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Comparison of three test methods for determination of water absorption and wet density of intact rock

Forsmark, drill hole KFM01A Simpevarp, drill hole KSH01A

Matz Sandström SP Swedish National Testing and Research Institute

June 2004

Svensk Kärnbränslehantering AB

Swedish Nuclear Fuel and Waste Management Co Box 5864 SE-102 40 Stockholm Sweden Tel 08-459 84 00 +46 8 459 84 00 Fax 08-661 57 19 +46 8 661 57 19



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Keywords: Water absorption, Density, Comparison testing, Rock mechanics, Petrophysics, Porosity.

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

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Abstract

Water absorption and wet density on twenty specimens of drill hole KFM01A, Forsmark, and twenty specimens of drill hole KSH01A, Simpevarp, were measured. The rock type in KFM01A is a Quartz monzodiorite or Fine-grained dioritoid. The rock type in KSH01A is an isotropic medium-grained red granite.

Measurements were in principle performed according to SKB's method description SKB MD 160.002 version 1.0 (SKB internal controlling document), with the following nonconformities. The water saturation is performed according to three different methods, ISRM 1979, SS-EN 13755 and EN 1936.

Generally, the difference between the methods is on the same level as the measurement uncertainty. The ISRM method seems however to give higher value of the porosity.

Contents

1	Introd	uction and objective	7
2	Equip	ment	9
3 3.1 3.2	Gener Descri Test pr 3.2.1	al information ption of the samples rocedure Nonconformities	11 11 12 13
4 4.1 4.2	Result Test re Compa 4.2.1 4.2.2	s sults arison of test methods Wet density Porosity	15 15 21 21 22
5	Concl	usions	25
6	Refere	ences	27
Арр Арр	endix 1 endix 2	Calculation of uncertainty in determining wet density by hydrostatic method Calculation of uncertainty in determining porosity by using water absorption and hydrostatic method	29 31
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1 Introduction and objective

The objective of this investigation was to estimate the difference between three methods for determination of wet density and water absorption of intact rock.

The Swedish Nuclear Fuel & Waste Management Co (SKB) delivered the rock samples to Swedish National Testing and Research Institute (SP). The rock cores from KFM01A arrived to SP Feburary 2003 and the cores from KSH01A arrived to SP May 2003. The testing was performed during July to September 2003.

Density was determined in accordance with SKB MD 160.002 version 1.0 (SKB internal controlling document) at department of Building Technology at SP with the following deviations. The water saturation was performed according to three different methods.

The density of natural stone is normally tested according to SS-EN 13755, water immersion in atmospheric pressure. According to ISRM 1979 the water immersion should be performed in a vacuum of less than 800 Pa. This is hard to achieve in normal laboratory climate, 20°C. According to earlier experience at SP, the difference of the measured density is insignificant for samples with low water absorption.

A third method EN 1936 can also be used to measure the density. According to this method the samples is placed in vacuum (2000 Pa) and then saturated. This method is assumed to give higher water absorption due to more efficient saturation and therefore more accurate when the water absorption is very low.

The difference between the methods must however be established in order to compare results from different measurements. The difference between the methods SS-EN 13755, ISRM 1979 and EN 1036 was estimated according to ISO 3301:1975.

2 Equipment

Following equipment has been used for the analyses:

- Vacuum chamber according to Figure 2-1. The minimum pressure that can be reached is 250 Pa.
- Thermometer (inv no 102080) for measurement of water temperature. Calibrated 2003-01-07. Uncertainty of measurement ± 0.4 °C.
- Scale (inv no 102084) for weight measurement. Calibrated 2003-03-19. Uncertainty of measurement $\pm 0.2g$.
- Heating chamber (inv no 102065) for drying the specimens. Calibrated 2003-02-21. Uncertainty of measurement ± 5°C.
- A covered plastic box filled with water for water saturation of the samples.
- A dessicator for cooling samples in.



Figure 2-1. Photo of equipment for water immersion.

