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Survey of Swedish buffer material candidates and methods for characterization

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SURVEY OF SWEDISH BUFFER MATERIAL
CANDIDATES AND METHODS FOR CHARACTERIZATION

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SUMMARY

The major criteria for natural clayey materials to qualify as candidate buffer materials are concluded to be a high content of 2:1 clay minerals - smectite, hydromica - and accessibility of very large quantities of fairly homogeneous raw material. Only four major source materials appear to fulfil these requirements, namely the Vallåkra, Kågeröd and Fyledalen clays, as well as the Quaternary moraine clay from the Lund-Landskrona area. The three first-mentioned materials represent the Mesozoic formations in Scania. The Vallåkra clay has smectites and kaolinite as major clay minerals. The beds have a very large extension and offer possibilities of finding substantial quantities of very smectite-rich portions. This is the case at Margreteberg but in this area iron compounds appear to coat the smectite aggregates and prevent them from expanding spontaneously. Attempts have been made to remove this cementing substance and there is good hope to find an effective industrial process of low cost.

The Kågeröd clay has evolved as a major candidate because the clay fraction, which forms 20-30 % of the bulk material, is almost exclusively composed of smectites. The extension of the beds is enormous and a further, detailed prospection is recommended in order to find areas which are particularly rich in clay.

The Fyledalen clay has a high clay content with kaolinite and hydromica as major minerals. The conclusion is that this is the most easily accessible large and rich source of hydromica in Sweden.

In addition to these major, relatively pure materials, weakly smectitic moraine clays should also be considered as potential candidates since their granular composition make them ideal for backfilling purposes.

One of the major questions in characterizing clays for use as buffer materials is to make reliable quantitative determinations of the clay mineral content. A major goal of the present study was to identify the most accurate method that is used internationally, and Reynolds' technique was focussed on early in the project. It has turned out that his method is very accurate when using a small quantity of pyrofyllite as reference substance, and a refined version of the method is therefore suggested as an SKB standard in future prospecting and characterization of buffer materials.

1   SCOPE OF STUDY

Clay materials need to be characterized for judging their possible use in future repositories as canister envelopes, and as plugs in boreholes, shafts and tunnels, as well as for backfilling of tunnels and shafts. The key point is to define the functional requirements of the various buffers since this determines the required properties and, in turn, what the composition and density should be like. Once this has been settled it is possible to identify the major types of buffer materials that may be considered for practical use. The remaining question is then to select sufficiently stringent but also simple ways of characterizing the materials. This line has been followed in the present report, which serves as a pilot survey of major Swedish clay candidates with special reference to their composition and physical state, without specifying their detailed physical properties.

Quantitative clay mineral analysis offers particular difficulties since there is no unanimous, generally accepted laboratory technique for such determinations and much of the work reported here has involved testing of possible methods for such analyses. A promising method for determination of the content of clay minerals in general, and smectites in particular, has been found suitable for standard analyses. It is fully described in the report.

In summary, the study is focussed on the following two major objects:

i)   A survey of potential Swedish buffer material candidates characterized with respect to their potential usefulness as barrier materials as indicated by their mineralogy, granulometry, stratigraphy and accessibility. Since most available geological data refer to the southernmost part of Sweden, i.e. Scania (Skåne), the survey mainly covers that part.

ii)   Quantitative clay-mineral analysis. A review of various quantification methods and definition of a suitable standardized method which is suggested as a suitable SKB standard.

2   PHYSICO-MECHANICAL CRITERIA OF BUFFER MATERIAL
        CANDIDATES

2.1   GENERAL

Functional criteria naturally form the basis of the choice of clay barriers for
repositories. They are related to three main applications of clay technology: 1)
near-field shielding of canisters with high level wastes, 2) backfilling of shafts and
tunnels, 3) plugging of boreholes, shafts and tunnels, and 4) sealing of rock frac-
tures.

2.1.1   Near-field shielding of canisters in boreholes

The functional requirements of canister-embedding buffer materials, sometimes
termed "overpack", are listed below together with the required properties of the
clays (cf. Fig 1).
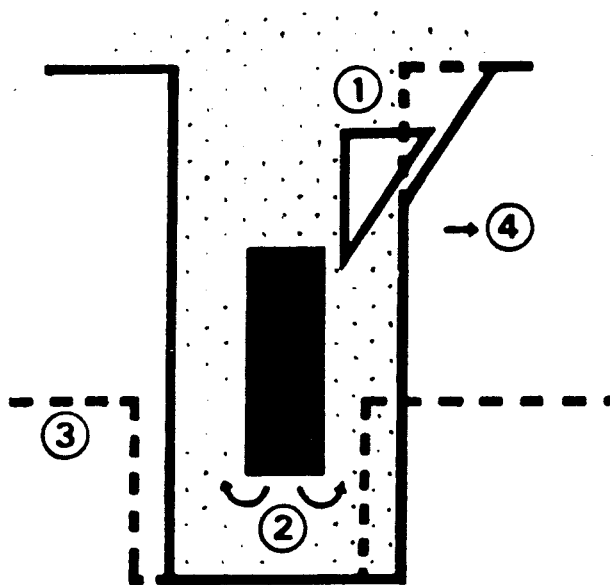
Fig 1.   Schematic section through deposition hole with canister. Support of
            loose rock ① and small settlement ② requires stiffness. Minimum
            stresses in canisters at rock shear ③ calls for soft backfill. Low
            hydraulic conductivity and diffusivity are basic requirements ④

Function	Required properties
* Sufficient strength to support rock and minimize settlement	● High bulk density ● Clay mineral composition not critical
* Sufficient ductility to reduce impact of rock displacement on canisters	● Moderate bulk density ● Clay mineral composition not critical
* Low hydraulic conductivity to delay canister corrosion and minimize migration of radio-nuclides by flow	● Very high bulk density for non-smectite clay ● Moderate to high density of smectitic clays
* Low ion diffusion rate to delay canister corosion and minimize migration of radionuclides by diffusion	● Very high bulk density for non-smectite clay ● Smectite-rich clays for cation uptake through ion exchange ● Kaolinite-rich clays for anion uptake through ion exchange
* Sufficient heat conductivity to minimize canister and clay temperature	● Quartz as accessory mineral
* Expandability to fill up the space between canister and rock and establish integrated clay/rock contact	● Very high density of non-smectite clays ● Moderate to high density of smectitic clays

In addition, it is considered to be valuable if the embedding substance has the ability of retaining radionuclides emerging from the canisters, through ion exchange mechanisms. It should be mentioned that the listed requirements apply also to concepts with different geometry.

2.1.2   Backfilling

The functional requirements of far-field buffer materials, i.e. backfills in tunnels and shafts (cf. Fig 2), or slots like that of the WP cave concept, are less rigorous

than those of the canister-embedding substance. The major ones are listed below together with the required properties.

Function	Required properties
* Sufficient strength to support rock and to prevent compression under its own weight	● High bulk density ● Mineral composition not critical
* Lower hydraulic conductivity than of the surrounding rock	● Very high bulk density of non-smectite backfill ● High bulk density of smectitic backfill
* Expandability to fill up the space and establish integrated clay/rock contact	● Very high bulk density for non-smectite clayey backfill ● High bulk density of smectitic backfill
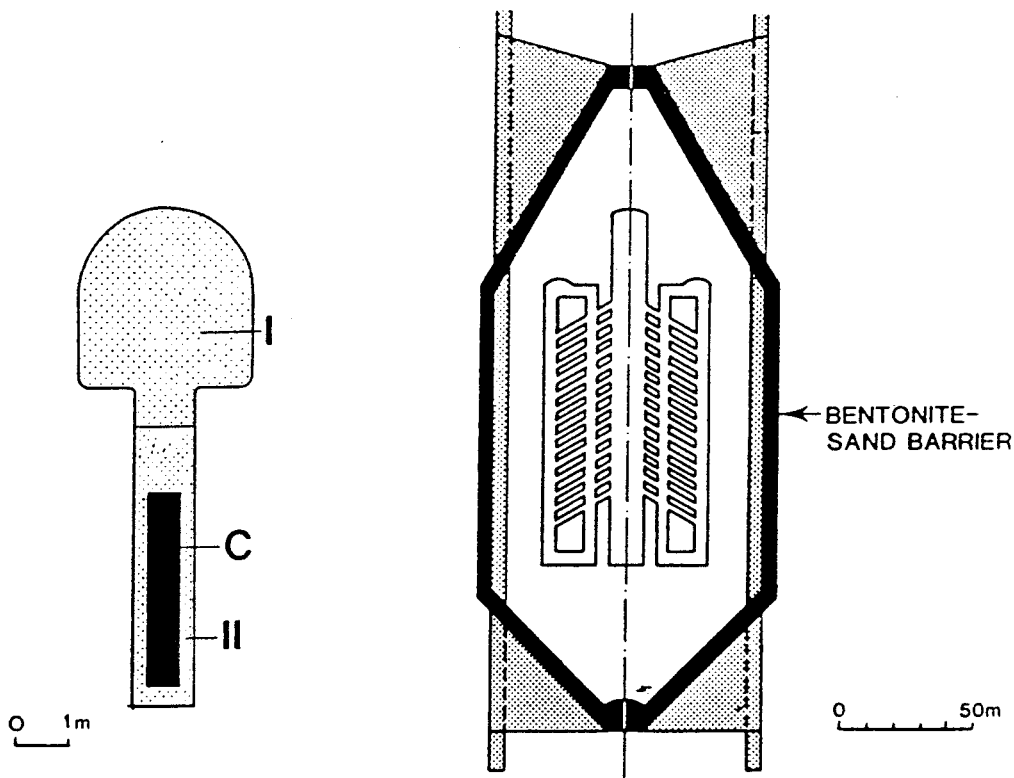
Fig 2.   Schematic picture of backfilled tunnel with deposition hole according to the KBS3 concept (left), and the backfilled slot of the WP-cave

2.1.3   Borehole, shaft-, and tunnel plugging

Boreholes as well as tunnels and shafts may act as short-circuits for groundwater driven by regional hydraulic gradients if they are not properly plugged. Even if they are effectively sealed and given a lower hydraulic conductivity than the surrounding rock, they may still serve as water conduits because of the disturbed zone that is created in the rock mass adjacent to the excavation as a result of blasting and stress redistribution, and special arrangements may be required to shunt off local groundwater currents.

Effective plugging is required to seal off local penetrations of high-conductivity rock zones and this may involve the cutting of slots (Fig 3).
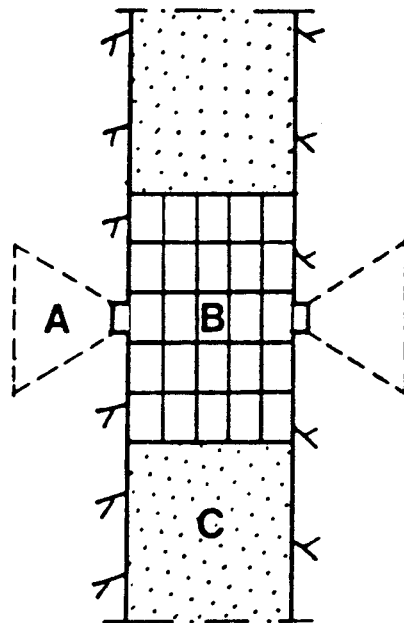
Fig 3.   Shaft or tunnel plugging. A) Grouting, B) Effective, expansive sealing in the form of highly compacted smectite-rich clay, C) Backfill (1)

The functional requirements are listed below together with the required properties.

Function	Required properties
* Low hydraulic conductivity	● Smectitic composition
* Sufficient expandability to fill up the space and establish integrated clay/rock contact ("bond strength")	● Smectite-rich composition ● High bulk density
* Low erodability where erosion is expected	● Grain size composition according to suitable "filter criteria" ● Certain amount of angular ballast material

2.1.4   Rock grouts

Clay grouts should serve as effective seals and need to be low-viscous to be groutable. Also, they must resist erosion by flowing groundwater. Functional requirements are given below together with required properties.

Function	Required properties
* Low viscosity while being injected and high viscosity after injection	● Thixotropic behavior, smectite-rich substances
* Low hydraulic conductivity	● Smectite-rich compositions
* Low erodability	● Certain content of fine-grained quartz powder

2.1.5   Conclusions

If the various criteria are put together we see that the most essential physical properties are the hydraulic conductivity and the swelling potential. It was concluded early in the Swedish search for suitable buffer substances that certain clays or clay-based materials would best fulfill these requirements and smectites were preferred from the very start of the investigation, especially for use as

canister envelopes and for plugging purposes. However, the limited domestic resources has called for a closer examination of other clay types as well. Thus, clays rich in hydromica may be acceptable for certain functions since they have a reasonably low hydraulic conductivity and some swelling ability. Kaolinitic clays have a relatively high hydraulic conductivity but serve as rather effective anion exchangers, which is a valuable property for catching critical radionuclides. The final choice may well involve mixing of several components or the use of natural soils of low purity.

It is very essential to realize that if smectite is the major constituent of the buffer, the bulk density does not need to be very high to yield the required sealing and expansive properties, while a smectite-poor buffer material must be given a very high bulk density. In principle, therefore, the search for smectitic clays should have a very high priority, which indicates that the most important point in the characterization of potential candidate clays is to get a quantitative measure of the smectite content.

3   SWEDISH BUFFER MATERIAL CANDIDATES

3.1   GENERAL

Clays occur in a wide range of environments and are considered as the most important industrial minerals that are quarried. They form a complex group with several mineral constitutents which reflect the environment and geologic conditions under which they were formed and which are determinants of their practical use. In Sweden clays have played a less important role as industrial minerals than in other countries like USA, West Germany and Italy. A much increased technical application is foreseen, however, particularly in the field of engineering geology.

Since the physical properties and thus their potential usefulness as buffer materials is very much dependent on the type of clay minerals we will start here by defining the major representatives of this group.

3.2   MAJOR CLAY MINERALS

3.2.1   General

The clay minerals contain tetrahedral and octahedral sheets as their basic structural units. The tetrahedral sheets consist of Si and Al ions surrounded by four oxygens. The octahedral sheet consists of Mg, Fe or Al ions surrounded by six oxygens and hydroxyls. The coordination and organization of these main units are somewhat different in most clay minerals.

We will only consider four major clay types in this report: the kandite group (especially kaolinite) the illites or, rather, the hydromicas, the smectite group and the chlorite group. These four clay types are predominant in almost all natural clay formations and represent different properties of practical importance.

3.2.2   Kandites

The kandite group, kaolinite, dickite, nacrite, and several hydrous forms, consist of a 1:1 layer dioctahedral structure with minimum substitution within the structure. This means that there is little deviation from the ideal formula, $Al_2Si_2O_5(OH)_4$ but occasionally some Fe^{3+} substitutes for Al^{3+}. The limited lattice substitution also requires very little cation adsorption in interlayer sites to

balance charge deficiencies. The greatest difference among the members of the kandites is the mode of stacking and disorder of the 1:1 layers:, one layer per unit cell results in triclinic kaolinite while two stacked layers per unit cell produces the monoclinic structural varieties of dickite and nacrite.

Kaolinite is formed in nature by acid reactions in an anaerobic medium. It is most abundant in warm moist climates and is a prominent constituent of oceanic sediments in the equatorial belt. The genesis of the kaolinite group of clay minerals is thus usually related to intensively weathered soil profiles in subtropical to subhumid climates.

Temperatures below 200°C, low pH, moderate silica activity, and low alkali activity favors the stability of kaolinite.

3.2.3   Hydromicas ("illite")

The hydromicas represent the 2:1 organization structure of the crystal lattice, i.e. one octahedral and two tetrahedral sheets bound together. If one of the silicons in each group of four tetrahedrons are replaced by aluminum, leaving the octahedral layer intact, we obtain the muscovite structure, provided that the induced lattice charge is balanced by interlayer potassium ions. If the muscovite structure of the type $K_2Si_6O_{20}(OH)_4$ is altered so that part of the potassium is replaced by hydronium ions (H_3O+) we obtain the mineral hydromica. It has long been known that this mineral does not have any definite chemical composition but obeys the general rule:

$$M_y^ISi_{(8-y)}Al_yO_{20}(OH_4) \ (M^{III}, M^{II})_y \tag{1}$$

This formula, originally given by Maegdefrau, has been suggested for the rock-forming micas, the hydrous micas and the smectites. M^I represents monovalent cations, while M^{II} stands for magnesium and divalent iron and M^{III} for aluminum or trivalent iron. For $y=0$ the formula represents the smectite group and for $y=z$ it is valid for rock-forming micas. The hydrous micas have an y-value between 1 and 2, which well illustrates that this mineral represents an intermediate, not well defined stage between micas and smectites.

The hydromicas are the most common clay minerals found in sedimentary rocks and those undergoing low-grade metamorphism. Along with variations in

chemical composition, there are several variations in structural type. They are also commonly associated with chlorite, biotite, and quartz rocks that have not been exposed to temperatures exceeding 200°C according to Velde (2), who also stated that hydromicas also dominate the argillic zone of hydrothermal alteration, generally very close to the heat source. Authigenic hydromica is also found in deep sea sediments and in sandstones that have not experienced high temperatures. All this demonstrates that these minerals are found over a wide range of environments.

3.2.4 Smectites

The smectite group includes some of the most important clay minerals in natural soils. Its structure can be described with the formula given by Maegdefrau. Aluminum and magnesium occupy the cation sites in the octahedrons of the most common smectite mineral montmorillonite. Substitution of Si^{4+} in the tetrahedrons by Al^{3+} results in the smectite member beidellite (3).

Substitutions of this sort result in a large capacity to adsorb cations, water and organic compounds due to the negative net charge of the lattice structure.

Smectites are very common at and near the earth's surface, in fault zones, on the ocean floors, as authigenic cement in shallow-buried sandstones and shales, and in certain hydrothermal zones. However, in most cases the smectites are not pure but have a small percentage of other clay minerals interstratified within the smectite packs. Thus, various investigations of sediments and diagenetic smectites indicate that nonexpandable sheets are frequent. The only pure smectites appear to be associated with freshly devitrified volcanic glass in recent bentonites, recent authigenic cement in very porous sandstones, and recent hydrothermal alteration products. Given enough time it is likely that even these clays begin to incorporate other layers within the smectite packs.

It is generally claimed that smectites form stable phases at the earth's surface and at slightly elevated temperatures. It is clear, also, that they can be formed at higher temperatures as demonstrated experimentally, though equilibrium has not been certified and reaction reversibility has not documented (4). In very short hydrothermal experiments (runs usually are less than 14 days), smectites are identified at temperatures of 300-400°C (3) but in experiments of longer duration the maximum temperatures at which smectites are still stable decrease signifi-

cantly (5). This is in agreement with observations made in many low-grade diagenetic sequences in which smectites begin to disappear at temperatures of about 100°C.

A successive increase in the frequency of non-expandable hydromica layers of I/S clays with increased burial depth and temperature is observed in sedimentary basins of all ages throughout the world. The transformation of a smectite layer to a layer of hydromica requires an increase in the negative charge in the structure and the incorporation of potassium in the interlayer which thereby collapses to a 10 Å spacing and irreversibly expels interlayer water. The increased negative charge is usually created by Al^{3+} for Si^{4+} substitution in the smectite tetrahedral sheet, though substitution within the octahedral sheet of Mg^{2+} for Al^{3+} or the reduction of Fe^{3+} to Fe^{2+} can also increase the structure's charge deficiency (4). A generalized equation for the "illitization" of smectite is:

$$Smectite + Al^{3+} + K^+ -Si^{4+} -OH^{-1} \rightarrow Hydromica + Si^{4+} + H_2O$$
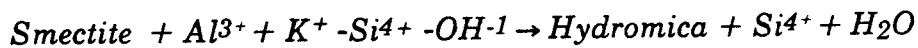
A classical question is whether alteration of smectite to hydromica is controlled by a chemical equilibrium criterion or by reaction kinetics. If the process is controlled by the first-mentioned criterion, the degree of transformation, i.e. the percentage of hydromica layers in the I/S mixed-layer clay, is determined by the prevailing temperature, pressure, and chemical conditions. The kinetic argument implies that hydromica is an inevitable reaction product of smectite and that the rate at which the end product is formed only depends on rate constants. Determining factors include temperature, thermal history of the sediment, and the presence of inhibiting ions in the interstitial fluid. An often cited support of the kinetic hypothesis is Grims statement in 1951 that there is relatively less smectite in ancient sediments. However, montmorillonite is quite common in Jurassic and Permian sediments of up to 1500 m thickness and is present as I/S minerals in Middle and Lower Orodovician and Upper and Middle Cambrian sediments. Volcanic material is the major source of smectite and it has been demonstrated that there has been less volcanism during the early Paleozoic than in the Tertiary when much smectite were formed. Furthermore, many of the older Paleozoic bentonitic beds were altered by regional metamorphism.

As pointed out by Meyer and Howard, plots of I/S expandability against temperature reveal that there is a wide range in I/S clay mineral expandability at any given temperature in different basins and also within one and the same basin. The

first occurrence of illite layers in the smectite clays appears to be around 50-80°C (although this cannot be safely evidenced), while some expandable layers seem to persist at temperatures as high as 250°C. The major change in I/S expandability between 90 % to 20 % expandable layers takes place in a fairly short temperature range. There is actually substantial support of the hypothesis of a minimum reaction temperature associated with the activation energy that is required to break lattice bonds, which, in turn, is the first step in the creation of excess layer charge and subsequent collapse of the expandable layer.

