# Rectorite-Pyrophyllite-Chlorite-Illite Assemblage in Pelitic Rocks from Fómeque Formation, Central Eastern Cordillera, Colombia

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Palabras claves: Clay Mineral Analysis, Fómeque Formation, Eastern Cordillera-Colombia, Illite Crystallinity, High Diagenetic-Anchizonal Metamorphism,

#### RESUMEN

La investigación aquí descrita se realizó sobre muestras de shale de la Formación Fómeque, cerca a Bogotá, Colombia. Se encontró que la pirofilita ocurre junto con ilita/esmectita, clorita e ilita. Otros minerales incluyen cuarzo, feldespato potásico, dolomita y pirita. La evaluación de los patrones de difracción de rayos-X muestra que el silicato en láminas representa una rectorita de orden R1 con 80-90% de capas de ilita.

La microfábrica i.e. la foliación, se desarrolla como un clivaje estrechamente espaciado en las rocas abundantes en filosilicatos, el cual pasa a a un clivaje de fractura a medida que aumenta el tamaño del grano. En las intercalaciones de limolitas no se observa clivaje. Se sugiere que la etapa de microfábrica desarrollada en estas rocas, reprresenta condiciones de diagénesis alta a angimetamórfica.

La cristalinidad de la ilita se ha medido en muestras tratadas con glicol, de pizarra y pasa de 0.47 a  $0.55^{\circ}\Delta 2\theta$ , con una media de  $0.52^{\circ}\Delta 2\theta$ .

En base a la estabilidad de la rectorita de orden R1, a la cristalinidad de la ilita y al desarrollo de la microfábrica, se postula que las rocas han sido sometidas a condiciones de diagénesis altas i.e. una temperatura de cerca de 200°C a baja presión. A esta temperatura, la pirofilita sólo se estabiliza a expensas de caolinita y cuarzo si  $a_{\mu 20} << 1$ .

## ABSTRACT

The research described in this paper was performed on samples of slate from the Fómeque formation near Bogotá in Colombia. Pyrophyllite was found to occur together with mixed layered illite/smectite, chlorite, and illite. Other minerals were quartz, K-feldspar, dolomite and pyrite. Evaluation of the x-ray diffraction patterns reveals that the mixed layered sheet-silicate represents an R1 ordered rectorite with 80-90% illite layers.

The microfabric i.e. the formation of cleavage is

developed as a closely spaced cleavage in the phyllosilicate-rich rocks, which grades into a fracture cleavage as the grain size becomes coarser. In the interbedded siltstone no cleavage is observed. It is suggested that the stage of microfrabric developed in these rocks represents high diagenetic to anchizonal conditions.

The illite crystallinity has been measured on glycolated samples from the slate and ranges from 0.47 to 0.55° $\Delta$ 20, with a mean of 0.52° $\Delta$ 20.

Based on the stability of R1 ordered rectorite, the illite crystallinity and the microfabric development, it is proposed that the rocks have been subjected to high diagenetic condition i.e. a temperature of about 200°C at low pressure. At this temperature pyrophyllite can only be stabilized at the expense of kaolinite and quartz if  $a_{\mu 20} << 1$ .

## **1. INTRODUCTION**

The object of this paper is to draw attention to the occurrence of pyrophyllite in association with rectorite in rocks of pelitic composition belonging to the diagenetic and/ or very low grade metamorphic zones. In the late 1960's pyrophyllite was considered to occur at the beginning of the greenschist facies i.e. at the beginning of low grade metamorphism (WINKLER 1967). Since then, it has been shown that the mineral is formed during very low grade metamorphism (WINKLER 1979). In FREY (1987) more than sixty references are listed, dealing with the occurrence of pyrophyllite during incipient regional metamorphism. For many of these references illite "crystallinity" data are available. Based on these works FREY (1987) concluded that pyrophyllite is an index mineral of the anchizone and low grade epizone in very low grade metamorphic rocks of pelitic composition. However, the temperature stability range is strongly dependent on the  $a_{_{\rm H2O}}$  and therefore no 'pyrophyllite facies is defined.