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

- Density $\pm 4 \text{ kg/m}^3$.
- Porosity $\pm 0.09\%$.
- Water absorption $\pm 0.05\%$.

The calculations were performed in Microsoft Excel.

3 General information

3.1 Description of the samples

Twenty cores were sampled from drill hole KFM01A, Forsmark, and KSH01A, Simpevarp, respectively. Each sample was cut into two specimens A and B so that fifty specimens, with a thickness of 25 mm each were achieved from the samples at SP. The diameter of the specimens is app. 50 mm depending on the drill hole, KSH01A or KFM01A. Table 3-1 shows the rock type, from the overview mapping, and identification marks of the specimens. For a more detailed description see SKB database Sicada.

Rock type	Identification	Sampling depth (Sec low)
Granite to granodiorite	KFM01A-90V-5A	227,02
Granite to granodiorite	KFM01A-90V-5B	227,02
Granite to granodiorite	KFM01A-90V-6A	389,04
Granite to granodiorite	KFM01A-90V-6B	389,04
Granite to granodiorite	KFM01A-90V-7A	389,15
Granite to granodiorite	KFM01A-90V-7B	389,15
Granite to granodiorite	KFM01A-90V-8A	389,27
Granite to granodiorite	KFM01A-90V-8B	389,27
Granite to granodiorite	KFM01A-90V-9A	389,38
Granite to granodiorite	KFM01A-90V-9B	389,38
Granite to granodiorite	KFM01A-90V-10A	389,68
Granite to granodiorite	KFM01A-90V-10B	389,68
Granite to granodiorite	KFM01A-90V-21A	494,32
Granite to granodiorite	KFM01A-90V-21B	494,32
Granite to granodiorite	KFM01A-90V-22A	494,43
Granite to granodiorite	KFM01A-90V-22B	494,43
Granite to granodiorite	KFM01A-90V-23A	494,62
Granite to granodiorite	KFM01A-90V-23B	494,62
Granite to granodiorite	KFM01A-90V-24A	494,74
Granite to granodiorite	KFM01A-90V-24B	494,74
Quartz monzodiorite	KSH01A-090V-2A	300,33
Quartz monzodiorite	KSH01A-090V-2B	300,33
Quartz monzodiorite	KSH01A-090V-3A	300,45
Quartz monzodiorite	KSH01A-090V-3B	300,45
Quartz monzodiorite	KSH01A-090V-5A	306,01
Quartz monzodiorite	KSH01A-090V-5B	306,01
Fine-grained dioritoid	KSH01A-090V-6A	306,22
Fine-grained dioritoid	KSH01A-090V-6B	306,22
Fine-grained dioritoid	KSH01A-090V-10A	404,12
Fine-grained dioritoid	KSH01A-090V-10B	404,12
Fine-grained dioritoid	KSH01A-090V-11A	404,24
Fine-grained dioritoid	KSH01A-090V-11B	404,24

Table 3-1. Rock type and identification marks.

Rock type	Identification	Sampling depth (Sec low)	
Fine-grained dioritoid	KSH01A-090V-14A	483,02	
Fine-grained dioritoid	KSH01A-090V-14B	483,02	
Quartz monzodiorite	KSH01A-090V-19A	703,49	
Quartz monzodiorite	KSH01A-090V-19B	703,49	
Quartz monzodiorite	KSH01A-090V-20A	703,61	
Quartz monzodiorite	KSH01A-090V-20B	703,61	
Quartz monzodiorite	KSH01A-090V-23A	709,24	
Quartz monzodiorite	KSH01A-090V-23B	709,24	

Table 3-1 (cont). Rock type and identification marks.

3.2 Test procedure

The measurement procedure followed the prescription in SKB MD 160.002 version 1.0 (SKB internal controlling document).

