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MINERAL AND ISOTOPIC COMPOSITION OF MIOCENE LIMESTONES FROM WYDRZA SULPHUR DEPOSIT

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Abstract. The paper deals with the results of mineralogical and isotopic study of limestones overlying sulphur-bearing series and corresponding to it supra-gypsum zone. One has studied also sulphur-bearing limestones and Lithotamnium ones underlying the former deposits. It was found that limestones overlying the so-called *chemical series* within the areas containing sulphur deposits are enriched in light carbon isotope in comparison with those ones overlying the zones of gypsum occurrence. On the basis of the results of isotopic composition analysis, sulphur-bearing limestones have been assigned to post-gypsum ones. It was also found that carbon isotope ratio ($\delta^{13}C$) in them is different from the ratio in Miocene, Carboniferous and Devonian hydrocarbons which could have been the source of carbon for post-gypsum sulphur-bearing limestones. Lithotamnium limestones, underlying the so-called chemical series, display a negligible variation of carbon isotope ratio, indicating their primary character. On the other hand, sulphur occurring in them is unquestionably of secondary origin.

INTRODUCTION

Examination of isotopic composition of carbonate rocks and sulphur was initiated about 25 years ago. One of the first results were reported by Craig (1953). In Poland the studies of this type were carried on by e.g. Zboiński (1966). More detailed examination has been carried out by Żuk *et al.* (1970, 1973a, 1973b), Hałas (1973a, 1973b) and Nielubowicz (1975). Besides, studies of isotopic composition of hydrocarbons occurring in the region of sulphur deposits (Głogoczowski, 1975, 1976) as well as paleotemperature examinations of the conditions of formation of post-gypsum minerals, based on isotope ratios (Bowen, 1966) are of unquestionable importance for the problem under consideration.

In this work determination of mineral composition of carbonate rock samples collected from six boreholes drilled within the Wydrza sulphur deposit was ac-

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companied by estimation of carbon and oxygen isotope ratios $\delta^{13}C$ and $\delta^{18}O$. The samples were so selected as to represent infra-tuffitic limestones overlying the so-called chemical series, sulphur-bearing carbonate deposits of this series and Lithotamnium limestones underlying the chemical deposits in question.

GEOLOGY OF WYDRZA DEPOSIT

Similarly to other deposits of the Tarnobrzeg region, its bottom part consists of Lower Badenian Lithotamnium limestones showing variable content of arenaceous fraction and locally intertonguing with Baranów sands. The upper part of the so-called chemical series display variable lithology. It consists of sulphur-bearing limestones grading into gypsum deposits. These limestones show considerable variety of structural and textural features. Their average total thickness amounts to ca. 20 m (Fig. 1). Gypsum deposits, contacting with sulphur-bearing series, are more than 30 meter thick. In their bottom part there occur coarse-crystalline gypsum crystals, up to 50 cm in size. They are porous and, sometimes, in bottom part contain native sulphur intercalations and in the upper one – pyritic concentrations. The middle part of gypsum deposits consists of slightly smaller crystals, 20 – 30 cm in size. From the bottom to the top these deposits are intergrown with several layers of fine-crystalline gypsum. Total thickness of the complex in question amounts to approx. 15 m. It is overlain by fine-crystalline gypsum deposits containing phenocrysts of this mineral up to 15 cm in size. The top part consists of coarse-crystalline gypsum. Total thickness of the top part of this deposit does not exceed 5 m. Gypsum sediments are contacting with sulphur-bearing limestones showing no distinct transition zone. However, in southern part of the deposit, we observe both in the top and bottom parts of limestones their intertonguing with gypsum sediments. Usually, the thickness of these intercalations does not exceed 7 m. Sulphur-bearing limestones and gypsum sediments are overlain by mudstones and limestones containing one or two tuffitic interlayers, being a correlation key-horizon for this so-called chemical series. In this part of the deposit they occur 1 – 2 m above the top of the latter series. It should be emphasized that the form of tuffitic layers and their distance from the top of the so-called chemical series is considered to be a good index of sedimentation conditions and epigenetic alteration processes. As follows from the present author's observations, this tuffitic horizons above sulphur-bearing limestones show secondary disturbances while overlying gypsum sediments are regular in type.

