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## ILLITE AS INDICATOR OF POSTSEDIMENTARY ALTERATION OF THE PERMIAN SEDIMENTS FROM THE NORTHERN GEMERICUM

(11 Figs., 2 Tabs.)



**Abstract:** Illites of pelitic sediments from the Northern Gemericum were studied by XRD, IR spectroscopy and chemical analysis. The  $< 2 \mu\text{m}$  and  $< 0.2 \mu\text{m}$  fraction was separated for the analyses. Presence of expandable layers from 4 to 6 % was established in illite. Individual characteristics of illites from the Lower and Upper Permian were compared. The most pronounced differences were manifested in  $d(060, \bar{3}31)$  value and in IR spectra. Differences in the K u b l e r and W e b e r indices were not so distinct. The Upper Permian illites are characterized by weaker postsedimentary alteration than the Lower Permian illites.

**Резюме:** Иллиты пелитовых осадков северного гемерикума были исследованы рентгенографическим методом, инфракрасной спектроскопией и химическим анализом. Для анализов была отсепарирована фракция  $< 2 \mu\text{m}$  и  $< 0,2 \mu\text{m}$ . В иллите были обнаружены разбухающие межслои с 4 по 6 %. Были сравнены отдельные характеристики иллитов нижней и верхней перми. Самые яркие отличия были в значении  $d(060, \bar{3}31)$  и инфракрасных спектрах. Отличия в индексах К у б л е р а и В е б е р а не были такими яркими. Иллиты верхней перми характерны признаками более слабого послеосадочного изменения чем иллиты нижней перми.

**Key words:** illite, illite crystallinity, Permian, burial metamorphism.

### Introduction

Illite is one of the most frequent minerals of clay sedimentary rocks. It is true also about fine clastic sediments from the northern part of the Western Carpathian Gemericum. Though this dioctahedral mica mineral is called very simply illite, its detailed characterization is not so simple. But more detailed knowledge of illite enables, on the other hand, to become closely acquainted with postsedimentary history of the sediments. Illite of fine sediments is from two sources. Detrital illite comes from the weathered rocks. The second type – authigenic illite comes from transformations of some clay minerals. Authigenic illite is most often formed in a greatest amount by transformation of smectite (Perry – Hower, 1970). Especially this authigenic illite is important for the study of evolutionary series smectite  $\rightarrow$  mixed-layered illite/smectite  $\rightarrow$  illite  $\rightarrow$  muscovite, because it reflects change of thermodynamic conditions during the postsedimentary alteration (Dunoyer de Segonzac, 1970; Hower et al., 1976; Eslinger et al., 1979; Šrodoň – Eberl, 1984; Weaver – Broekstra, 1984; Hunziker et al., 1986).

### Geological setting

The samples under study are from the Kropachy Group in the northern part of the Western Carpathian Gemericum (Bajaník et al., 1981). The Kropachy Group – an