The tests were performed in the following order:

- 1. The samples were cut and polished.
- 2. Testing according to SS-EN 13755:
 - a) Water immersion in 1 atm. The wet density was determined by weighing in air and in water according to SS-EN 13755 after 72 h and 96 h saturation.
 - b) After drying and weighing the porosity was determined according to ISRM.
- 3. Testing according to ISRM 1979:
 - a) Water immersion according to ISRM 1979. The wet density was determined by weighing in air and in water according to SS-EN 13755.
 - b) After drying and weighing the porosity was determined according to ISRM.
- 4. Testing according to EN 1936:
 - a) Water immersion according to ISRM 1979. The wet density was determined by weighing in air and in water according to SS-EN 13755.
 - b) After drying and weighing the porosity was determined according to ISRM.
- 5. The difference between the methods, two methods at a time, was estimated by paired t-test according to ISO 3301:1975.

The main difference between the methods is that SS-EN 13755 prescribes saturation in 1 atm for 72 h followed by weighing in water and thereafter weighing surface dry. The dry mass is determined after drying in 105°C.

ISRM prescribes saturation by water immersion in a vacuum of less than 800 Pa followed by weighing in water and thereafter weighing surface dry. The dry mass is determined after drying in 105°C.

EN 1936 prescribes drying in 70°C to constant weight and thereafter evacuation in 2 kPa for 24 h before water saturation. The pressure shall be maintained for 24 h before weighing in water and thereafter weighing surface dry.

3.2.1 Nonconformities

According to ISRM the water saturation shall be performed at a pressure of 800 Pa. The vacuum pump used has a capacity to reach 250 Pa in dry condition. When the vacuum chamber is filled with water it is only possible to reach 1100 Pa due to boiling of water.

4 Results

4.1 Test results

Wet density and porosity measurements according to SS-EN 13755, ISRM 1979 and EN 1936 were performed during July–September 2003. The results from the tests are shown in Table 4-1–4-8.

Identification	Weight in	Surface dry	Dry	Porosity	Wet
	water Msub (g)	weight Msat (g)	weight Ms (g)	n (%)	density (g/cm³)
KFM01A-90V-5A	84,50	135,25	135,08	0,33	2,659
KFM01A-90V-5B	85,09	136,12	135,95	0,33	2,662
KFM01A-90V-6A	81,73	131,07	130,92	0,30	2,651
KFM01A-90V-6B	82,74	132,59	132,45	0,28	2,654
KFM01A-90V-7A	82,66	132,35	132,19	0,32	2,658
KFM01A-90V-7B	82,31	131,81	131,66	0,30	2,657
KFM01A-90V-8A	83,60	133,73	133,58	0,30	2,662
KFM01A-90V-8B	81,78	130,81	130,67	0,29	2,662
KFM01A-90V-9A	83,31	133,49	133,32	0,34	2,655
KFM01A-90V-9B	83,23	133,30	133,10	0,40	2,657
KFM01A-90V-10A	83,32	133,40	133,20	0,40	2,658
KFM01A-90V-10B	83,44	133,61	133,41	0,40	2,658
KFM01A-90V-21A	84,01	134,33	134,18	0,30	2,664
KFM01A-90V-21B	83,98	134,43	134,30	0,26	2,659
KFM01A-90V-22A	81,28	130,04	129,90	0,29	2,661
KFM01A-90V-22B	83,58	133,79	133,64	0,30	2,659
KFM01A-90V-23A	83,55	133,67	133,52	0,30	2,661
KFM01A-90V-23B	83,33	133,22	133,07	0,30	2,665
KFM01A-90V-24A	83,85	134,05	133,89	0,32	2,665
KFM01A-90V-24B	82,74	132,69	132,52	0,34	2,651