In studies carried out by BRATTLI & BROCH (1988) on rock samples of Cretaceous and Tertiary age from the Bogotá area in Colombia, pyrophyllite was found to coexist with rectorite, a 1:1 regular interstratification of a dioctrahedral mica and a dioctahedral smectite. The appearance of pyrophyllite in association with rectorite has been described from a few places in the world; i.e. western Sahara (DUNOYER DE SEGONZAC 1969; CHENNAUX *et al.* 1979), the Glarus Alp (KISCH 1983), Devonian deposits in the Pyrenees (DUNOYER DE SEGONZAC & HEDDEBOUT 1971), north central Utah (HENDERSON 1970) and from Asturias in Spain (ALONSO & BRIME 1970). As far as this author knows, the rectoritepyrophyllite association has not been previously reported from the Andes in South America.

## 2. MATERIALS

The materials used in this investigation are from the eastern part of the Andes Cordillera, some 20 km east of the city of Bogotá in Colombia. The rocks belong to the so called Fómeque Formation which is dominated by Cretaceous shale with interbedded silt- and limestones. In outcrop the bedding varies from horizontal to near vertical. The inclined strata are caused by folding which has resulted in large scale, tight to open folds with amplitudes measurable in metres or tens of metres.

In order to describe the unusual rectorite - pyrophyllite assemblage, rock samples from the Fómeque Formation (from now on called Fómeque rocks) have been collected and studied for general mineralogical content (the samples were obtained during field inspection of some extensive fallouts and slides in a water tunnel supplying Bogotá with freshwater). All the samples studied in this work are from the shale which is the dominating rock type of the Fómeque Formation.

In hand specimen the rock exhibits a certain ability to split up into very thin, planar slabs. However, the cleavage may vary considerably across the strata and over short distances. The most pronounced cleavage is found in the very fine grained parts (phyllosilicate-rich slate) and is characterized by a closely spaced cleavage with smooth to rough surfaces which sometimes appears to be microfolded. The ability to split along the cleavage is usually good. This grades into a fracture cleavage in layers where the grain size become coarser, probably as a result of differences in initial composition (clastic grains). Contrary to theshale, the interbedded siltstone and limestone totally lacks any kind of parallel fabric or cleavage development.

When rubbed across an unglazed porcelain tile the samples give a black streak, indicating that the rocks contain carbonaceous remains.

## **3. ANALYTICAL TECHNIQUES**

In characterizing the mineralogy and micro texture, thin sections have been studied both by transmitted and reflected light microscopy. In order to identify the matrix minerals and to make a semi-quantitative analysis of the mineral content, the rocks were subjected to X-ray diffraction technique. A mineral identification scan from 2-60°20 was made on all samples. Both unoriented mounts (packed into an aluminium holder) and mounts filtered on millepore filter by means of a vacuum filter apparatus were used for the investigation.

The following fast method was used for identification of expandable clay minerals. It involves spraying the mount surface with a mixture of 40% ethyl alcohol -30% ethylene glycol -30%  $H_2O$ . The preparation is then allowed to stand for some minutes until the translucence of the clay indicates penetration of the liquid.

In order to distinguish between kaolinite and chlorite several methods are available (DIXON & WEED 1977; MOORE & REYNOLDS 1989). In this case an aliquot of the samples was heated for about 6 hours in 3N HCI at 80°C. Such a treatment dissolves most chlorites, and any residual peaks at 12.6°20 (7Å) and 25°20 indicates the presence of kaolinite.

The quantitative analysis was carried out by measurement of peak intensity. The method is based upon the assumption that the intensity of a peak from a particular mineral is related to the abundance of that mineral in the mixture (MOORE & REYNOLDS 1989).

For detailed investigation of the illite crystallinity, material from the rock samples were crushed down in a mortar by hand. The crushed rock was then transferred to a plastic container for ultrasonic treatment for a few minutes (2-3min). The fraction <  $2\mu m$  was decanted, pipetted and dried at room temperature on glass slides to produce thinlayer, highly oriented preparates with density of at least  $3mg/cm^2$ .

The "crystallinity" of illites was measured by scanning the 4.0-12.0°20 range at 2°20/min. and a time constant of 2s, using a normal focus CuK $\alpha$  radiation at a tube setting at 40KV and 20mA. A Phillips PW 1775 instrument was used with an automatic divergence slit, 0.2mm receiving slit and graphite monochromator. Peak width at half height was calculated in terms of ° $\Delta$ 20 and measured from patterns of Ca-saturated glycolated samples. The sample preparation and X-ray diffraction setting are strictly after the recommendations given by KISCH (1991).