Meyer & Howard correctly stated that the chemistry of the interstitial fluids also plays a significant role in the rates of "illitization". They demonstrated that within intercalated sandstones and shales buried at the same depth and with equal thermal history (inferred from sedimentation rates) the I/S clays found in sandstone laminae are more expandable than those found in adjacent shale lami-nae. The only difference between the two environments is the greater salinity in the sand porewaters, which indicates that the presence of abundant Na^+ and Ca^{2+} in interlayer space effectively competes with K^+ and retards the formation of hydromicas.

3.2.5   Chlorites

The chlorites exhibit a high degree of atomic substitution, the general formula being:

$$(Mg, Fe, Al)_6 (Al, Si)_4 O_{10}(OH)_8 \qquad\qquad (2)$$
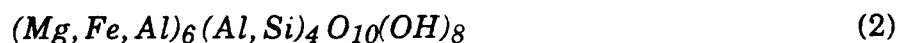
In this general expression Mg and Fe are mutually replaceable and ferric iron is often present. The mica layer is negatively charged, the charge being neutralized by a positive charge of the $Mg(OH_2)$, i.e. a brucite layer in the chlorites thus corre-sponds to the potassium ions in the micas.

The chlorite group of clay minerals is associated with a wide range of depositional environments and diagenetic grades. In shales, their appearance as authigenic minerals is first noted around 75°C and they persist beyond 350°C (4). The only generalizations about chlorite composition and chemical stability that can be made at present are that Fe-rich and aluminum dioctahedral chlorites are more stable at low temperatures, while Mg-rich trioctahedral chlorites tend to be more stable at higher temperatures. Chlorite composition otherwise appears to be more

dependent upon host rock and interstitial fluid chemistry than the low temperatures found during diagenesis.

In many sandstones at shallow burial depths, chlorite is present as an authigenic cement coating quartz grains (6). The occurrence of authigenic chlorite cement is often associated with the disappearance of various unstable components in the sandstone: rock fragments, plagioclase feldspar, and heavy minerals. Chlorite is also observed replacing feldspars and rock fragments in some sandstones. With increasing diagenetic grade, authigenic cements are replaced by another chlorite species which is often a major component of graywacke matrix.

In shales, non-detrital chlorite appears at temperatures around 75°C along with a loss of kaolinite and progressive illitization of smectite (4). The association of chlorite appearance with illitization is evident in Tertiary sediments of the Gulf Coast where authigenic chlorite is observed at 70°C in some Oligocene shales and at 80°C in Miocene shales. The chlorites and smectites in these shales have different Fe/Mg ratios, indicating that chloritization is a solution-reprecipitation reaction and that Mg- and Fe- release rates from dissolving smectite are unequal.

The appearance of chlorite in many shales has also been correlated with the reduced amount of kaolinite over a fairly wide range of temperatures. The kaolinite-to-chlorite reaction, though never actually observed to be a simple one-step process, is concluded to begin at temperatures as low as 100°C, while the high end of the temperature range for this reaction is believed to be around 150-200°C (4). Chlorite found in the kaolinite-rich claystones of Australia is associated with temperatures as high as 200-250°C. The formation of corrensite (mixed-layer smectite/chlorite) from smectite is believed to occur around 170-220°C. We conclude from this that chlorite has a wide range of composition and is found under quite different environments, and is therefore not easily placed on activity diagrams. Sedimentary chlorites are found in primarily alkaline waters, hence high cation/ H+ ratios, and are usually not associated with free silica.

3.2.6   Comments

The low permeability and self-healing properties that are required for buffer materials are best offered by clays rich in smectite because of the strong gel-forming properties of these minerals. Since hydromicas are intermediate to ordinary micas and smectites, both with respect to their crystal lattice constitu-

tion and colloid-chemical properties, they serve as second-best constituents. Kandite and chlorite particles have physical properties that do not differ very significantly from those of very fine mica, feldspar and quartz grains. It is just their small size that make them form narrow, tortuous passages and therefore a relatively low hydraulic conductivity when densely layered, but a very effectively compacted mass of feldspar or quartz particles with a grain size distribution that is characteristic of moraine clays would also yield a very low hydraulic conductivity. Smectites and hydromicas are thus of particular importance and the clay characterization that we are concerned with should refer to the content of these constituents in the first place. It is obvious, however, that also the granulometry is of substantial importance and this brings moraine clays into focus, particularly if the contain smectite minerals.

3.3 METHODS OF STUDY

3.3.1 General

Prospection of Swedish clay buffer materials is a two-step procedure, the first part being to identify suitable types of materials as concluded from available general geological data concerning the mineral composition, homogeneity, accessibility, and need of processing. Such an initial survey is made in this report. Once geological strata of potential use have been identified, detailed regional investigations should be made comprising systematic geophysical measurements including drillings, and systematic sampling and laboratory determination of relevant data for characterization with respect to the most important properties, i.e. the mineral composition in general and the smectite content in particular. This activity requires access to suitable techniques which are presently not standardized but for which a set of laboratory tests are suggested in this report.

3.3.2 Present survey

A standard investigation program has been applied for the general survey, the major steps being those shown below:
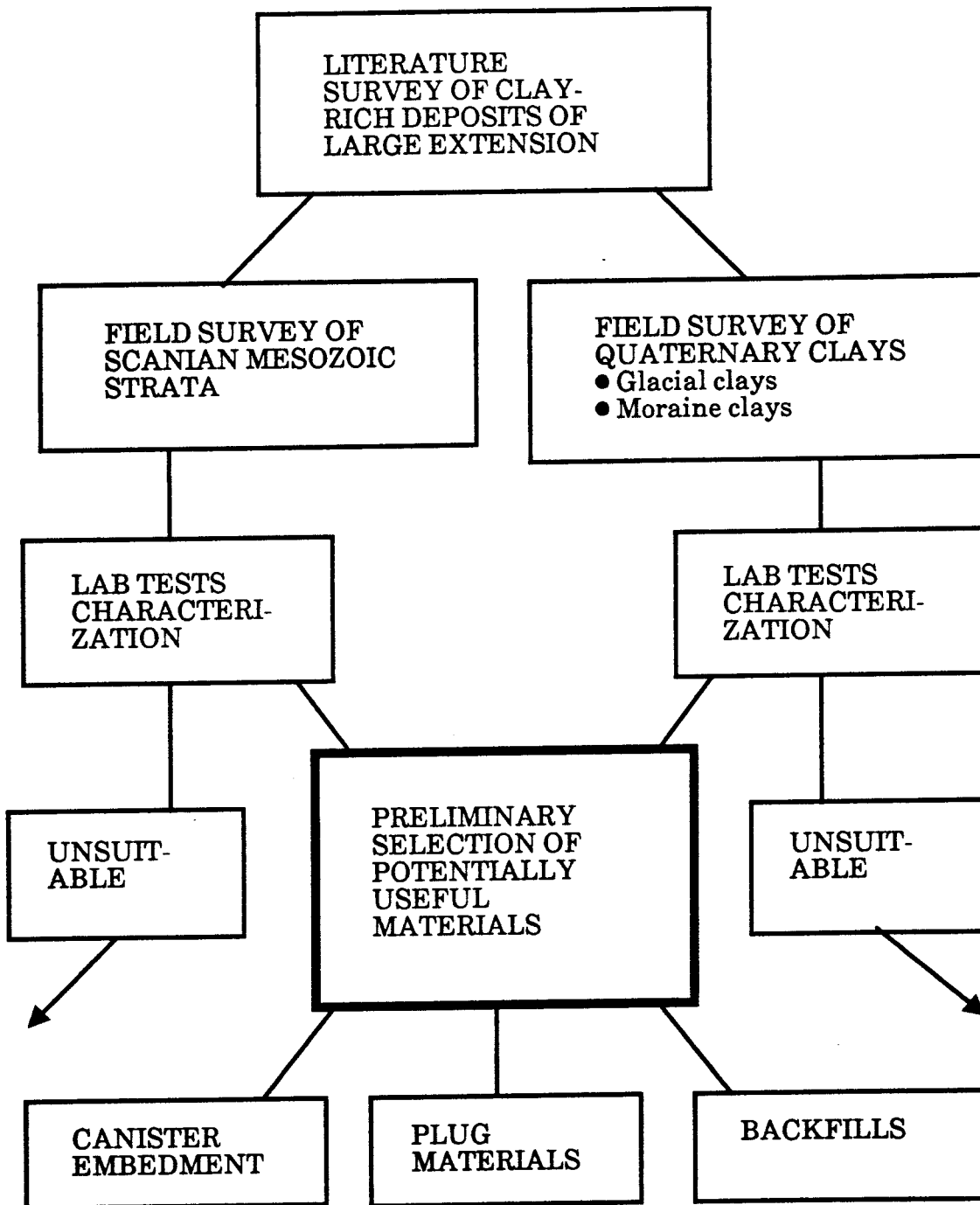
3.3.3   Review of previous work

The clays of Scania have been investigated in different ways, mainly strathi-graphically. Relatively few publications deal with clay mineralogy and petro-

physical properties (7, 8), but a substantial amount of facts is available through the companies which utilize the various substances.

We see from this scheme that clay materials for rock grouting have not been included and the reason for this is that the required amount of such particularly smectite-rich clays is very small and can be obtained by refining smectitic materials for canister embedment or plugging.

Many of the publications are unfortunately more than 35 years old and therefore not accessible. Still, it has been possible to make a rather comprehensive litera-ture review of the stratigraphy, lithology, geochemistry, and petrophysical character of a number of potential candidates.

A survey of different methods of analysing clays using a standard program was also conducted. More than eighty more or less interesting references emerged from a computer-aided search based on the six most comprehensive literature reference lists in the world, such as Georef and Geoarchive.

3.3.4   Field investigations

An inventory of the field accessibility of the different candidates was performed before any sampling of clay was carried out. A number of promising outcrops were chosen of each candidate material and they were subsequently examined and sampled in connection with field excursions. Sampling was only made from origi-nal material which was unaffected by surface weathering and contamination by Quaternary soils. In some instances rather deep excavation by hand had to be made.

Core material (Margreteberg) was used for one candidate because suitable out-crops were not available.

3.3.5   Laboratory analyses

Petrophysical characters related to the natural state of the materials, e.g. moisture, bulk density and liquid limit, were determined prior to more detailed mineralogical analyses. These initial tests served to characterize the materials sufficiently well to make a first critical selection. The various tests are described below in chronological order.

3.3.5.1 Natural water content and bulk density

The natural water content w (%) is defined as the ratio of the mass of porewater to that of the solid minerals, expressed in weight percent. The water content per se is not a measure of the clay mineral composition but with due respect to the clay content (see par. 3.3.5.3) it reflects the stress history and the overconsolidation ratio, which are in turn indicative of the clay content. The hydrophilic character of organic constituents can raise the water content, and this needs to be considered in forthcoming, detailed analyses.

The tests were made according to the standard procedure. Thus, a 30-50 g bulk sample was dried for approximately 24 hours at 105°C before the subsequent weighing and evaluation of the water content.

The bulk density (ρ), i.e. the ratio of total mass to total volume, yields similar information as the water content and is an easily determined property. Knowing the bulk density, the water content and the unit weight of the solid constituents, the degree of water saturation can be determined. This is of considerable importance in the case of porous clay sediments with partly gas-filled voids. It should be noticed that a high bulk density may result from cementation processes and not necessarily from heavy preconsolidation.

3.3.5.2 Liquid limit

By determination of the liquid limit (w_L) a rather safe estimate can be made of the major clay mineral if the clay content is also known.

The liquid limit is defined as the water content at which a remolded clay-sample has a consistency that yields a depth of penetration of a 60 g/60° fall-cone of 10 mm.

This consistency state is measured by using a standard fall-cone apparatus. In the present study, about 100 cm^3 of clay was saturated with distilled water and thoroughly remolded to uniform consistency. The sample was then placed in a cup and the surface smoothed. The depth of penetration of the 60 g/60° fall-cone was recorded by multiple measurements and the liquid limit calculated according to the standard procedure.

$$w_L = M\ w_i + N$$

where M and N are variables depending on the depth of penetration and w_i is the natural water content of the sample.

The evaluated liquid limit represents the water-sorbing capacity of the clay, the following data being representative of different clay types:

	w_L (%)
- illitic clays	60-120
- kaolinitic clays	40-60
- mixed layer clays	60-150
- montmorillonite clays (Ca^{2+})	80-150
- montmorillonite clays (Na^+)	300-600

3.3.5.3   Grain size analysis

The amount of particles <2 µm, i.e. the clay content of a bulk sample, is of essential importance since the clay mineral crystallites are almost always found in this fraction. Most petrophysical characters are related to the granulometry since it reflects the mineralogy.

Clayey sediments are analysed by using sieve and hydrometer analyses. The silt and clay fraction (<0.06 mm) can also be analyzed with automatic "sedigraph" techniques by which the intensity of a beam of light penetrating through a suspension of clay is recorded and related to the grain size composition. The sedigraph and hydrometer methods are both based on Stoke's law of the settling rate of particles in suspension. The strong deviation from spherical particle shape that is characteristic of smectite flakes implies a considerable difference between the "Stoke diameter" and the actual crystal size and the evaluated particle size is therefore fictitious (9).

3.3.5.4   Chemical analysis

The chemical composition of the investigated material is essential when considering potential deteriorating effects arising from chemical attack by extreme pH conditions produced by contacting or intermixed compounds like concrete, carbonates and sulphates or by percolating groundwater. The analysis is preferably

performed on both the bulk material, which may contain such compounds, and on the clay fraction.

The analyses presented in this report have all been made by the Swedish Geological Co in Luleå. A standard X-ray spectrometer method has been applied on ground and dried samples. The ground sample (2 g) was homogeneously mixed with spectral pure cellulose ($C_6H_{10}O_5$) and epoxy resin and subsequently pressed and formed into a briquette. The prepared sample was mounted in a X-ray spectrometer (Rigaku Simultik UI) with 21 preset channels. All recorded intensity data were compared and controlled with the preset channels offering intensity references by which the percentage of the respective chemical component was obtained.

The standard program included several chemical elements which are of minor interest for the characterization of clayey material, e.g. rare earth elements and it is recommended to design a special program for future tests. The following program of chemical analysis is recommended:

Compound	Origin
SiO_2	Quartz, main component of clay minerals
K_2O	Feldspars, mica, clays
Na_2O	Feldspars, clays
CaO	Feldspars, karbonates, clays
MnO	Clays
TiO_2	Rutile, clays
P_2O_5	Apatite
MgO	Carbonates, clays
Fe_2O_3	Carbonates, clays
FeO	Sulphides, clays
CO_3	Carbonates
S	Sulphates, sulphides, sulphate
Org cont	Organic mtrl (coal, fossil plants, fossils)

3.3.5.5 X-ray diffraction analysis (XRD)

The best method to obtain data concerning the clay mineral composition of a material is by the use of X-ray diffraction technique. Analyses of this kind can be

performed in many ways, the method of preparation being a determinant of the accuracy. Thus, the choice of dispersing agents and methods (ultrasonic treatment, stirring), are particularly important variables which have to be considered in order to obtain representative results from XRD studies.

Another major problem of sample preparation is the orientation of clay particles onto a glass slide or some other kind of sample holder. Several strategies have been applied, including sedimentation from suspensions under gravity, suction, smearing, application under pressure etc (cf. 10, 11, 12, 13, 14).

A technique of quantitative XRD-analysis, recently presented by Reynolds (15), implies suction to obtain orientated, homogeneous and sufficiently thick samples. The method was first described by Drever (16) and has been found to give the most representative X-ray diffraction patterns of clayey materials. Characteristic intensity signals are necessary for qualitative identification of the mineral constituents and a defined particle orientation is a prerequisite for any accurate quantitative interpretation.

The preparation is made by applying vacuum and filtering of the suspension through a membrane filter (ϕ 0.47 μm). The obtained filter cake is then transferred to a glass slide. A thickness corresponding to at least 20 mg/cm^2 is required to give reproducible peaks and intensities over the investigated 2θ-interval (3-40°).

Ethylene-glycole, Mg^{2+}, K and HCl treatments are made when different clay mineral peaks coincide or when swelling of smectites is to be investigated in qualitative determinations, and additional preparation has to be applied in quantitative analyses. The latter are discussed separately in Chapter 4.

3.3.5.6 Additional

Certain natural smectite-rich materials, such as the Kinnekulle bentonites, are slightly cemented by silicious or carbonate precipitations, which prevents them from expanding although they may have a high content of smectite. This can be checked by running creep tests of undisturbed samples since the creep behavior is fundamentally different for stresses below and above the critical stress at which the cementation breaks down. Also, ultrasonic dispersion can be used to determine whether the granulometry is altered towards finer grain size by breaking

cementation bonds. If the diagnosis is "cementation" there are options of chemical treatment to remove the cementing substances as shown later in the text.

3.4 OCCURRENCE OF SWEDISH CLAY STRATA RICH IN ONE OR MORE CLAY MINERALS

3.4.1 General

The main three types of clay minerals (smectite, illite and kaolinite) occur more or less regularly and commonly in sediments as demonstrated by strathigraphic recordings. They have been quarried for a wide spectrum of applications, i.e. for use as drilling muds, for pottery, foundry sand bonding, filtering, refining, decolorizing etc (17).

Major sources of the different clay types are found in sediments of Quaternary and pre-Quaternary age. Reasonably pure clays are those represented by sediments in stable depositional environment such as lakes, sedimentary marine basins of nearshore origin or offshore marine environments and by weathered rock. The clay mass has to be very large for exploitation and this means that a number of natural processes must have combined to form deposits of potential interest:

i) Areas of source rock exposed to weathering and clay mineral formation, preferably crystalline, feldspar-rich source rock

ii) Transporting agents or a natural process for separation and concentration of small particles

iii) Environments in which significant amounts of clay were accumulated, e.g. flood plains, tidal flats, marine shelves, lakes etc

iv) Uniform conditions over a significant period of time to allow for the development of thick deposits

The above stated criteria are valid under most conditions with one exception, i.e. residual kaolinitization where transport and sorting have played a minor role.

During post-Eocambrium all the criteria have been fulfilled occasionally but homogeneous clay deposits of potential commercial interest are confined to a few areas in Sweden, namely:

- Scania
- Öland
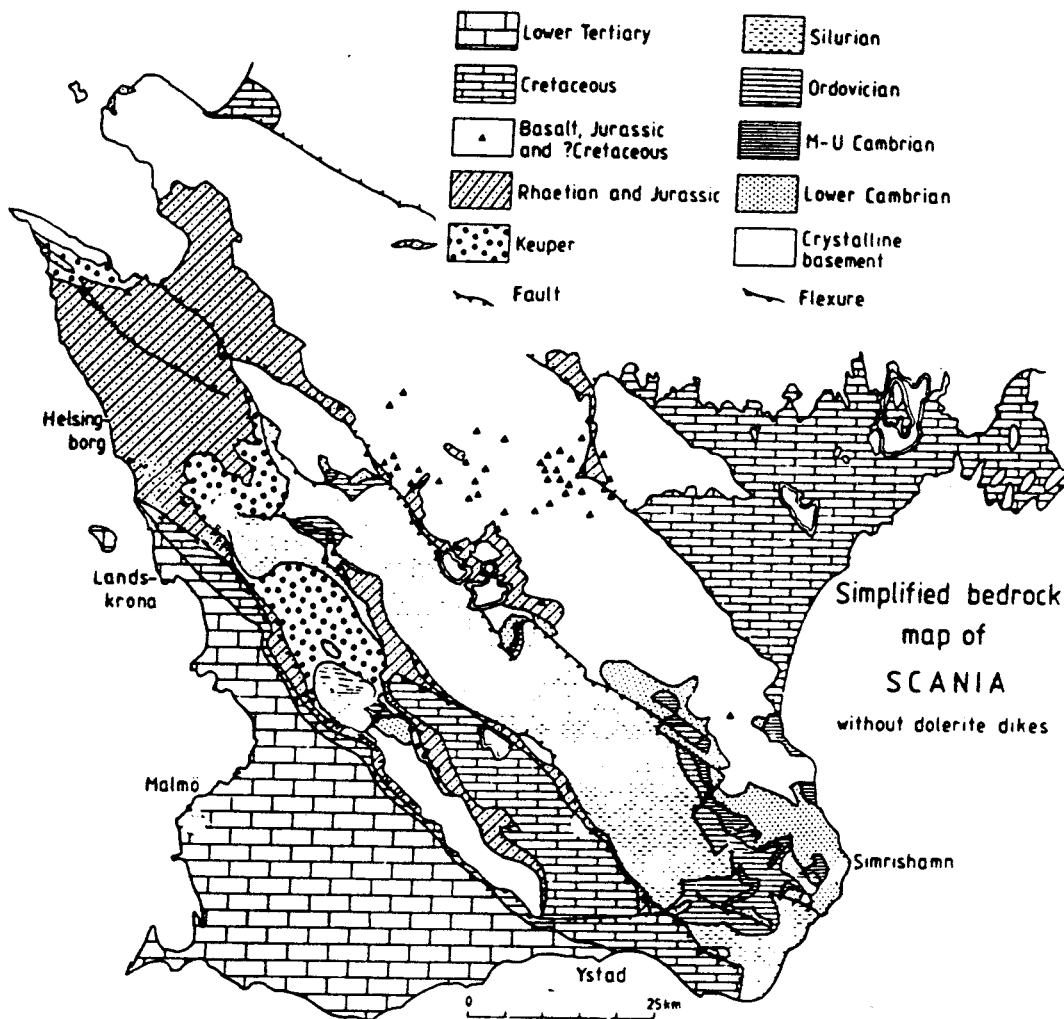- Gotland
- Västergötland
- Dalarna
- Jämtland

Fig 4.   Simplified bedrock map of Scania [Modified from Bergström (18)]

Many of these areas only bear early Paleozoic strata (Cambrium, Ordovicium and Silurian) with thin clay beds or intercalations of very limited use. Scania, on the other hand, has a wide spectrum of rocks of different age and lithology and the

best possibilities of identifying deposits of potential use for the present purpose. In particular, the Mesozoic strata are of great potential importance and they will be discussed in detail in this report. In principle, these strata (Trias-Cretaceous) represent a large number of sediment-types formed and deposited during different climatic conditions and depositional environments. These variations resulted in different types of clay minerals and also in large differences in layer thickness. Beds rich in kaolinitic and smectitic clays have been formed under widely different physico-chemical environmental conditions.