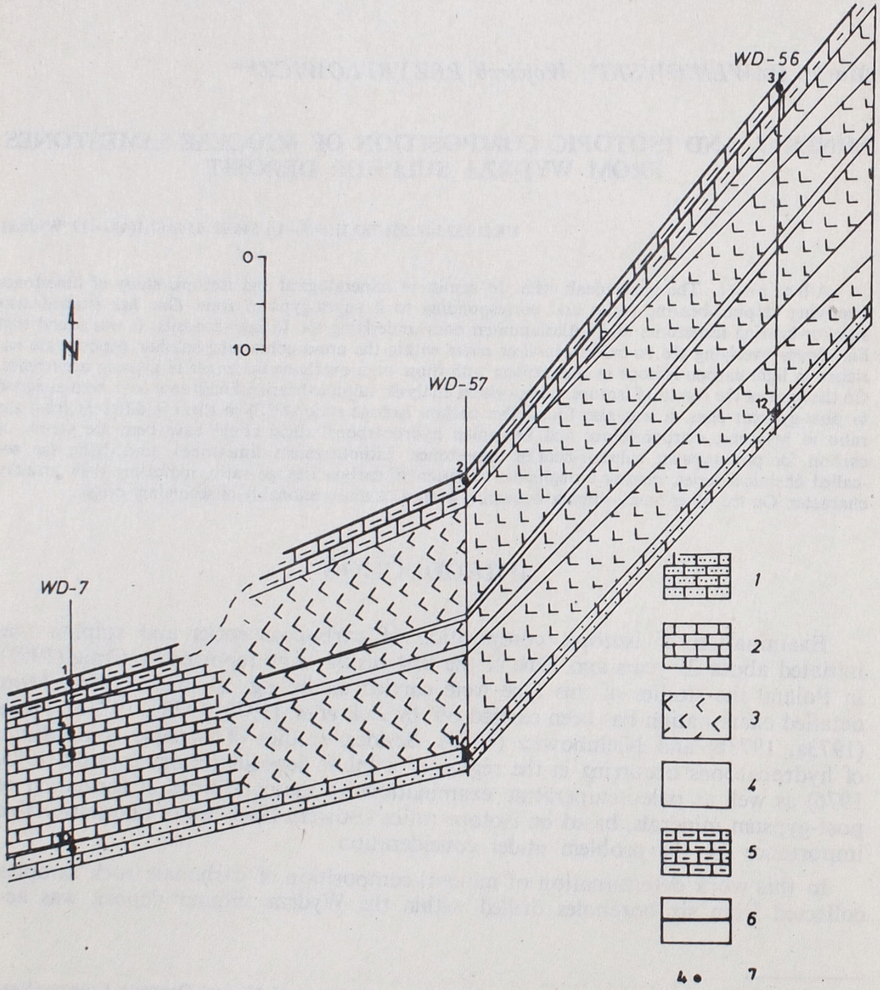

Fig. 1. Geological cross-section of Wydrza sulphur deposit

1 – Lithotamnium limestones, *2* – Sulphur-bearing limestones, *3* – Coarse-crystalline gypsum, *4* – Fine-crystalline gypsum, *5* – Limestone between tuffites, *6* – Tuffites, *7* – Sampling sites

METHODS OF EXAMINATION

Among 12 samples selected for mineralogical and isotopic study, three of them (No. *1*, *2* and *3*) represented limestones occurring between tuffitic intercalations overlying the so-called chemical series. They are coming from the boreholes WD-7 (depth 178.6 m), WD-57 (depth 193.7 m) and WD-56 (depth 181.3 m). Samples Nos. *4*, *5*, *6*, *7*, *8* and *9* represent various types of sulphur-bearing limestones and are coming from the following boreholes: WD-7 (depth 192.7 m and 195.0 m), PT-76 (depth 188.2 and 192.0 m), WD-12 (depth 215.6 m) and WD-61 (depth 205.2 m). Samples Nos. *10*, *11* and *12* were collected from the boreholes WD-7 (depth 205.2 m), WD-57 (depth 215.0 m) and WD-56 (depth 214.5 m), and represent Lithotamnium limestones underlying the so-called chemical series.