	Table 4-1.	Test results	from testing s	amples from K	FM01A accordin	g to ISRM 1979.
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Identification	Weight in	Surface dry	Dry	Porosity	Wet
	water Msub (g)	weight Msat (g)	weight Ms (g)	n (%)	density (g/cm³)
KSH01A-090V-2A	89,87	140,03	139,80	0,46	2,786
KSH01A-090V-2B	87,87	137,03	136,79	0,49	2,782
KSH01A-090V-3A	90,27	140,86	140,61	0,49	2,778
KSH01A-090V-3B	88,88	138,65	138,39	0,52	2,780
KSH01A-090V-5A	90,83	141,45	141,31	0,28	2,788
KSH01A-090V-5B	89,72	139,87	139,69	0,36	2,783
KSH01A-090V-6A	86,33	134,43	134,31	0,25	2,789
KSH01A-090V-6B	86,07	133,89	133,78	0,23	2,794
KSH01A-090V-10A	85,24	132,95	132,87	0,17	2,781
KSH01A-090V-10B	85,07	132,91	132,80	0,23	2,772
KSH01A-090V-11A	85,68	133,85	133,73	0,25	2,773
KSH01A-090V-11B	85,53	133,12	132,99	0,27	2,791
KSH01A-090V-14A	87,80	136,42	136,34	0,16	2,800
KSH01A-090V-14B	87,30	135,57	135,51	0,12	2,803
KSH01A-090V-19A	90,22	139,20	139,10	0,20	2,836
KSH01A-090V-19B	90,75	140,03	139,91	0,24	2,836
KSH01A-090V-20A	88,53	137,43	137,25	0,37	2,805
KSH01A-090V-20B	89,06	138,40	138,28	0,24	2,799
KSH01A-090V-23A	93,46	143,02	142,90	0,24	2,880
KSH01A-090V-23B	91,13	139,55	139,42	0,27	2,876

 Table 4-2. Test results from testing samples from KSH01A according to ISRM 1979.

Identification	Weight in	Surface dry	Dry	Porosity	Wet
	Msub (g)	Msat (g)	Ms (g)	n (%)	(g/cm ³)
KFM01A-90V-5A	84,67	135,53	135,45	0,16	2,659
KFM01A-90V-5B	85,21	136,33	136,25	0,16	2,661
KFM01A-90V-6A	81,89	131,27	131,20	0,14	2,653
KFM01A-90V-6B	82,89	132,81	132,74	0,14	2,655
KFM01A-90V-7A	82,81	132,59	132,52	0,14	2,658
KFM01A-90V-7B	82,50	132,07	131,99	0,16	2,658
KFM01A-90V-8A	83,74	133,94	133,86	0,16	2,662
KFM01A-90V-8B	81,94	131,09	131,02	0,14	2,661
KFM01A-90V-9A	83,44	133,71	133,61	0,20	2,654
KFM01A-90V-9B	83,43	133,57	133,46	0,22	2,658
KFM01A-90V-10A	83,44	133,56	133,45	0,22	2,659
KFM01A-90V-10B	83,54	133,77	133,66	0,22	2,657
KFM01A-90V-21A	84,18	134,59	134,44	0,30	2,664
KFM01A-90V-21B	84,09	134,59	134,45	0,28	2,659
KFM01A-90V-22A	82,10	131,37	131,25	0,24	2,660
KFM01A-90V-22B	83,73	134,01	133,88	0,26	2,659
KFM01A-90V-23A	83,74	134,00	133,87	0,26	2,660
KFM01A-90V-23B	83,48	133,44	133,32	0,24	2,665
KFM01A-90V-24A	83,97	134,21	134,09	0,24	2,666
KFM01A-90V-24B	82,84	132,82	132,69	0,26	2,652

Table 4-3. Test results from testing samples from KFM01A according to EN13755, saturation for 72 h.