3.4.2   The Mesozoic of Scania

The Mesozoic strata are dominated by sediments deposited in fairly nearshore regions, such as deltas, tidal flats, beaches, shallow marine environments. Lateral variation of the type of sediment and thereby also the vertical distribution of the stratal sequence is evident. As illustrated in Fig 5, sands, limestones and more or less clayey deposits alternate and form a complex stratigraphic pattern, still reflecting the nearshore origin of deposition.

Most of the Cretaceous strata are known from deep boreholes in the southwestern part of Scania. Here, calcareous deposits are found to have a significant thickness, i.e. up to 2000 metres. The Cretaceous strata are less rich in thick clay beds than other deposits of Mesozoic age and since they are located relatively deep down they can hardly be economically exploited.

The Triassic and Jurassic are far better known from outcrops, shallow boreholes, and quarries in a zone extending from NW to the SE through Scania (cf. Fig 4). Lower and Middle Triassic are known mainly from deep boreholes in SW Scania.

The relatively easily accessible strata are more or less indicative of tectonic movements (inversion) during early Cretaceous time in Scania. Thus, faulting, tilting, uplifting, and compression of earlier deposited strata gave a complex structure of the deposits. Tilted strata are found for instance in Fyledalen, where a Jurassic sequence is standing vertically, next to horizontally lying strata of Cretaceous age.

A number of clayey sedimentary units of potential use are exposed and relatively wellknown with respect to their extention and composition. Three of them, repre-senting large volumes, i.e. more than 200.000 tons, were chosen for further inves-

tigation. They are termed Kågeröd, Vallåkra, and Fyledalen and are located as shown in Fig 5. In addition to these "primary" units there are vast amounts of Quaternary morain clays derived from Mesozoic strata, and although their clay content is relatively low they should still be considered as possible candidate materials for certain buffer material functions.

CHRONOSTRATIGRAPHY			MAIN LITHOLOGY	m	INVESTIGATED CANDIDATE
CRETACEOUS	UPPER	MAASTRICHTIAN	CHALK	+2000 m	
			LIMESTONE, SAND, CLAY		
		CAMPANIAN	SAND, CLAYEY LIMESTONES		
		SANTONIAN			
		CONIACIAN			
		TURONIAN	LIMESTONE		
			//////////		
		CENOMANIAN	SAND, LIMESTONE		
			//////////		
	LOWER	ALBIAN	SAND		
		APTIAN	SHALE		
		BARREMIAN	SHALE, SANDSTONE		
		NEOCOMIAN	SANDSTONE SAND CLAY SHALE, CONGLOMERATE		
JURASSIC		UPPER	CLAYS	70	←FYLEDALE N
			SAND, SANDSTONE	40	
			CLAYS MARL	145	
		MIDDLE	SAND, SILT	30	
			SAND, CLAY, COAL	120	
		LOWER (LIAS)	SHALE, CLAYSTONE SANDSTONE, CLAYS COAL, SILTSTONE, CALCAREOUS SEDIMENTS	635	
TRIASSIC	UPPER	RHAETIAN	COAL, CLAY	50	←VALLÅKRA
			CLAYS		←KÅGERÖD
			SHALE		
			SANDSTONE	270	
		MIDDLE	SANDSTONE, SHALE MARLS	120	
		LOWER	SANDSTONE, SHALE	40	

Fig 5. Schematic illustration of the Mesozoic sedimentary record of Scania [Modified from Lidmar-Bergström, (19)] and the stratigraphic position of the chosen candidates for closer investigation

3.5   CHOICE OF POTENTIAL CANDIDATES

The buffer material candidates chosen and investigated in this report all originate from Scanian sedimentary rocks and Quaternary deposits. The initial choice was mainly based on the review of literature dealing with clayey deposits of Sweden. The following criteria were then defined for a first screening:

i)   sufficient clay bed thickness (>1 m) and lateral extension (>1km^2)

ii)   easy access i.e. the clay should not be situated below thick deposits of other origin, such as Quaternary morain

iii)   a clay content of at least 20 % and at least 30 % of this fraction being either smectite, hydromica or kaolinite

Four major candidates emerged out of this initial review:

- The Vallåkra clays
- The Kågeröd clays
- The Höganäs clays
- The Fyledalen clays

The candidates are stratigraphically distributed from the Upper Triassic to the Upper Jurassic (Fig 5). They have quite different petro-physical properties under natural field conditions and were expected to represent a spectrum of clay-mineral assemblages. They are described in detail in the following chapters.

3.5.1   The Vallåkra clays

3.5.1.1   General

The Vallåkra member including both clayey and sandy beds was originally described by Troedsson (20). He located the beds stratigraphically as early Rhaetian deposits and characterized the sediments as dark grey to light reddish, non-laminated clays interbedded by siderite and greenish sandstones, occasionally rich in fossil plants.

The clays were considered to be montmorillonitic by Norin (7, 8) who indicated mineralogical variations both laterally and vertically. Most of the clayey deposits

are also considered to be redeposited sediments originating from the older Kågeröd beds (cf. Fig 5). This has caused the obvious variation in lithological character of the Vallåkra beds.

3.5.1.2   Outcrops, thickness, and lateral extension

The Vallåkra beds do not have many outcrops since any new and freshly cut pit is covered or collapsed after a short period of time. The best clay pits are those at Vallåkra, Lunnom and Skromberga.

The Vallåkra clays immediately underly the Quaternary morain in a narrow zone bordering the Rhaetian-Liassic depositional basin in NW-Scania (Fig 6). Further out in the central part of this basin the same beds are found at considerable depths.

There are some uncertainties about the exact vertical extension and location of the clays since it is difficult to distinguish them from over- and underlying Rhaetian - Liassic beds (cf. Fig 5). Reasonably reliable data are collected in Table 1.

Table 1   Depth below ground level and thickness of the Vallåkra beds in NW Scania (cf. Fig 7) in selected boreholes

Borehole	Depth, m	Thickness, m
A N. Vallåkra 3	15-25	5
B Kvistofta 59	- 50	(150)
C Fleninge 266	170-179	9
D Fleninge 265	62-75	13
E Oregården 271	314-	-
F Nya Wilhelmsfält	387-407	20
G Farhult 270	98-124	26
H Mörarp schact	53-	-
I Bjuv schact III	51-	-
J Bjuv 1	44-66	22
K Bjuv 2	35-48	13
L Gustav Tornerhjelms schact	96-104	8
M Schact consul	21-22	1
N Truedstorp 4	21-	-

Borehole	Depth, m	Thickness, m
O Skromberga 273	16-20	4
	- 20	20
P (Borehole without name)	42-57	15
Q Selleberga, concession 150	45-	-
R Selleberga, concession 154	64-	-
S Charlottenborg, concession 112	10-	-
T Skromberga, concession 96	20-	-
U Skromberga, concession 4A	- 51	51
V Assarstorp 19A	- 56	56
X Mörarp, concession 8	103-	-
Y Schact, Oscar II	39-39.2	0.2
Z Schact, Ljungsgård	- 55	55
Å Norra Vram, Concession 4		

It is seen from the borehole data that the area where the clay is within reach for quarrying is rather limited. Only a narrow approximately 2-3 km broad zone constitutes the outcrop area. An exact thickness of the clay is difficult to derive from borehole data and because of the fact that the area in NW Scania is frequently faulted and tectonically disturbed. The thickness is, however, generally more than 5 m.

3.5.1.3   Lithology

The Vallåkra unit is mainly composed of clays and clayey sandstones. The clays are grey to slightly reddish in color for samples being taken in the "Vallåkra North" old clay pit where the clay have been commercially quarried since the 17th century. The clay here is found approximately 3 m below 6-8 m of sandstone layers with interbedding coal seams and thin shaly beds. The locality had to be dug out by firstly removing old debris from overlying beds and then exposing fresh clay which was sampled.

The samples consisted of dark grey and grey, slightly reddish silty clay, occasionally organic. It was somewhat shaly in appearence but only weakly lithified (cemented). The clay beds are relatively thick in the Vallåkra area but the lateral
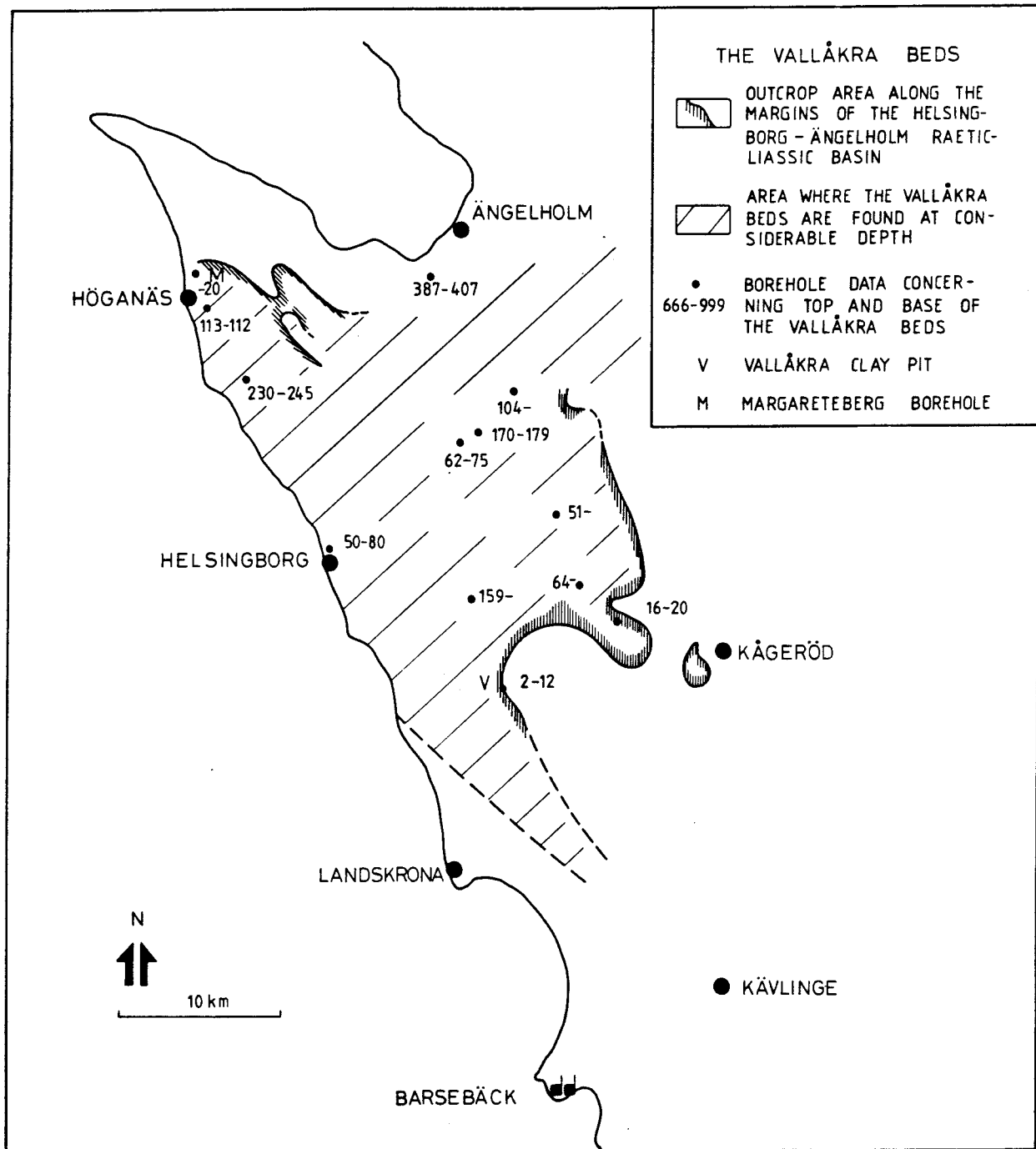
Fig 6.   Occurrence and borehole data of the Vallåkra beds [Modified after
           Troedsson (21)]

extension of the clays is uncertain. The lithology changes laterally as shown in
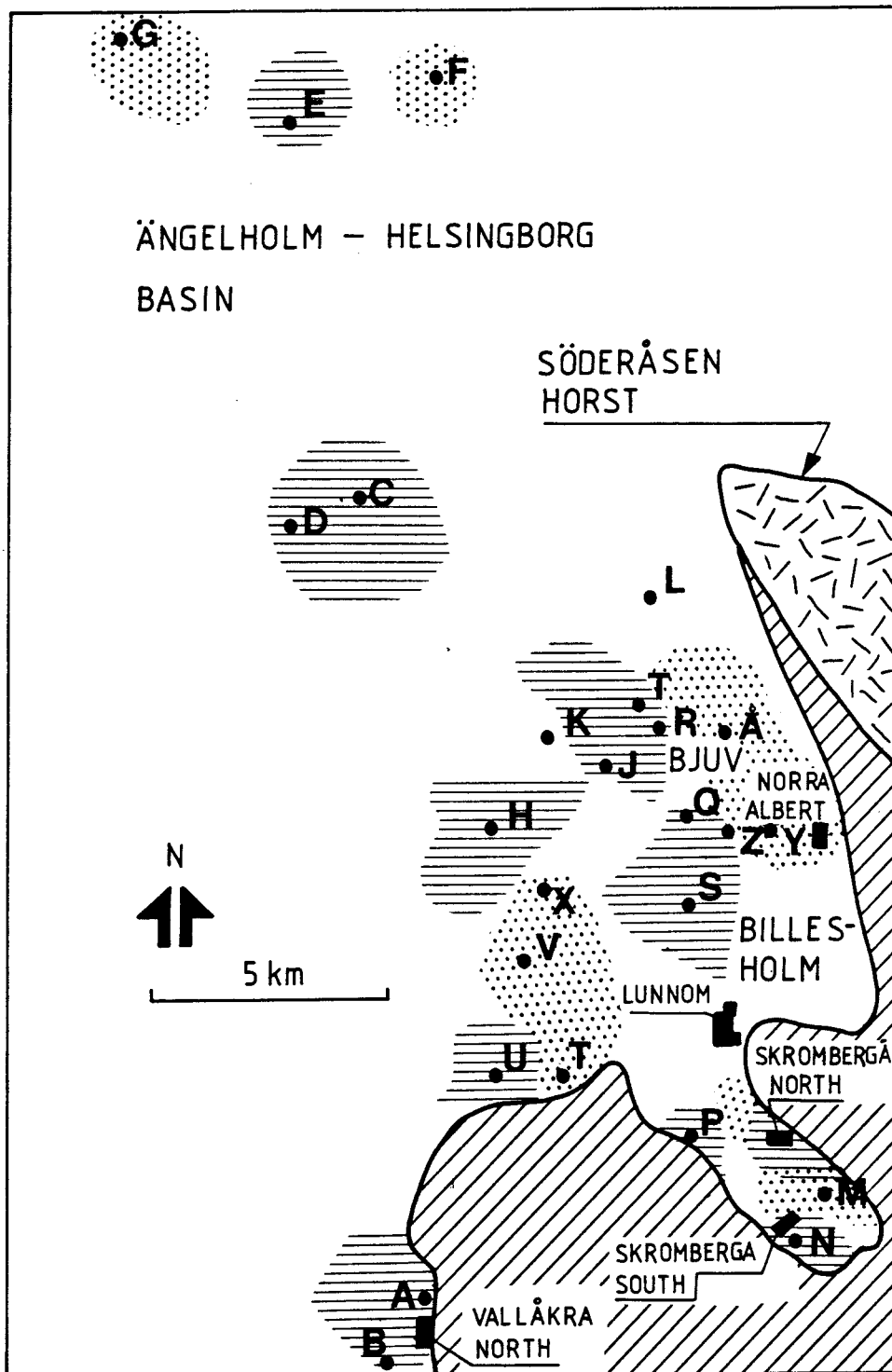Fig 8.

Fig 7.   Sketch-map showing the main types of lithology in the Vallåkra
          member [Modified after Troedsson (21)]. Letters refer to boreholes
          presented in Table 1

KÖPINGE Nr.3   VALLÅKRA   SKROMBERGA   FLENINGE
                          Nr.273   Nr.265
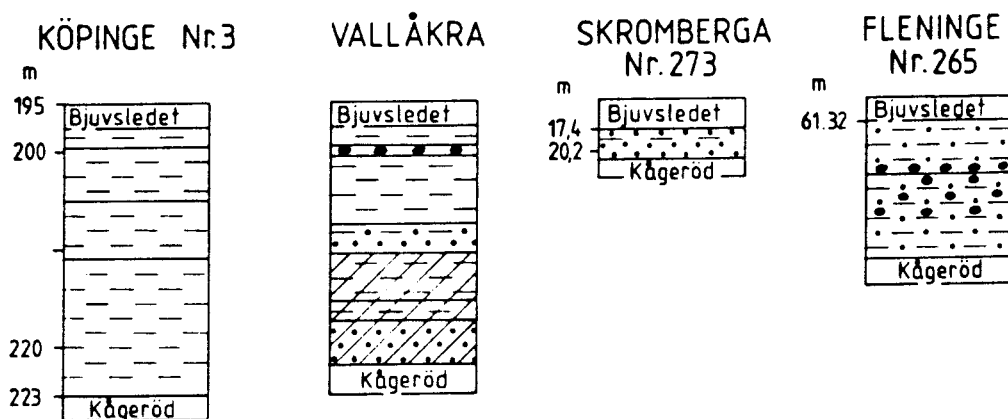
Fig 8.   Boreholes penetrating the Vallåkra [member after Sivhed (22)]

The Vallåkra beds are generally more clayey and fine-grained in the south-western part of the investigated area, compared to the dominantly sandy lithology close to the Söderåsen horst (Fig 8). This is most likely due to the type of sediments underlying the Vallåkra beds (22). This correlation should be considered in future, more detailed investigations for possible exploitation purposes.

The samples were analyzed mainly with respect to their granulometry and type of clay-minerals.

A standard hydrometer analysis was performed on the samples and this gave a clay content between 50 to 60 percent. The remaining part of the sample consisted of 30-40 % silt and 10 percent fine sand, i.e. with a grain size of 0.063-0.125 mm (Fig 9). The liquid limit was found to be 80 %.

3.5.1.4   Chemical composition

The chemistry of the Vallåkra unit varies considerably. Characteristic compositions are given in Table 2, the Margreteberg clays being located close to Höganäs and the "Lower Vallåkra clay" originating from Vallåkra.
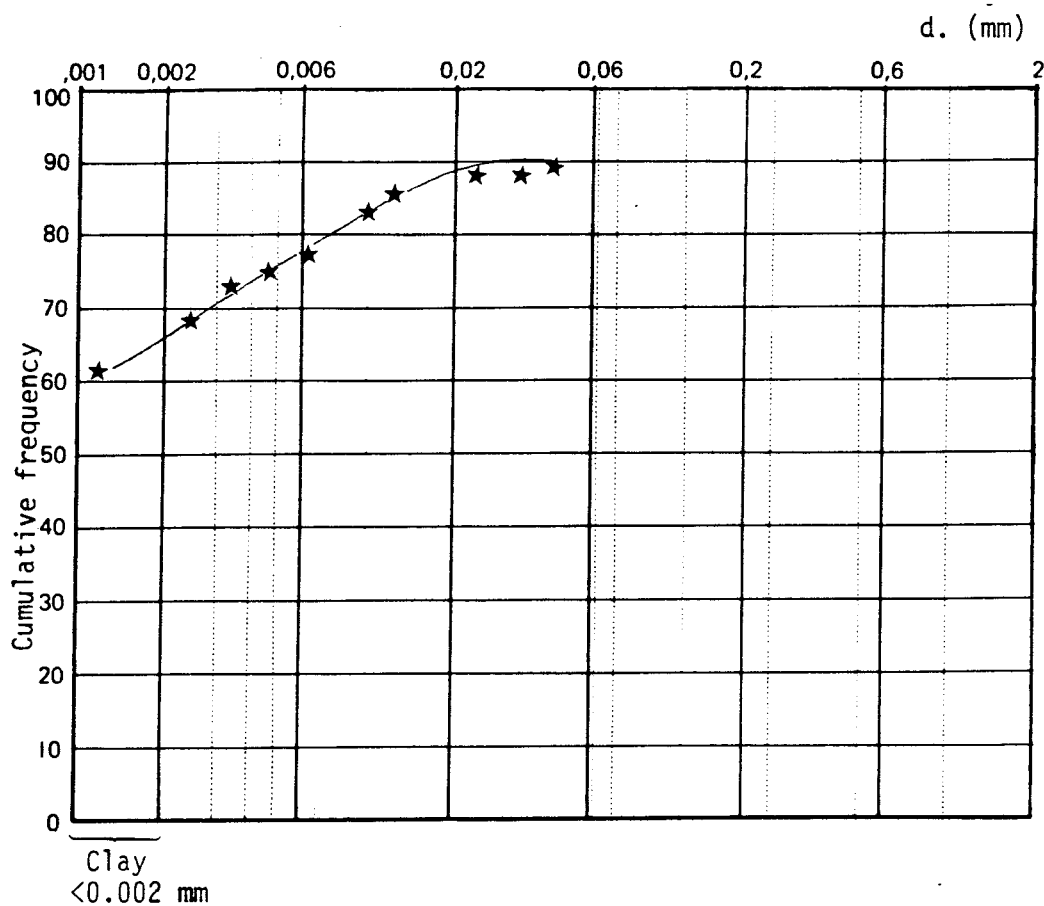
Fig 9.   Grain size frequency diagram of Vallåkra clay sampled at "Vallåkra North" old clay-pit

3.5.1.5   Stress and temperature history

The Vallåkra strata appear to be almost unaffected by tectonic forces, except for local strong disturbances caused by faulting. Since the estimated maximum sediment overburden did not exceed 300 m the temperature cannot have exceeded 15-20°C in the presently exposed layers. It may have been considerably higher in very local areas due to Tertiary volcanism but the larger part of the strata, including the investigated samples, were not exposed to high temperatures.

