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PRELIMINARY REPORT ON PRECIPITATION OF MINERALS FROM NATURAL CO_2-SATURATED SPRING WATERS (SIVA BRADA, SLOVAKIA)

Abstract. Simple experiments concerning evaporation of natural CO_2-rich water were carried out to get precipitates and define their mineral composition. As a result, some rare minerals appeared including such as nesquehonite, picromerite, thenardite, arcanite and konyaite, apart from calcite, aragonite, and halite. While Mg/Ca ratio was low, calcite was the first mineral phase to precipitate. When the ratio grew up, aragonite began to crystallize. Calcite occurred as rhombohedra and sphaerulites, aragonite formed also sphaerulites and thin needles. The third mineral phase was nesquehonite as concretions and rosettes built up of radially arranged well developed crystals. Halite and sulphates formed at the end stage of evaporation. Halite appeared as dendritic and cubic crystals, while sulphates formed efflorescences and sphaerulites. Wavy extinction was typical of all crystals.

Key-words: CO_2-rich water, precipitates, calcite, aragonite, nesquehonite, halite, sulphates.

INTRODUCTION

Siva Brada hill (Fig. 1) is situated about 2.5 km west of Spisske Podhradie (Slovakia). The hill is 20 m high and is built up of postglacial and recent travertines (Ložek and Prošek 1957). Of the six springs on this hill, two are of special interest: the „gayser" and the small „living" crater filled up with CO_2-saturated water. The recurrent eruptions in gayser are caused by high CO_2-pressure: fountains of water may reach up to 10 m in height (Kovanda 1971). Water in the crater (about 30 cm deep) is agitated violently and its surface is crowded with gas bubbles. Temperature of the water depends to a high degree on the air temperature, as it is heated by the sun. At the beginning of September '93 when samples of water were taken, it reached 17.9°C (air temperature = 22°C). Temperature of the water in the spring intake is 12.3° (*cf.* Franko et al. 1975).

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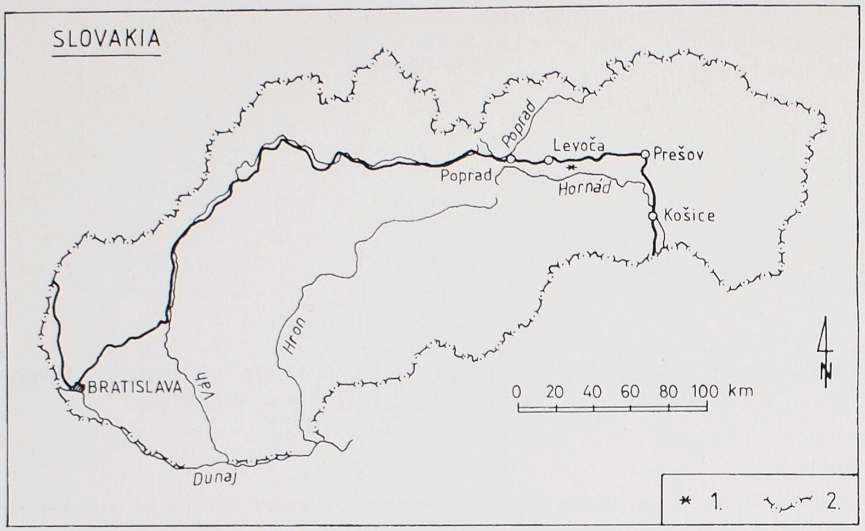

Fig. 1. Index map of Slovakia showing location of investigated area
1 — Siva Brada area; 2 — state boundary

The CO_2-saturated water is highly mineralized (>7000 mg/l), and contains free carbon dioxide up to 1351.6 mg/l. Its main components are: Ca^{2+}, Na^{+}, Mg^{2+}, K^{+}, HCO_3^{-}, SO_4^{2-}, and Cl^{-}, other ions such as Sr, Li, Mn, Fe, Br, I, and F appear in minor quantities. About 10 cm beneath the water table in the crater the pH reached 6.2, but it changed toward the surface to 6.6. Around the crater and on its walls travertine is deposited.