Identification	Weight in	Surface dry	Dry	Porosity	Wet
	water Msub (g)	weight Msat (g)	weight Ms (g)	n (%)	density (g/cm³)
KSH01A-090V-2A	90,35	140,78	140,67	0,22	2,785
KSH01A-090V-2B	88,23	137,63	137,51	0,24	2,780
KSH01A-090V-3A	90,48	141,22	141,11	0,22	2,777
KSH01A-090V-3B	89,24	139,23	139,12	0,22	2,779
KSH01A-090V-5A	91,28	142,14	142,06	0,16	2,789
KSH01A-090V-5B	90,18	140,60	140,51	0,18	2,782
KSH01A-090V-6A	86,94	135,36	135,29	0,14	2,789
KSH01A-090V-6B	86,49	134,53	134,46	0,15	2,794
KSH01A-090V-10A	85,50	133,35	133,30	0,10	2,781
KSH01A-090V-10B	85,27	133,23	133,16	0,15	2,772
KSH01A-090V-11A	86,30	134,81	134,73	0,16	2,773
KSH01A-090V-11B	86,23	134,21	134,12	0,19	2,791
KSH01A-090V-14A	87,94	136,63	136,57	0,12	2,800
KSH01A-090V-14B	87,46	135,83	135,78	0,10	2,802
KSH01A-090V-19A	90,82	140,13	140,07	0,12	2,836
KSH01A-090V-19B	91,21	140,74	140,66	0,16	2,835
KSH01A-090V-20A	89,43	138,84	138,75	0,18	2,804
KSH01A-090V-20B	89,45	138,98	138,91	0,14	2,800
KSH01A-090V-23A	93,89	143,70	143,60	0,20	2,879
KSH01A-090V-23B	91,55	140,16	140,08	0,16	2,877

Table 4-4. Test results from testing samples from KSH01A according to EN13755, saturation for 72 h.

Table 4-5. Test results from testing samples from KSH01A according to EN13755, saturation for 96 h.

Identification	Weight in	Surface dry	Dry	Porosity	Wet
	Msub (g)	Msat (g)	Ms (g)	n (%)	(g/cm ³)
KSH01A-090V-2B	88,25	137,63	137,51	0,24	2,781
KSH01A-090V-3A	90,51	141,24	141,11	0,26	2,778
KSH01A-090V-10A	85,49	133,36	133,30	0,13	2,779
KSH01A-090V-10B	85,28	133,23	133,16	0,15	2,772
KSH01A-090V-11A	86,30	134,82	134,73	0,19	2,772
KSH01A-090V-11B	86,24	134,22	134,12	0,21	2,791
KSH01A-090V-19A	90,82	140,14	140,07	0,14	2,835
KSH01A-090V-23A	93,89	143,70	143,60	0,20	2,878
KSH01A-090V-23B	91,55	140,17	140,08	0,19	2,876

Identification	Weight in water	Surface dry weight	Dry weight	Porosity	Wet density
	Msub (g)	Msat (g)	Ms (g)	n (%)	(g/cm³)
KFM01A-90V-6A	81,89	131,27	131,20	0,14	2,652
KFM01A-90V-9A	83,45	133,71	133,61	0,20	2,654
KFM01A-90V-21A	84,16	134,57	134,44	0,26	2,663
KFM01A-90V-23A	83,75	134,00	133,87	0,26	2,661
KFM01A-90V-23B	83,48	133,45	133,32	0,26	2,664
KFM01A-90V-24A	83,96	134,23	134,09	0,28	2,664
KFM01A-90V-24B	82,82	132,81	132,69	0,24	2,651

Table 4-6. Test results from testing samples from KFM01A according to EN13755, saturation for 96 h.

Table 4-7. Test results from testing samples from KSH01A according to EN1936.

Identification	Weight in	Surface dry	Dry	Porosity	Wet
	water Msub (g)	Msat (g)	Ms (g)	n (%)	(g/cm³)
KSH01A-090V-2A	89,83	139,96	139,82	0,279	2,786
KSH01A-090V-2B	87,80	136,98	136,81	0,346	2,780
KSH01A-090V-3A	90,19	140,80	140,63	0,336	2,776
KSH01A-090V-3B	88,79	138,57	138,41	0,321	2,778
KSH01A-090V-5A	90,79	141,40	141,31	0,178	2,788
KSH01A-090V-5B	89,63	139,79	139,70	0,179	2,781
KSH01A-090V-6A	86,30	134,40	134,32	0,166	2,789
KSH01A-090V-6B	86,05	133,86	133,78	0,167	2,794
KSH01A-090V-10A	85,21	132,93	132,87	0,126	2,780
KSH01A-090V-10B	85,04	132,89	132,80	0,188	2,772
KSH01A-090V-11A	85,63	133,82	133,74	0,166	2,772
KSH01A-090V-11B	85,48	133,11	133,00	0,231	2,790
KSH01A-090V-14A	87,74	136,38	136,35	0,062	2,799
KSH01A-090V-14B	87,27	135,54	135,51	0,062	2,803
KSH01A-090V-19A	90,16	139,16	139,11	0,102	2,835
KSH01A-090V-19B	90,71	139,98	139,92	0,122	2,836
KSH01A-090V-20A	88,45	137,35	137,28	0,143	2,804
KSH01A-090V-20B	89,00	138,34	138,30	0,081	2,799
KSH01A-090V-23A	93,40	142,98	142,91	0,141	2,879
KSH01A-090V-23B	91,11	139,53	139,43	0,207	2,876

