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Saline Aquifer CO_2 Storage Project, SACS, Phase II

Task 1.4: Evaluation of cap rock sealing the reservoir Clay mineralogical investigation of core and cuttings from the Ekofisk and Sleipner areas

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1. Introduction

As a partner in the Saline Aquifer CO_2 Storage (SACS) project, GEUS Core Laboratory participated in subtasks 1.4 "Evaluation of cap rock sealing the reservoir". The reservoir is the Miocene to Pliocene Utsira sand unit of the Utsira Formation, which is capped by the Nordland Shale. The objective was to test the sealing and transport capacity of the cap rock with respect to the injected CO_2, i.e. to measure the liquid permeability of the cap rock and the entry pressure to CO_2 – the pressure where gas/super critical CO_2 displaces the liquid phase from the pores of the cap rock and therefore invades the cap rock. This information is required to assess the chances of CO_2 leaking through the cap rock.

During Phase I of the SACS Project it finally became clear that no full core material was available from the Nordland Shale for cap rock testing. Only wet samples were kept from a number of Sleipner wells from which cuttings of the Nordland Shale might be obtained. At a Technical Meeting in Orleans in September 2000 it was decided to advance the cap rock evaluation. A possible analogue to the Nordland Shale may be found further to the south in the Ekofisk area where a Pliocene clay/shale section has been cored in the 2/4-C11 well. If the clay mineralogy in the Ekofisk area is comparable to the Nordland Shale this well could be a potential candidate to a proper cap-rock test.

Permission was obtained from the Norwegian Petroleum Directorate (NPD) to inspect and sample the Pliocene clay core. At the following visit to NPD's core facility GEUS took 3 small clay samples for mineralogical analysis. No equipment was available to take unconsolidated plug size samples on site and GEUS therefore requested the core be transferred to a local core laboratory for plugging. Unfortunately permission to transfer the core was not granted by NPD, and the objective to carry out a cap rock sealing capacity test finally had to be given up.

The present report therefore is not a presentation of the cap rock sealing properties of the Nordland Shale, but only collects the relatively few clay mineralogical analyses performed on cuttings from the Nordland Shale in the Sleipner area and its presumed Pliocene analogue in the Ekofisk area.

2. Sampling

2.1 Cuttings samples.

SINTEF forwarded 10 wet samples of Nordland Shale from 3 wells in the Sleipner area for mineralogical characterization, table 2.1. The study carried out at GEUS was focused on the clay mineralogy of the cap rock solely from an investigation of cuttings washed from the wet samples. Separation of cuttings from the drilling mud in the wet samples is a challenge because the soft shale is washed away if preparation is carried out using normal routine methods. Because of the fragile cuttings, the mud was removed by soaking and gently stirring the samples in distilled water and decanting the dispersed drilling mud until the remaining samples appeared to consist predominantly of clean cuttings. Cuttings were successfully washed from all 3 wells, table 2.1.

Table 2.1. List of cuttings washed from 3 wells in the Sleipner Field. In the selected size fractions a minor number of whitish and rusty cuttings appeared, indicating cementation with carbonate and siderite. The weight of the wet sample is given in the table, but only a few grams of clean cuttings remained for analysis after washing and sieving.

Well no.	Formation	Depth (MD) meter	Wet sample, g	Description
15/9-15	not given	650-660	45	approx. 1 mm in size, flaky
15/9-16	Nordland Group	700-710	80	approx. 1 mm in size, flaky to sub rounded
15/9-18	Nordland Group	850-860	70	1-4 mm in size, flaky or slightly rounded

2.2 Core samples.

GEUS visited NPD's core facility in Stavanger to sample the Nordland Shale equivalent from the Ekofisk 2/4-C11 well. Four 5” diameter full core sections preserved in glasfiber sleeves were on display for description and sampling. The sleeves had been cut longitudinally but were sealed airtight with a heavy-duty sticky tape. The clay cores were still humid and found to be in a good condition, and 3 small clay samples were taken for mineralogical analysis, table 2.2. From the table it appears that every other 3-feet core box is missing; no record was kept of the missing boxes, however.

Table 2.2. Description and list of samples from the Ekofisk well 2/4-C11, core # 3.