The maximum overburden pressure, about 10 MPa, was exerted by Pleistocene glaciers.

A qualitative XRD-analysis was performed on the fraction <2 μm as to characterize the type of clay minerals present. From the diffractogram (Fig 10) it is seen

Table 2. Chemical composition of Vallåkra clay

Compound	Margreteberg Reddish clay %	Margreteberg Yellow, weathered reddish clay %	Vallåkra %
SiO_2	57.7	54.7	42.7
Al_2O_3	19.3	23.4	27.1
Fe_2O_3*	8.8	5.5	13.2
MgO	1.2	1.2	1.5
CaO	0.6	0.8	1.7
Na_2O	1.5	0.6	1.5
K_2O	1.4	0.6	1.5
TiO_2	1.0	1.5	1.4
H_2O	9	12	8.6

* The figure refers to the total Fe content

that kaolinite dominates in the analysed sample. Hydromica is present in minor amounts and smectite does not make up more than a few percent of the total mineral amount.

3.5.1.6 Summary

The investigation of the Vallåkra unit shows that clays constitute a major part of the beds and that they have a varying lithological character over relatively short distances. The clay is furthermore found at considerable depth (>40m) except for a narrow zone (<1 km) irregularly bordering the Rhaetian-liassic basin. Here, the Vallåkra clay is found at shallow depth (approximately 15 m) below the Quaternary deposits. Actually, there are significant areas (>1 km2) with a clay bed thickness of about 2 m in the vicinity of Vallåkra, Skromberga and N. Albert, in which areas the clay has been quarried locally. The analysed samples were from Vallåkra and consist of dominantly kaolinitic clay with a clay content of 50-60 % and a liquid limit of 60-80 %.

SAMPLE: V1
A = Untreated
B = EG-treated

9.94 A Hydromica (001) ?

10.05 A Hydromica (001)

Kaolinite (001)
7.20 A

7.14 A

Kaolinite (001)

B.

4.95 A Hydromica (002) ?

4.44 A (Kaolinite)

4.25 A Quartz

3.56 A
Kaolinite (002)

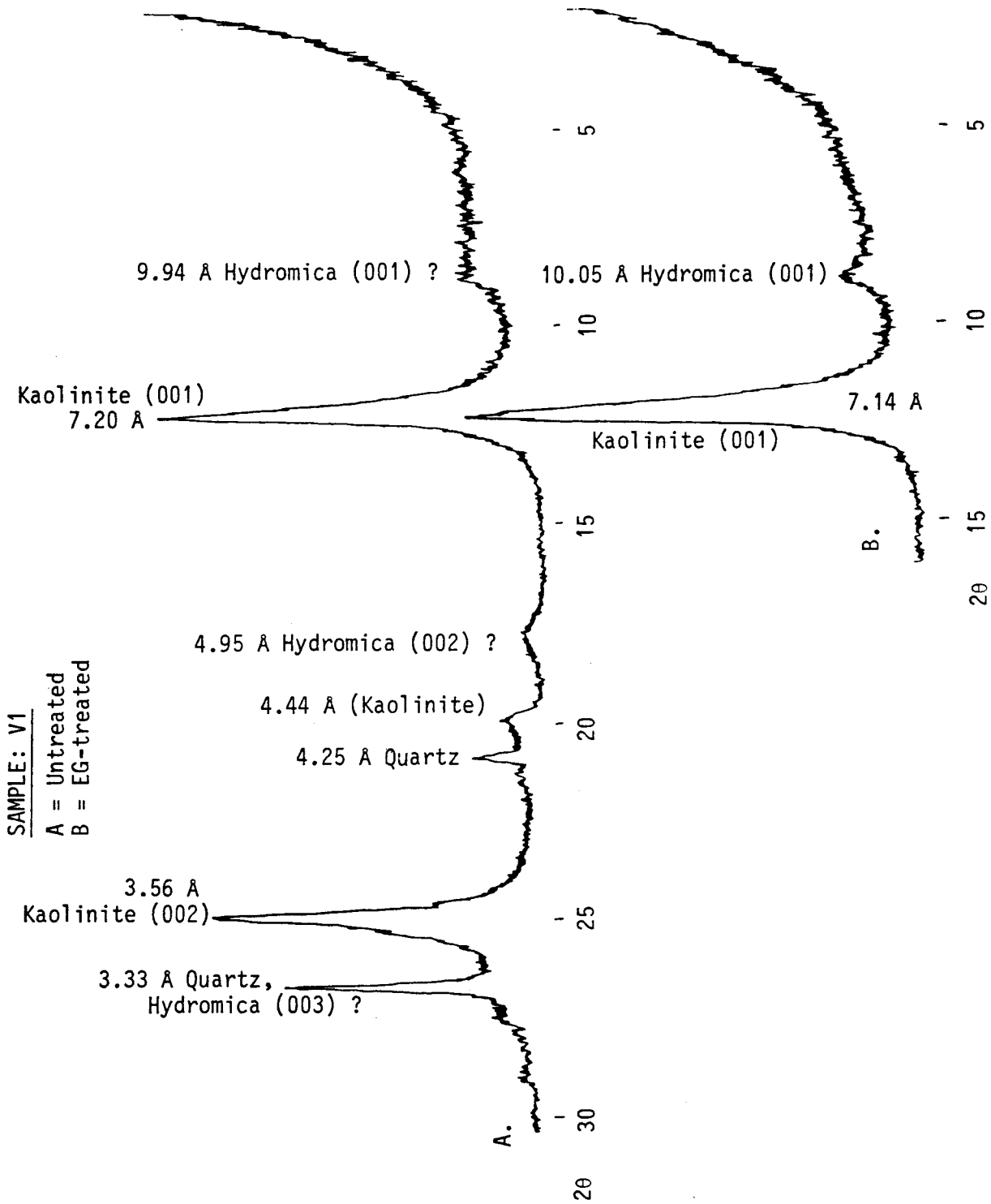3.33 A Quartz,
Hydromica (003) ?

A.

2θ

Fig 10.   X-ray diffractogram of Vallåkra clay <2 µm, untreated and EG-treated sample

3.5.2 The Höganäs clay (Margreteberg)

3.5.2.1 General

The Höganäs clay, which belongs to the Vallåkra unit described in a previous chapter, has been exploited commercially for many years by the Höganäs AB company. The material included in the recent study originates from a borehole located in Margreteberg just outside Höganäs (Fig 6). The core has been investigated earlier with respect to its petrophysical and mineralogical properties (3).

The rather "inactive" behavior of the slightly reddish clay, which appears to have the highest content of smectites of the three horizons (red, grey, yellow), is clearly contradictory to the richness in smectite that the X-ray diffraction pattern of this clay indicates. This is probably explained by precipitated matter acting as cement. The main object of this earlier study was to determine whether precipitated compounds, presumably iron oxides, could be removed as to "activate" the clay but it could neither be demonstrated by certainty what the nature of the cementitious matter really is, nor whether it is possible to desarm it. Three additional attempts have been made, two by SGAB, and one by the Höganäs AB, and they are summarized below.

In the first of the studies, activation has been tried by treating the clay with dilute hydrochloric acid, the intention being to dissolve the iron oxides that were assumed to cover large domains of montmorillonitic flakes and to "open" the pathways of water into the clay-mineral interlayer. The clay has also been treated with soda (Na_2CO_3) in order to promote substitution of the interlayer cation which appears to be largely Ca^{2+}. However, the experiments have not yielded positive results which demonstrates that the precipitated compounds are strongly and tightly bound to the clay particles. A more intense acid treatment cannot be applied without comprehensive breakdown of clay mineral lattices.

A technique for removal of free iron oxides from soil material has been described by Mehra and Jackson (29) and it has recently been tested by SGAB in a second study. The procedure was originally designed as to increase the sensitivity of XRD-analyses since iron oxides are known to suppress the diffraction intensity and it involves treatment with buffered neutral citrate-bicarbonate-dithionite (CBD). The dithionite ($Na_2S_2O_4$) is employed for the reduction, the sodium bicarbonate ($NaHCO_3$) ($Na_3C_6H_5O_7, 2H_2O$) being a buffer, and sodium citrate serving

as a chelating or complexing agent for the iron. The study was never completed because of the positive outcome of the third attempt made by the Höganäs AB. This company reported that an improved swelling ability is obtained by a rather simple and rapid and therefore relatively cheap method that can also be applied on an industrial scale. Thus, washing in 5 % acetic acid buffered with sodium acetate to pH5 and adding sodium carbonate to a fairly dense consistency (water content 30-50 %) doubled the liquid limit to the final value 140 %. No detailed information or written report is available but it seems as if an industrial process is now available. Further tests on smaller and larger scale are required to certify this.

3.5.2.2   Outcrops, thickness, and lateral extension

The Margreteberg clay belongs stratigraphically to the Vallåkra unit. The material identified in a series of boreholes at Margreteberg consists of approximately 10 m of grey to slightly reddish clay, resting on reddish Kågeröd clay of unknown thickness. The lateral extension of the Margreteberg clay is difficult to describe due to the faulted and disturbed bedrock but it is believed that it covers an area of a few square kilometers with a bed thickness that ranges between 2 and 15 m.

3.5.2.3   Lithology

The X-ray diffractogram (Fig 11) shows that smectite, quartz, and kaolinite are the main constituents of the clay fraction. EG-treatment shows a diagnostic smectite swelling of the 13 Å peak to 16.4 Å.

3.5.2.4   Summary

The Margreteberg clays have a thickness of 2-15 m and a lateral extension of 1-10 km^2 in the Höganäs area. It appears to be possible to "activate" the clay, which has restricted swelling capacity because of iron-compound coating of the clay domains.
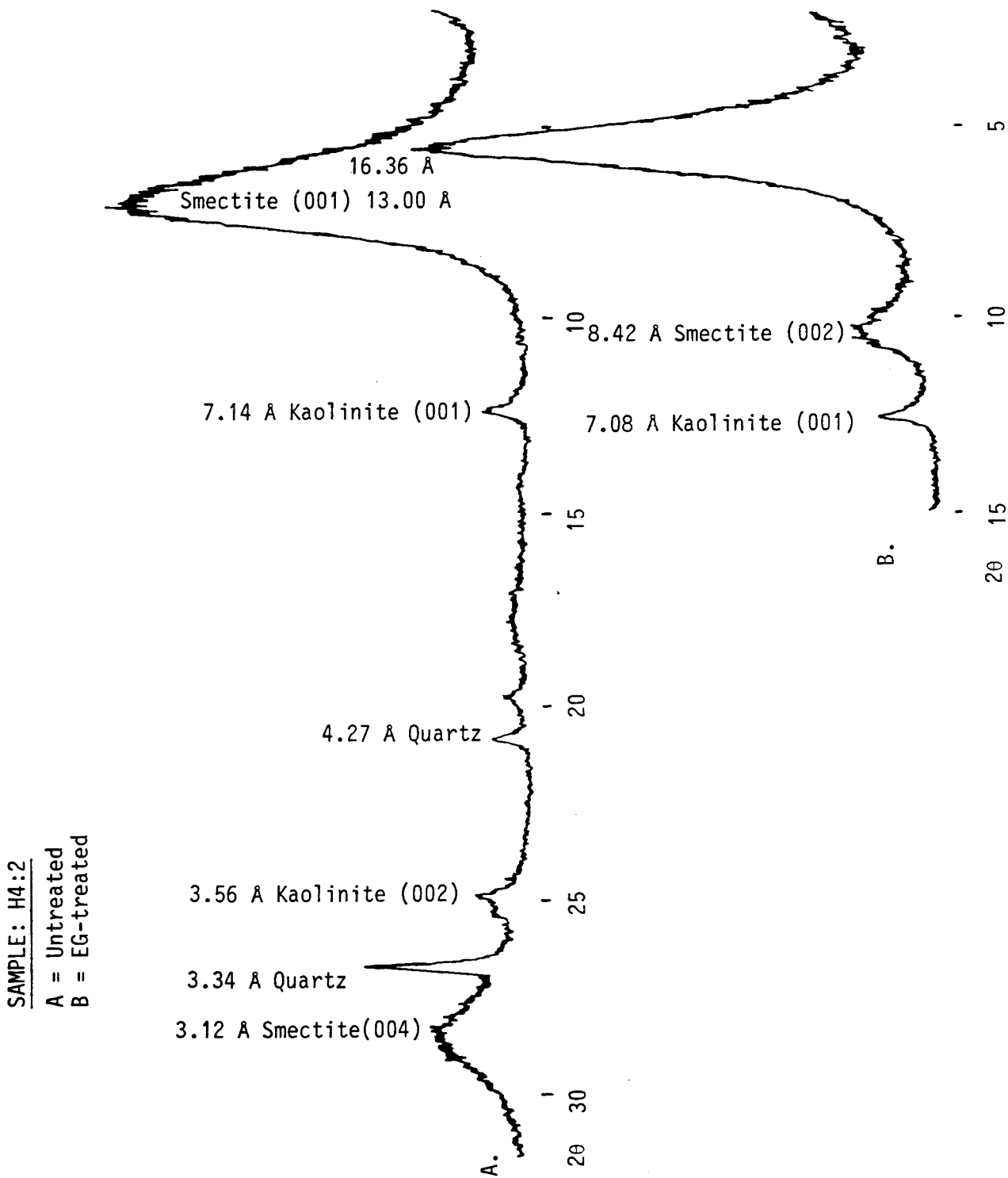
Fig 11. X-ray diffractogram on the Margreteberg clay, untreated and EG-treated fraction <2 μm

3.5.3 The Kågeröd clays

3.5.3.1 General

The Kågeröd Formation has frequently been mentioned in the geological litera-
ture (18, 22, 23, 24, 25, 26, 27). It is mainly known for being composed of poorly
sorted arkosic sandstones and conglomerates. However, clay beds are dominating
in the upper part of the formation, which has a wide extension both laterally and
vertically. The composition and thickness are varying, the maximum thickness
being found in boreholes in the vicinity of Bjuv where the Kågeröd Formation is
approximately 180 m thick. Here, it is covered by thick deposits of lower Jurassic
sediments (Vallåkra unit). The Kågeröd Formation is of upper Triassic age (28).

3.5.3.2 Outcrops, thickness and lateral extension

The Kågeröd Formation is found in a large, irregularly shaped outcrop extending
from the town of Lund to the Söderåsen horst (Fig 12). The lithological variation
within this area is poorly known except for the northern part of the area where
there are regions of dominantly clayey or sandy lithology. The clay thickness in
this area is more than 5 m. It has been found that the Kågeröd formation extends
down to considerable depth below younger Mesozoic deposits in the Rhaetic-
Liassic basin. The thickness is poorly known since sporadic investigations were
mainly made to get data concerning the coal seams of the Upper Triassic - lower
Jurassic.

Outcrops of the Kågeröd Formation are mainly composed of coarse- grained
sandstones and conglomerates, while fresh outcrops of clayey beds are not
frequently found. The samples analysed in this study were collected at Ottarp (Fig
13), where a relatively recent cut is available for sampling.

Known data concerning the thickness of the Kågeröd clay are compiled in Table 3.

3.5.3.3 Lithology

The Kågeröd strata were formed during a period of dry climate when desert-like
conditions prevailed in Scania. Intense, periodic rain falls eroded and transported
material into the depositional area, which was located close to the site where the

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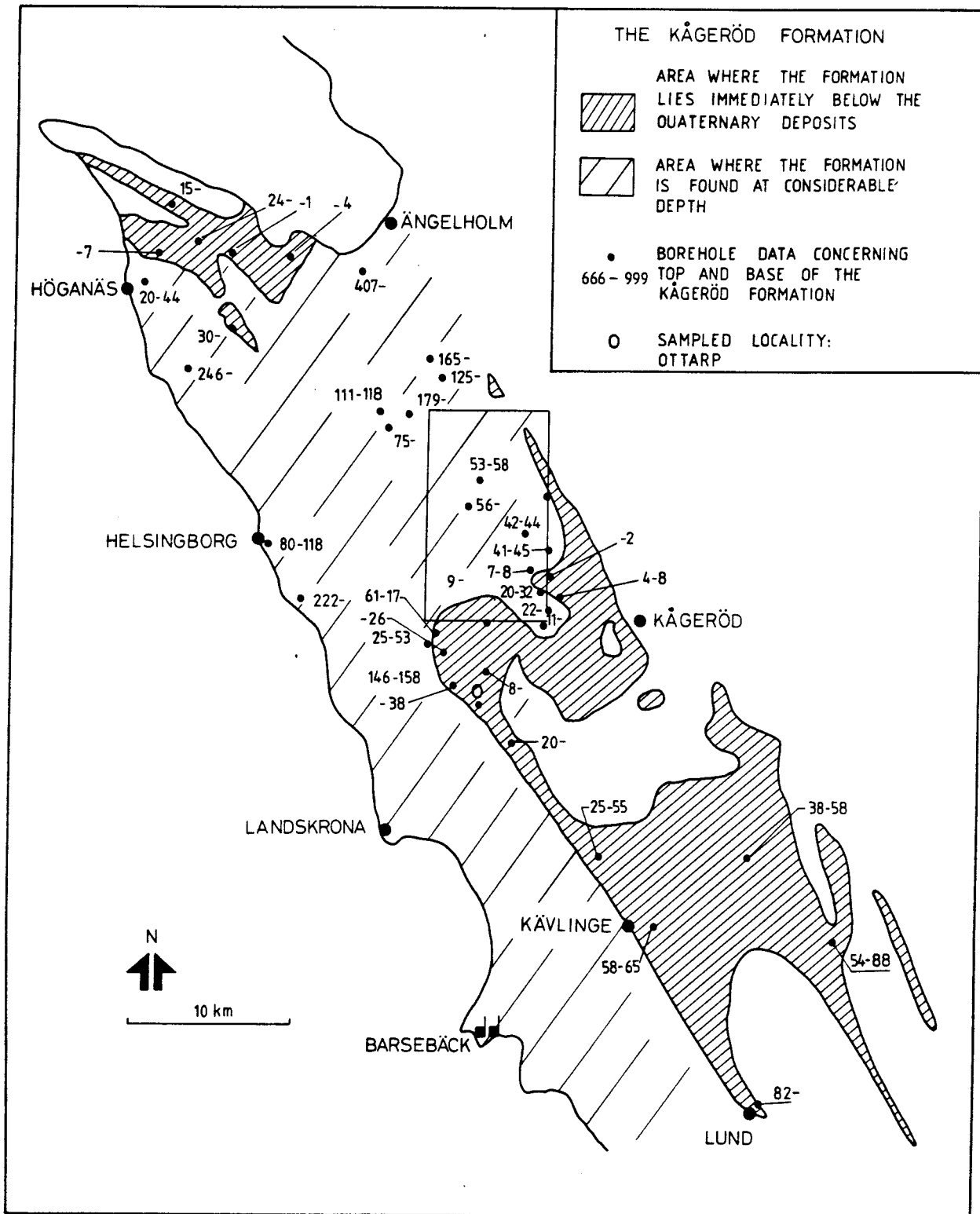
Fig 12. Lateral extension of the Kågeröd Formation in northwestern Scania. Framed area refers to enlarged area in Fig 13

THE KÅGERÖD FORMATION

DOMINANTLY SANDY AREAS OF THE KÅGERÖD FORMATION

DOMINANTLY CLAYEY AREAS OF THE KÅGERÖD FORMATION

THICKNESS OF THE KÅGERÖD CLAY

AREA WHERE THE KÅGERÖD FORMATION IS COVERED BY THICK YOUNGER DEPOSITS

SÖDERÅSEN HORST

BJUV

MÖRARP

RISEKATSLÖSA   BILLESHOLM

N

2.5 km

EKEBY

Fig 13.   Enlarged area of the Kågeröd Formation and its extension within the framed area of Fig 12

Table 3. Data concerning thickness and depth below ground level of the
         Kågeröd clays