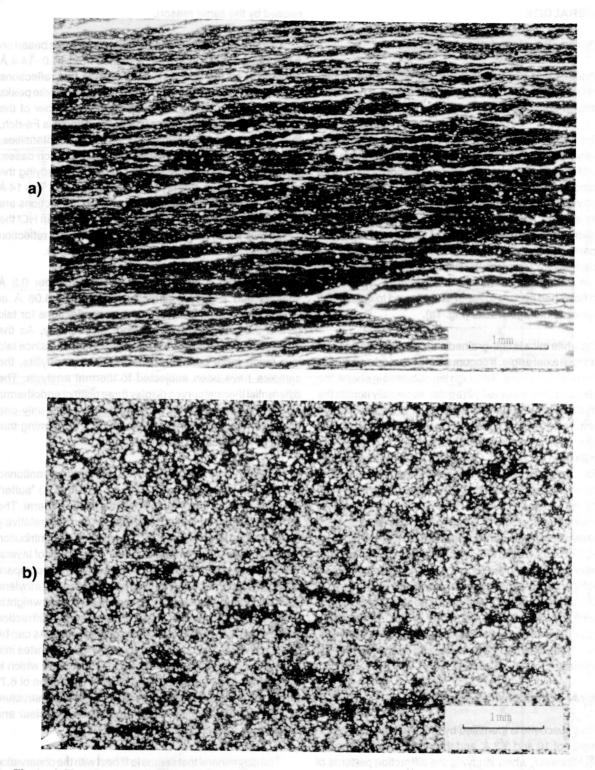


Fig.1. a) Closely spaced cleavage in the phyllosilicate-rich slate. Thin section prepared using water for grinding process. The rocks split up along the cleavage as the water dries. b) Thin section of the interbedded siltstone. No cleavage is developed. The two examples represent end members of cleavage development. All gradation seem to exist between these two as a function of the grain size.

## 4. MINERALOGY

#### 4.1. Thin section description

Minerals that can be identified are white mica, pyrite, carbonate, and very small amounts of quartz. The matrix constitutes about 50%.

The dominant feature of the shale is a closely spaced cleavage with parallel to semi-parallel fabric within the microlithons. The cleavage surfaces are mostly rough, but also smooth. Especially the sheet-silicates (white mica and pyrophyllite?) seem to have a preferred orientation parallel to the cleavage. A slight bedding represented by alternating light (white mica, and aggregates of carbonate, represent the coarser layers within the rock, see materials description) and dark layers (cryptocrystalline matrix) is usual. These may be parallel or inclined to the cleavage. Specimens which have had access to water and allowed to dry, exhibit wide open shrinkage cracks (Fig. 1a).

The white mica is fine-grained and poorly crystalline and with a small axial angle. It occurs as widespread and mostly parallel oriented grains. Although lepidoblastic in shape, the grain boundaries are usually irregular, especially across the length direction of the mineral. Some of what is thought to be mica, could be pyrophyllite. Because of the fine grain size and the poor crystallinity it is difficult to distinguish the minerals from each other. Carbonate usually appears both as idiomorphic crystals and as hypidiomorphic grains. The first type is even grained (c. 0.1 mm) and seems to grow across the S-plane, while the latter varies from very fine to fine grained and occurs in elongate aggregates along the cleavage. Most of the carbonate seemed to be dolomite as no reaction occurred in 10% HCI. The only ore mineral that has been observed in the rock is pyrite which constitutes a rather high percentage. Its grain size varies between 0.01 -0.15 mm and it appears mostly as aggregates of framboids, randomly distributed throughout the matrix. Some pyrite grains are also seen as idiomorphic inclusions in carbonate. All the pyrites seem to have sharp and well defined grain boundaries.