Box no.	Depth interval, (MD) feet	Sample depth, feet	Description
1	1733’ – 1736’	1735’4”	preserved, still humid, homogeneous plastic clay; dark and bright spots of mm to cm size are visible, but lamination is not discernible.
2	1739’ – 1742’	1741’0”	preserved, homogeneous plastic clay; dark and bright spots of mm to cm size are visible, no lamination; humid but superficial drying cracks; 45° fracture in mid of box.
3	1745’ – 1748’	1747’4”	preserved, homogeneous plastic clay; more silty 10 cm thick layer approx. 20 cm b.t.; slightly humid but with superficial drying cracks.
4	1751’ – 1754’	not sampled	preserved, but core surface mechanically damaged; still humid, homogeneous plastic clay; dark and bright spots of mm to cm size are visible, no lamination.

3. Analytical methods

3.1 Clay mineralogy

Cuttings and clay samples were disintegrated and separated into size fractions using a particle size centrifuge. The coarse and fine clay (size fractions 2-0.2 µm and <0.2 µm respectively) were investigated by X-ray diffraction (XRD) using a Philips 1050 instrument and Co-Kα radiation. Oriented specimens were prepared by the pipette method as follows:

- Mg-saturated analysed air dry (all samples)
- Glycolated (Ekofisk Field cores) or
- Glycerolated (Sleipner cuttings)
- K-saturated samples analysed air-dry and after heating to 300 ºC.

The minerals were identified from peak positions as follows:

Mineral	Glycol/glycerol	Mg-air	K-300°C
Illite	10Å, 5Å	10Å, 5Å	10Å, 5Å
Smectite	17Å/18Å	14Å	10Å
random illite-smectite	17Å/18Å+10Å	10-14Å	10Å
ordered illite-smectite	>20Å	>20Å	
Kaolinite	7Å, 3.5Å	7Å, 3.5Å	7Å, 3.5Å
Chlorite	14Å, 4.8Å	14Å, 4.8Å	14Å, 4.8Å
Vermiculite	14Å	14Å	10Å

No attempt was made to quantify the mineral phases because the degree of similarity between the sample minerals and available standard clay minerals is unknown.

3.2 Gas adsorption, specific surface area.

Adsorption isotherms were determined on a Coulter SA 3100 gas adsorptometer using liquid N_2. The specific surface area was calculated according to the BET model.

4. Results

4.1 Nordland Group cuttings, Sleipner area

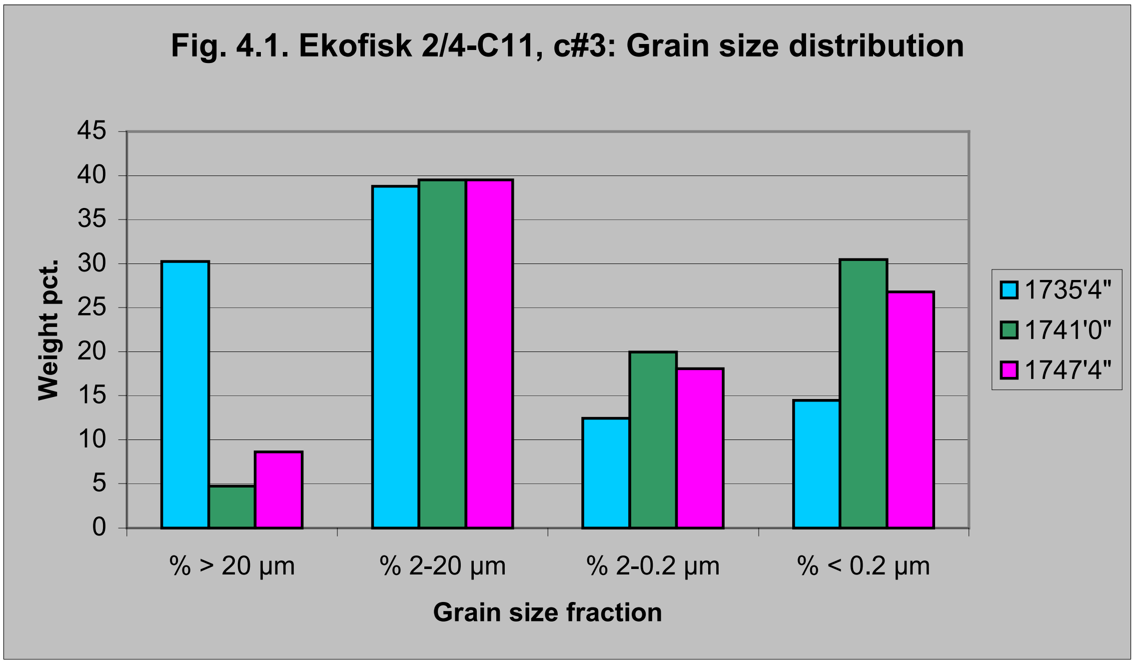
Diffractograms of the different treatments are shown in Fig. 4.2 and 4.3, and for the Mg-air dry specimens in Fig 4.4. The samples from the different Sleipner wells have approximately the same clay mineralogy, namely a mixture of approximately equal amounts of smectite, vermiculite, illite and kaolinite, with traces of chlorite. Small amounts of quartz are present in the clay fraction as well. Mixed-layer illite-smectite is not detected.