Identification	Weight in	Surface dry weight Msat (g)	Dry	Porosity	Wet
	water Msub (g)		weight Ms (g)	n (%)	density (g/cm³)
KFM01A-90V-5A	84,42	135,20	135,08	0,24	2,657
KFM01A-90V-5B	85,05	136,07	135,96	0,22	2,662
KFM01A-90V-6A	81,70	131,07	130,92	0,30	2,650
KFM01A-90V-6B	82,67	132,56	132,46	0,20	2,652
KFM01A-90V-7A	82,61	132,30	132,20	0,20	2,657
KFM01A-90V-7B	82,28	131,77	131,67	0,20	2,657
KFM01A-90V-8A	83,55	133,69	133,58	0,22	2,661
KFM01A-90V-8B	81,74	130,78	130,67	0,22	2,661
KFM01A-90V-9A	83,26	133,45	133,32	0,26	2,654
KFM01A-90V-9B	83,19	133,26	133,11	0,30	2,656
KFM01A-90V-10A	83,25	133,35	133,21	0,28	2,657
KFM01A-90V-10B	83,35	133,55	133,43	0,24	2,656
KFM01A-90V-21A	83,97	134,31	134,18	0,26	2,663
KFM01A-90V-21B	83,95	134,41	134,28	0,26	2,659
KFM01A-90V-22A	81,26	130,03	129,89	0,29	2,661
KFM01A-90V-22B	83,56	133,77	133,64	0,26	2,659
KFM01A-90V-23A	83,49	133,66	133,52	0,28	2,659
KFM01A-90V-23B	83,30	133,21	133,07	0,28	2,664
KFM01A-90V-24A	83,83	134,04	133,90	0,28	2,665
KFM01A-90V-24B	82.70	132.66	132.53	0.26	2.651

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4.2 Comparison of test methods

4.2.1 Wet density

The different methods give very analogous results, see Figure 4-1.



Figure 4-1. Measured wet density of samples from KSH01A and KFM01A. The wet density was measured by hydrostatic method after different methods for saturation in water. The figure shows the wet density after saturation according to ISRM and EN 1936 respectively as function of the wet density after saturation according to SS-EN 13755. The dashed line shows the function Y = X, that is that the results are the same.

The uncertainty of measuring wet density by hydrostatic method is 2 kg/m^3 , see appendix 1. The investigation shows a significant difference between the methods in the range of 1 kg/m³, see Figure 4-2, which is less than the uncertainty of the methods. The methods could therefore be considered as equivalent, especially as the wet density often is rounded to the nearest 10 kg/m³.



Figure 4-2. Difference in measured wet density of samples from KSH01A and KFM01A. The wet density was measured by hydrostatic method after different methods for saturation in water. The figure shows the mean values 95% confidence intervals of the differences, calculated according to ISO 3301:1975 (paired t-test). The dashed line shows the expanded measuring uncertainty of the method.

4.2.2 Porosity



The differences between the methods are larger for porosity than for wet density as shown in Figure 4-3.

