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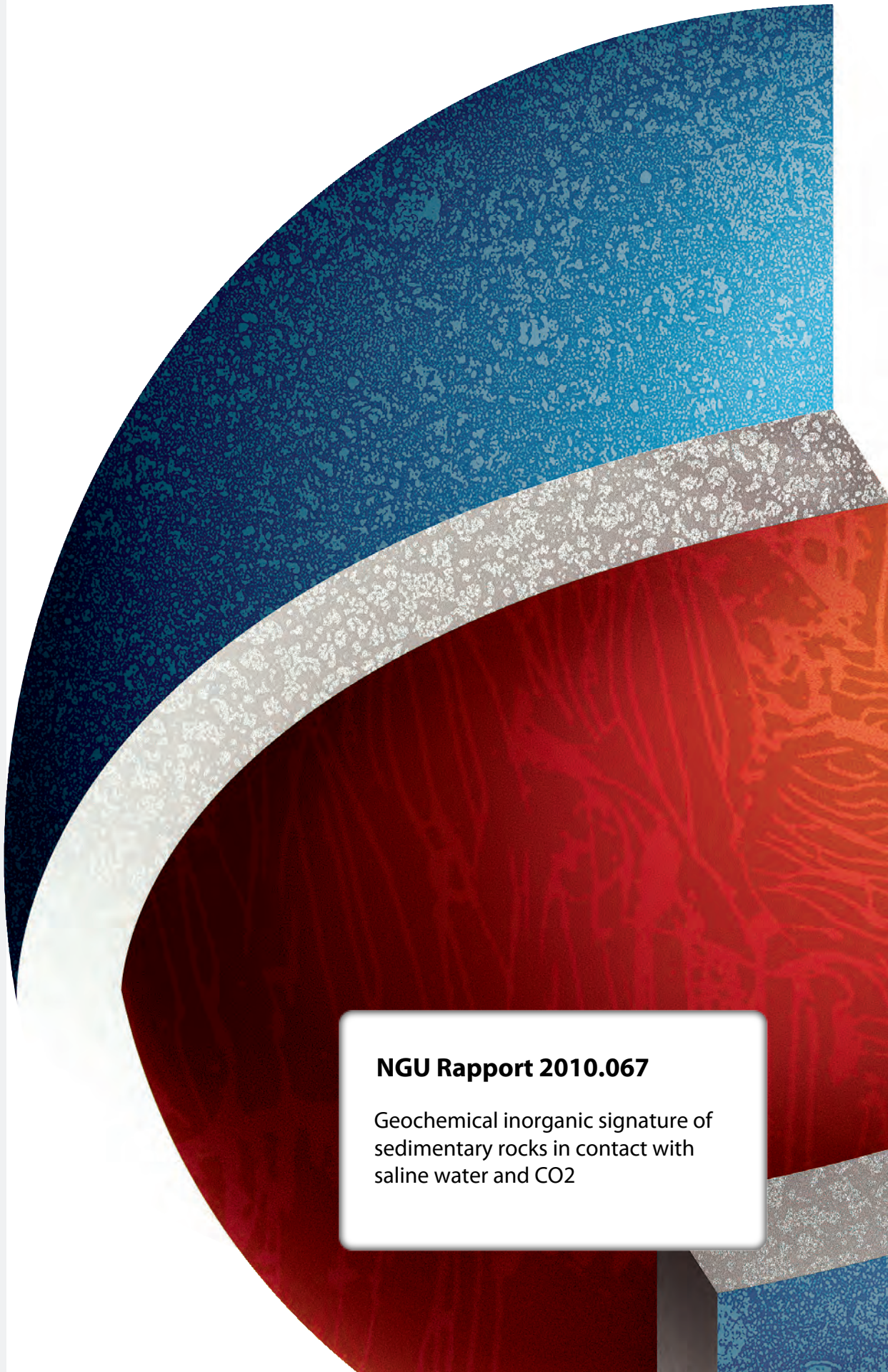

NGU Rapport 2010.067

Geochemical inorganic signature of sedimentary rocks in contact with saline water and CO2

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REPORT

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Title:
Geochemical inorganic signature of sedimentary rocks in contact with saline water and CO_2

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Summary:

A pilot study was carried out with the intention of establishing a geochemical inorganic signal or "fingerprint" of soluble major, minor, and trace elements when different types of reservoir and cap rocks come in contact with saline water with and without CO_2. The focus here is the value of the elemental concentrations in relation to each other and not the absolute value of each elemental concentration in itself or in relation to the thermodynamic state of the carbonate system. The results are very preliminary and the experimental set-up is not stringent enough to draw any permanent conclusions about solubility of major, minor, and trace elements dissolved when mixtures of these chosen sedimentary rock types are put in contact with saline water with and without CO_2.