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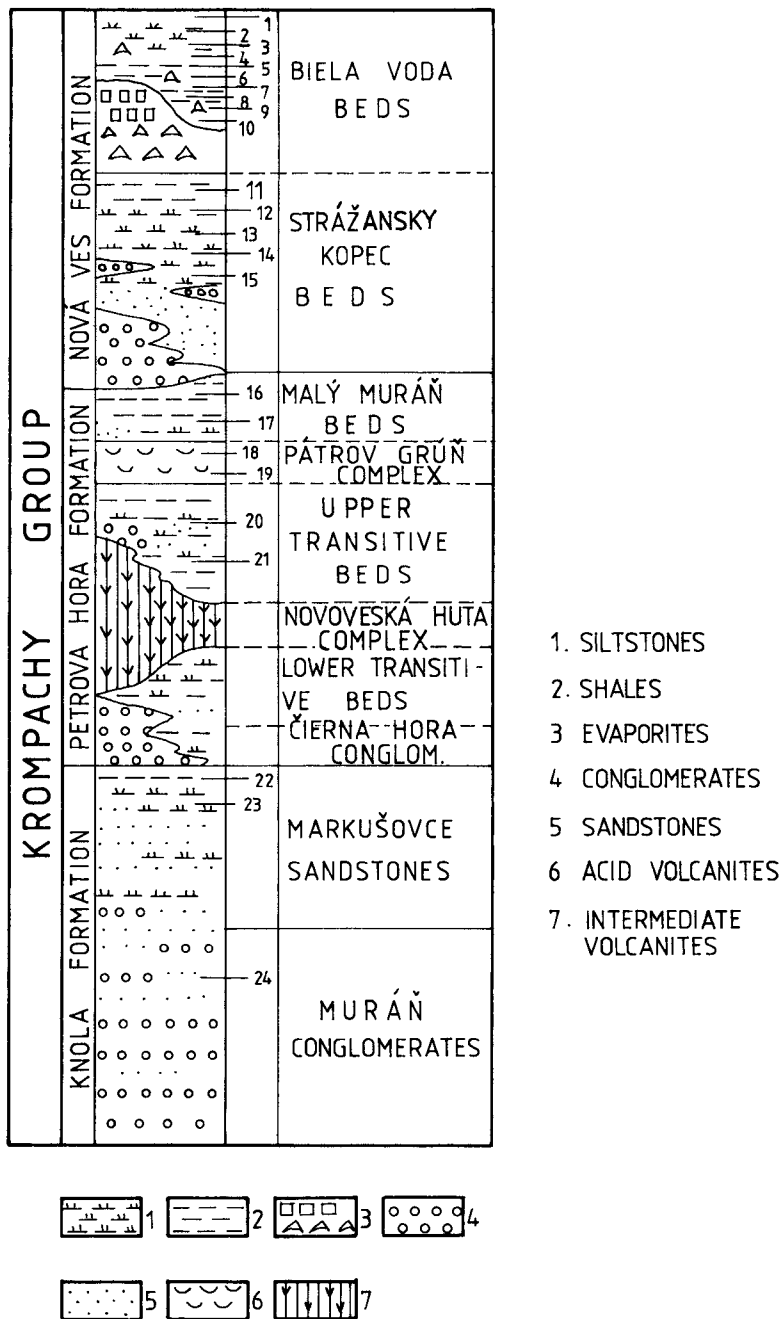


Fig. 1. Lithostratigraphic column of the Kropachy Group in the northern part of Gemericum with denotation of the studied samples. Taken over and adapted from Novotný–Miháľ (1987).

intermediate unit between the subjacent Dobšiná and Rakovec Groups and the Lower Triassic – is divided into three formations: Knola, Petrova Hora – the Lower Permian and Nová Ves – the Upper Permian (Novotný – Mihál, 1987). According to the above authors who added some lithostratigraphic units to the Krompachy Group, lithological content of individual Permian formations is as follows: the Knola Formation is built mainly of conglomerates and breccias, sandstones and aleurolites are less frequent. The Petrova Hora Formation is typical by presence of acid and intermediate volcanic material, predominantly pyroclastic rocks, less volcanites. The Nová Ves Formation contains mainly in the lower part a lot of conglomerates, but it is typical especially by pelites and aleurolites. The upper part of the Nová Ves Formation – Biela Voda beds are characteristic by occurrence of evaporites.

For our study especially fine, pelitic sediments were chosen. Aleurolites were chosen only in some cases. This fact limited our possibilities in the Lower Permian. The samples were taken from the boreholes of Geological Survey, Spišská Nová Ves and Uranium Survey, Liberec, branch Spišská Nová Ves. Majority of the samples were from the boreholes situated south of Matejovce.

### *Methods*

#### *Separation of samples*

The study is based on analyses of the clay fraction less than 2  $\mu\text{m}$  which was separated by sedimentation in distilled water from grinded rocks disintegrated by ultrasonic. Content of smectite or of expandable layers was determined from the  $< 0.2 \mu\text{m}$  fraction which was obtained by centrifugation. Before separation of the fraction, carbonates were removed from the rock by acetate buffer, organic matter by  $\text{H}_2\text{O}_2$  and free Fe-oxides by citrate-dithionite method (Jackson, 1975). Chloride anions were removed by dialysis after changing the samples to Na form by 1 M NaCl. Oriented specimens were prepared on glass slides using 10 mg of the sample per  $1 \text{ cm}^2$  (Eberl et al., 1987). Specimens were saturated in ethylene glycol vapour at the temperature of 60 °C. Separation of fine fractions represses detrital illite effect (and amount) which is probably present in the samples.