Area	Overlying deposits	Depth below ground level, m	Thickness m
Vallåkra	Quaternary, Rhaetian	6-25	10.9-27.8
Billesholm	Rhaetian	18 - 51	2.5-20
Söderåsen horst (W)	Rhaetian	35-115	1-6
Helsingborg	Rhaetian	80-220	18-38
Höganäs (S)	Rhaetian	10-30	20
Kävlinge	Quaternary	17- 89	1-50

erosion took place. The basal clay is interpreted as an in situ residual product
from weathered crystalline bedrock (22).
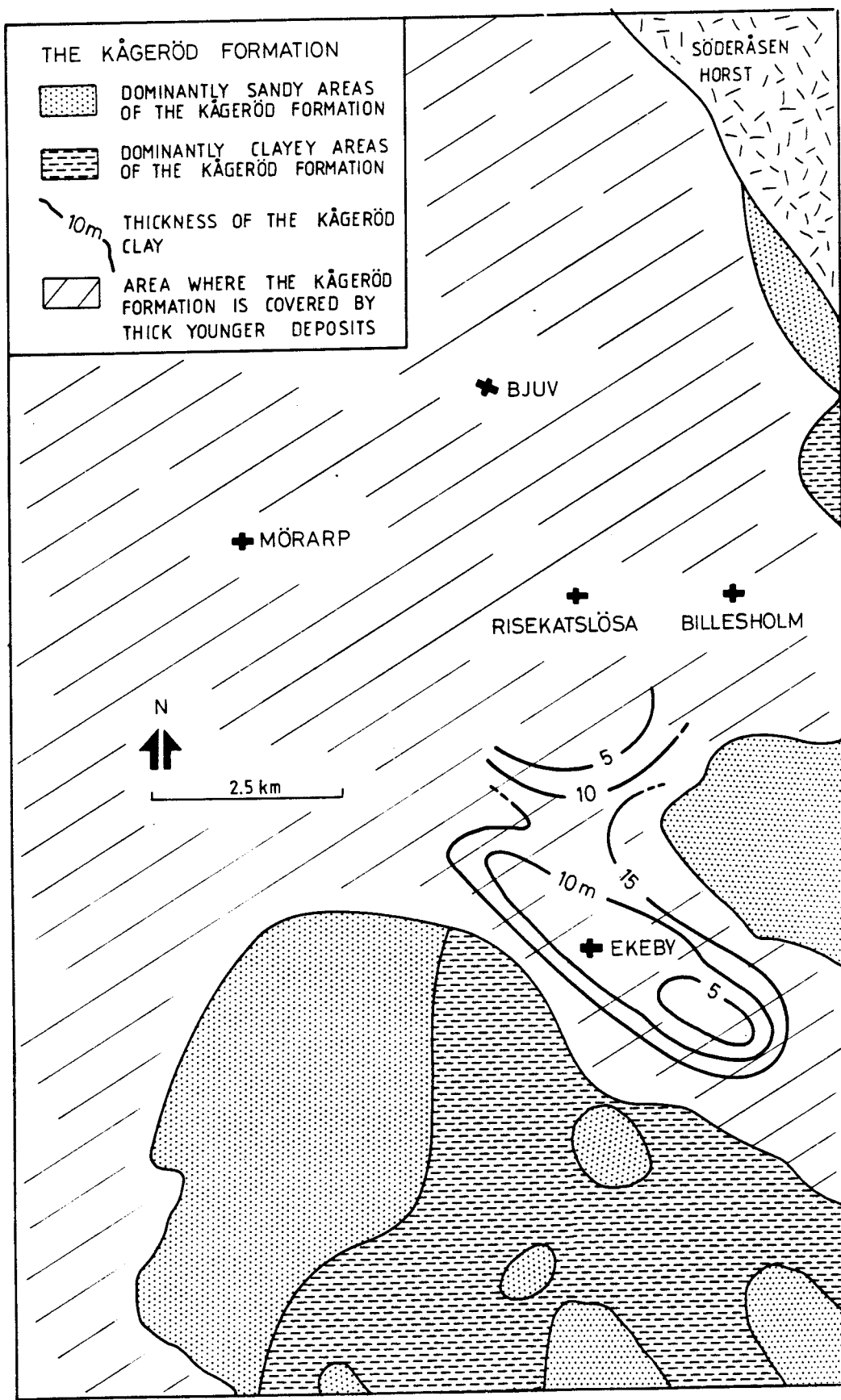
The upper clays, which are included in the present survey, are thought to be
deposits formed in stable, shallow marine environments.

The lithology is typical of the type of source rock of the Kågeröd Formation.
Where Silurian shales served as a source, mainly clayey deposits are found. The
dominant lithology, particularly of the basal parts, is characterized by poorly
consolidated sandstones and conglomerates, which are mainly non-lithified. The
upper part of the Formation is generally composed of a reddish clay with a clay
content of 20-30 % according to hydrometer analyses made of samples collected at
Ottarp (Fig 14).

The liquid limit ranges between 40-60 % for the bulk sample. A value of 85 % was
obtained when the coarser material >0.063 mm had been removed from the
sample and this indicates a considerable content of smectite.

The XRD-analysis of the clay fraction was performed in the same way as for the
Vallåkra clay. It showed that the clay fraction primarily consists of swelling
smectites with a minor amount of kaolinite (Fig 15).
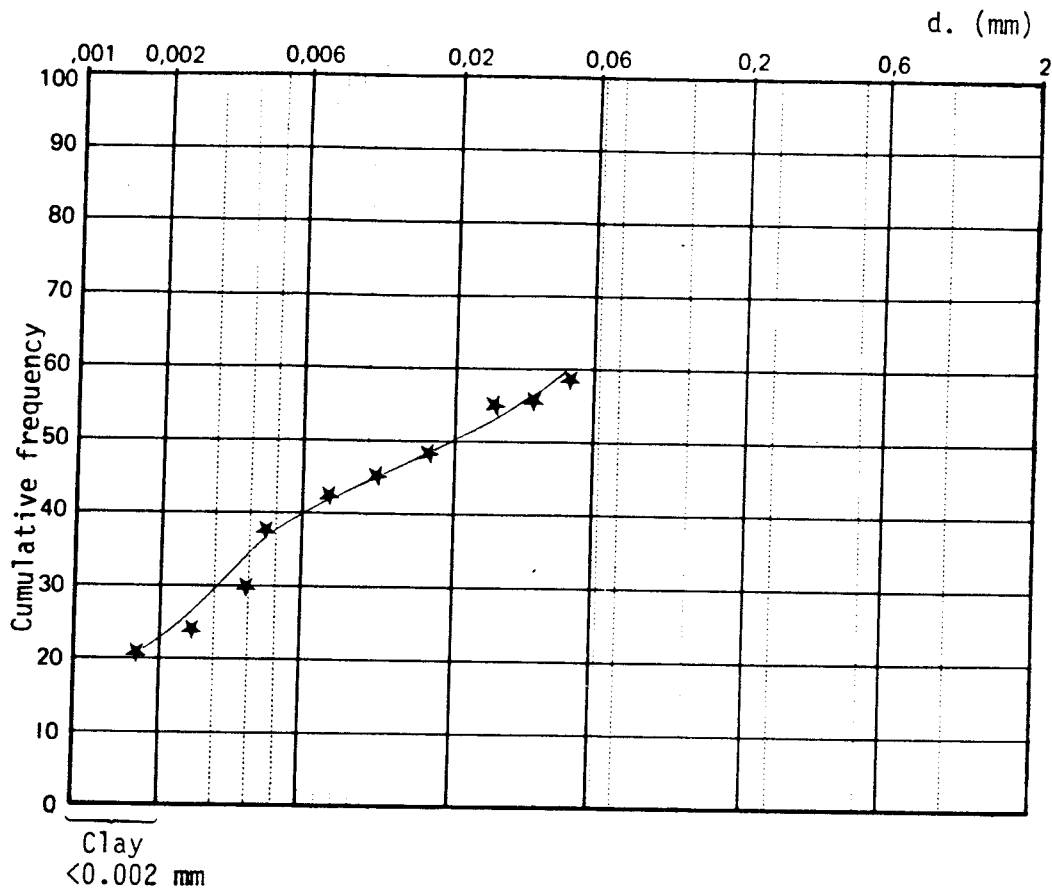
Fig 14.   Grain size frequency diagram of Kågeröd clay sampled at Ottarp

3.5.3.4   Chemical composition

The chemistry of the Kågeröd Formation is concluded to vary approximately as much as the Vallåkra unit. No analyses were made of the extracted samples.

3.5.3.5   Stress and temperature history

Like the Vallåkra strata the Kågeröd Formation is largely unaffected by tectonics, with the exception for large local disturbances produced by faulting. Similar conditions also prevailed with respect to temperature and stress.

3.5.3.6   Summary

The Kågeröd clay has been found to have a large lateral extension in the investigated area. The thickness is significant, frequently more than >10 m over areas of 1-10 km^2.

Fig 15. X-ray diffractogram on Kågeröd-clay <2 μm, untreated and EG-treated samples

The clay fraction consists of smectite minerals and a minor amount of kaolinite. This fraction was found to be 20-50% of the bulk material, which makes this clay the primary buffer material candidate.
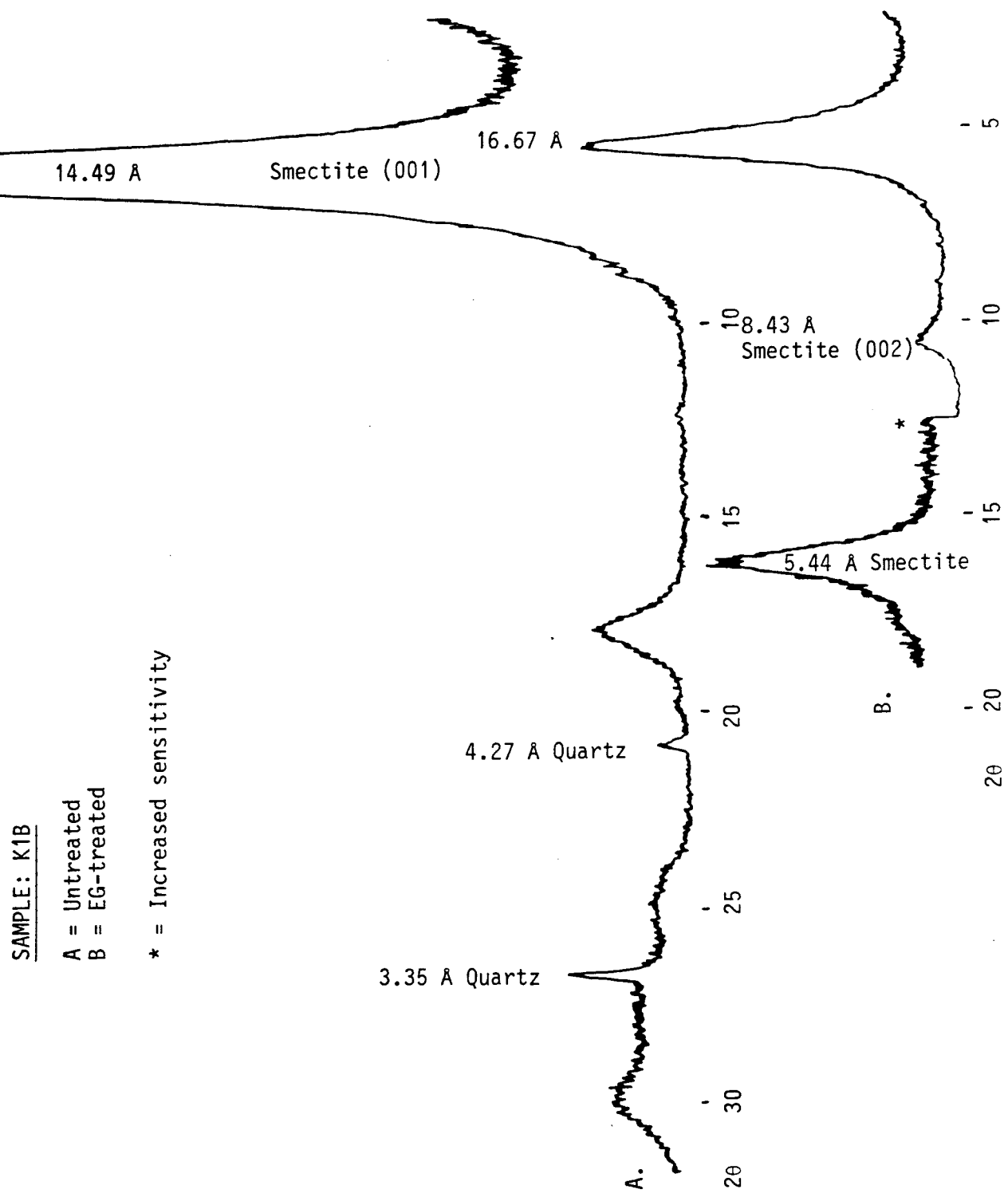
3.5.4   Fyledalen clay

3.5.4.1   General

The Upper Jurassic Fyledalen clay (Fig 5) is the most thoroughly investigated one of the potential candidates included in this study. This is because the clay has been used as a reference material in the current review of different XRD analyses. The clays were found to be very suitable as reference substances since unweathered samples are currently available at the Fyleverken Co quarry in the Fyledalen valley. Samples for the present study were obtained from an approximately 80 m thick sequence of clay.

At present the clays are exploited to a very small extent by Fyleverken Co, which mainly utilizes the glass sand in the area.

3.5.4.2   Outcrops, thickness and lateral extension

The clay is exposed in a 1-2 km wide zone which runs along the east side of the Vomb Basin (Fig 16) and extends from Sandhammaren in the SE to the town of Eslöv in the NW. Thus, the total length of this zone is approximately 75 km. The clay represent a sequence of sediments which comprise ferrogeneus sandstones, coals seams and quartz sand (glass sand) in addition to the clay strata. It should be mentioned that this sequence has also been found in boreholes south of Helsingborg at 78-138 m depth (22).

An excellent locality for studying the clay sequence is close to the Fyleverken Co sand quarry in the northeastern end of the Vomb Basin. Here, the sequence is in an upright, slightly overturned position and the strata can be sampled and exploited down to at least 200 m depth. The upright position (Fig 17) is caused by strong lateral compression of the earth crust during early Cretaceous.

The lateral extension of the clay in the Fyleverken area is approximately 1.5 km^2. This yields a volume of 75x10^6 m^3 clay if it is quarried to a depth of 50 m.

Rhaetic and Jurassic sediments at the base of the Pleistocene

Rhaetic and Jurassic sediments covered by pre Pleistocene sediments
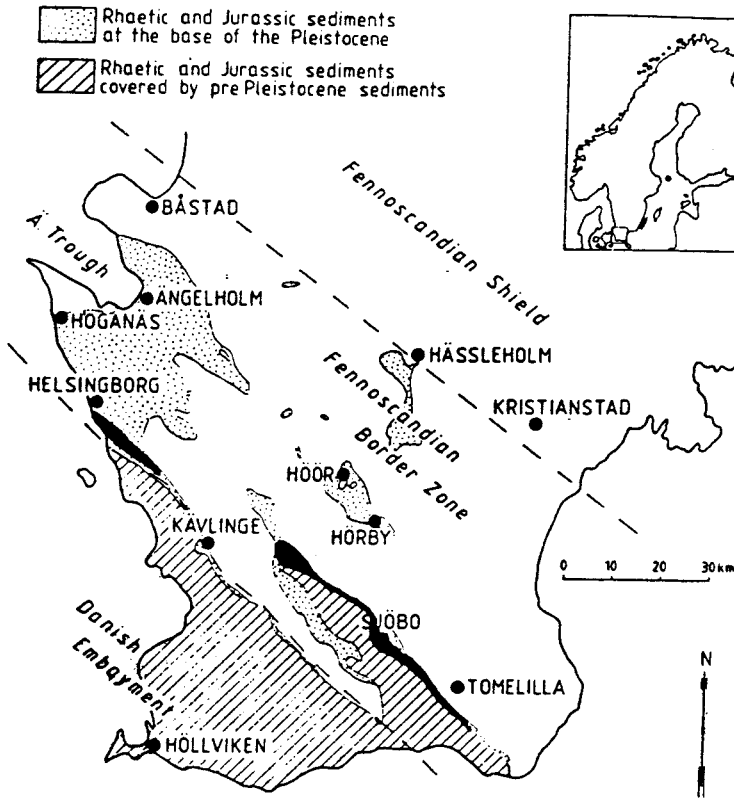
Fig 16. Dark shaded area indicates where the Fyledalen clays are located

SW   NE

FYLEVERKEN SAND PIT   KURREMÖLLA VALLEY

Fyledal clay	Glass sand	"Eriksdal beds"	Ferruginous sandstones
Port.-Kim.	Bathonian	Bajocian	Lower Jurassic

Clay

Coal

Sand(stone) Silt (stone)

Sideritic sandstone

Chamositic sandstone

0   50   100 m
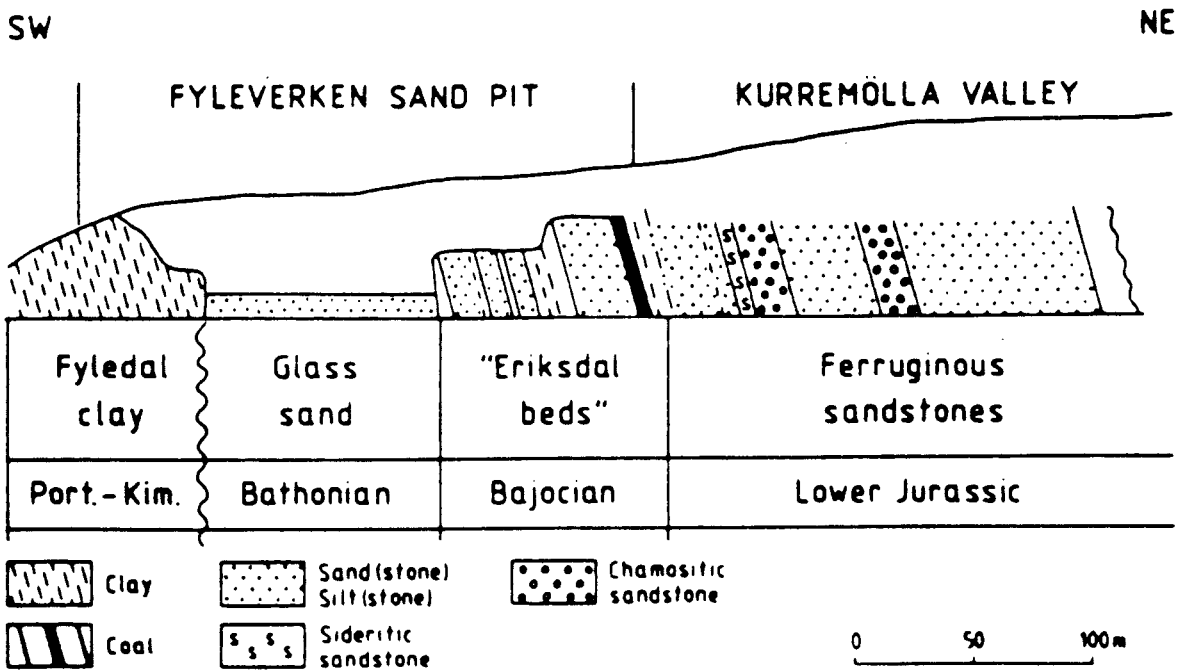
Fig 17. Schematic illustration of the position of the Jurassic sedimentary sequence at the Fyleverken sand pit
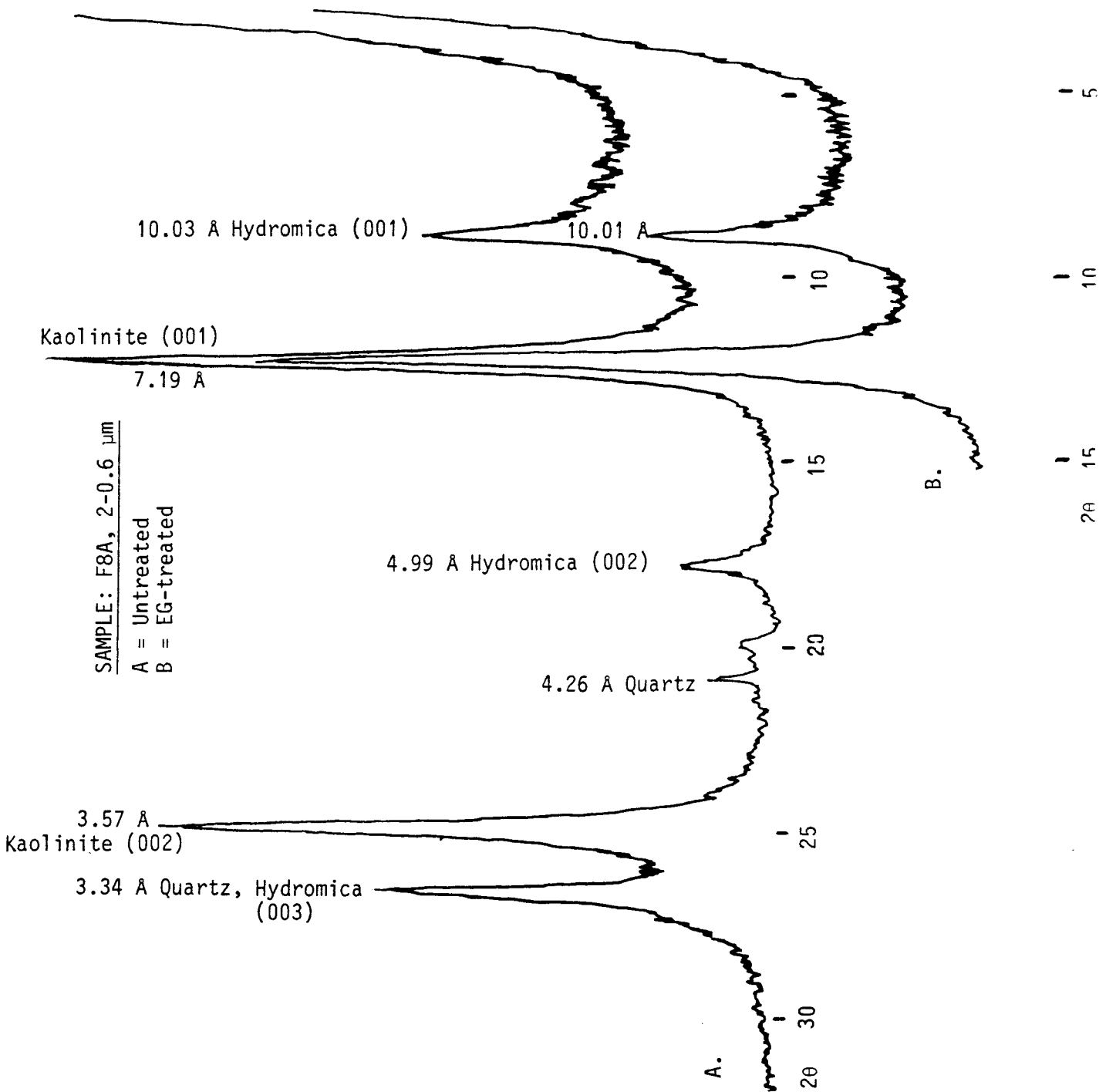
Fig 18.   X-ray diffractogram of Fyledalen clay (F8A). Untreated and EG-treated

9.98 Å
Hydromica (001)

7.19 Å

Kaolinite (001)

4.99 Å Hydromica (002)

4.24 Å Quartz

SAMPLE: F 11, 2-0.6 µm

A = Untreated
B = EG-treated

3.57 Å
Kaolinite (002)

3.33 Å Quartz,
Hydromica (003)

Fig 19.   X-ray diffractogram of Fyledalen clay (F11). Untreated and EG-treated

3.5.4.3   Lithology

The clay is characteristically composed of alternating beds of varying green to almost black, coloured clays. They are commonly referred to as the "green clays of Fyledalen" and belong, stratigraphically, to the uppermost Jurassic period (Fig 5).

