

# MINERALOGICAL COMPOSITION OF TWO SILURIAN BENTONITE BEDS FROM SUNDVOLLEN, SOUTHERN NORWAY

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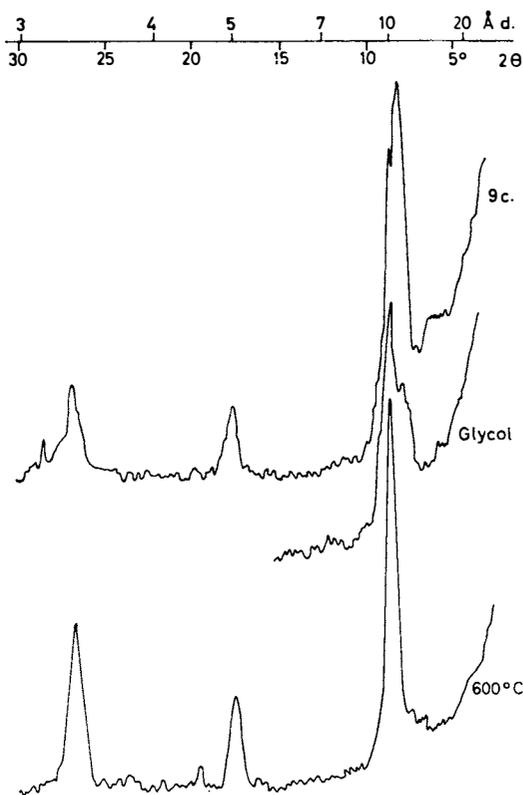
**Abstract.** X-ray, infrared and thermal investigations of the clay-sized fractions showed that the beds mainly consist of dioctahedral illite and a mixed-layer mineral with randomly distribution of illitic and montmorillonitic layers, the sample 9c contained montmorillonite and 9f/g contained chlorite and very fine grained calcite.

## **Introduction and methods**

Two new bentonite beds, found by dr. Nils Spjeldnæs at "riksvei 20" between Vik and Sundvollen have stratigraphical positions in 9c (lower Ludlow) and between 9f and g (lower or middle Ludlow). The purpose of this paper is to describe the mineralogical composition of the clay-sized fractions from these beds.

From each of the two beds, which are a few cm thick, two hundred g was selected for analysis. The beds have a distinct cleavage parallell to the layers, and possess a grey/white (9c) or yellow/green (9f/g) colour.

The minus two micron fractions were oriented by placing films on glass slides before X-raying, using a Philips diffractometer (PW 1050/PW 1051) having Ni-filtered  $\text{CuK}\alpha$  radiation and a scanning speed of  $2^\circ 2\theta$  per minute. After the X-ray diffraction effects were recorded at room temperature, the sample was treated with ethylen glycol, using the method of BRUNTON (1955), and then examined for new diffraction effects. Following this procedure the sample was heated



Diffractometer records of sample 9c.

Fig. 1.

to 100, 350 and 600° C for one hour, air quenched and X-rayed immediately afterwards. All diffraction curves are shown in Figs. 1 and 2, except the curves obtained after heating to 100 and 350° C, which are not included because they do not contribute pertinent information.

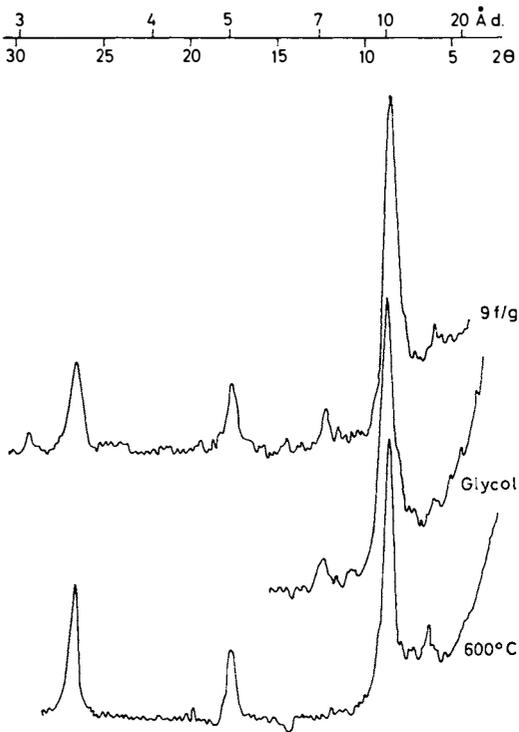
A Geonor apparatus, having a heating rate of 10° per minute was used for the DTA investigations. Chromel/Alumel thermocouples were used together with calcined  $\text{Al}_2\text{O}_3$  as a thermally-inert substance. The DTA curves in Fig. 3 were obtained from bulk samples, and the minus two micron fractions untreated, and after treatment with piperidine (ALLAWAY,

1948). After piperidine treatment the samples were dried on a water bath. For the detection of calcite, the samples were treated with 0.1N HCl for ten minutes at room temperature. Infrared curves were obtained from a Perkin Elmer Spectrophotometer (Model 21).