EXPERIMENTS

A few simple experiments were carried out to investigate what minerals would be precipitated from natural CO_2-saturated water. Water from the crater was subjected to evaporation: 1) under normal pressure conditions and temperature about 30°C during the day and 17°C at night; 2) under normal pressure and room temperature (21.5—23°C); 3) under normal pressure and high room temperature (24.5—26°C) during a hot summer. The water was placed into a small glass cylinder and was not stirred during evaporation. Almost each day a small quantity of precipitate was taken off the water surface and a sample was prepared on a glass plate for investigation under the petrographic microscope and/or the SEM. No geochemical analyses of the solution were done during evaporation. When the evaporation was completed, the precipitate was subjected to X-ray diffraction and infrared analyses. Moreover, EDS method was used under the SEM. Because

precipitates were of very small sizes and strongly joined together, separation of particular mineral phases was impossible; hence identification of these minerals under the petrographic microscope and comparison with X-ray data presented a real problem not completely resolved.

RESULTS

Precipitation of minerals from CO_2-saturated water was very rapid. During the first experiment, 10 minutes after the glass cylinder was filled with the water, the water surface became covered with sparkling minute crystals. The first mineral phase was calcite in the form of very small crystals (3—5 μm in size), joined into ramified rafts. A day later, calcite spherulites and hemispherulites appeared growing on calcite crystals. Calcite was not, however, the main phase. A day later, aragonite became prevailing as minute needles and spherulites growing on and around calcite crystals. The next mineral phases that had succeeded aragonite were not investigated until evaporation came to the end. Then X-ray diffraction (sample I), and infrared

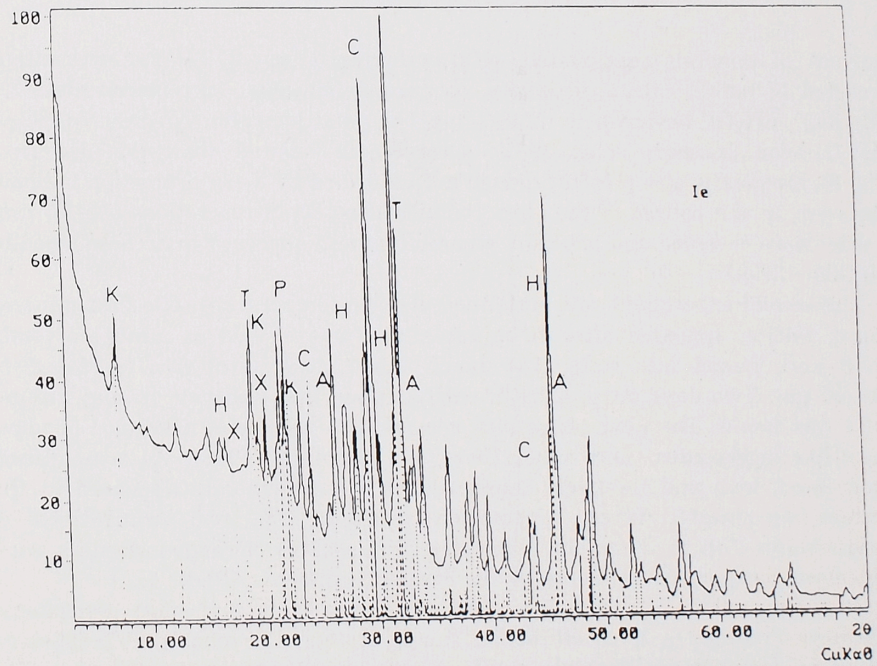

Fig. 2. X-ray diffraction pattern of minerals from sample I.
H — halite; C — calcite; A — aragonite; T — thenardite; P — picromerite; X — hexahydrite; K — konyaite