Figure 4-3. Measured porosity of samples from KSH01A and KFM01A. The wet density was measured by hydrostatic method after different methods for saturation in water. The figure shows the porosity after saturation according to ISRM and EN 1936 respectively as function of the porosity after saturation according to SS-EN 13755. The dashed line shows the function Y = X, that is that the results are the same.

The uncertainty of measuring porosity by saturation in water is 0.09%, see appendix 2. The investigation shows a significant difference between the methods, see Figure 4-4. The difference between EN 1936 and SS-EN 13755 is less than the uncertainty of the methods and could therefore be considered as equivalent. Saturation according to ISRM leads to higher water absorption than the other two methods, in the range of the uncertainty of the method for porosity.



Figure 4-4. Difference in measured porosity of samples from KSH01A and KFM01A. The porosity was measured after measuring the water absorption with different methods for saturation in water. The figure shows the mean values and the 95 % confidence intervals of the differences, calculated according to ISO 3301:1975 (paired t-test). The dashed line shows the expanded measuring uncertainty, 0.09%, of the method.

Another way to evaluate the difference between the methods is to compare the results with the expected variance of the samples thus the uncertainty of sampling. Each sample was cut into two specimens A and B. The difference between these pair of samples can be a measure of the sampling uncertainty. For the samples from KFM01A the differences were insignificant. For the samples from KSH01A differences between the results were measured as shown in Figure 4-5. The mean value of the difference is less than 0.02% by volume but single results can differ up to 0.05% by volume. If this variation is added (sum of squares) to the uncertainty (0.09%) of the method, the possible difference between two samples will increase to 0.12% by volume which is in the range of the difference between the methods, se Figure 4-4. The methods could therefore be considered as nearly identical in practice. One way to reduce the difference between SS-EN 13755 and the other methods is to extent the time of immersion.



Figure 4-5. Difference in measured porosity between consecutive samples A and B of samples from KSH01A. The porosity was estimated by measuring the water absorption for different methods for saturation in water. The figure shows the mean values and the 95% confidence intervals of the differences between the specimen A and B, calculated according to ISO 3301:1975 (paired t-test).

5 Conclusions

According to ISRM the density shall be rounded to the nearest 10 kg/m³. The largest differences between the results are 0.3 kg/m³ for samples from KSH01A. The conclusion is therefore that the difference between the methods is insignificant in comparison with the rounding and the measuring uncertainty. The main source of the uncertainty is the uncertainty of the balance.

The largest difference between the results from porosity is about 0.12 vol-% when SS-EN 13755 is compared with the ISRM method. This is slightly more than the uncertainty of the method which is 0.09 vol-%. If the variation of the samples is taken into consideration, the difference is insignificant.

Water absorption in vacuum shall according to ISRM be performed in water at 800 Pa pressure. This pressure is very near the triple point of water, 611 Pa at 0°C. This is hard to achieve at normal laboratory temperature. When the pressure is lowered, the water will boil. In practice it is possible to achieve a pressure of 1000–2000 Pa.

A practical way is to establish the pressure in a first step and then introduce the water still under vacuum as described in EN 1936. This is also a method which is used in many other applications as sample preparation of thin sections for microscopic examination of concrete. In this case, the sample will dry in the first step which could cause entrapping air void. This is minimised by introducing the water in steps. In a first step the sample should be covered with water to the half and the after a sufficient time totally covered with water.

6 References

ISRM, 1979. Suggested methods for determining water content, porosity, density, absorption and relayed properties and slake-durability index properties (saturation and Buoyancy techniques), International Journal of Rock Mechanics and Mining Sciences & Geomechanics Abstracts, Vol. 16, pp 141 to 156, Pergamon Press Ltd., 1979.

SIS, SS-EN 13755. Natural stone test methods – Determination of water absorption at atmospheric pressure, 2002.

CEN, EN 1936. Natural stone test methods – Determination of density and apparent density, and of total and open porosity, 1999.

ISO. Guide To The Expression Of Uncertainty In Measurement, Geneva, Switzerland 1993 ISBN 92-67-10188-9.