### X-ray results

Sample 9c showed a strong double reflection at 10.3 and 10.8 Å. The glycol treatment caused a shift in position and a reduction in intensity of the 10.8 Å reflection. After heating only one strong reflection occurred at 10.4 Å. The interpretation of these effects is that the sample contained illite and a mixed-layer mineral of composition

between illite and montmorillonite. In the mixed-layer mineral there was a random distribution of the two types of layers (JACKSON et. al. 1952). In addition to the (001) and (001/001) reflections, the (002) or (002/003) and (003) or (003/004) reflections (BROWN and MACEWAN, 1950) from these minerals are clearly shown. The intensity ratio between the first and second order reflections indicated that the phyllosilicates are of the dioctahedral Al type (GRAFF-PETERSEN, 1961), and the position of the (001/001) reflection gives an approximate proportion of 1:4 for the expandable : non-expandable layers (BROWN and MACEWAN, 1950). The untreated sample had a broad and diffuse reflection-band between 5 and 7° 2θ. This reflection-band appears lost in the background blackening after glycol treatment, indicating that the sample contained a certain amount of montmorillonite. In the untreated sample there was a weak but distinct reflection at 3.14 A, which has not been explained.



Diffractometer records of sample 9f/g.

Fig. 2.

Sample 9f/g, like sample 9c, contained probably dioctahedral illite and a small amount (clearly less than in 9c) of the mixed-layer mineral. The untreated sample had a broad reflection at 14.4 A, and a more distinct reflection at 7.2 A. There was no change in these two reflections following the glycol treatment. After heating the 7 A reflection disappeared and the 14.4 A reflection had changed in position to 13.8 A, while at the same time increased in intensity, which demon-

strates that the sample contained chlorite (BRINDLEY and ALI, 1950 and WEISS and ROWLAND, 1956).

### DTA results (Fig. 3.)

DTA curves of the bulk samples are similar to those described by GRIM and ROWLAND (1942), WEAVER (1953), BYSTRÖM (1956) and HAGEMANN and SPJELDNÆS (1955). These authors state that the first endothermic peak after 100° is caused by the loss of adsorbed or inter-layer water, and the endothermic and exothermic peaks between 900 and 1000° C are caused by the destruction of the 2:1 structure (illite or montmorillonite) and recrystallization, respectively.

Different explanations are given for the endothermic peak(s) in the temperature region between 500 and 800° C.

Weaver found that samples containing chlorite have double hydroxylendothermic peaks in this temperature region. One of the samples (9f/g) contained chlorite (X-ray), but because the DTA curve for this sample had only one distinct peak and the curve for the other sample (9c) had two, this explanation is not plausible. Grim and Rowland, on the other hand, have shown that synthetic mixtures of illite and montmorillonite gave peaks at about 600 and 700° C, which are characteristic of illite and montmorillonite, respectively. They believed that illitic and montmorillonitic layers in a mixed-layer mineral can lose their hydroxyl-water at two different temperatures. Byström found that Grim and Rowland's explanation was not plausible for bentonite material from Kinnekulle, "since the relative sizes of the two effects show no relation to the proportions of the layers found by other methods". Byström's explanation was that the reaction is dependent upon the kind of exchangeable ions in the interlayer positions. It is worth mentioning that the curve (9c) with two peaks in this temperature region also has two peaks in the low temperature region (100–300° C). According to HENDRICKS *et.al.* (1940), the second peak about 200° C is caused by the loss of water adsorbed to the divalent Ca- or Mg-ions.

HCl treatment of sample 9f/g shows that calcite impurities enlarged the third endothermic peak at 900° C. The DTA curves for the less than two micron fraction showed that dehydration and destruction temperatures decrease with decreasing particle size.

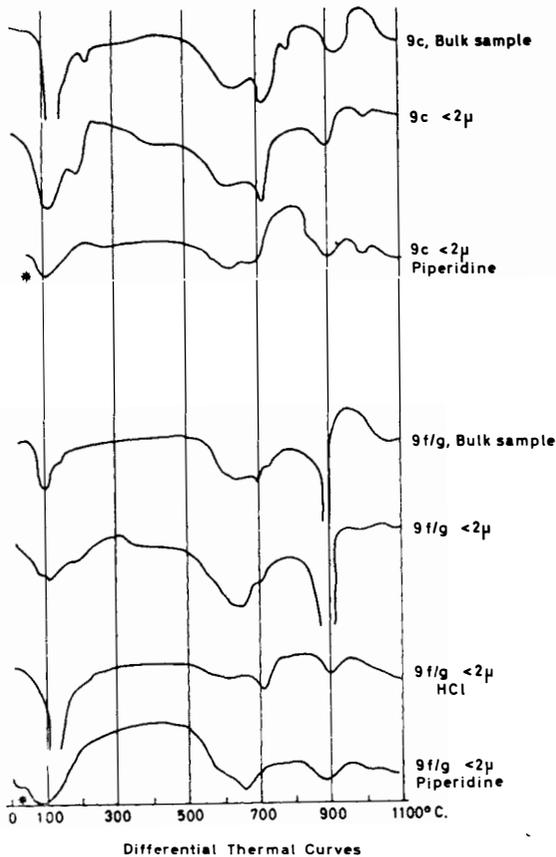


Fig. 3.