#### *XRD analysis*

XRD analyses were performed with a Philips PW 1050/25 diffractometer using  $\text{CuK}_\alpha$  radiation. The width at half height of illite reflection was measured by goniometer rate of  $1^\circ 2\Theta/\text{min}$ , paper shift rate of 1200 mm/h and quartz as an external standard. Goniometer and paper shift rates were identical in measurement of  $d(060, \bar{3}31)$  values. Quartz was used as an internal standard.

#### *IR spectroscopic analysis*

Analyses were performed with a Perkin Elmer 597 spectrophotometer. Clay fraction was pressed to KBr disks. 0.7 and 1.5 mg of the sample was used for 300 mg of KBr.

### *Results*

#### *Illite crystallinity*

This term covers different ways of geometry changes study of (001) illite reflection (Weaver, 1961; Kubler, 1967, 1968; Weber, 1972). Substance of reflection geometry changes is in “ordering” of illite structure, namely amount of coherently diffracting domains (Árkai – Tóth, 1983). These changes are influenced by other factors too

(K i s c h , 1983). Certain rules were preserved for their elimination. The same fraction ( $< 2 \mu\text{m}$ ), homoion Na form of the samples and external standards were used. The width at half height of (001) reflection in  $2\Theta$  degrees (K u b l e r index) was observed separately and then also in comparison with the width at half height of (100) reflection of quartz (W e b e r index).

$$Hb_{rel} = \frac{Hb \text{ illite (001) (mm)}}{Hb \text{ quartz (100) (mm)}} \times 100$$

Results of the width at half height and  $Hb_{rel}$  measurements were plotted in histograms for the whole Permian of the Northern Gemicum (Figs. 2, 3) and for the Upper and Lower Permian each (Fig. 4).

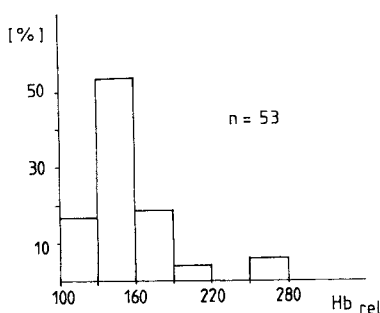


Fig. 2. Histogram of  $Hb_{rel}$  Weber index values without division of the samples into the Upper and Lower Permian.

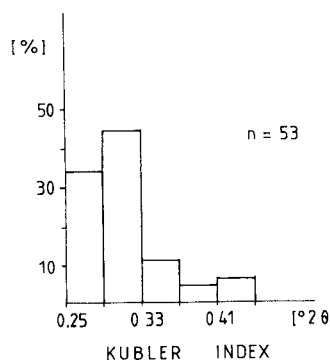


Fig. 3. Histogram of Kubler index values without division of the samples into the Upper and Lower Permian.

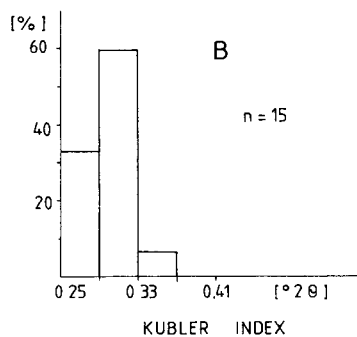
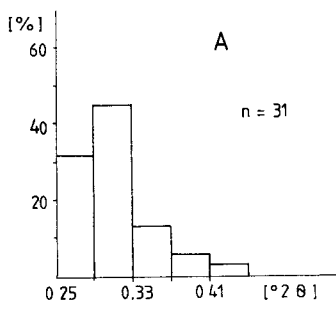


Fig. 4. Histograms of Kubler index values. A – samples from the Upper Permian, B – samples from the Lower Permian.

*b*-parameter

$d(060, \bar{3}31)$  values in illite depend on the presence of several elements in the structure (Radoslovich – Norrish, 1962). The greatest changes of  $d(060, \bar{3}31)$  value are, however, caused by the changes in Mg and Fe contents in the illite structure. Mg and Fe

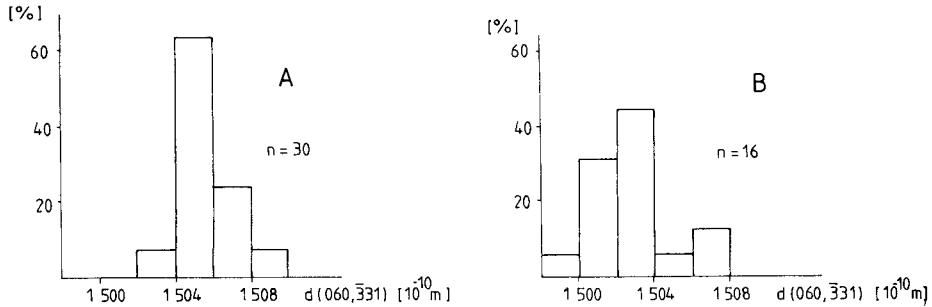


Fig. 5. Histograms of  $d(060, \bar{3}31)$  values.