Keywords: Marine geology	Sedimentary rocks	Salt water
CO_2	Elements	Cations
pH	Electrical conductivity	

CONTENTS

TABLES

1. INTRODUCTION

A pilot study was carried out with the intention of establishing a geochemical inorganic signal or "fingerprint" of soluble major, minor, and trace elements when different types of reservoir and cap rocks come in contact with saline water with and without CO_2. The focus here is the value of the elemental concentrations in relation to each other and not the absolute value of each elemental concentration in itself. The results are very preliminary and the experimental set-up is not stringent enough to draw any permanent conclusions about solubility of major, minor, and trace elements dissolved when mixtures of these chosen sedimentary rock types are put in contact with saline water with and without CO_2.

2. METHODS

The effect of CO_2 on mechanical stability of the reservoir-caprock system using the Brazilian test (Aydin & Basu 2006) was performed on five different sedimentary rock types after ageing; Bentheimer and Castlegate sandstones, Austin and Lixhe chalk, and Pierre shale (Ojala 2010). The mineralogical composition of these five sedimentary rocks and a sixth, the Saltwash North Sandstone, which also was subjected to aging, is shown in Table 1.

The ageing procedure before the Brazilian test consisted of placing mixtures of from 7 to 41 specimen discs (each disc size approximately D=381 mm and H=254 mm, assumed) of these six types of rock (Table 2a) in a 2 liter steel container and fill it up with saline water (i. e. $\approx$ 3.5% NaCl) saturated with CO_2 (see Table 2b under "Rock mixture" I, II, and III, with "Aqeous samples" 1-6), or solely saline water (i. e. $\approx$ 3.5% NaCl) (see Table 2b "Rock mixture" IV and V, with "Aqeous samples" 7-10).

The ageing process was carried out at T = 80 °C and P_{Pore} = 25 MPa in order to simulate reservoir conditions. This corresponds to a depth of 2.5 km assuming a geothermal gradient of 32 °C/km and water density of 1 g/cm^3. While such depth condition may be greater than some CO_2 sequestration projects these values were chosen to accelerate the chemical reactions between the CO_2-saturated saline water and the minerals that make up the rock samples.

The ten samples of aqueous solutions (labeled #101519 - #101528 in the analytical reports and aqueous sample 1-10 under "Results") were analyzed for their content of chemical elements by ICP-AES at the chemical laboratory of NGU, in addition to electrical conductivity (*norwegian:* ledningsevne) and pH (see Tables 3, 4, and 5). Analyses of anions were not performed on these aqueous solutions due to the expected predominance of the chloride ion.

3. RESULTS

The total content of chemical elements in the ten samples of aqueous solutions (labeled #101519 - #101528, see Table 2b) after one week of "ageing" are presented in Table 3, the electrical conductivity in Table 4, and the pH in every other sample in Table 5.

In general, the results of the chemical analyses are above the detection limit for two or more aqueous samples for Si, Mg, Ca, Na, K, Mn, Sr, P, Cu, Zn, Ni, V, Ba, B, and Li.

The Na-concentrations in the ten aqueous solutions after one week (approximately168 hours) of contact with the five different rock mixtures are pair-wise comparable, i.e. at the same level, since the amount of Na released from the rock mixtures would be insignificant compared to the concentration in the ≈ 3.5% saline solutions. However, this is true within the analytical error for most of the other elements analyzed, except:

a) Ca, which is relatively lower in aqueous sample 2 and 3,

b) Cu, which is fairly high in samples aqueous 9 and 10,

c) Mg and Mn, which are both fairly high in aqueous samples 7 and 8,

d) Zn, which is fairly high in aqueous samples 1, 2, 3, and 4,

e) Ba, which is fairly high in aqueous samples 1 and 2, but also in samples 3 and 4, and 9 and 10,

f) Sr, which is fairly high in aqueous samples 7 and 8, but also samples 5 and 6,

g) B, which is fairly high in aqueous samples 7 and 8, and

h) Li, which is fairly high in aqueous samples 1 and 2.