The curves for material treated with piperidine are characteristic for samples with montmorillonite (ALLAWAY, 1948), but it is not possible to state whether it is pure montmorillonite, or a mixed-layer mineral having this type of layers.

In conclusion it can be said that the curves are consistent with the presence of illite and montmorillonite, either as a mixture or a mixed-layer mineral. In sample 9f/g is calcite easily detected.

### Infra-red results

The infra-red curves, obtained from samples diluted in Nujol, are typical for phyllosilicates (Fig. 4). The band with the highest wave-

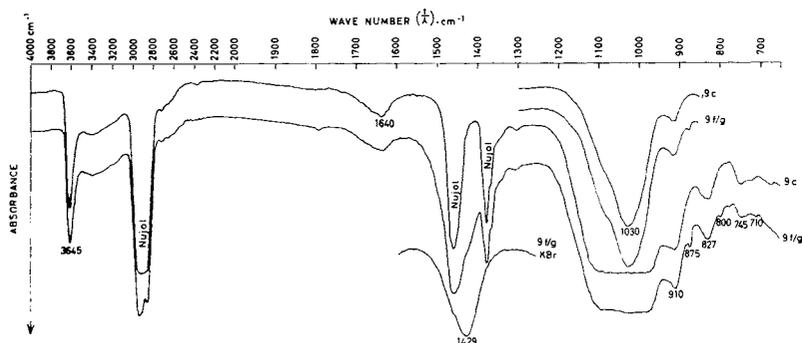


Fig. 4.

number,  $3645\text{ cm}^{-1}$ , is probably caused by excitation of structural OH groups in the phyllosilicates. A broad band about  $3400\text{ cm}^{-1}$  can be attributed to adsorbed water, which also causes another band, corresponding to the deformation vibration of water, about  $1640\text{ cm}^{-1}$  (SERRATOSA, 1962) that is also clearly seen in the two curves. The strongest band, at  $1030\text{ cm}^{-1}$ , is probably caused by the  $\text{SiO}_4$  groups, and its position indicates that the phyllosilicates are of the dioctahedral type (STUBICAN and ROY, 1961). This structure is also indicated by the presence of one absorption band at  $910\text{ cm}^{-1}$ , because this absorption can be assigned to the vibration of a proton associated with dioctahedral layers (STUBICAN and ROY, 1961 and SERRATOSA, 1962). There is one distinct difference between the two samples. Sample 9f/g has a very strong band at  $1429\text{ cm}^{-1}$  and two very weak bands at  $875$  and  $710\text{ cm}^{-1}$ . These bands, which are not present in sample 9c, are probably caused by the activation of  $\text{CO}_3$  groups (ADLER, 1951). DUYCKAERTS (1959) has shown that when the dimensions of the carbonate particles are reduced below 10 microns the relative values of apparent extinction are changed rapidly (the band at  $1429\text{ cm}^{-1}$  is increasing very rapidly in apparent extinction, while the other two bands are increasing more slowly). The relative values of apparent extinction for these bands in sample 9f/g shows that the carbonate particles are clearly much less than one micron.

### Summary and conclusions

X-ray, DTA and infra-red results show the presence of dioctahedral phyllosilicates, which are of two different types: illite and a mixed-

layer mineral having a random distribution of illitic and montmorillonitic layers. The approximate proportion for expandable: non-expandable layers is 1:4.

The presence of calcite in sample 9f/g was established by DTA- and infra-red methods. Relative values of apparent extinction showed that the calcite grains had a very small size, consequently line broadening may be an explanation of why calcite reflections were not found by the X-ray analysis. A low DTA destruction temperature (900° C) can also be explained by the small size of the particles.

Chlorite was proved to be present in sample 9f/g and montmorillonite in sample 9c by X-ray.

The same type of mixed-layer mineral has been found in Ordovician K-bentonites from Pennsylvania (WEAVER, 1953) and Billingen, Sweden (BYSTRÖM, 1957).

Byström says: "One of the reactions which seems to have occurred in these bentonite beds is a formation of muscovite layers where potassium has been available. The magnesium set free has formed chlorite layers with the remaining montmorillonite".

This is supported by my results since I found chlorite, illite and only a small amount of mixed-layer mineral in sample 9f/g, while sample 9c, which did not contain chlorite, had a high content of mixed-layer mineral and a small amount of montmorillonite. DTA showed that the dioctahedral phyllosilicates in sample 9c had Mg- or Ca-ions in interlayer positions.

Weaver, Byström and I found the same ratio, 1:4 for expandable: non-expandable layers, in the mixed-layer mineral, indicating that this is a relatively stable intermediate composition in the formation of illite from montmorillonite.

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