A – samples from the Upper Permian, B – samples from the Lower Permian.

Table 1

Some results of XRD and IR spectroscopy analyses

Sample	I	CH	K	Q	Ir	BB1	$d(060, \bar{3}31)$	Wave number [ $\text{cm}^{-1}$ ]		E 750 E 830
1	●	○	○	○	N	N	1.505	525	1075	1.41
2	●	—	—	○	1.28	3.4	1.5058	520	1078	N
3	●	○	—	○	N	N	1.5051	526	1071	1.61
4	●	○	—	○	1.15	3.2	1.5041	525	1078	1.28
5	●	○	—	○	N	N	1.5045	528	1075	1.18
6	●	○	—	○	1.09	2.9	1.5041	526	1078	N
7	●	○	—	○	1.53	2.8	1.5076	N	N	N
8	●	○	—	○	1.46	2.6	1.5088	518	1075	0.44
9	●	○	—	○	N	N	1.5076	520	1075	0.89
10	●	○	—	●	N	N	1.5076	516	1075	0.64
11	●	·	—	○	1.33	2.8	1.5055	520	1071	0.73
12	●	—	—	○	1.74	2.6	1.5043	529	1070	0.97
13	●	—	—	·	1.66	2.9	1.5033	528	1075	0.75
14	●	—	—	○	N	N	1.505	526	1068	0.80
15	●	—	—	○	N	N	1.5045	530	1068	0.85
16	●	—	—	○	1.44	2.5	1.5063	520	1073	0.50
17	●	—	—	○	1.33	2.6	1.5086	520	1076	0.78
18	●	○	—	○	1.19	2.4	N	N	N	N
19	●	—	○	○	N	N	1.5030	530	1080	1.34
20	●	○	—	○	1.22	2.5	N	N	N	N
21	●	○	—	○	1.12	2.1	N	N	N	N
22	●	—	—	○	N	N	1.5021	532	1062	2.12
23	●	—	—	○	N	N	1.5015	535	1068	1.98
24	●	—	—	○	N	N	1.4995	535	1060	2.39

Note: ● = major phase, ○ = minor phase, · = trace phase, — = absent phase, N = not analyzed, I = illite, CH = chlorite, K = kaolinite, Q = quartz.

contents increase in octahedral sheet is manifested by  $d(060, \bar{3}31)$  value increase. At the same time, higher  $d(060, \bar{3}31)$  values are characteristic of illites less affected by the postsedimentary alteration (Maxwell – Hower, 1967; Frey et al., 1983; Hunziker et al., 1986; Lindqvist – Widmark, 1986). Our results were used for construction of histograms and, in such way, difference between the illites from the Lower and Upper Permian became very apparent (Fig. 5). Higher values prevail in the Upper Permian samples. Majority of them are within an interval ranging from  $1.504$  to  $1.506 \cdot 10^{-10}$  m (some of them even to  $1.510 \cdot 10^{-10}$  m).

### *IR spectra of illite*

20 samples of the  $< 2 \mu\text{m}$  fraction were analyzed (Tab. 1). All spectra are typical of dioctahedral micas similar to muscovite in their composition (Farmer, 1974; Veldé, 1978). IR spectra reflect many changes taking place in the structure due to the changes in chemical composition. Flehmig–Gehlken (1988) dealt in detail with reflection of octahedral Fe and Mg contents changes in IR spectra. They used relation between the shift Si-O bond vibration wave number at  $510\text{--}540 \text{ cm}^{-1}$  and the ratio of Al-O-Si vibration at  $750 \text{ cm}^{-1}$  and Al-Mg-OH vibration at  $830 \text{ cm}^{-1}$  absorbancies. This relation was used in our samples and a clear dependence was obtained (Fig. 6). According to Flehmig–Gehlken (1988), this dependence enables to estimate content of octahedral  $\text{Fe}_t + \text{Mg}$ . But the values obtained from the chemical analyses added to the above-mentioned plot do not prove this statement.