#### 4.2. X-ray diffraction analysis (XRD)

Illite/muscovite is identified by the two intense peaks in the region of 10 and 3.3 Å and the relatively weaker peak at 5 Å. However, when studying the diffraction patterns of the untreated samples it is obvious that both the 10 Å and the 5 Å peaks are poorly defined (Figs. 2b, d). Especially the 10 Å peak "suffers" from a broad shoulder of a superimposed peak structure belonging to a mixed layered clay mineral (see below). The broad tail of the 5 Å peak is probably

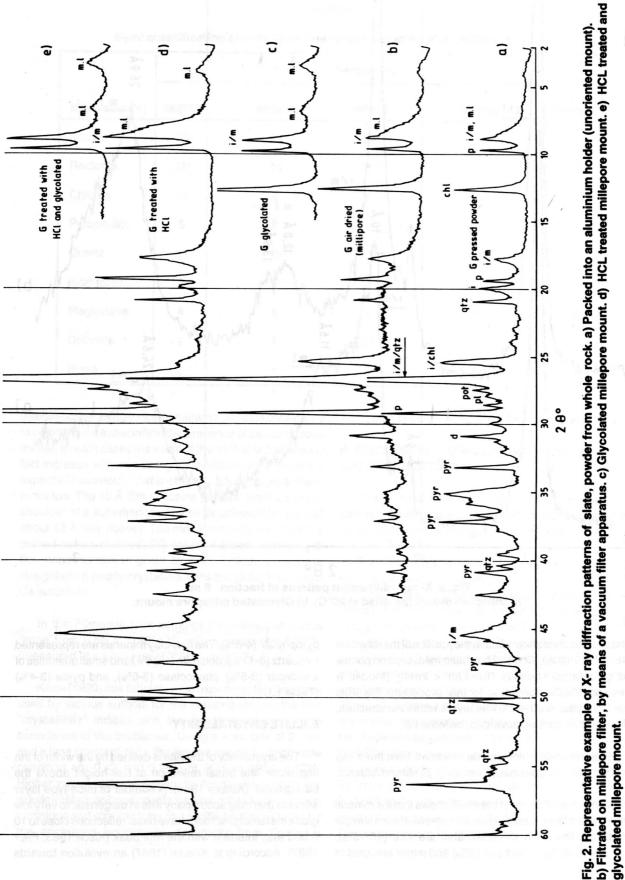
#### caused by the same reason.

Chlorite has a basal series of diffraction peaks based on a first order reflection at 14.2 Å (varies from 14.0 -14.4 Å depending on the species), and kaolinite has reflections based on a 7.1 Å structure. In fact, even-order chlorite peaks superimpose or nearly superimpose on a number of the kaolinite 001 series. In cases where the chlorite is Fe-rich, the "odd" (001, 003) reflections have weak intensities, whereas the "even" reflections are strong. In such cases, the chlorite could be mistaken for kaolinite. Studying the diffraction patterns, this is just what can be seen. The 14 Å peak is missing or very weak, while the 002 reflections are strong (Figs. 2a, b, c). After heating the samples in HCI the 7 Å peak is totally missing, indicating that the 7 Å reflection represent chlorites and not kaolinite (Figs. 2d, e).

*Pyrophyllite* is distinguished by its first order 9.2 Å spacing and a strong diffraction maxima of 3.06 Å at 29.15°20 (Fig. 2). However, the same reflections for talc give a 9.3 and a 3.10 Å spacing, respectively. As the reflections for the two minerals are so close, and since talc is a much more common mineral than pyrophyllite, the samples have been subjected to thermal analysis. The differential thermal curves display three distinct endotherms which are diagnostic for pyrophyllite (talc has only one endothermic peak, between 900 -1000°), confirming that the XRD identification is correct.