4.2 Pliocene core, Ekofisk area

Diffractograms for Mg-air dry specimens are shown in Fig 4.5. The different samples have approximately the same clay mineralogy, namely a mixture of approximately equal amounts of smectite, illite and kaolinite, with minor amounts of vermiculite and traces of chlorite. Small amounts of quartz are present in the clay fraction as well. Mixed-layer illite-smectite is not detected.

Table 4.1. Ekofisk well 2/4-C11; grain size analysis and specific surface area of Pliocene clay core samples. Bulk sample approx. 10 g of each core section.

Core / Depth	**Grain size fractions, wt-%**					**Clay fraction**	**BET surface area**
	> 20 µm	2-20 µm	2-0.2 µm	< 0.2 µm	Σ		
						wt-%	sq.m/g
C#3 1735'4"	30	39	12	14	96	26	20
C#3 1741' 0"	5	40	20	30	95	50	40
C#3 1747'4"	9	40	18	27	93	45	35



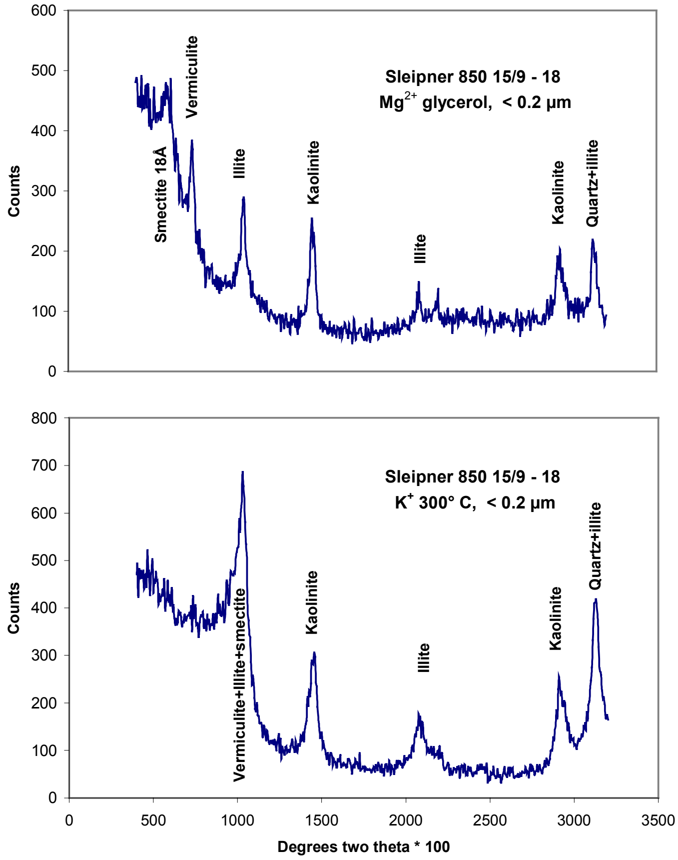


Fig. 4.2. Sleipner well 15/9-18; fine clay fraction oriented mount X-ray diffraction traces showing identified minerals.