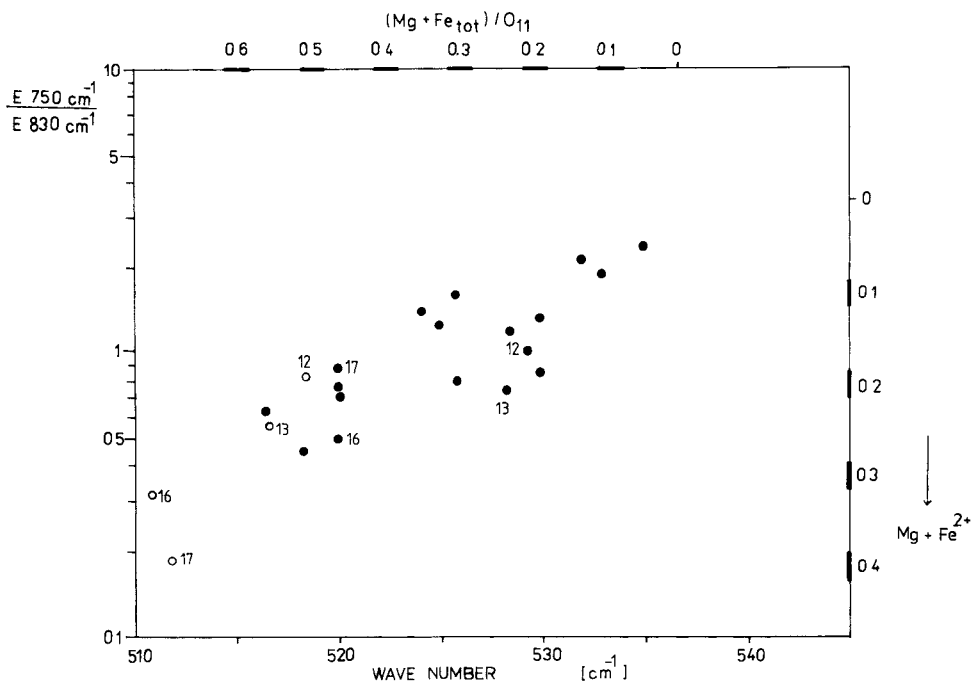


Fig. 6. Nomogram for estimation of octahedral  $\text{Fe}_t + \text{Mg}$  content on the basis of IR spectra according to Flehmig–Gehlken (1988) supplied with data on chemical analyses (o).

Relation between shift of Si-O bond vibration and Fe and Mg contents was observed by several authors, e. g. Stubičan-Roy (1961), Hunziker et al. (1986), Eberlet et al. (1987). Since we had not a crystallochemical formula of all illites at our disposal, we could not study the similar dependence in our samples. But we tried to compare dependence of wave number range from 510 to 540  $\text{cm}^{-1}$  with change of  $d(0,60, 331)$  value which depends on octahedral Fe, + Mg content too (Fig. 7). In this way we obtained a very interesting linear indirect-proportional dependence with high correlation coefficient ( $r = -0.92$ ). In our opinion, the relation between octahedral Fe and Mg content and XRD and IR spectroscopy manifestations may be proved also in this indirect way. At the same time, it refers to a good

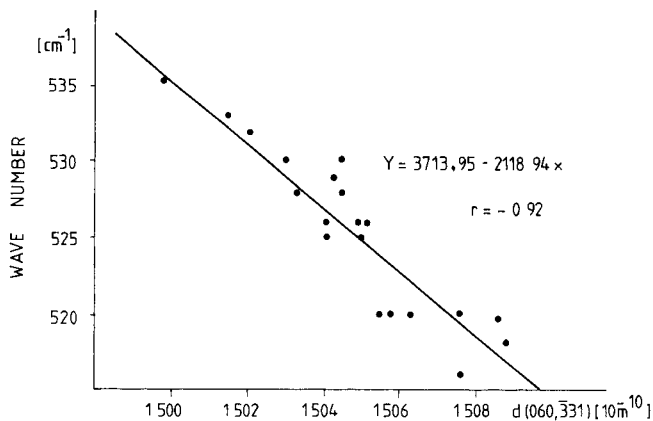


Fig. 7. Relation between Si-O vibration wave number and  $d(060, 331)$  value.