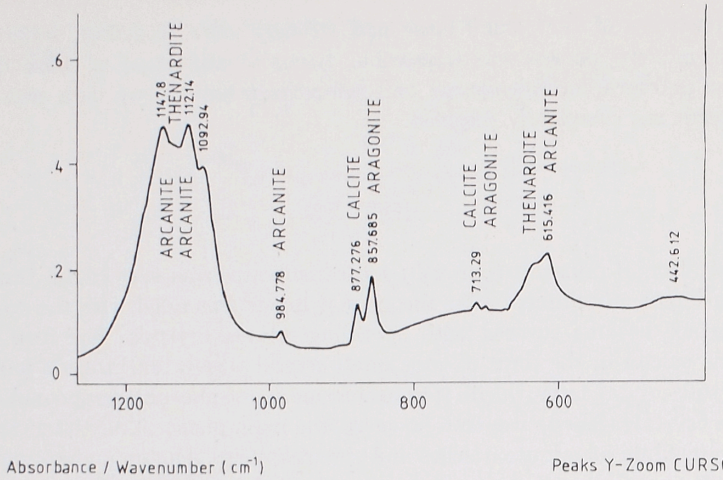

Fig. 3. Infrared absorption spectrum of some minerals from sample I

analyses of minerals precipitated were performed (Figs. 2, 3). The precipitate consisted of halite, calcite, aragonite, thenardite (Na_2SO_4), picromerite ($K_2SO_4 \cdot MgSO_4 \cdot 6H_2O$), hexahydrite ($MgSO_4 \cdot 6H_2O$), and konyaite ($Na_2SO_4 \cdot MgSO_4 \cdot 5H_2O$) (van Doesburg et al. 1982). According to infrared absorption spectrum (Fig. 3), arcanite is also present, though not confirmed by X-ray diffraction method. Moreover, at the bottom of the glass cylinder some small concretions (1—1.5 mm in size) were observed and analysed separately: these appeared to be nesquehonite ($MgCO_3 \cdot 3H_2O$).

The second experiment was performed at room temperature. The first mineral phase, calcite, appeared after 10 minutes, and was formed as minute crystals (3—8 μm), joined into rafts. Two hours later, the crystal size increased to 15—20 μm. Two days later, a lot of calcite spherulites growing on and around rafts was found. The next phase was aragonite in the form of minute needles, sheaf-like aggregates, and spherulites (*cf.* Lippmann 1973). As evaporation progressed, long and relatively thick needles of nesquehonite appeared in the surface precipitate. When evaporation came to the end, concretions of nesquehonite (up to 2 mm in diameter) with radially arranged crystals were the most conspicuous elements of the bottom sediments (Phot. 1).

In the next stage of evaporation picromerite, thenardite, and halite precipitated and were identified by X-ray diffraction (sample II) (Fig. 4). Probably, (?) langbeinite has been found under the petrographic microscope, because isotropic, glass-like mineral is occasionally observed in sediment. At the present stage of investigation it is impossible to determine the sequence of crystallization of the minerals.

In the third experiment, the room temperature reached 24.5—26°C. The first precipitate was calcite in such a small quantity, that it was concealed by aragonite

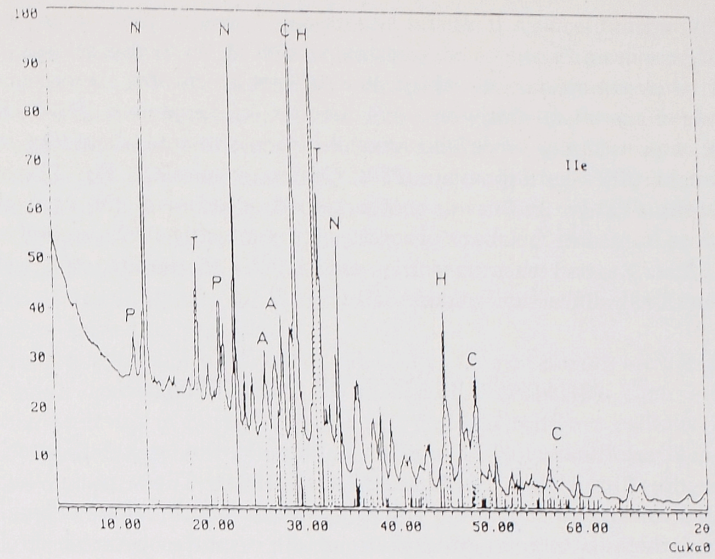

Fig. 4. X-ray diffraction pattern of minerals from sample II
N — nesquehonite; H — halite; P — picromerite; T — thenardite; A — aragonite; C — calcite