All the above samples have been examined using a microscope and X-ray powder methods. The latter study was carried out by means of TUR-61 diffractiometer by applying CoK_α radiation (filtered) under the following conditions: voltage – 34 kV, current 1 A, moving rate of the counter 1° per min. and that of registration paper 1200 mm per hour. Interpretation of X-ray diffractometer patterns was carried out using Mikhejev's tables (1957).

Determination of stable carbon and oxygen isotope ratios were carried out using "Micromass 602C" mass-spectrometer at the Mass-Spectrometry Laboratory of the Institute of Physics and Nuclear Techniques. In the spectrometer we can analyse the isotopic composition of C and O only in gas-CO_2 form.

Sample preparation procedure was identical to that described by Barański (1976). After disintegration in an agate mortar they were treated with phosphoric acid to decompose calcium carbonate:

$$3\,CaCO_3 + 2\,H_3PO_4 = 3\,CO_2 + Ca_3(PO_4)_2 + 3\,H_2O$$

Reactions has been effectuated in high vacuum conditions (10^{-3} Tr). Thus formed carbon diaxide was dehydrated and introduced into CO_2 – container.

The measurements have been carried out using automatic compensation method relative to laboratory standard – Marianna Biała marble form Stronie Śląskie. This standard has been calibrated versus international standard PDB (Craig, 1953, 1957) by Barański (1976).

All the results obtained by the authors are related to PDB as well as the cited ones. Standard deviation of single determination of $\delta^{13}C$ and $\delta^{18}O$ is ± 0,1 °/₀₀. Necessary calculations have been carried out using "Odra 1304" computer.

MINERALOGICAL AND PETROGRAPHICAL DATA

Limestones occurring between tuffitic interlayers, represented by the samples *1*, *2* and *3*, are compact and greyish in colour, showing fine-crystalline textures and poorly parallel structures. They consist of calcite, a little quartz and subordinate amounts of clay minerals. Local appearance of volcanic glass is connected with the presence of accompanying tuffite intercalations (Phot. 1). These rocks are cut by veinlets, up to 40 μm thick, filled with sulphur. Calcite grains are usually hipidiomorphous, being on the average 20–40 μm in size. X-ray examinations (Fig. 1) have shown that calcite in these limestones is accompanied by quartz, illite and probably some chlorite admixture. Besides, the samples *1* and *2* contain a little sulphur (reflexion d_{hkl} = 2.86 Å*).

Sulphur-bearing limestones are represented by the samples *4*, *5*, *6*, *7*, *8* and *9*. Their grey varieties contain nests and veins of pulverized and wax-like sulphur (samples *4* and *5*). In the latter rock sulphur also occurs in spheroidal forms. The sample *6* represents a light gray limestone showing coarse-crystalline gypsum structure and containing small amounts of pulverized and crystalline sulphur, while the sample *7* abounds in wax-like variety of this mineral occurring in form of spheroids up to 10 mm in size. Sample *8* consists of dark grey calcitic pelite laminated with pulverized sulphur, while the sample *9* represents compact porous limestone displaying medium-crystalline gypsum structure and containing wax-like and crystalline sulphur.

Microscope examinations of limestones of sulphur-bearing zone have shown them to consist mainly of microcrystalline calcite. Their grains are less than 30 μm in size and accompanied by sulphur of the same grain size. Only in the samples *6*, *7* and *9* the grains of the latter mineral are larger than those of calcite, being up to 150 μm in size. Euhedral strontianite crystals (Phot. 2) and individual flaky clay mineral aggregates were observed in sample *6* (Fig. 2). Moreover, in all the examined samples, concentrations of poorly transparent minerals occur at the contacts of calcite and sulphur. The sample *8* represents a peculiar petrographic variety. Apart from sulphur, it consists of microcrystalline calcite grains, 10–15 μm in size. The rock is not compact and thus it can be called a calcareous clay showing very disadvantageous exploitation properties. X-ray study confirmed that the rocks in question consist essentially of calcite and sulphur, sometimes accompanied by quartz and pyrite (sample *5*) or illite (sample *6*).

