D-6A and B.

Sample	Calculated volumetric heat capacity [MJ/(m ³ , K)]	Specific heat capacity [J/(g, K)]
KFM07A-90D-6 Sec low: 379.92	2.00	0.755

Specimens KFM08A-90D-7A and B



Figure 5-7. Specimens KFM08A-90D-7A and B (KFM08A-90V-01).

Table 5-13. Porosity, wet and dry density of specimens KFM08A-90D-7A and B, average values.

Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM08A-90D-7 Sec low: 200.89	2,650	2,650	0.5

Table 5-14.	Heat capacit	y of specimens	KFM08A-90D-7A	and B.

Sample	Calculated volumetric heat capacity [MJ/(m ³ , K)]	Specific heat capacity [J/(g, K)]
KFM08A-90D-7 Sec low: 200.89	2.02	0.763

Specimens KFM08A-90D-8A and B



Figure 5-8. Specimens KFM08A-90D-8A and B (KFM08A-90V-04).

Table o Tel Telebily, net and ally denoty of opconnent interviewe of and b, average values	Table 5-15.	Porosity, wet and	dry density of	of specimens	KFM08A-90D-8A a	nd B, average values
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Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM08A-90D-8 Sec low: 350.19	2,660	2,660	0.3

Table 5-16. Heat capacity of specimens KFM08A-90D-8A and B.

Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM08A-90D-8 Sec low: 350.19	2.05	0.772

Specimens KFM08A-90D-9A and B



Figure 5-9. Specimens KFM08A-90D-9A and B (KFM08A-90V-09).

Table 5-17	Porosity,	wet and dry	density	of specimens	KFM08A-90D	-9A and B	average	values

Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM08A-90D-9 Sec low: 503.61	2,660	2,650	0.3

Table 5-18.	Heat capa	acity of spe	cimens KF	M08A-90D-9A	and B.
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Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM08A-90D-9 Sec low: 503.61	2.06	0.775

Specimens KFM08A-90D-10A and B



Figure 5-10. Specimens KFM08A-90D-10A and B (KFM08A-90V-11).

	Table 5-19.	Porosity, wet and dr	y density o	of specimens	KFM08A-90D-10A and B	, average values.
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Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM08A-90D-10 Sec low: 650.05	2,660	2,650	0.3

Table 5-20. Heat capacity of specimens KFM08A-90D-10A and B.

Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM08A-90D-10 Sec low: 650.05	2.08	0.783

Specimens KFM08A-90D-11A and B



Figure 5-11. Specimens KFM08A-90D-11A and B (KFM08A-90V-05).

Table 5-21, P	orosity, wet and dr	v density of s	specimens KFN	108A-90D-11A and	B. average values
	orosity, wet and a	y density of a	pecimens in w		D, average values

Sample	Density, wet [kg/m³]	Density, dry [kg/m³]	Porosity [%]
KFM08A-90D-11 Sec low: 424.29	2,650	2,650	0.3

Table 5-22.	Heat capacit	y of specimens	KFM08A-90D-11A and	B.
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Sample	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM08A-90D-11 Sec low: 424.29	2.12	0.801

5.2 Results for the entire test series

Table 5-23 displays the calculated volumetric heat capacity as well as the specific heat capacity of the samples measured by the calorimetric method. The specific heat capacity ranged between 0.743 and 0.830 J/(g, K).

Present and initial sample identification	Calculated volumetric heat capacity [MJ/(m³, K)]	Specific heat capacity [J/(g, K)]
KFM01A		
KFM01A-90D-1 (KFM01A-90V-10)	1.98	0.743
KFM01A-90D-2 (KFM01A-90V-12)	1.98	0.746
KFM01A-90D-3 (KFM01A-90V-14)	2.02	0.761
KFM07A		
KFM07A-90D-4 (KFM07A-90V-02)	2.20	0.830
KFM07A-90D-5 (KFM07A-90V-04)	2.09	0.787
KFM07A-90D-6 (KFM07A-90V-05)	2.00	0.755
KFM08A		
KFM08A-90D-7 (KFM08A-90V-01)	2.02	0.763
KFM08A-90D-8 (KFM08A-90V-04)	2.05	0.772
KFM08A-90D-9 (KFM08A-90V-09)	2.06	0.775
KFM08A-90D-10 (KFM08A-90V-11)	2.08	0.783

Table 5	-23. §	Specific	heat ca	pacity	measured	bv	calorimeter.
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6 References

- /1/ SKB, 2001. Site investigations. Investigation methods and general execution programme. SKB TR-01-29, Svensk Kärnbränslehantering AB.
- /2/ Adl-Zarrabi B, 2004. Forsmark site investigation. Drill hole KFM01A. Thermal properties: heat conductivity and heat capacity determined using the TPS method and Mineralogical composition by modal analysis. SKB P-04-159, Svensk Kärnbränslehantering AB.
- /3/ Adl-Zarrabi B, 2005. Forsmark site investigation. Borehole KFM07A. Thermal conductivity and thermal diffusivity determined using the TPS method. SKB P-05-214, Svensk Kärnbränslehantering AB.
- /4/ Adl-Zarrabi B, 2005. Forsmark site investigation. Borehole KFM08A. Thermal conductivity and thermal diffusivity determined using the TPS method. SKB P-05-219, Svensk Kärnbränslehantering AB.
- /5/ **SP-Method 4221, 2006.** Determination of specific heat capacity using calorimetric measurements, SP Sveriges Provnings- och Forskningsinstitut.