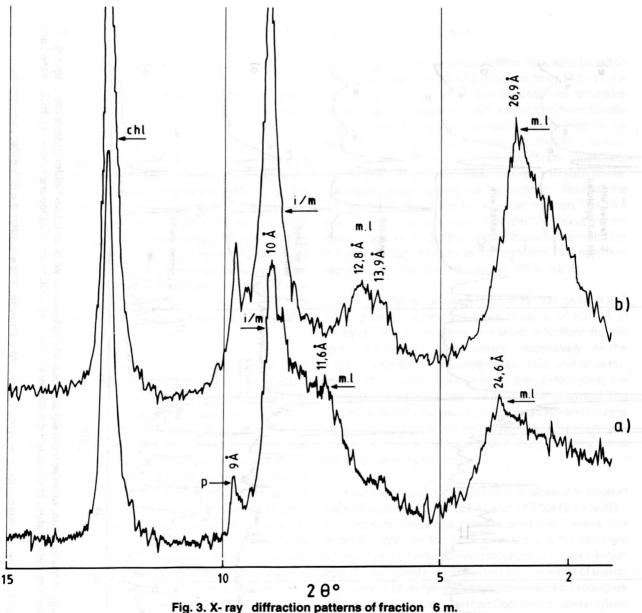
Mixed layered illite/smectite (I/S). As already mentioned the illite peaks at 8.8°20 (10 Å) and 17.7°20 (5 Å) "suffer" from the occurrence of a mixed layered clay mineral. The broad shoulder on the 10 Å structure and the relatively poorly defined 5 Å reflection indicate an important contribution from a sheet silicate formed of two or more kinds of layers. The best way of studying interlayered minerals is to compare diffraction patterns produced from both air dried and ethylene glycol-solvated preparations (Hower 1981). Most weight is given to the interpretation of the "glycol solvated" diffraction patterns because these are the most diagnostic. As can be seen from Figs. 2a, b, d, the peak at 8.8°20 separates into two peaks upon glycol solvation, one 10 Å peak which is very well defined and one broad peak in the region of 6.7° 20 (13-14 Å) (Figs. 2c, e). The weak, but visible overstructure at 3°20 (23 -24 Å) (Figs. 2b, d) becomes more clear and expands to a 26 -27Å structure (Figs. 2c,e).

The clay mineral that seems to fit best with the observation is an illite/smectite interstratification, that means a 10 + 14Å stacking, which expands to a 10 + 17 Å stacking upon glycol solvation. The position of the reflection at  $6.8^{\circ}2\theta$ (peak 12.8 Å, Figs. 3a, b) indicates an R1 ordered illite/ smectite [12]. In determining the illite/smectite composition



Abbreviations: m.l. = mixed layered sheet silicates; i /m = illite / muscovite; p = pyrophyllite; chl = chlorite; qtz = quartz; d = dolomite; pyr = pyrite.

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a) Millepore mount (air dried at 20°C). b) Glycolated millepore mount.

of the interstratified sheet silicate, the position of the reflection at 16 -17° 20  $[(005)_{27}/(003)_{17}]$  has been measured on normal and glycolated samples (fraction < 2mm) (MOORE & REYNOLDS 1989). According to this procedure the illite/ smectite contains 80 -90% illite layers within the structure, indicating a rectorite (allevardite) ordered I/S.

Other minerals which can be identified from the X-ray diffraction patterns, are *quartz*, *dolomite*, *pyrite* and *feldspar*.

A semi-quantification of the shale shows that the mineral content with a few exceptions varies somewhat from sample to sample. The main sheet silicates are illite (49-18%), rectorite (10-26%), chlorite (2-22%) and minor amounts of

pyrophyllite (4-8%). The non-clay minerals are represented by quartz (6-17%), dolomite (1-24%) and small quantities of k-feldspar (3-5%), plagioclase (3-6%), and pyrite (3-4%) (Table 1.).

## 5. ILLITE CRYSTALLINITY

The crystallinity of the illite is defined by the width of the first order illite basal reflection at half-height above the background (KÜBLER 1964). A number of mica-type layer silicates that may accompany illite in diagenetic to very low grade metamorphic rocks have basal reflections close to 10 Å and may interfere with the illite peak (KISCH 1983; FREY 1987). According to KÜBLER (1967) an evolution towards

## TABLE 1

Semi quantification of slate (bulk) based on X-ray diffraction (millepore mount)

Minerales (%)	Sample No.				
	960252	960253	960254	960255	960256
Illite	28	49	19	18	27
Rectorite	25	10	20	26	22
Chlorite	17	2	6	17	22
Pyrophyllite	5	8	4	4	6
Quartz	13	15	17	10	6
K-feldspar	3	5	3	4	4
Plagioclase	4	6	4	4	3
Dolomite	2	1	24	14	6
Pyrite	3	4	3	3	4

illite/smectite during incipient metamorphism is considered to be common, especially in the presence of carbonaceous matter. In such cases the width of the 10 Å structure may in fact increase with increasing temperature, contrary to the expected behaviour. That is what actually happens in these rocks too. The 10 Å illite structure "suffers" from the broad shoulder of a superimposed rectorite (allevardite) peak at about 12 Å (see above). The measurements are therefore carried out on air-dried, EG-solvated slides, according to the recommendation given by KISCH (1980). In order to straighten up poorly crystalline illites the slides have been Ca-saturated.