Lithotamnium limestones (samples *10*, *11*, *12*), underlying the so-called chemical series contain, apart from fragments of these fossils, quartz and sulphur as well as small amounts of glauconite and clay minerals (Fig. 3). Lithotamnia consist of grains of fine crystalline calcite up to 20 μm in size. It was observed that some part of Lithotamnia fossils are cracked and the fissures filled with sulphur or secondary coarser crystalline calcite. Generally, however, sulphur and other minerals are located between Lithotamnia bodies. Besides, some of latters contain euhedral piryte aggregates which is also present within inter-Lithotamnia matrix, suggesting its primary origin.

X-ray examinations have shown the presence of other components as e.g. of illite in the sample *11* and probable lepidocrocite, showing reflection corresponding to d_{hkl} = 6.4 Å.

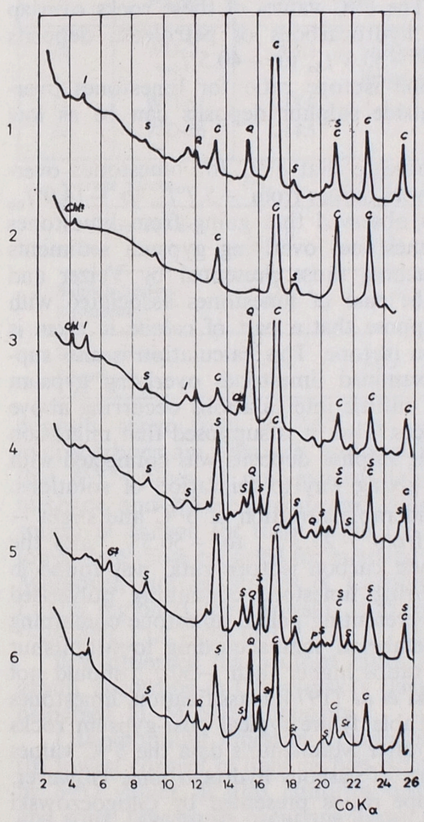

Fig. 2. X-ray diffraction patterns of samples *1–6*

C – calcite, *S* – sulphur, *Q* – quartz, *I* – illite, *Chl* – chlorite, *G* – gypsum, *Sr* – strontianite, *P* – pyrite

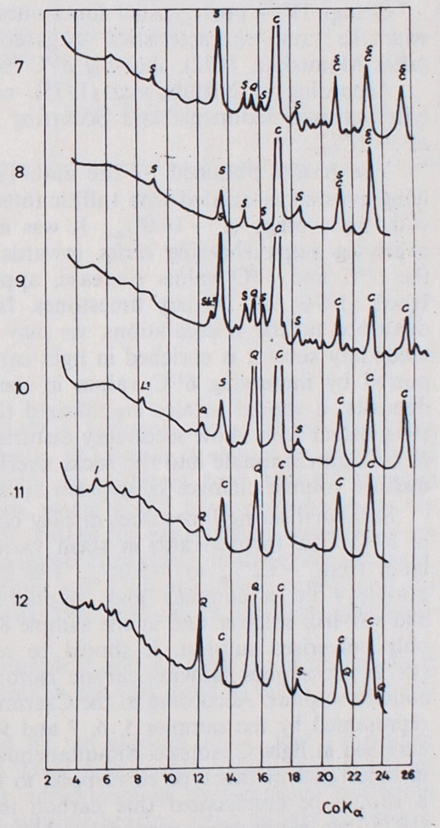

Fig. 3. X-ray diffraction patterns of samples *7–12*

C – calcite, *S* – sulphur, *Q* – quartz, *I* – illite, *L?* – lepidocrocite

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[*] 1 Å = 0.1 nm (SI).

RESULTS OF ISOTOPIC COMPOSITION STUDY

As follows from more than 1000 data of isotope analysis for Tertiary carbonate rocks (Veizer, Hoefs, 1976) the majority of $\delta^{13}C$ values in them ranges from $-8^{\circ}/_{oo}$ to $+3^{\circ}/_{oo}$, while the values of $\delta^{18}O$ are grouped within limits from $-7^{\circ}/_{oo}$ to $+1^{\circ}/_{oo}$. When considering these two isotopic compositions in Polish sulphur deposits, we have to accept the classification of carbonates proposed by Czermiński and Osmólski (1974) who distinguished four groups of these rocks differing in the $\delta^{13}C$ isotope ratio:

Group I – limestones showing the highest content of light carbon isotope connected with gaseous hydrocarbons (after Grinienko, 1972), namely $\delta^{13}C = -52.5^{\circ}/_{oo}$ to $-59.9^{\circ}/_{oo}$.