The suppressed concentrations of Ca in aqueous samples 2 and 3 could possibly reflect a net precipitation of $CaCO_3$ during the one week of contact.

The elevated concentrations of Cu in aqueous samples 9 and 10 could possibly reflect a contribution from Cu in the Saltwash North Sandstone.

The fairly high concentrations of Mg, Mn, B, and Sr in aqueous samples 5, 6, 7 and 8 could possibly reflect the presence of these elements in Pierre shale.

The variations in the concentrations of Zn, Ba, and Li are not obvious from the data collected.

4. CONCLUSIONS

This is a pilot study assessing the variation in the concentration of chemical major, minor, and trace elements released from assemblages of typical reservoir rocks and one shale (Pierre) that have been allowed to react (to "age") with saline water saturated with CO_2 and solely saline water at T = 80 °C and P_{Pore} = 25 MPa in order to simulate reservoir conditions. The results of the "ageing" and subsequent chemical analysis of inorganic elements, electrical conductivity, and pH, show that there are large variations in the measured variables in some of the samples compared to others when considering major, minor and a few (i.e. Cu, Zn, and V) trace

elements. Most of the trace elements are below the detection limit of the analytical method applied in this study. The focus in this study is the numerical value of the elemental concentrations in relation to each other and not the absolute value of each elemental concentration in itself or in relation to the thermodynamic state of the carbonate system. However, the results can be used as a reference and starting-point for designing future experiments in which saline formation pore water with and without CO_2 is allowed to react with different types of reservoir and cap rocks.

5. REFERENCES

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Table 1: Mineralogical composition of sedimentary rocks subjected to ageing.

Rock	Por %	Name	Minerals (volume%)	Literature source
A	27	Austin chalk	Calcite$CaCO_3$(88%), Qtz(12%)	Lee and Wiltschko (2000)
L	40	Lixhe chalk	Calcite$CaCO_3$(98%), Qtz(2%)	Homand and Shao (2005)
P	13	Pierre shale	Calcite$CaCO_3$(15%), Qtz & Fspar(32%), Clays(41%)	Bailey et al. (1994)
B	25	Bentheimer ss	Qtz(95%), Fspar(2%), Clay(3%)	Dautriat et al. (2009)
C	29	Castlegate ss	Qtz(70%), Fspar(30%), Clay(<1%)	Fjær et al. (2005)
S	29	Saltwash North ss	Qtz(85%), Fspar(8%), Silicified tuff & chert(7%)	Thamm et al. (1981)

Table 2a. Specific properties of the five rock mixtures subjected to ageing (source Ojala 2010).

Ageing agent	Rock mixture	No. of sedimentary rock specimen discs						Specimen Size	Average Porosity	Pore Volume of rock sample	Water volume container	Ratio Porevolume/Watervolume	Geometric surface area	Rock volume	PoreSurface area
		Aust	Lix	Pie	Bent	Cast	Salt	cm3	%	cm3	mL		cm-1	cm3	cm2
Saltwater + CO_2	I	10	10		2	6	2	20.43	31,7	194	1472	0.13	205	613	125492
"	II				8	4	8	20.43	27,4	112	1594	0.07	218	409	88972
"	III			7				20.43	13,0	19	1766	0.01	261	143	37317
Saltwater	IV	1		12	11	1	1	20.43	19,4	99	1479	0.07	242	510	123347
"	V	9	9		2	10	11	20.43	30,8	258	1311	0.20	208	837	173900

Table 2b. Results of visual inspection of aqueous solutions (source Ojala 2010).