Appendix 1

Calculation of uncertainty in determining wet density by hydrostatic method

Calculation of uncertainty: Density

Date 2003-12-19 Responsible Matz S

	Estimatior	_	Standard	Assess-	Distri-	Standard uncertai	nty	Sensibility		Uncertainty	
Source		Unit	deviation	ment	bution	of source	Jnit		Unit		Unit
Dry weight (MS)	138,10	5 g									
Balance uncerts	ainty	g	0,0050	В	u*2	0,010 9		0,0367	1/cm ³	0,3666	kg/m ³
Balance resolut	ion	g	0,0050	В	Rekt	0,002886751 g		0,0367	1/cm ³	0,1058	kg/m ³
Vikt i v. (Msub)	88,912	2 0									
Tara		g	0,0071	В	Rekt	0,01040833 g		0,0569	1/cm ³	0,5927	kg/m ³
Balance uncerts	ainty	g	0,0050	В	u*2	0,010 0		0,0569	1/cm ³	0,5694	kg/m ³
Balance resolut	ion	g	0,0050	В	Rekt	0,002886751 g		0,0569	1/cm ³	0,1644	kg/m³
incompleted sat	uration	g	0,0500	В	Rekt	0,028867513 g		0,0569	1/cm ³	1,6438	kg/m³
Density of water	3266'0	3 g/cm ³	0,0002	В	Rekt	0,000115216 9	1/cm ³	2,8074	1	0,3235	kg/m ³
Provvariation											kg/m³
Combined uncert	ainty									1,9118	kg/m3
Expanded uncer	tainty									3,8236	kg/m3

Utrustning

Balance No: 102084 Digital thermometer No:100820, 285109, 285110 Variation of temperaturvariation 1 grad C Variation of the density of water 0,2 kg/m³ per degree The uncertainty at weighing is twice the uncertainty at calibration

Notes

	Evaluation of senibility factor by partial derivation
Rekt:	Rectangle distributed uncertainty
ïZ	Normal distributed uncertainty
n	Uncertainty at calibration

Appendix 2

Calculation of uncertainty in determining porosity by using water absorption and hydrostatic method

Calculation of uncertainty; porosity

Date 2003-12-19 Responsible Matz S

	Estimation		Standard	Assess-	Distri-	Standard uncerta	linty	Sensibility		Uncertainty	
Source		Unit	deviation	ment	bution	of source	Unit		Jnit		Unit
Dry weight (MS)	138,023	g									
Balance uncertainty		б	0;0050	В	u*2	0,0100	D	2,0328	1/g	0,0203	vol %
Balance resolution		D	0,0050	В	Rekt	0,0029	0	2,0328 1	1/g	0,0059	vol %
Wet wight (Msat)	138,105	g									
Balance uncertainty		б	0,0050	В	u*2	0,0100	0	2,0294 1	1/g	0,0203	vol %
Balance resolution		D	0,0050	В	Rekt	0,0029	0	2,0294 1	1/g	0,0059	vol %
incompleted saturation		б	0,0500	В	z	0,0167	0	2,0294 1	1/g	0,0338	vol %
Vikt i v. (Msub)	88,912	g									
Tara		б	0,0071	В	Rekt	0,0104	9	0,0034 1	1/g	0'0000	vol %
Balance uncertainty		б	0,0050	В	u*2	0,0100	6	0,0034 1	1/g	000000	vol %
Balance resolution		б	0,0050	В	Rekt	0,0029	0	0,0034 1	1/g	0000'0	vol %
incompleted saturation			0,0100	В	Rekt	0,0058		0,0034		0'0000	vol %
Sample variation											
Combined uncertainty										0,0451	vol %
Expanded uncertainty										0,0903	vol %

Utrustning Balance no:102084

Digital thermometer: No: 100820, 285109, 285110 The uncertainty at weighing is twice the uncertainty at calibration

NotesEvaluation of senibility factor by partial derivationB:Evaluation of senibility factor by partial derivationRekt:Rectangle distributed uncertaintyN:Normal distributed uncertaintyuUncertainty at calibration