Group II – limestones connected with isotopes having originated from heavy hydrocarbons (after Hałas, 1973a) and showing $\delta^{13}C$ from $-18.6^{\circ}/_{oo}$ to $-27.4^{\circ}/_{oo}$.

Group III – limestones of most probably marine origin (distinguished after Grinienko, 1972) displaying $\delta^{13}C$ from $-8.1^{\circ}/_{oo}$ to $-11.6^{\circ}/_{oo}$.

Group IV – post-gypsum limestones. The $\delta^{13}C$ values of these rocks overlap with the same characteristics of gaseous hydrocarbons of petroleum deposits (after Mamtshur, 1972), showing $\delta^{13}C$ from $-35.8^{\circ}/_{oo}$ to $-40.5^{\circ}/_{oo}$.

According to Nielubowicz (1975), carbon isotope ratio for limestones overlying gypsum sediments and occurring outside sulphur deposits can be as low as $-27^{\circ}/_{oo}$.

The results obtained by the authors indicate that $\delta^{13}C$ for limestones overlying gypsum associated with tuffitic interlayers ranges from $-5.7^{\circ}/_{oo}$ to $-14.9^{\circ}/_{oo}$ with the average of $-10.0^{\circ}/_{oo}$. It was also observed that going from limestones overlying sulphurbearing series towards limestones overlying gypsum sediments the $\delta^{13}C$ and $\delta^{18}O$ values increase, approaching those presented by Veizer and Hoefs (1976) for Tertiary limestones. In the case of limestones associated with disturbed tuffitic intercalations, we may suppose that a part of calcite in them is secondary since it is enriched in light carbon isotope. This calculation is also supported by increasing $\delta^{13}C$ values in the examined limestones overlying gypsum deposits. It should be also emphasized that tuffitic intercalations occurring above the gypsum zone show secondary disturbances. Thus it is supposed that migration of calcium carbonate into the rocks overlying sulphur deposits was connected with distinct volume changes of gypsum rocks, giving way to migration of solutions.

Sulphur-bearing limestones display considerable variation of $\delta^{13}C$ and slight – of $\delta^{18}O$. The former ratio in them varies from $-25.3^{\circ}/_{oo}$ to $-50.5^{\circ}/_{oo}$ and the latter from $-3.2^{\circ}/_{oo}$ to $-5.6^{\circ}/_{oo}$. The highest carbon isotope ratio was found in sample *4* i.e. a compact grey sulphur-bearing limestone containing pulverised and vax-like sulphur and in the sample 8 representing pelitic limestone containing only pulverised sulphur. It should be remembered that according to Mamtshur (1972), limestones showing carbon isotope ratios higher than $-30^{\circ}/_{oo}$ should not contain sulphur. According to the Czermiński *et al.* (1974) classification, limestones represented by the samples *5*, *6*, *7* and *9* (Table 1) are typical post-gypsum rocks enriched in light C isotope. Simultaneously, after Mamtshur's data the $\delta^{13}C$ values in limestones of this type correspond to those of gaseous hydrocarbons. However, it should be emphasized that carbon isotope ratios presented by Głogoczowski (1976) for methane in gases from Miocene deposits of the Carpathian foredeep range from $-61.57^{\circ}/_{oo}$ to $-71.01^{\circ}/_{oo}$ and their migration direction was found to be from SE to NW. Consequently the isotopic composition of the Miocene methane is different from that characteristic of typical post-gypsum limestones. Besides, the $\delta^{13}C$ values obtained for dispersed bitumina occurring in Miocene deposits range from $-27.6^{\circ}/_{oo}$ to $-23.4^{\circ}/_{oo}$ (Głogoczowski, 1976). So, these hydrocarbons are relatively enriched in heavy C isotope. It should be emphasized that $\delta^{13}C$ values of post-gypsum limestones are different from that of Miocene methane and Miocene dispersed bitumina which could have probably been the source of carbon for them. Thus the $\delta^{13}C$ values for post-gypsum limestones are lower than those reported for methane and higher than those for dispersed bitumina. Moreover, isotope studies of Carboniferous and Devonian hydrocarbons of the Lublin region (Głogoczowski, 1975) indicate that $\delta^{13}C$ values for them vary from $-26.20^{\circ}/_{oo}$ to $-33.34^{\circ}/_{oo}$.