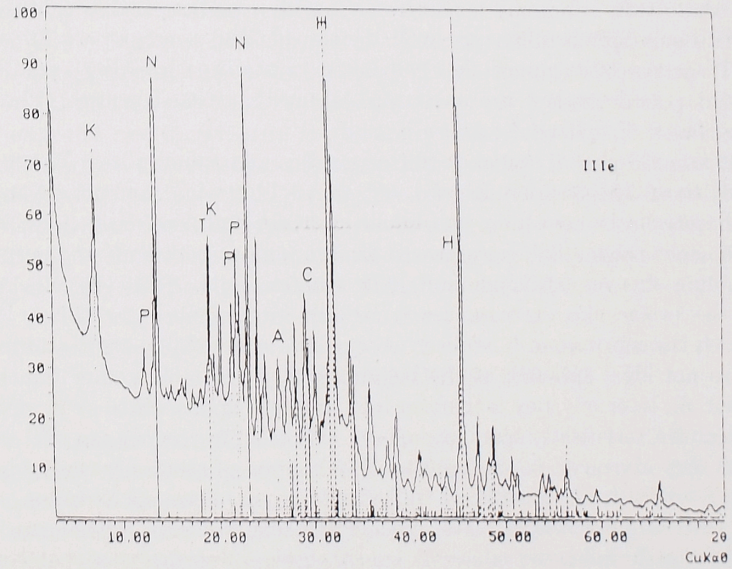

Fig. 5. X-ray diffraction pattern of minerals from sample III
H — halite; N — nesquehonite; K — konyaite; P — picromerite; T — thenardite; A — aragonite; C — calcite

crystallizing spontaneously. It should be stressed, however, that water was already slightly degassed, and some calcite crystals settled at the bottle bottom. Aragonite has occurred as minute needles which tend to form broom-like excrescences on one or two ends of crystals growing in c-axis direction (*cf.* Lippmann 1973). During the next step of growth the broom-like crystals transform into sheaf-like forms and then into spherulites (*cf.* Lippmann 1973; Chafetz et al. 1991, Fig. 12), or directly into spherulites (Phot. 2). Just as in the second experiment, the next phase was nesquehonite formed in the shape of rosettes and concretions with radially arranged crystals (Phot. 3), and then konyaite, picromerite, thenardite, and halite were detected by X-ray diffraction (sample III) (Fig. 5).

CHARACTERISTICS OF PRECIPITATES

Calcite forms rhombohedral crystals built up of flat spikes (Kostecka 1994), that join into thin plates (*cf.* Chafetz et al. 1991). These plates are slightly misoriented relative to each other (Phot. 4), and in result calcite shows undulate extinction, which is a common feature of all crystals observed. Most of the calcite crystals create steep-sided rhombohedra.

Calcite crystals are joined into rafts floating on the water surface. The upper surface of a raft is flat, and crystals grow downward into the water column (*cf.* Hanor 1978; Chafetz et al. 1991; Kostecka 1992). When precipitation proceeds, the rafts become overgrown with calcite sphaerulites (or hemisphaerulites). Sphaerulites are well developed, and reach up to 70 μm in diameter. They are built up of closely packed crystallites growing radially, so that uniaxial pseudocrosses are well visible under crossed polars (Phot. 5). External surfaces of sphaerulites are smooth.

The next mineral phase, aragonite, forms needles and sphaerulites. The needles are relatively short (15—30 μm in length), very thin (<1 μm wide), and polycrystalline, and even the smallest ones show, like calcite crystals, undulose extinction. During their development broom-like excrescences form on one or both ends of the needles thus resembling sheaves (*cf.* Lippmann 1973; Chafetz et al. 1991, Fig. 12). At the last stage the broom-like excrescences transform into sphaerulites (Phot. 2). All stages of such transformation have been observed in slides. Sphaerulites formed in this way are not ideal spheres; at the beginning they resemble rather bunches of flowers (Phot. 6), later on, they transform into irregular sphaerulites or hemisphaerulites with characteristically spicular surfaces (Phot. 7). In comparison with calcite sphaerulites they appear to be darker, probably because of relatively loosely packed crystallites. Pseudocrosses are not so well visible as in calcite sphaerulites.