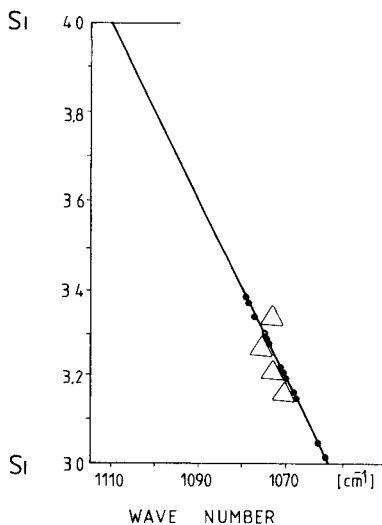


Fig. 8. Estimation of tetrahedral Si content (o) using IR spectra controlled by results of chemical analysis ( $\Delta$ )

After Veldé (1978).

agreement of the results of the both methods. Coming out from the experimental Veldé's works (1978), we tried to estimate content of tetrahedral Si on the basis of Si-O bond vibration shift within an interval ranging from 1060 to 1110  $\text{cm}^{-1}$ . Our values vary from 1060 to 1078  $\text{cm}^{-1}$  what should correspond approximately to tetrahedral Si content: 3.0–3.4. Si value varies most often around 3.2 (Fig. 8). Values obtained from the crystallochemical formulae recalculated from the chemical analyses were plotted to the graph. These values correspond well with the Veldé's line too.

#### *Expandability measurement*

Expandability, i. e. content of expandable layers in the illite structure, is a quite important parameter giving an information about the stage of postsedimentary development in the smectite  $\rightarrow$  mixed-layered illite/smectite  $\rightarrow$  illite series. Some authors state that the expandable layers disappear as soon as in the end of diagenesis (Frey, 1986; Kisch, 1983), the others set disappearance of the expandable layers as far as beyond the border of anchimetamorphism (Weaver–Broekstra, 1984). Presence of the expandable layers was verified on the basis of identification procedures of Šrodón (1984) and Eberl et al. (1987). Ir index (Šrodón, 1984, p. 339) representing a ratio of air-dried and EG-saturated reflections intensities was used for detection of presence of the expandable layers in the structure:

$$I_r = \frac{(001) / (003)_{\text{air-dried}}}{(001) / (003)_{\text{EG}}}$$

If  $I_r > 1$ , the expandable layers are present, if  $I_r \leq 1$ , the expandable layers are not present.  $I_r > 1$  in all analyzed samples from the Northern Gemic Permian (Tab. 1). It means that the expandable layers are present here. BB1 index (Šrodón, 1984, p. 339) lower than  $4^{\circ}2\Theta$  in all samples was used too. It means that number of the expandable layers is smaller than 15% and interstratification type  $R = 3$  (Reynolds, 1980). More precise determination of content of the expandable layers within an interval (0; 15%) is complicated. Two possibilities were used. The first, estimation of the content according to (002) and (003) reflections using computer-modelled X-ray records (Eberl et al., 1987) (see Fig. 9). In such way, the content was determined at about 4–6%. We are aware of the problems caused by presence of detrital illite. The second possibility is in application of an equation of  $I_r$  and the expandable layers content dependence gained by Eberl et al. (1987, p. 923). Substituting our  $I_r$  values to the equation  $y = -18.53 + 25.96x + 7.88x^2 + 0.78x^3$ , an interval ranging approximately from 2 to 6% of the expandable layers was obtained.