Table 1

Carbon and oxygen isotope ratios in rocks of the Wydrza deposit

Sample number	Borehole	Depth m	Rock	Stratigraphy	$\delta^{13}C^{\circ}/_{oo}$	$\delta^{18}O^{\circ}/_{oo}$
1	WD-7	178.6	limestone	upper Badenian	−14.9	−6.9
2	WD-57	193.7	,,		−9.5	−3.0
3	WD-56	181.3	,,		−5.7	−0.2
4	WD-7	192.7	sulphur-bearing limestone		−25.3	−3.8
5	WD-7	195.0	,,		−42.0	−5.6
6	PT-76	188.2	,,		−45.0	−3.2
7	PT-76	192.0	,,		−46.5	−2.5
8	WD-12	215.6	,,		−31.8	−3.7
9	WD-61	205.2	,,		−50.5	−3.2
10	WD-7	205.2	lithotamnium limestone	lower Badenian	−6.8	−0.5
11	WD-57	215.0	,,		−4.2	−0.2
12	WD-56	214.5	,,		−5.8	−0.3

Average values	$\delta^{13}C^{\circ}/_{oo}$	$\delta^{18}O^{\circ}/_{oo}$
Limestone between tuffites	−10.0	−3.4
Sulphur-bearing limestones	−40.2	−3.7
Lithotamnium limestones	−5.6	−0.3

It is observed that isotope ratios of sample *4* and *8* (Table 1) belonging to II group, according to Czermiński's classification, are very close to those of bitumina dispersed in Miocene deposits.

Lithotamnium limestones, represented by the samples *10*, *11* and *12*, display the lowest content of light carbon isotope. Their primary character is also indicated by $\delta^{18}O$ values. It also confirms distinct enrichment of post-gypsum limestones and some limestones occurring above the sulphur-bearing series in light C isotope. This conclusion is important if we take into account that sulphur occurring locally within Lithotamnium limestones is unquestionably of post-gypsum origin.

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SKŁAD MINERALNY ORAZ IZOTOPOWY WAPIENI MIOCEŃSKICH ZE ZŁOŻA SIARKI WYDRZA

Streszczenie

Wykonano badania mineralogiczne i izotopowe wapieni występujących ponad serią siarkonośną i odpowiadającą jej strefą nadgipsową. Prześledzono zmiany składu mineralnego i izotopowego wapieni osiarkowanych oraz podścielających je wapieni litotamniowych.

W wapieniach występujących ponad serią chemiczną stwierdzono wzbogacenie w lekki izotop węgla w strefie nadsiarkowej w porównaniu z wapieniami występującymi ponad gipsami. W wyniku analizy izotopowej zaliczono wapienie osiarkowane do wapieni pogipsowych stwierdzając równocześnie odmienny skład izotopowy zawartego w nich węgla w stosunku do węglowodorów mioceńskich oraz karbońskich i dewońskich. Badania izotopowe podścielających serię chemiczną wapieni litotamniowych wykazały ich słabe zróżnicowanie izotopowe. Potwierdziły także ich pierwotny charakter przy równoczesnym występowaniu siarki mającej niewątpliwie wtórny charakter.