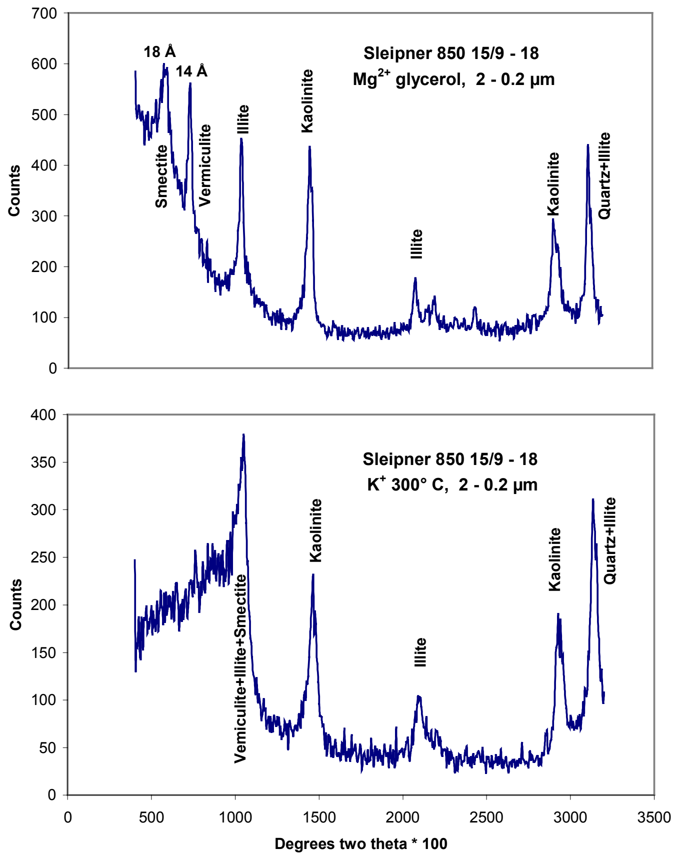
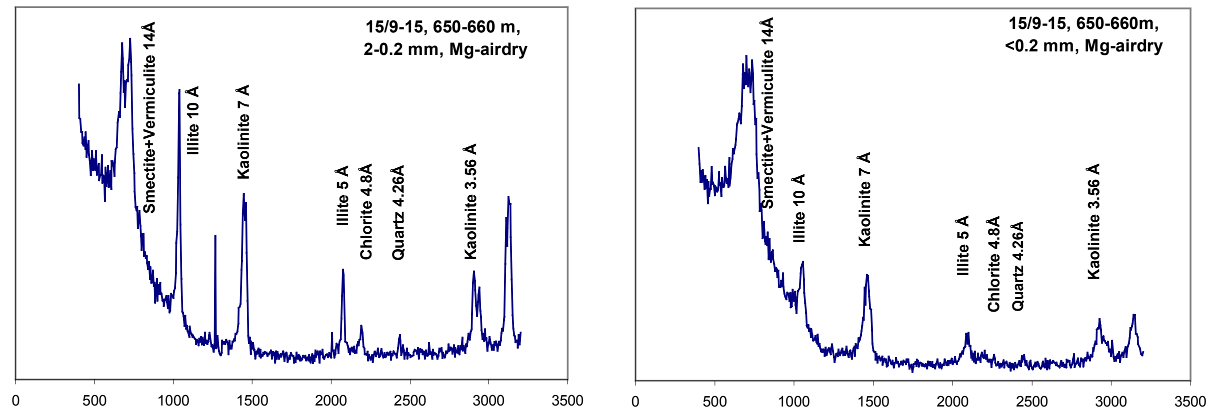


Fig. 4.3. Sleipner well 15/9-18; coarse clay fraction oriented mount X-ray diffraction traces showing identified minerals.

Fig. 4.4. Sleipner wells; coarse and fine clay fractions oriented mount X-ray diffraction traces showing identified minerals and their diagnostic d-spacings.