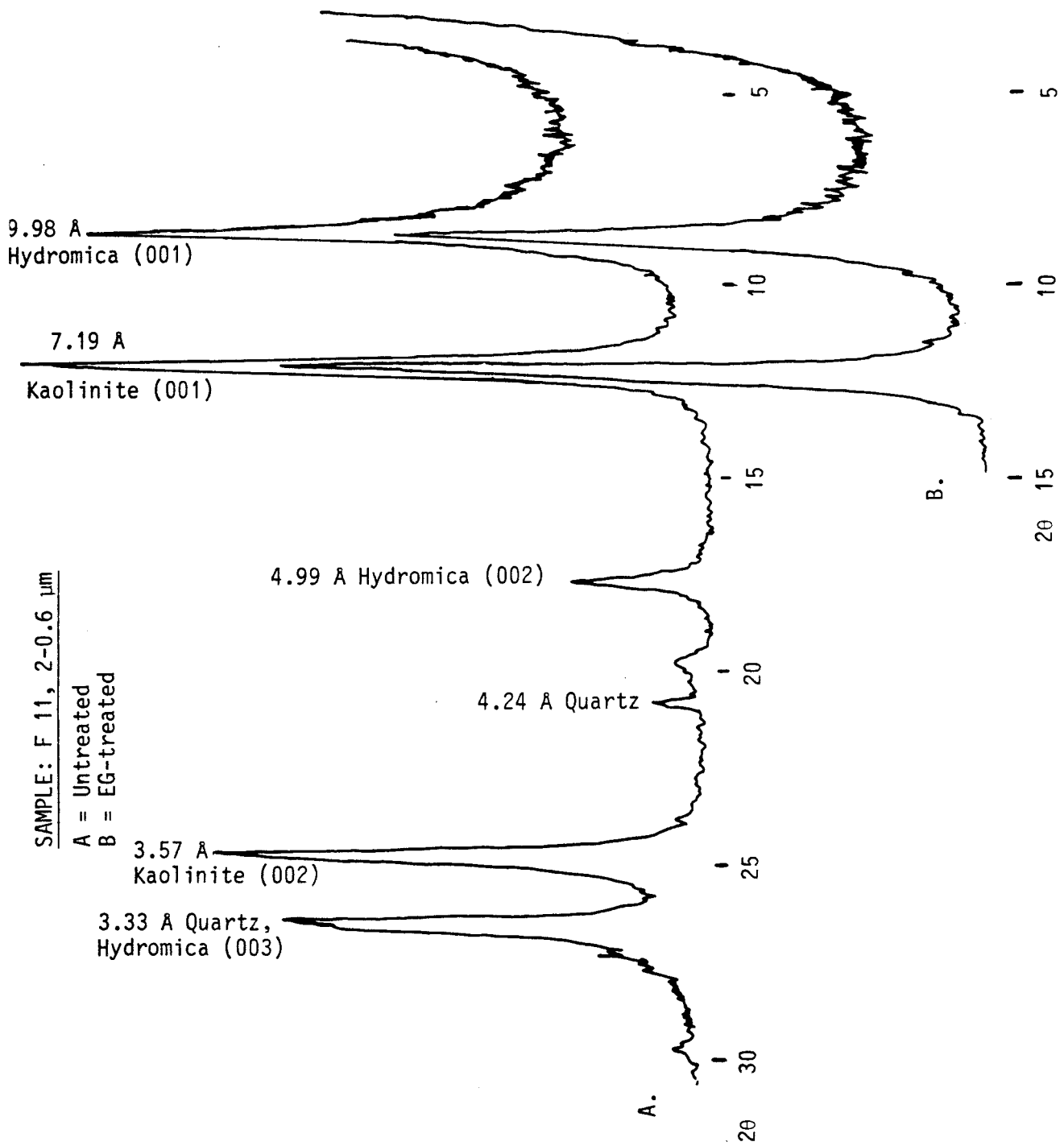The clays of the investigated 80 m profile consist of greenish grey, and blackish grey, occasionally slightly reddish material. Clay-rich beds have an individual thickness of 0.5-2 m.

The clay content is generally very high. Thus, the hydrometer analyses show that it exceeds 66 %, and that it is frequently as high as 90 %. Hydrometer analyses of samples F8A and F11, which are described in detail in a subsequent chapter, are presented in Fig 20 and 21. The liquid limit ranges between 40 and 65 %.
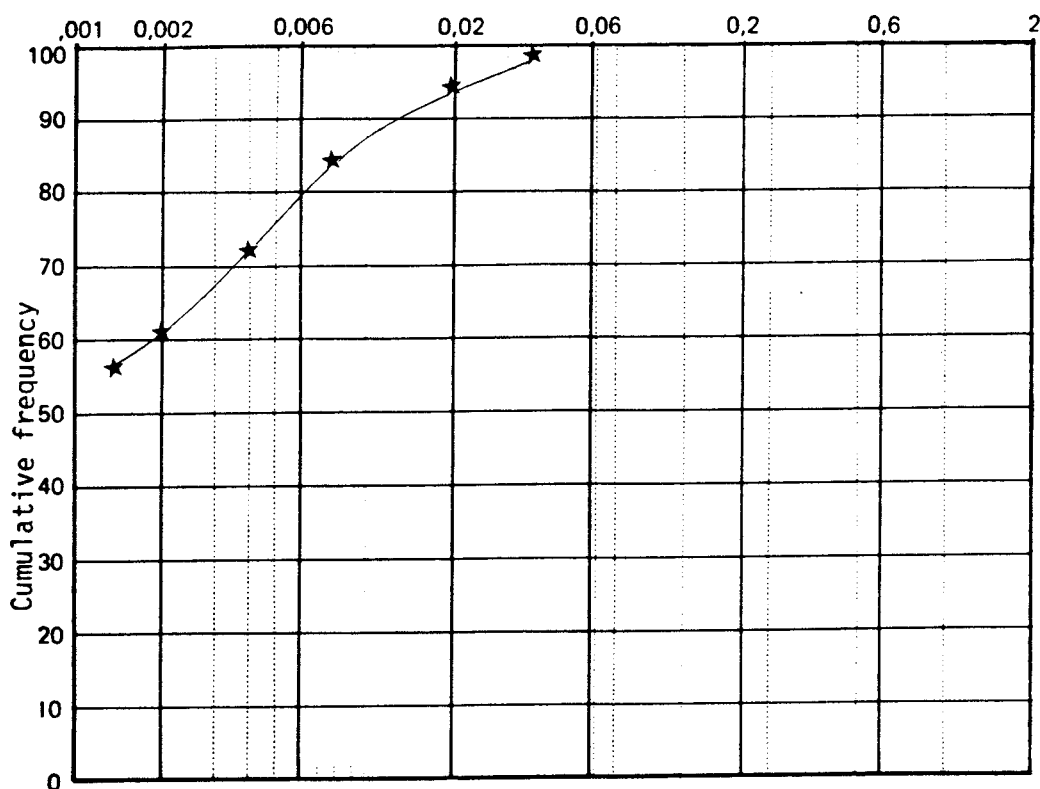

Fig 20.   Grain size frequency diagram of Fyledalen clay, sample F8A

The XRD-analyses showed that hydromica and kaolinite are the dominating clay minerals (Fig 18, 19). The samples F8A and F11 were taken as reference materials in a separate study of the applicability of Reynolds' technique for quantitative clay mineral analysis (cf. Chapter 4.3) and this detailed study demonstrated
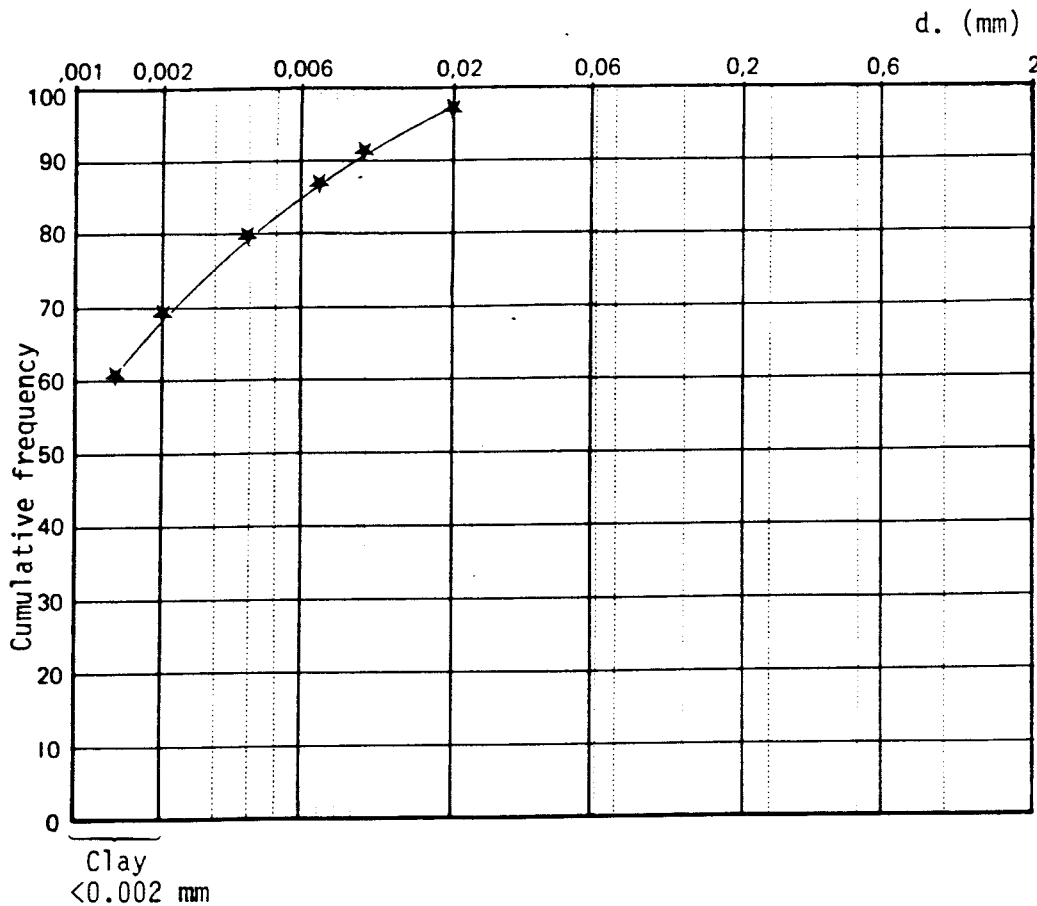
Fig 21.   Grain size frequency diagram of Fyledalen clay, sample F11

that there are two types of clay mineral associations in the Fyledalen strata, one with kaolinite as dominant mineral (F11), and one with hydromica as major constituent (F8A).

3.5.4.4   Chemical composition

The chemistry of the Fyledalen clays varies considerably also within apparently homogeneous strata although this is probably due to the variation in the amount of accessory minerals like quartz. Thus, the content of clay minerals is relatively constant within each stratigraphical unit. Main chemical data are shown in Table 4.

Analyses of the organic content of the Fyledalen clay gave values ranging between 3 and 9 %. The higher content was found for the blackish beds. The carbonate content was generally very low but occasional, thin horizons with nodules of carbonate were found to occur in the upper part of the investigated sequence.

Table 4.   Bulk chemical composition of samples representative of the
Fyledalen clay. KA = kaolinite, HM = hydromica

Compound	F 4	F 6-7	F 8 A	F 11
SiO_2	57.4	77.9	67.7	55.8
TiO_2	1.01	0.90	0.95	0.96
Al_2O_3	24.3	13.3	20.4	23.1
Fe_2O_3	6.6	2.1	2.5	8.8
MnO	0.03	0.02	0.02	0.05
CaO	0.5	0.4	0.7	0.5
MgO	1.24	0.39	0.56	1.23
Na_2O	<0.10	<0.10	<0.10	<0.10
K_2O	3.7	1.2	1.5	4.2
BaO	0.08	0.08	0.14	0.07
CO_3	0.03	0.04	0.01	0.04
Total (%)	94.89	96.33	94.48	94.75
Clay cont (%)	78	65	70	65
Clay mineral	KA HM	KA HM	KA HM	KA HM

3.5.4.5   Stress and temperature history

While the previously discussed clays had only been insignificantly affected by
stress and temperature the Fyledalen sediments have been exposed to very high
stresses and a temperature of up to 80°C.

3.5.4.6   Summary

The Fyledalen clay sequence consists of an approximately 200-400 m thick series
of green and dark grey clay beds with an individual thickness of 0.5 m to 2 m. The
clay content is high (>65 %) and this fraction consists of hydromica and kaolinite
minerals in different strata. The lateral extension and thickness, at the

Fyleverken Co sand pit in the northeast corner of the Vong Basin, is >1.5 km^2 and this yields about $>75 \times 10^6$ m^3 of clay available for exploitation.

3.5.5 Other potential clay candidates

The survey of potential candidates also comprised other formations but none of them offer sufficient quantities to be considered as potential sources of buffer materials, except for a very promising Tertiary clay deposit just across the Öresund channel. It has been included here despite the fact that it is located in Denmark. This deposit and certain bentonite-type clay beds in southwestern Sweden as well as clay-weathered crystalline rock in general, are briefly described in the subsequent text (cf. 30).

The Kinnekulle/Billingen clays

The bentonitic clays of Middle Ordovician age in the area south of Lake Vänern represent a number of strata of a thickness varying from less than one centimeter to about two metres. They were originally deposited as volcanic ash which sedimented in sea water. The clay material is composed of mixed-layer clay minerals (montmorillonite-hydromica) in a random interstratification and usually with a low proportion of montmorillonite. The mineralogy of the layers and beds varies due to chemical alteration caused by groundwater percolation and diffusion of potassium. The location of the thicker beds is in high-productive agricultural land and in park areas and they are therefore hardly accessible for exploitation.

Clay-weathered zones in crystalline-rock

Weathered zones are commonly occurring in fractured and tectonically disturbed crystalline rock. The weathering processes have occasionally proceeded to a depth of more than 100 m in granite, forming irregular zones of clay-rich material. Typical zones of possible interest are the rocks of deep fault zones and those forming strongly tectonized areas, like the Karlshamn area in southern Blekinge. Here, smectite-rich clays can be found in relatively large quantities although the variation in composition and accessibility probably makes exploitation expensive.

A special case of altered crystalline rock is the large kaolinite deposits of Cretaceous age in northeastern Scania. The clay was formed by weathering of granite from which it was eroded and river-transported to where it is presently found. It is

being exploited industrially and can hardly be considered as a buffer material re-
source.

Quaternary clays

Soils of glacial origin may be of potential use in repositories. Thus, Ancylus and
Litorina clays, which form transients between older, varved deposits and younger
usually organic-rich post-glacial sediments, can possibly be used for repository
backfilling. They contain as much as 40-50% hydrous mica but since they usually
do not form thick beds - the average being 0.5-2 m- and represent productive
agricultural land exploitation is hardly practical. Also, the high water content
(60-100%) requires drying and grinding on an industrial scale and both excava-
tion and land restoration are expected to be very expensive.

The most promising Quaternary candidate materials are the smectitic moraine
clays of southwestern Scania since they have smectite contents of up to 5-10 % and
a granulometry that is suitable for backfilling and effective compaction. They are
further treated in a separate chapter (3.6).

Tertiary clay from Denmark

In the course of the present study certain Danish clays (Faxe) have been found to
be of considerable interest since they represent large quantities of rather homo-
geneous smectite-rich deposits. They are of Tertiary origin and have a clay con-
tent which is generally higher than 75 %. Smectite clay minerals and minor
amounts of hydromica and kaolinite form the clay fraction. Feldspars, sulphides
and carbonates are very rare but organic compounds represent approximately 1-9
%.

The attractive petrophysical character and the large amounts that are available,
i.e. 40 m bed thickness and a lateral extension of several tens of square kilo-
meters, make these deposits almost equivalent to the Sardinian bentonites, a
further similarity being the Ca saturation.

3.6.1 Quaternary clays

3.6.1.1 General

The present study has been focussed on the Scanian clay deposits since they have been exploited for the production of brick and ceramic products on a large scale for a very long time by which their location and general properties have been fairly well documented. Also, geological mapping has been made more detailed than in most other areas in Sweden. Four clay-rich areas have been identified (cf. Fig 22):

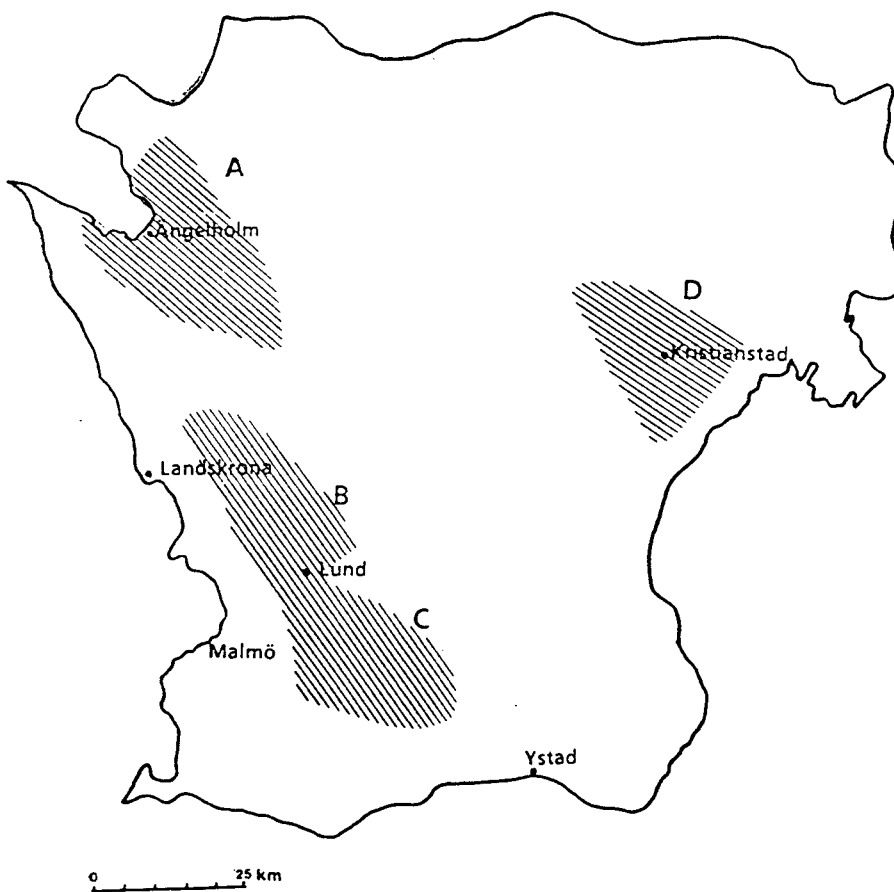A: Ängelholm
B: Lund-Landskrona
C: Svedala-Skurup
D: Kristianstad

Fig 22.   Location of promising clay-rich Quaternary deposits

3.6.1.2   The Ängelholm area

Glacial clays

Large parts of the flatlands of the Skälderviken bay area bear glacial clays with a clay content ranging between 20 and 70 %. These varved clay beds, which are 15-85 m thick, are covered by 1-5 m of sand. The clay beds are interfoliated by sand layers and boulder clay is relatively abundant in the series. The carbonate content in the clay-rich parts is usually 15-18 % while the clay mineral composition is not known with any certainty. There are indications that hydromica and chlorite are major clay fraction constituents and that expanding minerals are also present (vermiculite and smectite).

Postglacial clays

Postglacial clay beds are not very abundant and they are less than 1 m thick.

3.6.1.3   The Lund-Landskrona area

Glacial clays

Fine-grained glacial clays have been found only locally and they are often covered by thick moraines. Outcrops of glacial clay are exposed at Lödde river, the Borgeby brick plant and at Lomma. the thickness of the beds usually being less than 3 m.

Postglacial clays

Postglacial clays have not been reported in the area.