OBJAŚNIENIA FIGUR

Fig. 1. Przekrój geologiczny przez złoże siarki Wydrza

1 – wapienie litotamniowe, *2* – wapienie zawierające siarkę, *3* – gipsy wielkokrystaliczne, *4* – gipsy drobnokrystaliczne, *5* – wapienie pomiędzy tufitami, *6* – tufity, *7* – miejsca pobrania próbek

Fig. 2. Dyfraktogramy rentgenowskie badanych próbek (*1–6* numery próbek)

C – kalcyt, *S* – siarka, *Q* – kwarc, *I* – illit, *Chl* – chloryt, *G* – gips, *Sr* – stroncjanit, *P* – piryt

Fig. 3. Dyfraktogramy rentgenowskie badanych próbek (*7–12* numery próbek)

C – kalcyt, *S* – siarka, *Q* – kwarc, *I* – illit, *L?* – lepidokrokit ?

OBJAŚNIENIA FOTOGRAFII

Fot. 1. Szkliwo wulkaniczne (czarne) tkwiące w wapieniu. Pow. × 43, nikole X

Fot. 2. Kryształ stroncjanitu (s) tkwiący w wapieniu pogipsowym zawierającym siarkę. Pow. × 30, nikole X

Fot. 3. Fragment wapienia litotamniowego zawierającego piryt. Pow. × 25, nikole X

Мацей ПАВЛИКОВСКИ, Войцех ПШИБЫЛОВИЧ

МИНЕРАЛЬНЫЙ И ИЗОТОПНЫЙ СОСТАВ ИЗВЕСТНЯКОВ ИЗ МЕСТОРОЖДЕНИЯ СЕРЫ ВЫДЖА

Резюме

Были проведены минералогические и изотопные исследования известняков, которые залегают над серной серией и соответствующей ей надгипсовой зоной. Были прослежены изменения минерального и изотопного состава известняков с серой и подстилающих их литотамниевых известняков.

В известняках, которые залегают над химической серией, было обнаружено обогащение лёгкими изотопами углерода в надсерной зоне по сравнению с известняками расположенными над гипсами. В следствии изотопного анализа известняки с серой были причислены к послегипсовым известнякам и было обнаружено, что изотопный состав углерода, который в них содержится, другой, чем в миоценовых, угольного периода и девонских углеводородах. Изотопными исследованиями литотамниевых известняков, подстилающих химическую серию, была обнаружена их небольшая изотопная дифференциация. Подтвердился тоже их первичный характер с одновременным присутствием серы, которая несомненно вторичного происхождения.

ОБЪЯСНЕНИЯ К ФИГУРАМ

Фиг. 1. Геологический разрез через месторождение серы Выджа

1 — литотамниевые известняки, 2 — известняки с серой, 3 — крупнокристаллические гипсы, 4 — мелкокристаллические гипсы, 5 — известняки между туффитами, 6 — места отбора образцов

Фиг. 2. Рентгеновские дифрактограммы изучаемых образцов (*1—6* — номера образцов)

C — кальцит, *S* — сера, *Q* — кварц, *I* — иллит, *Chl* — хлорит, *G* — гипс, *Sr* — стронцианит, *P* — пирит

Фиг. 3. Рентгеновские дифрактограммы изучаемых образцов (*7—12* — номера образцов)

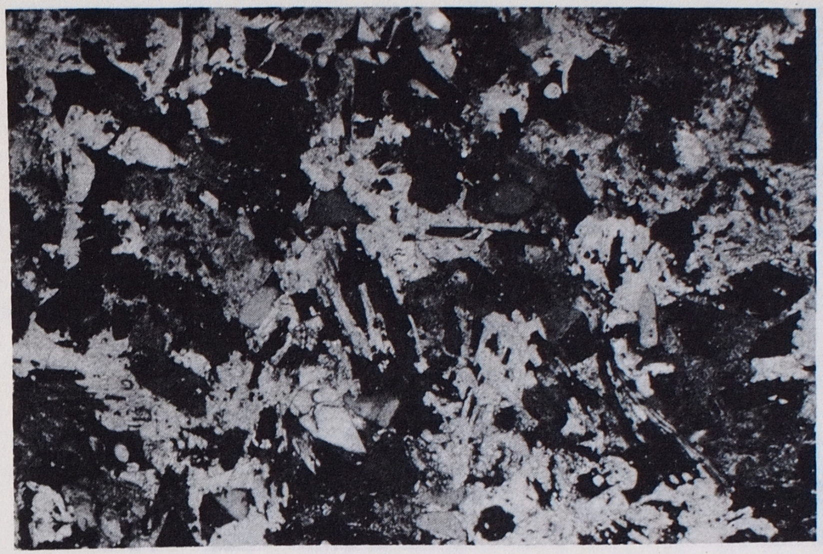
C — кальцит, *S* — сера, *Q* — кварц, *I* — иллит, *L?* — лепидокрокит?