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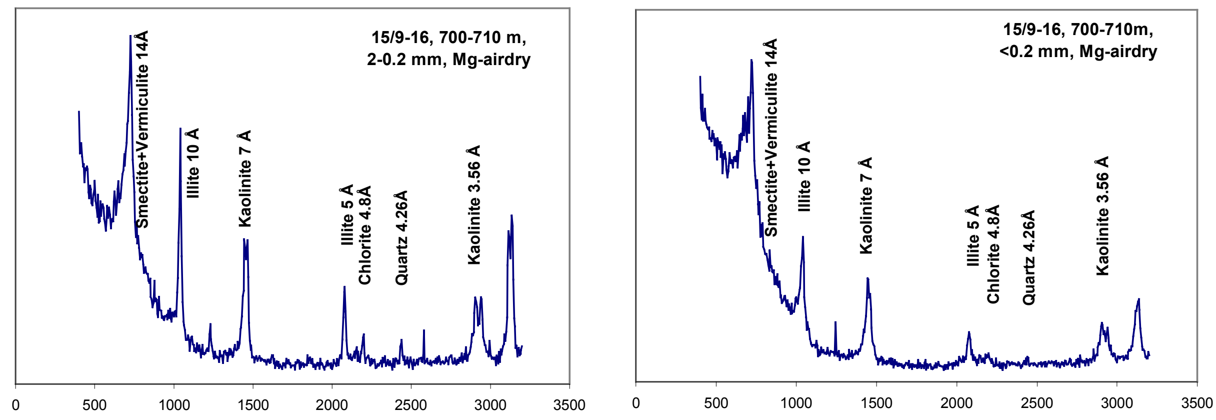
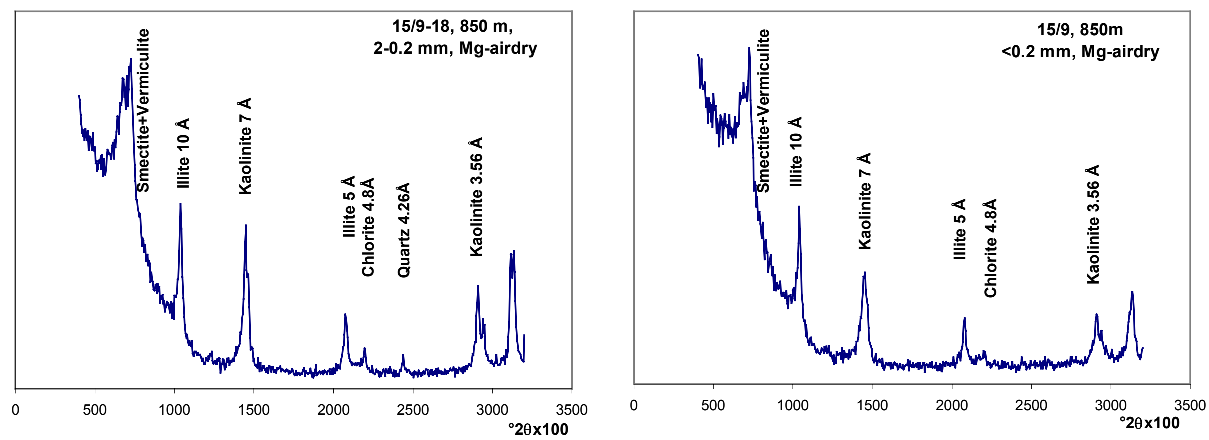
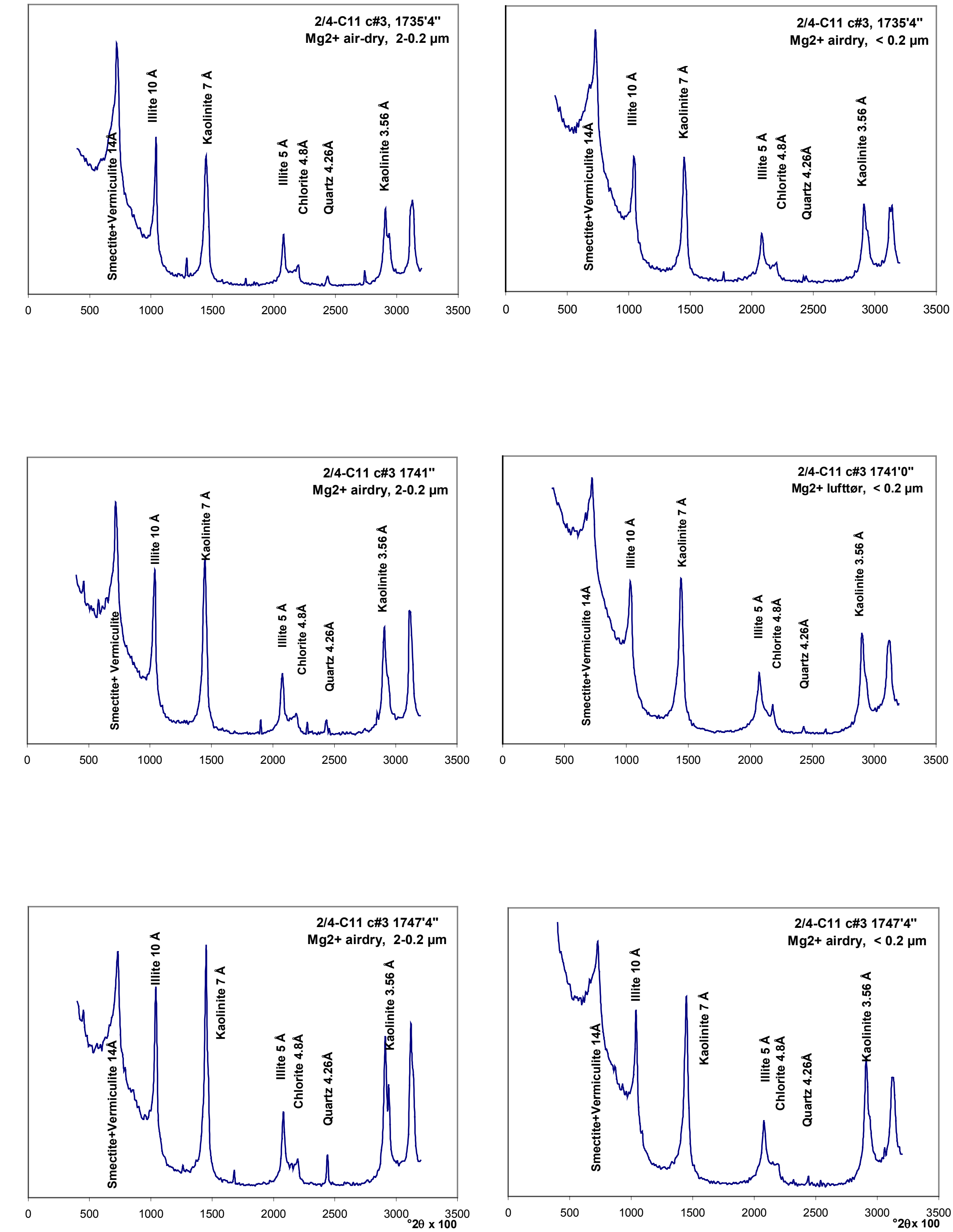