In the Fómeque rock samples the measured widths range from 0.55 to 0.48° $\Delta$ 2 $\theta$ , with a mean of 0.52° $\Delta$ 2 $\theta$  (measured at a scan rate of 2°/min. and TC= 2), Fig. 4.

KISCH (1990) has tabulated the experimental conditions used by various authors for the determination of the illite "crystallinity" indices and the adopted values for the boundaries of the anchizone. Using a scan rate of  $2^{\circ}$ /min and a time constant of 2s, the limiting values for anchizone condition are 0.42 and 0.25° $\Delta$ 20 (KÜBLER 1984, p.578). Higher values indicate diagenetic conditions, while lower values indicate epizone conditions. Using the same analytical technique and size fractions as specified in KIRCH (1990) for the anchizone boundaries, the rocks from Fómeque Formation should belong to the high diagenetic zone.

# 6. DISCUSSION: CORRELATION OF METAMORPHIC GRADE INDICATORS

The dominant silicate minerals in the examined rock samples are illite, chlorite, pyrophyllite and quartz. In addition, minor amounts of K-feldspar and plagioclase is usual.

Both rectorite and especially pyrophyllite have a fairly well established stability field with respect to temperature. HOFFMAN & HOWER (1979) suggested a sequence of mineral alteration during shale diagenesis from smectite -> I/S random -> I/S allevardite (rectorite, R=1) -> kalkberg (R 3) with increasing temperature. The primary controls on the smectite-to-illite reaction in shales are temperature, time and rock and fluid chemistry. Pressure seems not to be of great importance (Hower 1981). According to Eslinger & PEVEAR (1988) the kinetic factors are particularly important in the lower-temperature, higher-expendability portions of the diagenetic sequences. At temperatures close to 100°C, the I/S becomes ordered, this transition may correspond to the lower stability range of rectorite (50-50 proportioned I/ S). VELDE (1985) found that the transformation from R=0 to R=1 I/S took place at about 80°C in high geothermal gradient sequences and at 50°-80°C in deep drill holes. Temperature data for the smectite-illite transformation as

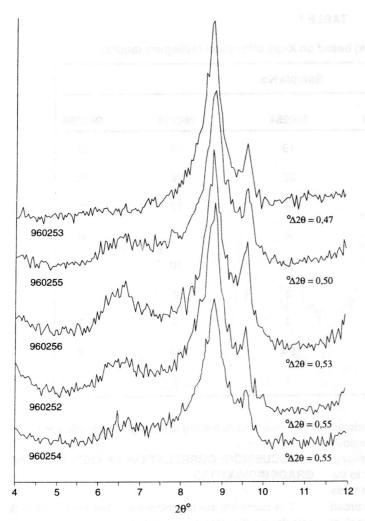


Fig. 4. X- ray diffraction patterns of fraction  $2 \mu$  m. Air dried glycolated and Ca- saturated samples. 9602XX = sample number. The crystallinity of illite is given in terms of  $^{\circ}\Delta 2\theta$ and measured as the peak width of the first order basal reflection at half height above background. For sample preparation and X- ray diffraction setting, see text.

obtained from laboratory experiments are much higher (for instance: EBERL & HOWER 1977; CHATTERJEE 1973). These differences are probably due to kinetic effects associated with the short duration of the experiments.

The second temperature of interest concerns the transition from R=1 to R=3 I/S with 85-97% illite. Data from STEINER (1968), Mc DOWELL & ELDERS (1980), JENNINGS & THOMPSON (1986), HOFFMAN & HOWER (1979) and ESLINGER & PEVEAR (1988) indicate that this change takes place at about180°-200°C.

In the Fómeque rocks the rectorite seems not to have been transformed from a R=1 to R= 3 I/S. Evaluation of the diffraction

patterns shows an ordered I/S with typically 80-90% illite layers. Using the stability of the R1 ordered rectorite as a temperature indicator for the Fómeque rocks, would suggest temperatures in the range of c.100°-200°C.