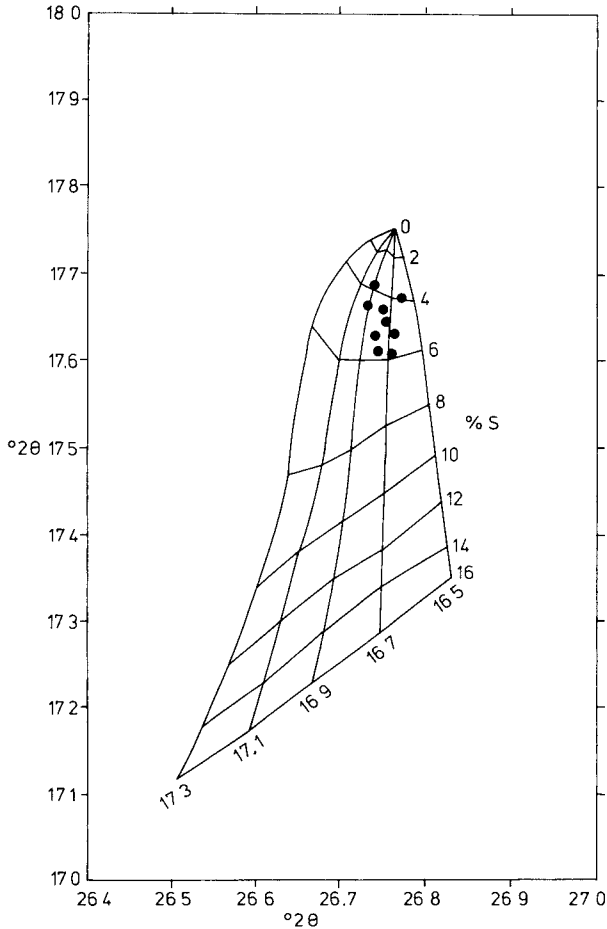


Fig. 9. Determination of contents of the expandable layers in illite according to (002) and (003) reflections of illite.

After Eberl et al. (1987).

### *Chemical analysis of illite*

Chemical analyses of 4 illite samples were carried out from the  $< 2 \mu\text{m}$  fraction. Results of analyses were recalculated to a crystallochemical formula (Tab. 2) by Kelley's method (in Číček, 1981). Recalculating the original results, a sum of elements in octahedral sheet was obtained, in three cases outside the interval  $3.96 \leq f_{\text{oct}} \leq 4.05$  what was caused, in all probability, by quartz admixture (Číček, 1979). Quartz presence was proved also by XRD analysis. A certain amount was subtracted from  $\text{SiO}_2$  content in a such way that a sum of octahedral atoms would be within a necessary interval after recalculation of a crystallochemical formula. Chemical analyses show that composition of the selected illites is close to that of

Table 2

Chemical composition of clay fraction of 4 selected samples and crystallochemical formulas of illites from these samples

Sample	12		13		16		18	
SiO <sub>2</sub>	49.80		46.78		51.75		53.73	
TiO <sub>2</sub>	0.55		0.56		0.81		0.43	
Al <sub>2</sub> O <sub>3</sub>	24.87		24.77		21.68		20.66	
Fe <sub>2</sub> O <sub>3</sub>	5.47		5.65		6.21		4.47	
FeO	0.32		0.45		0.34		0.58	
MnO	0.02		0.01		0.01		0.02	
CaO	0.85		0.94		1.08		0.99	
MgO	1.41		2.19		2.61		2.99	
K <sub>2</sub> O	8.39		8.70		8.70		7.19	
Na <sub>2</sub> O	0.30		0.23		0.27		1.06	
H <sub>2</sub> O <sup>-</sup>	1.92		2.64		1.24		1.84	
H <sub>2</sub> O <sup>+</sup>	5.76		6.70		5.16		5.90	
P <sub>2</sub> O <sub>5</sub>	0.28		0.27		0.22		0.17	
Total	99.94		99.88		100.08		100.03	
	A	B	A	B	A	B	A	B
Si	6.91	6.32	6.68	6.68	7.11	6.43	7.36	6.59
Al <sup>IV</sup>	1.09	1.68	1.32	1.32	0.89	1.57	0.64	1.41
Tetrahedral charge		1.68		1.32		1.57		1.41
Al <sup>VI</sup>	2.97	2.97	2.84	2.62	2.57	2.69	2.63	
Fe <sup>3+</sup>	0.57	0.65	0.61	0.61	0.64	0.76	0.46	0.56
Fe <sup>2+</sup>	0.04	0.04	0.05	0.05	0.04	0.05	0.07	0.08
Mg	0.29	0.33	0.47	0.47	0.53	0.63	0.61	0.74
Σ X	3.87	3.99	3.97	3.97	3.83	4.01	3.83	4.01
Octahedral charge		0.40		0.61		0.65		0.79
Ca	0.13	0.14	0.14	0.14	0.16	0.19	0.15	0.18
Na	0.08	0.09	0.06	0.07	0.08	0.28	0.34	
K	1.48	1.70	1.58	1.58	1.52	1.80	1.26	1.52
Σ Y	1.69	1.93	1.78	1.78	1.75	2.07	1.69	2.04
Interlayer charge		2.07		1.92		2.26		2.22

A — without subtraction of admixture, B — after subtraction of admixtures. Analyzed by Ing. E. Walzel.

muscovite. In comparison with ideal muscovite composition, all samples have higher Si, Mg, Fe<sup>3+</sup> contents and lower Al and K contents (Fig. 10). According to Porrenga's classification (1968), our illites fall to the upper part of the illite field and closely beyond the border towards Fe illites (Fig. 11).