Moraine clays

Moraine clays, fine-grained as well as coarse-grained, are very abundant. They are poor in coarse material and have a clay content of 10-30 %. The content of calcium carbonate is locally as high as 15-20 %. Literature data indicate that hydromica forms 25-65 %, smectites 10-50 %, kaolinite 35 % and chlorite less than 10 % of the clay fraction. The general grain size distribution is probably not very different from that of the artificially produced smectite-poor mixtures of bentonite

and graded ballast that were used in the BMT test and for the foundation of the SFR silo. For that reason it is felt that the huge quantities of moraine clays that are available in this area form a major source of backfill materials.

3.6.1.4   The Svedala-Skurup area

Glacial clays

A number of smaller deposits of rather uniform composition have been identified and they form up to 10 m thick beds (Torup) on moraine ridges and in valleys. The carbonate content is up to 20 % and the major clay mineral is hydromica. Smaller amounts of vermiculite and smectite have also been found.

Postglacial clays

Rather thick deposits of organic-rich postglacial clays are abundant but their lateral extension is small. The content of clay minerals is higher and the carbonate content lower than for the glacial clays.

Moraine clays

The moraine clays have a similar composition of the clay fraction as the glacial clays. The clay content is, however, not higher than 5-10 % and these materials therefore have a lower rank than those from the Lund-Landskrona area.

3.6.1.5   The Kristianstad area

There are large continuous areas of thick deposits of varved clays but a complete overview over the areal distribution and composition are not yet at hand since the mapping made by the Swedish Geological Survey is still not complete. It can be assumed, however, that the composition of the presumably huge masses is similar to that of the previously discussed areas since the general types of bedrock are not very different.

3.6.2 Comments

3.6.2.1 General

It can be concluded that there is only a limited number of potentially useful smectite-rich buffer material candidates in Sweden. The sediments of Scania represent the most promising materials. Certain Scanian smectitic moraine clays may serve well as backfillings.

The Vallåkra, Kågeröd and Fyledalen clays are those which appear to be most promising because of their clay mineralogy and since each of them represents a quantity of at least a few million tons. Further comments are:

*   The Vallåkra clay is composed of both kaolinitic and smectitic clay minerals and has a high clay-content. The beds at Margareteberg are of great potential interest but further work has to be made to find out a sufficiently cheap and effective way of "activating" the clay material. If this can be achieved a sufficiently good material for canister embedment and plugging purposes is at hand. At any rate, the material would serve very well as backfill also with no processing.

*   The Kågeröd clay consists of a reddish smectite clay which is found over large areas in Scania. The lithology varies both laterally and vertically but in general more than 2-10 m deep strata of uniform clay are found over areas covering several square kilometers. The clay content is generally in the range of 20-35 %, which makes this type of clay potentially useful for backfilling purposes. It can hardly be used for canister embedment unless the amount of clay minerals can be increased by some suitable large scale processing

*   The Fyledalen clays are the ones that are most easily mined. They represent homogeneous alternating strata which are rich in kaolinite and hydromica, the latter being suitable for backfilling

*   A promising candidate is the Danish smectite-rich clay which is common in large parts of Själland and which forms beds with a thickness of 30 m. The clay content is generally >70 % and the liquid limit ranges between 80 and 100 %, meaning that the clay is comparable to

the Sardinian bentonites with respect to the mineral composition. The Danish clay certainly represents a rich source of buffer materials for canister envelopes

* Smectite moraine clays are available in large quantities and deserve to be ranked as potential backfilling materials. Actually, the grain size distribution is very suitable for compacting the materials to a very high bulk density

In summary, this study has yielded the following major candidates:

	Clay type	Major clay minerals	Poss. buffer function
1)	Kågeröd clay	Smectite, hydromica	Backfill
2)	Danish Tertiary clay	Smectite, hydromica, kaolinite	Canister embedment and plugs
3)	Fyledalen clay	Hydromica, kaolinite	Backfill
4)	Fyledalen clay	Kaolinite, hydromica	Backfill
5)	Vallåkra clay (Vallåkra)	Kaolinite, hydromica	Backfill
6)	Vallåkra clay (Höganäs, Margreteberg)	Smectite, hydromica	Backfill (Canister embedment)
7)	Moraine clay (S Sweden)	Hydromica (+smectite)	Backfill

3.6.2.2 Practical aspects

While sedimentary postglacial and glacial clays contain so much water that drying and grinding is required in order to bring the material in a suitable condition for handling, application and compaction, the moraine clays and the candidate clays of pre-Quaternary origin may well be used as backfilling materials without processing. The present physical state is illustrated in Table 5.

Table 5. Natural water content (w, %), liquid limit (w_L, %),
and bulk density (> t/m^3) of the investigated clay
materials

Clay	ρ (t/m³)	w (%)	w_L (%)
Vallåkra	2.1	15	40-50
Vallåkra (Margreteberg)	1.9	20	60-80
Kågeröd	1.9-2.3	15	40-60
Fyledalen (green clay)	2.0-2.1	20	40-50
Fyledalen (black clay)	1.9-2.0	25	50-60
Tertiary (Danish clay)	1.6-1.8	35	80-100

It is essential to realize that all the materials described here probably need some
sort of processing or refinement. However, depending on the choice of geometry of
the canister embedment and plug arrangements such processing can be minimiz-
ed, a possible philosophy being to compensate a certain variation in smectite
content and distribution by an increase in barrier thickness.

4   QUANTITATIVE X-RAY DIFFRACTION ANALYSIS

4.1   GENERAL

Quantitative or rather semi-quantitative analyses of clays with respect to their
mineralogy have been applied for decades by several investigators but the diffi-
culties met with and the obvious, very low accuracy arrived at have not been
encouraging. Only recently more reliable methods have been introduced and they
will be discussed here with the aim to define a suitable technique for application
in future SKB buffer characterization.

In principle, the X-ray peak intensity of a clay-mineral is related to the amount of
the mineral that is present in the analysed mixture. However, the intensity is
strongly related to sample preparation and the conduction of the X-ray diffraction
test and it is therefore of great importance to standardize these procedures.

One major principle of simple estimation has been to compare the peak heights
and shapes of the diagram with those of various standard minerals (31). A more
common technique is the "internal standard X-ray diffraction method" according
to which the clay composition is determined by comparing the diagram with those
of mixtures of reference clays of various composition to which certain standard
minerals are added. The key parameter is the ratio of the integrated intensity of
characteristic diffraction peaks (32, 33, 34). Various investigations have suggest-
ed that the internal standard method yields results with an accuracy of 3 to 10 %
(35, 36, 37), but this seems to be an exaggeration.

Corundum (Al_2O_3), boehmite [$AlO(OH)$], fluorite (CaF_2) have ben proposed as
suitable standard substances but Mossman, Freas and Bailey (33) found that
pyrophyllite [$Al_2Si_4O_{10} (OH)_2$)] was the most suitable mineral for use as an inter-
nal standard in clay-mineral mixtures. The structure is similar to that of the most
common smectite montmorillonite, the crystal lattice of which is shown in Fig 23.

4.2   REYNOLDS METHOD OF QUANTITATIVE XRD-ANALYSIS

At the AIPEA conference in Denver, 1985, R.C. Reynolds specified the detailed
steps in a relatively simple procedure that can be used for the estimation of the
content of clay minerals in a polymineral soil sample. His method is referred to in
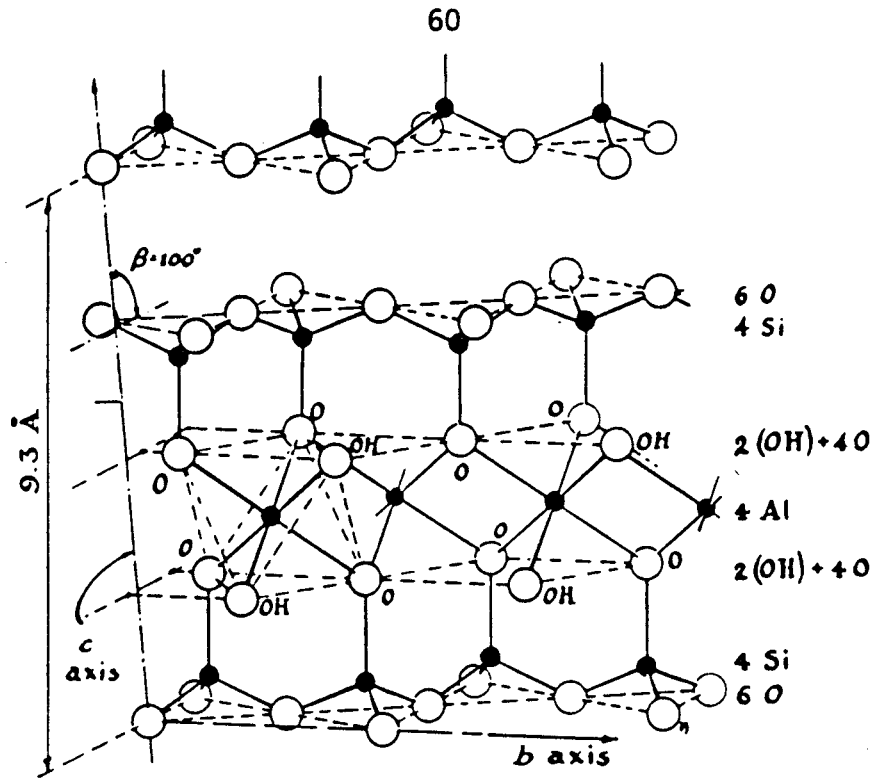the subsequent text.

Fig 23.   Lattice characteristics of the monoclinic mineral pyrophyllite [after Berry & Mason (38)]

4.2.1   Basic equation

In principle, there is a general equation that relates the intensity I of individual XRD peaks to crystallographic geometrical and instrumental properties (Eq. 3):

$$I = K \; W_f(1/V^2)(1/\rho) \; [F]^2(1 + cos^2\theta) \; (1/sin2\theta) \; (1/\mu^*) \tag{3}$$

where: K   = instrumental coefficient

W_f = Amount of a certain clay mineral

V   = Volume of unit cell

ρ = Specific density

F   = Scattering coefficient, related to the particle orientation

θ = Angle between beam direction and plane of lattice atoms

ψ = Divergence of beam:, notice: for random particle arrangement $1/sin\theta$

μ^* = Mass absorption coefficient (little importance for clay minerals)

Considering two clay minerals in a polymineral mixture and assuming K, ψ and μ^* to be identical, we obtain from Eq. (4):

$$\frac{I_a}{I_b} = \frac{W_{fa}}{W_{fb}} \cdot \frac{Q_a}{Q_b} \tag{4}$$

where I_a and I_b = Intensities of minerals $\underline{a}$ and $\underline{b}$, and W_{fa} and W_{fb} = Amounts of minerals $\underline{a}$ and $\underline{b}$

$$Q_a = (1/V_a)^2 \,(1/\rho_a)\, F_a{}^2 \,(1+\cos^2 2\theta_a)\, (1/\sin\theta_a)$$
$$Q_b = (1/V_b) \,(1/\rho_b)\, F_b{}^2 \,(1+\cos^2 2\theta_b)\, (1/\sin\theta_b)$$

From this we derive Reynolds' mineral intensity factor MIF:

$$\frac{Q_a}{Q_b} = MIF \tag{5}$$

It is obvious that if MIF is known for two minerals and the amount of one of them is known (reference material, W_{fb}), W_{fa} can be calculated if the intensity ratio I_a/I_b is derived from X-ray diffraction graphs.

4.2.2. Practical use

Pyrophyllite can preferably be used as a reference substance ("standard") since it is a flaky mineral with orientation and grain size properties similar to those of smectite. Quartz or feldspars cannot be used because of the quite different particle orientation behavior of such grains.

For any two clay mineral constituents $\underline{a}$ and $\underline{b}$, there is proportionality between the percentage of $\underline{a}$ and the intensity of characteristic diffraction peaks divided by MIF such that:

$$a \propto \frac{I_a}{MIF_a} \tag{6}$$

$$b \propto \frac{I_b}{MIF_b} \tag{7}$$

From this we obtain:

$$a = \frac{I_a}{MIF_a} \cdot b\left(\frac{MIF_b}{I_b}\right) \tag{8}$$

Now, taking $\underline{b}$ to be pyrophyllite we see that if MIF_a and MIF_b are known (with respect to the other mineral) and Ia and Ib are measured, $\underline{a}$ can be determined. For a 4 clay mineral assembly the result of such an analysis may then appear as in Table 5.

Table 5. Calculation of a mineral analysis (After Reynolds)

Mineral	Integrated intensity (I)	MIF	I/MIF	100 (I/MIF)/Sum % present
a	20	4	5	18.5
p (phyrophyllite)	15	1*	15	55.5
c	50	10	5	18.5
d	10	5	2	7.4
			27	101.9

*By definition

4.2.3 Validity, practical measures

Reynolds pointed out that there are two major sources of error which are specified and commented as follows:

1   The sample has to be sufficiently large (long) and not too thin
2   The sample must be properly prepared
3   Proper choice of peaks has to be made

Length of specimen

The diffraction intensity is proportional to the volume of the sample irradiated, which should therefore not be too small. At low diffraction angles and a small length of the sample a large part of the incident beam passes the "target". One way of minimizing errors is to use reflections that are close together on the diffractogram.

Preparation of samples

The best technique is to vacuum-filter clay suspensions through 0.45 µm membrane filters which are inverted - after partial drying - onto glass slides. The filter is stripped off leaving the clay film on the glass.

Although this mode of preparation yields homogeneous clay samples there are differences in orientation. Thus, smectite flakes tend to be particularly well oriented. This influence of varying degrees of orientation is effectively counteracted by using 2θ-values that are higher than 10°C.

Selection of peaks

According to Reynolds, the following peaks are the most suitable ones:

(Glycol) smectite	005	
Mg-vermiculite	005	
Kaolinite	002	
Chlorite	003	(The matter is very complex but Reynolds states that this selection is a useful analytical tool)

Recommended MIF-values

Reynolds listed mineral intensity factors with the intensity of the 003 peak of hydromica (illite) taken as unity (Table 6). MIF factors for other reflections can be calculated from those given, by making use of known relative values for the 001 series of a given mineral. For example, if a standard yields $002 / 003 = 0.5$, the MIF for the illite 002 is 0.5.

Mixed layer I/S in the glycol-solvated condition can be determined in two different ways according to Reynolds. The intensity of the illite (003) - smectite (005) mixed-layer peak is almost independent of composition and ordering (cf. Table 6) and is suitably used if there is no interference by hydromica. Alternatively, the hydromica (002)-smectite (003) reflection can be used although its intensity is very sensitive to the iron content (a very small ratio indicates a high iron content, and vice verse). It is recommended to check both alternatives.

Table 6. Mineral Intensity Factors (MIF) for selected reflections. Compositional data refer to numbers of atoms per $(Si,Al)_4O_{10}$ for 2:1 minerals and per $(Si,Al)_2O_5$ for the 1:2 types

Mineral	Fe	Reflection	MIF
(Glycol) Montmorillonite	0.1	005	1.06
(Glycol) Saponite	0.0	005	1.28
(Glycol) Saponite	0.5	005	1.43
(Glycol) Saponite	1.0	005	1.58
(Glycol) Saponite	1.5	005	1.75
(Hydrated-Mg) Vermiculite	0.0	005	1.05
(Hydrated-Mg) Vermiculite	0.5	005	1.17
(Hydrated-Mg) Vermiculite	1.0	005	1.29
(Hydrated-Mg) Vermiculite	1.5	005	1.42
Illite (0.75K)	0.2	003	1.00
Glauconite (0.6K)	1.8	003	1.83
Chlorite (Symm Fe Sub)		003	1.83
Chlorite (Total Fe)*	0.0	004	1.27
Chlorite (Total Fe)*	1.0	004	1.64
Chlorite (Total Fe)*	2.0	004	2.03
Chlorite (Total Fe)*	3.0	004	2.44
Kaolinite	0.0	002	1.97
Serpentine	0.0	002	2.26
Serpentine	0.5	002	2.56
Serpentine	1.0	002	2.90
(Glycol) Corrensite	0.0	25.8*20	1.11
(Glycol) Corrensite	1.0**	25.8*20	1.33

*(004) is independent of symmetry of substitution
** Symmetrical Fe substitution

Table 6 cont.

Mineral	Fe	Reflection	MIF
(Glycol) Corrensite	2.0**	25.8*20	1.56
(Glycol) Corrensite	3.0**	25.8*20	1.80
(Hydrated-Mg) Hydrobiotite	0.0	25.7*20	0.65
(Hydrated-Mg) Hydrobiotite	0.5	25.7*20	0.79
(Hydrated-Mg) Hydrobiotite	1.0	25.7*20	0.94
(Hydrated-Mg) Hydrobiotite	1.5	25.7*20	1.10

*(004) is independent of symmetry of substitution
** Symmetrical Fe substitution

The high-angle side of the latter peak contains an extra contribution from the interstratifications and is markedly asymmetrical for random types. Reynolds recommended to use the double integrated area of the low-angle at half height of this peak and stated that the MIF for this reflection is a linear interpolation - with respect to the percentage of expandable layers - between 0.5 for pure hydromica and 0.75 for pure smectite. Figure 24 illustrates this procedure.

4.3 APPLICATION OF REYNOLDS METHOD

The technique proposed by Reynolds appears to be very useful for quantification. However, the correlation between chosen peaks, the MIF:s, and the quantities of unknown minerals in the presece of the reference mineral has to be carefully investigated before the accuracy can be set.

Firstly, the MIF for pyrophyllite has to be determined before any quantification can be performed. In Reynolds' study no value was given for pyrophyllite and the first step in the present investigation was to determine the MIF constant for pyrophyllite. This was made by adding 5, 10 and 15 %, of pyrophyllite to Fyledalen clay samples that were mainly composed of kaolinite and illite. Particles larger than 0.6 µm were removed in order to eliminate quartz and feldspar. The materials were freeze-dried and then thoroughly mixed.

A=INTEGRATED AREA
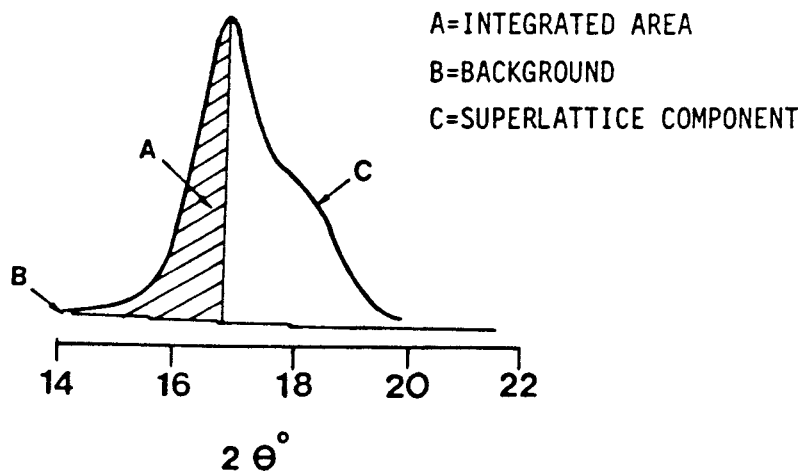B=BACKGROUND
C=SUPERLATTICE COMPONENT

Fig 24.   Measurement of I/S-peak intensity according to Reynolds

The mixed 200 mg sample was ultrasonically dispersed in 20 ml distilled water and then transferred to a membrane filter with a pore diameter of 0.45 μm vacuum filter apparatus. The oriented "clay-cake" with 47 mm diameter was then applied on a glass-slide and dried at room temperature. This technique of orientation of clay minerals is the most effective procedure for achieving preferred orientation.

The samples were analysed by use of a Philips PW1710 X-ray diffractometer with a scan speed of 1° 2θ/min.

The diffractograms (Fig 25) showed that the intensity of the recommended 003 pyrophyllite peak was too high to be suitable for analysis purposes and the 002 peak at 4.59 Å was therefore chosen as standard.