Ageing fluid	Rock mixture	No. of sedimentary rock specimen discs						Water-sample ID used in analytical report	Water sample no. referred to in this report	Color/characteristics of content
		Aust	Lix	Pie	Bent	Cast	Salt	ID#	Aqeuos Sample #	Visual inspection
Saltwater + CO_2	I	10	10		2	6	2	101519	1	pct, rust-coloured & white
"	I	10	10		2	6	2	101520	2	pct, rust-coloured & white
"	II				8	4	8	101521	3	pct
"	II				8	4	8	101522	4	pct
"	III			7				101523	5	crud/mud/filtrate
	III			7				101524	6	crud/mud/filtrate
Saltwater	IV	1		12	11	1	1	101525	7	clear
"	IV	1		12	11	1	1	101526	8	clear
"	V	9	9		2	10	11	101527	9	Clear water-coloured
"	V	9	9		2	10	11	101528	10	Clear water-coloured

Table 3: Concentrations of 32 elements in ten samples determined by ICP-AES (Analytical contract 2010.0153).

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ICP-AES ANALYSER
VANN
ANALYSEKONTRAKTNR. 2010.0153 FORELØPIG

NORSK AKKREDITERING TEST 020

INSTRUMENT: **ICP-AES type Perkin Elmer Optima 4300 Dual View**
METODE: Metodoppsettet er beskrevet i NGU-SD 3.1: ICP-AES -analyse av vann
NEDRE BESTEMMELSESGRENSER (LLQ) VED VANNANALYSER
(For vannprøver som fortynnes blir deteksjonsgrensene automatisk omregnet)

Si mg/l	Al mg/l	Fe mg/l	Ti mg/l	Mg mg/l	Ca mg/l	Na mg/l	K mg/l	Mn mg/l	P mg/l	Cu mg/l	Zn mg/l	Pb mg/l	Ni mg/l	Co mg/l	V mg/l
0,02	0,02	0,002	0,001	0,05	0,02	0,05	0,5	0,001	0,05	0,005	0,002	0,005	0,005	0,001	0,005

Mo mg/l	Cd mg/l	Cr mg/l	Ba mg/l	Sr mg/l	Zr mg/l	Ag mg/l	B mg/l	Be mg/l	Li mg/l	Sc mg/l	Ce mg/l	La mg/l	Y mg/l	As mg/l	Sb mg/l
0,005	0,0005	0,002	0,002	0,001	0,002	0,005	0,02	0,001	0,005	0,001	0,02	0,005	0,001	0,01	0,005

(1 mg/l = 1 ppm)

ANALYSEUSIKKERHET:

i) Nedre måleområde (LLQ - 5*LLQ):
± 50 rel. %: As, Sb (S, Se, Sn) ± 37.5 rel. %: K, Pb
± 25 rel. %: Ag, Al, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, La, Li, Mg, Mo, Mn, Na, Ni, P, Si, Sc, Sr, Ti, V, Y, Zn, Zr
ii) Øvre måleområde (> 5*LLQ):
± 20 rel. %: As, Sb (S, Se, Sn) ± 15 rel. %: K, Pb
± 10 rel. %: Ag, Al, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, La, Li, Mg, Mo, Mn, Na, Ni, P, Si, Sc, Sr, Ti, V, Y, Zn, Zr
Oppgitte usikkerheter har dekningsfaktor 2 (2 standardavvik), noe som tilsvarer et konfidensintervall på 95 %

PRESISJON: **Det analyseres rutinemessig kontrollprøver som føres i kontrolldiagram (X-diagram). Disse kan forevises om ønskelig.**

ANTALL PRØVER: 10

ANMERKNINGER: Dette er en midlertidig versjon. Prøvene ble fortynnet 20X.

Gjengivelse av analysedata skal skje på en slik måte at meningsinnholdet i rapporten ikke endres.