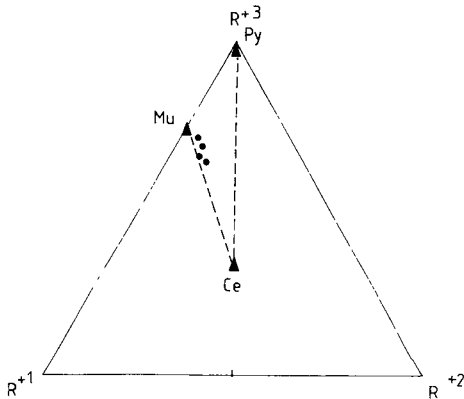


Fig. 10. Triangulate diagram for illite characterization according to chemical composition.

$$R^{+1} = K + Na, R^{+2} = Fe^{2+} + Mg,$$

$$R^{+3} = Fe^{3+} + Al.$$

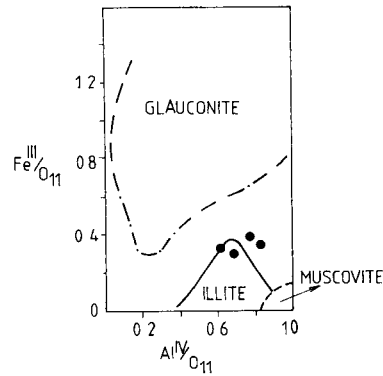


Fig. 11. Position of the Permian illites within the Porrenga's classification diagram (1968).

#### Discussion and conclusions

We tried to characterize dioctahedral mica minerals (called illites) from several points of view. Our aim was to create a certain picture of their structure, chemical composition and changes they underwent. According to lithostratigraphic position, illites were divided into the Upper Permian illites and the Lower Permian illites. Differences were shown in some of their features. The most pronounced differences were in  $d(060, 331)$  value, but also IR spectroscopy indicated lower  $Fe_t + Mg$  content in octahedral sheet in the Lower Permian samples than in the Upper Permian ones. Differences in the Kubler and Weber indices were just implied. In our opinion, it may be caused also by effect of evaporite environment in the Upper Permian which might shift the crystallinity index to lower values (Kubler, 1967). Comparing the Upper Permian and Lower Permian illites, the samples from Malý Muráň beds (Fig. 1) seem to be more similar to the Upper Permian illites. It is suggested especially by quite high  $d(060, 331)$  value and low Si-O vibration wave number value. There is no distinct difference between chemical compositions of two samples from the Upper Permian (Stražany Formation) and two samples from the Malý Muráň beds (Lower Permian). Constructing the histograms, 4 samples of the Malý Muráň illites were placed to the Lower Permian. They always belonged to the classes with higher Kubler index and  $d(060, 331)$  values. Measurement of presence and amount of the expandable layers brought interesting results too. Their presence enables us to consider illites as products of smectite transformation. Owing to the determined interval from 4 to 6%, we cannot place exactly a certain amount of the expandable layers to the Upper or to the Lower Permian. Illites were formed from the smectites probably by two ways. The first is the way of cyclic drying and wetting of smectite under the surface conditions at potassium presence (Decoinck et al., 1988). The second way is diagenetic formation by transformation of smectites due to temperature increase under the postsedimentary conditions.

On the basis of the presented results we assume that even if we admitted a possibility of illitization under the surface conditions, main process affecting the formation of illite in the Northern Gemericum region was, in all probability, the postsedimentary alteration. Its temperature may be estimated, on the basis of the Kubler index and content of the expandable layers (Kubler, 1968; Kisch, 1983; Frey, 1986; Hunziker et al., 1986; Eberl et al., 1987), at about 250° C what corresponds to the anchimetamorphic stage of alteration.

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