A total of 10 mixtures were X-rayed and the MIF for pyrophyllite measured. Firstly, the intensities of the 003 hydromica peak, the 002 kaolinite and the 002 pyrophyllite were calculated by multiplying the height by the width at half
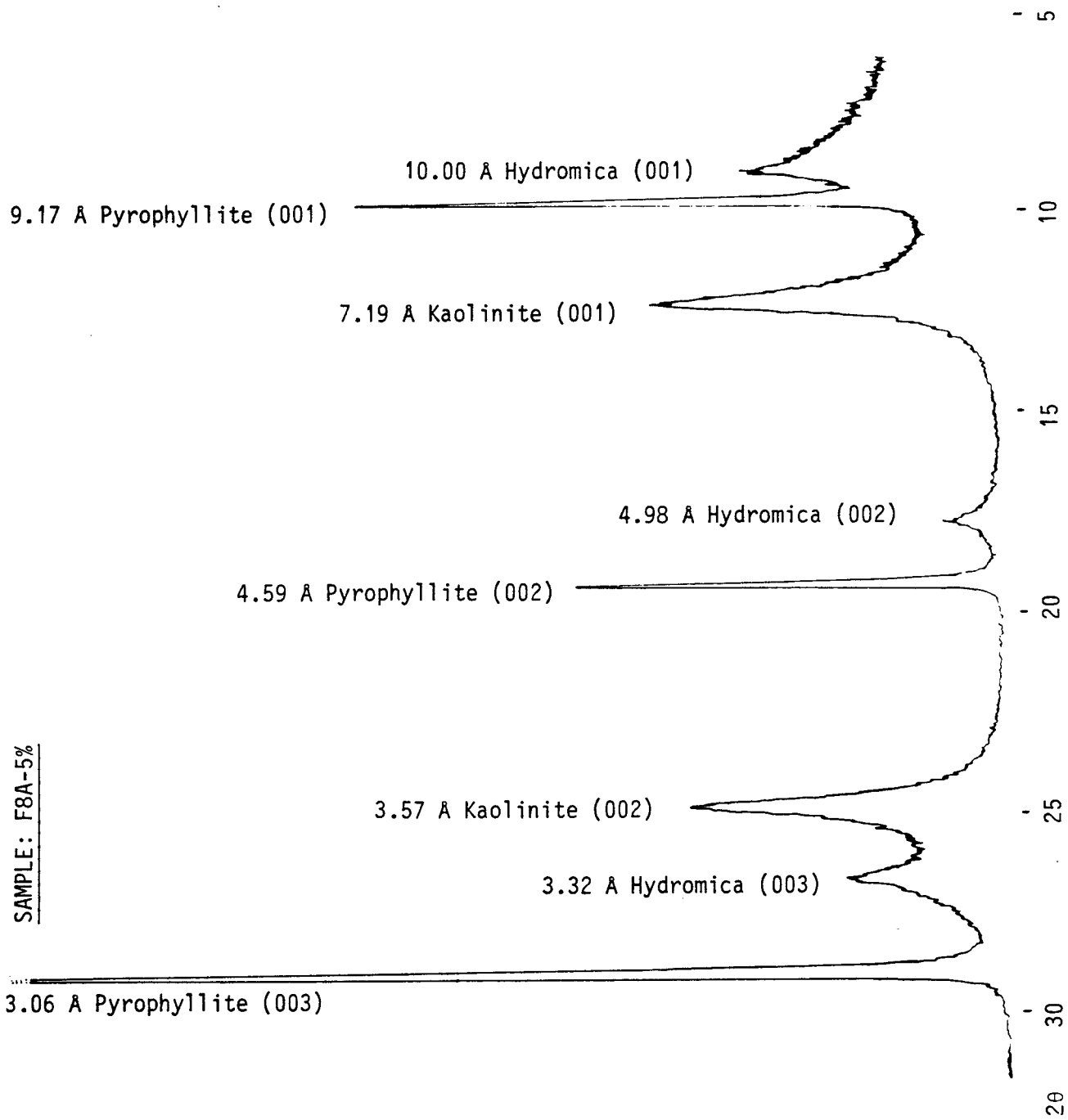
Fig 25.   Diffractogram of F8A - 5 % II with 5 % pyrophyllite added

maximum height of the respective peak. The measurements were made by use of low-magnitude microscopy in order to obtain maximum accuracy.

Table 7 illustates the MIF calculation for the diffractogram in Fig 26. Approximately 5 % pyrophyllite was added in this case and the I/MIF was calculated on the basis that the sum of the contents of hydromica and kaolinite is 95.04 %. Thus, the figure will be: (243.4+416.7)/0.9504-(243.4+416.7)=34.4. This results in a MIF of 3.99 for the 002 pyrophyllite peak (137.2/34.4=3.99) and 9.69 for the 003 peak (333.5/34.4=9.69). The peaks are easily distinguished from other clay mineral peaks over the investigated 2θ field (3-32°), which is a great advantage and makes pyrophyllite very suitable as reference substance. The MIF constants for kaolinite and hydromica originate from Reynolds' data.

Table 7.   Calculation of MIF on 002 and 003 peaks of pyrophyllite

Sample: F8A-5%II	Heigth	Width	Intensity	MIF	I/MIF	%
002 Pyrophyllite	88.5	1.55	137.2	3.99	34.4	4.96
002 Kaolinite	75.5	6.35	479.4	1.97	243.4	95.04
003 Hydromica	39.5	10.55	416.7	1.0	416.7	
003 Pyrophyllite	256.5	1.3	333.5	9.69	34.4	4.96

In F8A-5 % II, the 003 peak is also within a suitable sensitivity range (2000 cpm) and could therefore also be included in the MIF calculations. Similar measurements were performed on ten pyrophyllite-doped samples. Variations in added amounts of pyrophyllite were also made as to see if they would induce any significant change in the calculated MIF constants. This was expected because different amounts of pyrophyllite might lead to different degrees of homogeneity of the mixtures. The calculated MIF constants are given in Table 8.

The next phase of the analytic work was to calculate the amount of kaolinite and illite present in the samples. This was performed by making the assumption that the percentage $\underline{a}$ of the respective mineral was proportional to I_a/MIF_a and that the percentage $\underline{p}$ of added pyrophyllite was proportional to I_p/MIF_p. Dividing these two ratios (18) one arrives at Eq (9):

$$\% a = (I_a/MIF_a) \cdot \% p \, (MIF_p/I_p) \tag{9}$$

Table 8.   Calculated MIF-constants for pyrophyllite

Sample	002 Pyrophyllite	003 Pyrophyllite
F8A - 5 %	4.29	-
F8A - 5% I	3.93	11.30
F8A - 5 % II	3.99	9.69
F11 - 5 %	4.63	
F11 - 5 % II	3.95	11.80
F8A - 10 %	4.49	-
F11 - 10 %	4.38	-
F8A - 15 %	4.28	-
F11 - 15 %	4.40	-
F11 - 15 % I	3.79	-
MIF-average	4.21	10.93

This was made both with the hydromica and kaolinite and the sum should thus add to 100 %. Since the figures arrived at in the different tests (Table 9) are not very far from 100 % we conclude that Reynolds' method yields rather accurate results. We also draw the important conclusion that the amount of added pyrophyllite does not seem to affect the results.

A pilot test series resulted in MIF constants within the range of 3.79 - 4.63. A second series, comprising another three samples, e.g. F8A - 5% I, II and F11-5%II,which were prepared with very exact amounts of pyrophyllite, yielded MIF values of pyrophyllite of 3.93-3.99 which indicates that 3.96 can be taken as a representative value for this mineral. A quantitative analysis based on this MIF value is given in Table 10.

The correct amounts of hydromica and kaolinite in the investigated Fyledalen clays, i.e. without pyrophyllite, are given in Table 11. We conclude from this that the minus 0.6 µm fraction of these clays contain only hydromica and kaolinite. It is obvious, also, that they serve as a rich source of hydromica for backfilling pur-

Table 9.   Quantification by use of MIF = 4.21 for pyrophyllite

Sample	% Hydromica	Kaolinite	%
F8A - 5 %	59.9	33.2	93.1 (-1.9)
F8A - 5% I	55.4	46.5	101.9 (+6.9)
F8A - 5 % II	63.4	37.0	100.4 (+5.4)
F11 - 5 %	62.0	22.2	84.2 (-10.8)
F11 - 5 % II	75.7	24.5	100.2 (+5.2)
F8A - 10 %	48.6	35.5	84.1 (-5.9)
F11 - 10 %	84.4	26.1	110.5 (+20.5)
F8A - 15 %	48.9	34.7	83.6 (-1.4)
F11 - 15 %	61.5	19.8	81.3 (-3.7)
F11 - 15 % II	71.3	23.2	94.5 (+9.5)

Table 10.   Quantification by use of MIF = 3.96 for pyrophyllite

Sample	% Hydromica	Kaolinite	%
F8A - 5 %	56.3	31.2	87.5 (-7.5)
F8A - 5% I	52.1	43.6	95.7 (+0.7)
F8A - 5 % II	59.7	34.8	94.5 (-0.5)
F11 - 5 %	64.6	27.9	92.5 (-2.5)
F11 - 5 % II	71.2	23.0	94.2 (-0.8)
F8A - 10 %	45.8	33.4	79.2 (-10.8)
F11 - 10 %	73.9	23.0	96.9 (+6.9)
F8A - 15 %	48.7	34.2	82.9 (-2.1)
F11 - 15 %	60.9	18.4	79.3 (-5.7)
F11 - 15 % II	67.1	21.8	94.5 (+3.9)

poses. Large-scale compaction tests are recommended to find out how densely they can be compacted.

Table 11.   Quantities of hydromica and kaolinite in examined samples without pyrophyllite

Sample	% Hydromica	Kaolinite	%
F8A - 5 %	59.3	32.8	92.1
F8A - 5% I	54.8	45.9	100.7
F8A - 5 % II	62.8	36.6	99.4
F11 - 5 %	68.0	29.4	97.4
F11 - 5 % II	74.9	24.2	99.1
F8A - 10 %	50.9	37.1	88.0
F11 - 10 %	81.8	25.5	107.3
F8A - 15 %	57.3	40.2	97.5
F11 - 15 %	71.6	21.6	93.2
F11 - 15 % II	78.9	25.6	104.5

Samples which do not add up to 100% (+/-5%) were most likely prepared or mixed in an improper way. It should be mentioned that iron in the form of Fe_2 and Fe_2O_3 in the material yields lower intensities of certain peaks (cf Fig 26, 27). This can be observed in all F11 samples.

Naturally, there is a variation in composition also within small elements of any natural soil. It is illustrated in Table 12 for the two examined samples (F8A and F11).

A close analysis shows that the accuracy of quantitative estimates increases with dropping content of pyrophyllite. Thus, if only 5% of pyrophyllite is added the best results are obtained. This is because it is then possible to relate the pyrophyllite peaks (001-003) to each other with great accuracy and to measure peak intensities. Thus, a higher amount of pyrophyllite than 10% make the 001 and 003 peaks too large. A general conclusion is that the applied method yields quantitative estimates with an accurancy, of better than ±10%.

Table 12. Variation of hydromica and kaolinite in F8A and F11

Sample	% Hydromica	Kaolinite
F8A	50.9-62.8	32.8-45.9
F11	68.0-81.8	21.6-29.4

4.4 RECOMMENDED PROCEDURE

The application of Reynolds method of quantification of clay-minerals has been found to be most promising and it it is suggested as a SKB-standard for clay mineral quantification. Further analysis is required, however.

The experiments have shown that the MIF-constant for the added internal standard, pyrophyllite, has been identified and that it can be used as reference for quantification of the major clay minerals in a mixture. A program for sample preparation based on experiences from the experiments has been compiled. Thus, the following preparation has been found to be most accurate:

Step 1: Separation of the fraction minus 0.6 μm from the sample to be quantified.

Step 2: Freeze-drying of the minus 0.6 μm material

Step 3: Mixing the material with 5% freeze-dried pyrophyllite finer than 0.6 μm and 20-25 ml distilled water, and dispersing it by ultrasonic treatment for 15 minutes. The total weight of the freeze-dried mixture should be 200 mg.

Step 4: Preparation of an oriented sample by the use of suction technique and membrane filters. Transfer of the clay-cake to a glass slide for air-drying.

Step 5: XRD analysis covering the range of 3-32 degrees 2θ at a speed of 1 degree 2θ per minute (cps range about 2000).

Step 6: Checking that the MIF of pyrophyllite is about 4.0, and that the relation between the 002 and 003 peaks is correct.

SAMPLE: F11-5% 10.3

10.01 A Hydromica (001)

9.17 A Pyrophyllite (001)

7.18 A Kaolinite (001)

4.98 A Hydromica (002)

4.59 A Pyrophyllite (002)

3.57 A Kaolinite (002)

3.32 A Hydromica (003)
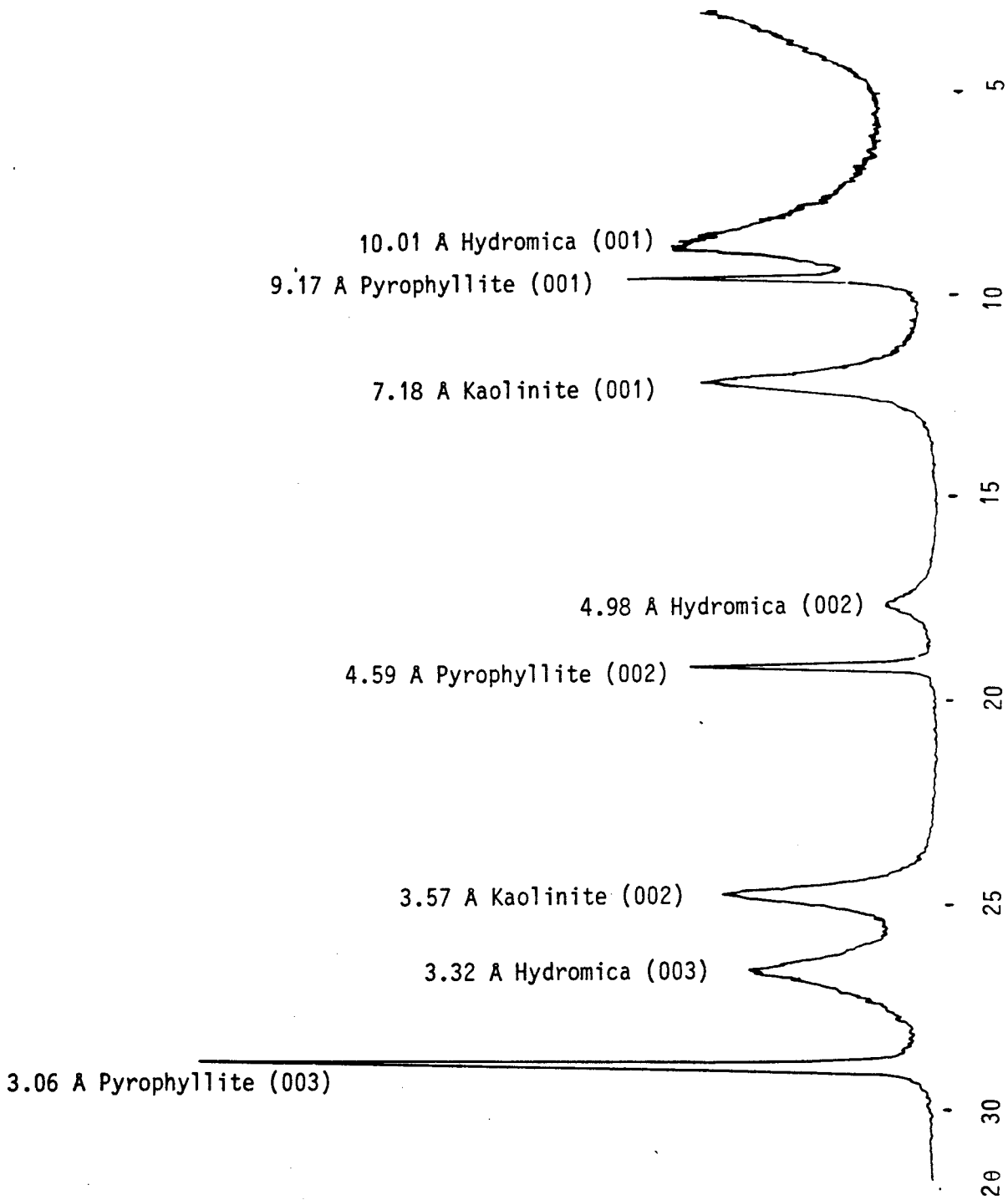
3.06 A Pyrophyllite (003)

5

10

15

20

25

30

2θ

Fig 26.   X-ray diffractogram showing suppressed peaks due to high content of iron oxides in the sample F11-10.3 (cf Fig 27). MIF for pyrophyllite was in this sample was found to be 3.18
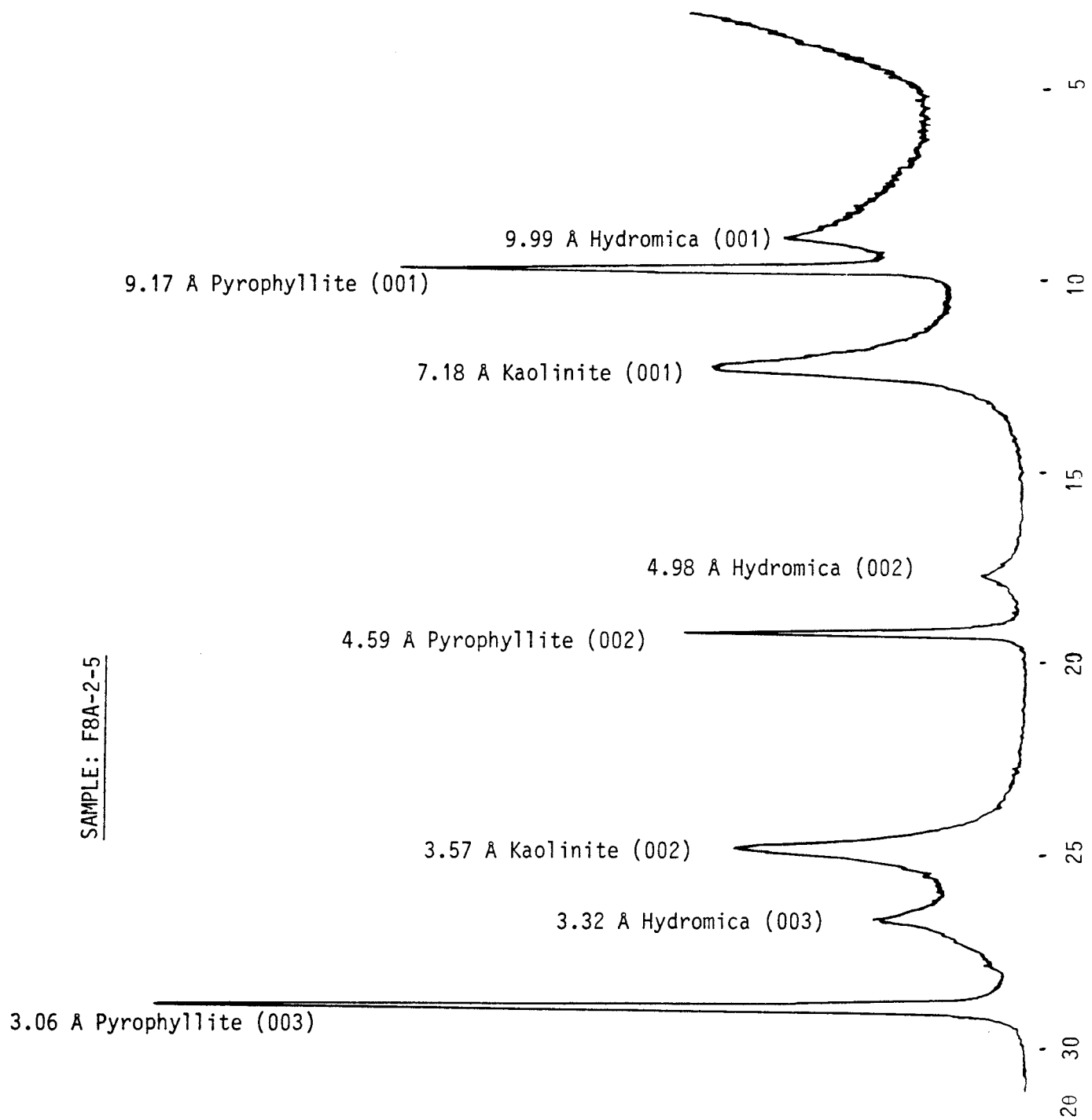
SAMPLE: F8A-2-5

9.99 A Hydromica (001)

9.17 A Pyrophyllite (001)

7.18 A Kaolinite (001)

4.98 A Hydromica (002)

4.59 A Pyrophyllite (002)

3.57 A Kaolinite (002)

3.32 A Hydromica (003)

3.06 A Pyrophyllite (003)

5

10

15

20

25

30

2θ

Fig 27.   X-ray diffractogram of F8A - 5%, with normal peak intensities of pyrophyllite (cf Fig 26). MIF for pyrophyllite 002 was found to be 4.29

Step 7: Calculation of the amounts of the different clay mineral compounds by the use of the following relationship: $\%a = (I^a/MIF^a) \, 5p(MIF^p/I^p)$. MIF for the other minerals can be taken from Reynolds data. The quantitative accuracy is estimated through the deviation from the theoretical total fraction which should be 95% for 5% pyrophyllite content. The amounts are then transformed to 100 percent by dividing the obtained totals for % a, b and c etc by 95%.

5   CONCLUSIONS AND RECOMMENDATIONS

5.1   CANDIDATE MATERIALS

The present study has given a good overview of potential clay buffer candidates in the part of Sweden that offers the best possibilities to find large accessible quantities of smectitic materials. The most promising Scanian materials are those in the Kågeröd and Vallåkra (Margreteberg) areas since they represent the most smectitic ones, which may serve as raw material for the production of canister embedment. The moraine clays in the Lund-Landskrona region seem to be useful for backfilling purposes.

Since the survey presented in this report is not very detailed and lacks information in various respects, a continuation is recommended and particular emphasis should be put on the following items:

- investigation of the lateral extension of the Kågeröd clay with special respect to the lithological variation and to the thickness of the clay-beds in the most promising areas

- continued laboratory investigations concerning "activation" of the Vallåkra clay.

- sampling of moraine clays in the Lund-Landskrona area for investigating their homogeneity and for laboratory tests concerning the hydraulic conductivity

5.2 CLAY MINERAL ANALYSIS FOR QUANTITATIVE CHARAC-TERIZATION

The quantification experiments by use of pyrophyllite as internal standard are very encouraging and indeed suggest this version of Reynolds' method to be adopted as SKB standard. However, the investigations should be extended in order to find out whether it is possible to identify also chlorite and vermiculite and possibly also different smectite species. The following additional investigations are recommended:

- Additional analyses to certify the MIF value of pyrophyllite. This should be performed by using mixtures with various standard clays e.g kaolinite, hydromica and smectite

- Further investigations concerning sample preparation in order to refine and improve the technique

- Comparison of the received results from other types of quantifications e.g transmission electron microscopy and chemical analyses

- Development of a complete clay analysis procedure for the SKB-standard characterization.

Parallel to this extended XRD study it is recommended to refine simpler methods for routine testing with respect to the usefulness of clay materials as buffers. Thus, emphasis should be put on a deepened study of the applicability of Skempton's "activity" number and similar practical tests.

6 ACKNOWLEDGEMENTS

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