The stability field for pyrophyllite is discussed in WINKLER (1979) and in FREY (1978, 1987). In shales of pelitic composition pyrophyllite is an index mineral for anchizonal and low grade epizonal conditions. The lower limit is defined by the formation of pyrophyllite. The most tenable starting material for the appearance of pyrophyllite is probably kaolinite and quartz according to the equation (FREY 1987);

$$1 \text{ kaol} + 2 \text{ gz} = 1 \text{ pyr} + 1 \text{ H2O}$$

The upper limit is defined by the formation of Alsilicates according to the equation;

$$1 pyr = 1 als + 3 qz + 1 H2O.$$

Under conditions where  $a_{H2O} = 1$  the first appearance of pyrophyllite takes place at about 290°C at 1kbar (FREY 1987, Fig 2.11). However, if organic materials are present, as often is the case in black shales and marls, these may generate CH<sub>4</sub> and/or CO<sub>2</sub>, causing a drastic decrease in the activity of water during the metamorphism. The limit reactions above will shift to lower temperatures.

The Fómeque rocks turned out to contain large quantities of methane gas (E. BROCH, personal communication, 1996) which caused severe problems during the construction of the tunnel. It is not known how great the influence of the  $CH_4$ -gas is upon the water pressure in this case. But it is reasonable to believe that  $p_{H2O}$  is considerably lower than  $p_{fluid}$ . In FREY (1987, Fig. 2.11) calibrated reactions for the formation of pyrophyllite at different  $a_{H2O}$  is given. If  $a_{H2O}$  equals about 0.8 the first appearance of pyrophyllite takes place at a temperature of about 260°C, at 1 kbar. With an  $a_{H2O}$  in the order of 0.1-0.2, the pyrophyllite is stabilized at the expense of kaolinite + quartz at a temperature of about 200°C (FREY 1991, Fig. 13).

Based on the discussion above and the assumption that the water activity was considerably lower than unity at the time when the mineralogical reactions took place, it is reasonable to suggest that formation of pyrophyllite took place at a temperature somewhere between a minimum of c. 200°C and a maximum of c. 260°C at low pressure<sup>1</sup>.

The appearance of various stages of cleavage in phyllosilicate-rich rocks associated with illite crystallinity and the grade of very low grade metamorphism have been compiled by KISCH (1991). Stating that the grade associated with the appearance of a well-developed, continuous slate cleavage is not uniform, he nevertheless pointed out a distinct relationship between the grade and the appearance of the various stages of cleavage development in different rocks.

In the phyllosilicate-rich Fómeque rocks a closely-spaced cleavage is more or less well developed. This grades into a fracture cleavage as the grain size become coarser. In the interbedded silt- and limestone no cleavage is observed. In the most fine grained layers the phyllosilicates are mainly parallel within the microlithons, while other minerals are not. The cleavage never becomes penetrative. Using the classification criteria of KISCH (1991) for the different stages of cleavage development, the microfabric of the Fómeque rocks most likely belongs to type B, or a transition between B and C, indicating high diagenetic to low anchizone conditions.

In conclusion the following characteristics can be summarized.

1) Mineralogy and illite "crystallinity" values have been determined on rock samples from the Fómeque formation located in the Andes Cordillera some 20 km east of the city of Bogotá in Colombia.

2) The mineral assemblage consists of illite (muscovite), chlorite, pyrophyllite and a mixed layered illite/smectite of rectorite type (R=1 with 80-90% illite layers). Other distinguishable minerals are quartz, dolomite, feldspar and pyrite.

3) The "crystallinity" values of illite have been measured (on glycolated samples) to range from 0.55 to 0.48° $\Delta$ 20, with a mean of 0.52° $\Delta$ 20, indicating high diagenetic conditions (in a sense of KÜBLER).

 The microfabric as represented by the stage of cleavage development is representative for the high diagenetic to low anchizone.

5) Based on the stability of R1 ordered rectorite, the illite crystallinity and the microfabric development, it is proposed that the rocks have been subjected to high diagenetic conditions i.e. a temperature of about 200°C at low pressure. At this temperature pyrophyllite can only be stabilized at the expense of kaolinite and quartz if  $a_{H20}$  is in the order of 0.1-0.2.

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<sup>&</sup>lt;sup>3</sup>The pressure must be low as the siltstone and limestone completely lack any kind of alignment.

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