Ferdig analysert	25.mai.10	Frank Berge/Ana Banica
	Dato	OPERATØR

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Prøve ID	Si [mg/L]	Al [mg/L]	Fe [mg/L]	Ti [mg/L]	Mg [mg/L]	Ca [mg/L]	Na [mg/L]	K [mg/L]	Mn [mg/L]	P [mg/L]	Cu [mg/L]	Zn [mg/L]	Pb [mg/L]	Ni [mg/L]	Co [mg/L]	V [mg/L]
101519	13,9	<0.4	<0.04	<0.02	7,95	456	10200	15,3	0,665	<1	<0.1	0,374	<0.1	0,412	0,0290	<0.1
101520	13,7	<0.4	<0.04	<0.02	7,39	138	10100	15,8	0,0840	<1	<0.1	0,536	<0.1	0,114	<0.02	<0.1
101521	23,0	<0.4	<0.04	<0.02	13,0	141	12500	20,9	<0.02	<1	0,113	0,457	<0.1	<0.1	<0.02	<0.1
101522	23,3	<0.4	<0.04	<0.02	14,0	283	12500	21,0	<0.02	<1	<0.1	1,60	<0.1	<0.1	<0.02	0,114
101523	3,88	<0.4	<0.04	<0.02	65,2	240	12700	26,8	0,290	<1	0,103	<0.04	<0.1	<0.1	<0.02	<0.1
101524	4,14	<0.4	<0.04	<0.02	71,0	300	13600	30,2	1,51	<1	0,102	0,154	<0.1	<0.1	<0.02	0,124
101525	32,8	<0.4	<0.04	<0.02	116	498	12300	31,4	0,541	1,08	<0.1	0,0662	<0.1	<0.1	<0.02	0,151
101526	34,6	<0.4	<0.04	<0.02	123	526	13000	32,9	0,555	1,23	<0.1	0,363	<0.1	<0.1	<0.02	0,201
101527	30,4	<0.4	<0.04	<0.02	8,81	231	13700	29,4	0,0243	<1	0,455	0,0499	<0.1	<0.1	<0.02	0,215
101528	29,4	<0.4	<0.04	<0.02	8,38	225	13300	30,0	0,0237	<1	0,459	0,0559	<0.1	<0.1	<0.02	0,195

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TEST 020

Prøve ID	Mo [mg/L]	Cd [mg/L]	Cr [mg/L]	Ba [mg/L]	Sr [mg/L]	Zr [mg/L]	Ag [mg/L]	B [mg/L]	Be [mg/L]	Li [mg/L]	Sc [mg/L]	Ce [mg/L]	La [mg/L]	Y [mg/L]	As [mg/L]	Sb [mg/L]
101519	<0.1	<0.01	<0.04	7,53	1,01	<0.04	<0.1	<0.4	<0.02	106	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101520	<0.1	<0.01	<0.04	6,77	0,881	<0.04	<0.1	<0.4	<0.02	104	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101521	<0.1	<0.01	<0.04	2,68	1,81	<0.04	<0.1	<0.4	<0.02	5,47	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101522	<0.1	<0.01	<0.04	2,89	1,97	<0.04	<0.1	<0.4	<0.02	5,44	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101523	<0.1	<0.01	<0.04	0,387	5,20	<0.04	<0.1	0,994	<0.02	0,106	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101524	<0.1	<0.01	<0.04	0,476	5,69	<0.04	<0.1	1,06	<0.02	0,110	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101525	<0.1	<0.01	<0.04	1,73	11,1	<0.04	<0.1	1,97	<0.02	0,109	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101526	<0.1	<0.01	<0.04	1,83	11,6	<0.04	<0.1	2,11	<0.02	0,165	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101527	<0.1	<0.01	<0.04	3,16	1,70	<0.04	<0.1	<0.4	<0.02	<0.1	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1
101528	<0.1	<0.01	<0.04	3,11	1,66	<0.04	<0.1	<0.4	<0.02	<0.1	<0.02	<0.4	<0.1	<0.02	<0.2	<0.1

Table 4: Values of electrical conductivity in ten samples determined by conductivity-meter (Analytical contract 2010.0153).

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BESTEMMELSE AV LEDNINGSEVNE
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ANALYSEKONTRAKTNR. 2010.0153

INSTRUMENT: **Radiometer Titralab 94 / CDM 210 Conductivity meter med 2-pole cells CDC749**

METODE: **Metodeoppsettet er beskrevet i NGU-SD 3.6: Bestemmelse av ledningsevne**
ANALYSENE UTFØRES ETTER NORSK STANDARD - NS-ISO 7888

NEDRE BESTEMMELSESGRENSE: **0.05 mS/m** **(1 mS/m = 10 µS/cm)**

ANALYSEUSIKKERHET:

Måleområde (mS/m)	Usikkerhet
0.05 - 1.0*	± 20 % rel.
1.0 - 2.0	± 8 % rel.
2.0 - 5.0	± 4 % rel.
> 5.0	± 2 % rel.