ОБЪЯСНЕНИЯ К ФОТОГРАФИЯМ

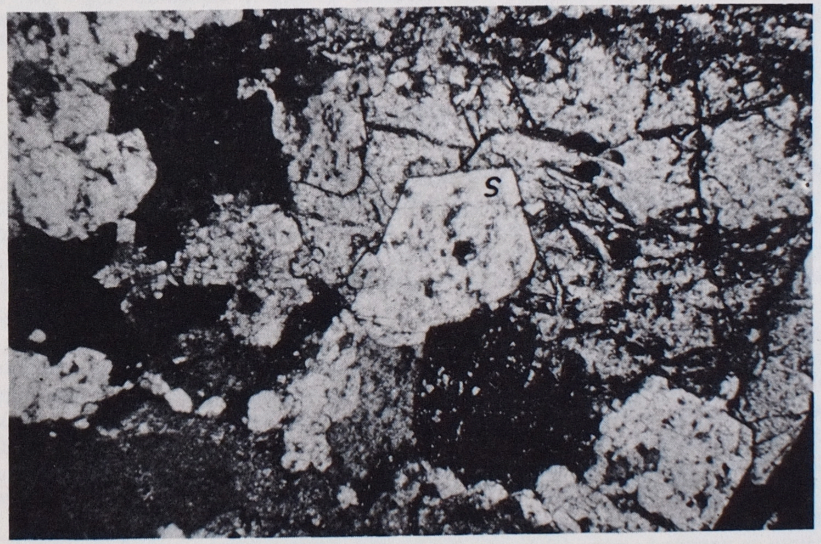
Фото 1. Вулканическое стекло (чёрное) в известняке. Увеличение × 43, скрещенные николи

Фото 2. Кристалл стронцианита (S) в послегипсовом известняке, содержащем серу. Увеличение ×30, скрещенные николи

Фото 3. Фрагмент литотамниевого известняка содержащего пирит. Увеличение × 25, скрещенные николи



Phot. 1. Volcanic glass (black) embedded in limestone. × 43, crossed nicols



Phot. 2. Strontianite crystal (s) embedded in post-gypsum sulphur-bearing limestone. × 30, crossed nicols

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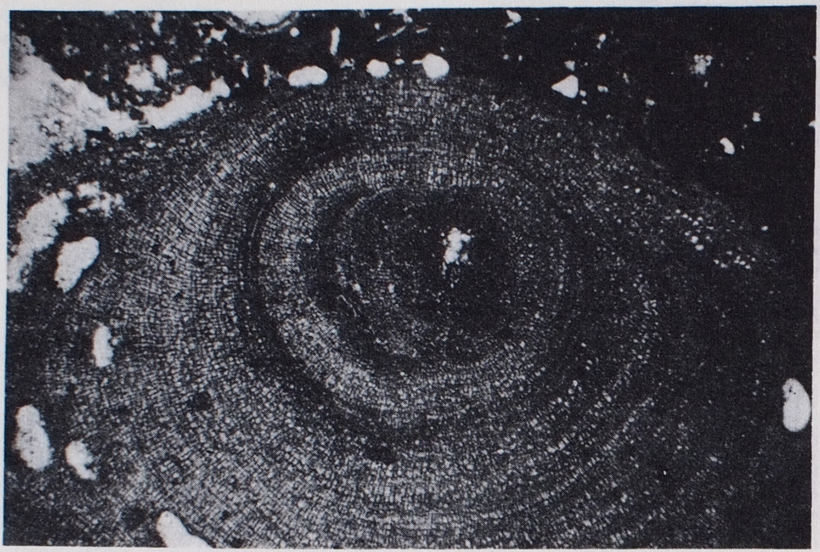

Phot. 3. Pyrite-bearing fragment of Lithotamnium limestone. × 25, crossed nicols

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