well: 15/9-16



well: 15/9-18

Fig. 4.5. Ekofisk well 2/4-C11; coarse and fine clay fractions oriented mount X-ray diffraction traces showing identified minerals and their diagnostic d-spacings



4.3 Observations

It cannot be completely ruled out that the cuttings from the Sleipner wells represent a more indurated interval of the otherwise soft Nordland shale or even cavings from above. Only 3 samples have been investigated, but it is conspicuous that 3 samples from 3 different wells have approximately the same clay mineralogical composition.

The core samples from the Ekofisk Field resemble clay mineralogically the cuttings samples from the Sleipner Field. The smaller amounts of vermiculite seen in the Ekofisk samples compared to the Sleipner samples are probably due to the fact that the Sleipner samples were investigated in Mg-glycerol treated condition in order to distinguish better between vermiculite and smectite and the Ekofisk samples were investigated in Mg-glycolated condition. Of these two methods the glycerol treatment is preferred for distinguishing between the two minerals, but the separation between vermiculite and smectite is not sharp mineralogically anyway.

In Mg-saturated air-dry specimens, vermiculite and smectite both have a peak at 14 Å. Of the three samples from the Ekofisk core especially the sample from 1735'4'' resemble the Sleipner samples, whereas the other two Ekofisk samples 1741' and 1747'4''contain slightly larger amounts of kaolinite.

The somewhat larger core samples allow a granulometric analysis of the Pliocene clay from the Ekofisk area. From Fig. 4.1 it appears that less than ⅔ of the bulk sample is silt, which justifies the term "shale" be used for the core samples in the sense of Lundegard & Samuels (1980). The two lower core samples have a significantly higher clay content than the upper sample, and this is also reflected in the $N_{2\text{-BET}}$ values, table 4.1. The clay percentage in the Pliocene core is close to the mean value for shale samples as determined by Patchett (1975).

The significant content of smectite (swelling clay) in the Nordland shale, Fig. 4.2-4.4, makes it questionable to perform mercury injection test on cuttings to determine the capillary displacement pressure as pointed out by Krushin (1997, p. 33). Thus it may appear that there is no short cut to estimating the seal capacity of the Nordland shale in the Sleipner area. The requirement is a fresh core sample of the Nordland shale and a petrophysical cap-rock test in the "classical sense". If this cannot be accomplished, it may be considered if the Pliocene shale in the Ekofisk well 2/4-C11 is a fair analogue to the Nordland shale, although there are minor differences in the clay mineralogical composition. Further, it should be observed that sampling from the existing preserved shale core seems to be a logistic challenge.

5. References

Lundegard, P.D. and Samuels, N.O. 1980: Field classification of fine-grained sedimentary rocks. J. Sedimentary Petrol., **50**, p. 781-786.

Patchett, J.G. 1975: An investigation of shale conductivity. SPWLA 16th Logging Symposium, Paper U, 41 p.

Krushin, J.T. 1997: Seal capacity of non-smectite shale, in R.C. Surdam (ed.): Seals, traps and the petroleum system. AAPG Memoir 67, p. 31-47.