Oppgitte usikkerheter har dekningsfaktor 2 (2 standardavvik), noe som tilsvarer et konfidensintervall på 95 %.

*. Resultater under 1 mS/m kan bli påvirket av atmosfærisk karbondioksyd og ammoniakk

PRESISJON: **Det kjøres rutinemessig kontrollprøver, som føres i kontrolldiagram (X-diagram). Disse kan forevises om ønskelig.**

ANTALL PRØVER: 10

ANMERKNINGER: 1. Målingene er ikke akkrediterte. Temperaturkorrigering utført ved å måle nedgang i ledn.-evne i std 0.01D KCl-løsning på 25.0 C. Deretter ble %-vis endring pr grad beregnet (2.92%) og korrigering utført for alle målinger. Oppgitt ledningsevne gjelder følgelig for 25.0 C-løsninger.

Gjengivelse av analysedata skal skje på en slik måte at meningsinnholdet i rapporten ikke endres.

Ferdig analysert	27. august 2010	Tomm Berg
	Dato	OPERATØR

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BESTEMMELSE AV LEDNINGSEVNE
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Analysedato	NGU Prøvenr.	Prøve id.	Ledn.-evne mS/cm	Temp. °C
26.08.2010	101519	1. Test 2 saltvann før flom	50,77	24,5
26.08.2010	101520	2. Test 3 ca.10:00 (3.5%)	50,25	24,8
26.08.2010	101521	3. Reslab test 1 før flom N 8.2	58,04	24,5
26.08.2010	101522	4. N 8.2 salt water (3.5%)	56,02	24,5
26.08.2010	101523	5. SK2.2(?)salt water (3.5%)	61,36	24,5
26.08.2010	101524	6. SK2.2(?)salt water (3.5%)	58,33	24,2
26.08.2010	101525	7. S2 12/3 salt water (3.5%)	52,88	24,0
26.08.2010	101526	8. S2 12/3 S-Ni til gm (3.5%)	104,6	24,0
26.08.2010	101527	9. 16.2() salt water (3.5%)	54,26	24,0
26.08.2010	101528	10. 16.2 SWSST salt water	58,66	24,0

Table 5: Values of pH determined on every other sample (i.e. five samples) by pH-meter (Analytical contract 2010.0153).

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BESTEMMELSE AV pH
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ANALYSEKONTRAKTNR. 2010.0153

NORSK AKKREDITERING
TEST 020

INSTRUMENT: **Radiometer Titralab 94 / Glasselektrode pHC 2701-8 "Red Rod"**

METODE: **pH: Metodeoppsettet er beskrevet i NGU-SD 3.5: Bestemmelse av pH. Utførelsen følger NS 4720**

ANALYSEUSIKKERHET: **± 0.20 pH**
Oppgitt usikkerheter har dekningsfaktor 2 (2 standardavvik), noe som tilsvarer et konfidensintervall på 95 %.

PRESISJON: **Det analyseres rutinemessig kontrollprøver som føres i kontrolldiagram (X-diagram). Disse kan forevises om ønskelig.**

ANTALL PRØVER: 5

ANMERKNINGER: Pga. lite løsning er kun 5 prøver målt

Ferdig analysert	8. juni 2010	Tomm Berg
	Dato	OPERATØR

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BESTEMMELSE AV pH
VANN
ANALYSEKONTRAKTNR. 2010.0153

Analysedato	NGU Prøvenr.	Prøve id.	pH pH
07.06.2010	101519	1) Test 2 saltvann før flom	7,39
07.06.2010	101521	2) Reslab test 1 før flom N 8.2	8,37
07.06.2010	101523	3) SK2.2(?)salt water	8,00
07.06.2010	101526	4) S2 12/3 S-Ni til gm	7,46
07.06.2010	101528	5) 16.2 SWSST salt water	7,45