Special kind of sphaerulites have appeared during the first experiment. They are honey-brownish and have relatively smooth external surfaces as visible under the petrographic microscope. They may form single sphaeres or twins and/or multi-individuals. When crossed polars are used, sphaerulites appear to be dark, though uniaxial pseudocrosses are slightly visible. The SEM observations show that sphaerulites are highly porous: their crystallites resemble a kind of honeycomb crystals that are empty inside (Phot. 8). This high porosity causes dispersion of light, and, probably, is responsible for the brown colouration of sphaerulites and their low interference colours under crossed polars. The honeycomb-like crystals resemble the „skeletal" aragonite that has been synthesized by Franke (pers. inform.,1993, Freie Universitat Berlin). However, according to EDS spot analyses, they contain Mg, S, K, Na and Cl. Cl and at least partly Na ions are connected with halite, that is associated frequently with sphaerulites, Mg, S, K, and/or Na enter probably into the composition of sphaerulites. EDS spot analyses are not precise, so the chemical composition of these sulphate sphaerulites in not fully known. Further investigations are needed to solve this problem.

Monoclinic nesquehonite ($MgCO_3 \cdot 3H_2O$) has formed during each experiment, creating small concretions (Phot. 1), or rosettes (Phot. 9). Concretions and rosettes are built up of perfectly developed crystals that are radially arranged (Phot. 3). Crystals of nesquehonite also show wavy extinction. This mineral phase is rarely found in nature (e.g., Palache et al. 1953; Fischbeck and Muller 1971), though relatively easy to precipitate.

Of the remaining minerals precipitated, halite is the commonest and the easiest to recognize. It forms dendritic or skeletal crystals (Phot. 10) and occasionally, small cubes, and is associated with sulphates, mainly thenardite (Phot. 10). Sulphates are, however, very difficult to recognize. They form efflorescences with amazing shapes (Phot. 11), intergrowing to each other. Their appearance on glass slide was quite fortuitous: when the precipitate was taken off the water surface, some drops of water together with the precipitate were put on the glass plate and then evaporated, thus leaving efflorescences. Sulphates have not their own crystallographic shapes, and all of them show wavy extinction.

Thenardite and picromerite are optically rather similar (*cf.* Łaszkiewicz 1967), especially when efflorescences are taken into account. In turn, konyaite, according to van Doesburg et al. (1982), belongs to very unstable minerals and decomposes easily (in a few days) at room temperature, giving rise to bloedite. In general, identification of these minerals is based mainly on X-ray diffraction and infrared analyses. EDS method gives only signal information.

REMARKS AND CONCLUSIONS

CO_2-saturated waters from Siva Brada are rich in Ca, Na, Mg, and K, being the most important components. The concentration of carbonate ions is also high, due to rapid CO_2 outgassing. According to Franko et al. (1975), Mg/Ca ratio of these waters is about 0.6, slightly higher than that (Mg/Ca = 0.3) in the experiments by Berner (1975), but low enough for calcite to be precipitated as low-magnesian phase (Fig. 2, 4, and 5). It is well known that adequate concentration of Mg ions in solution suppresses the precipitation of calcite in favour of aragonite (Monaghan and Lytle 1956; Simkiss 1964; Lippmann 1973), though other factors (e.g. amount

of carbonate ions) also control the nature of inorganically precipitated carbonate phases (Given and Wilkinson 1985). Unfortunately, no geochemical investigations were carried out during experiment duration. It must be stressed, however, that the first precipitate is always calcite, the next is aragonite and the third is nesquehonite. This means, that when calcite precipitation proceeded, Mg/Ca ratio grew higher, and at some moment, aragonite began to crystallize instead of calcite (*cf.* Simkiss 1964; Lippmann 1973; Berner 1975). The last carbonate phase was nesquehonite. This indicates, that during precipitation of calcium carbonates, all Ca ions had been exhausted, whereas Mg ions were still present in the solution, giving rise to crystallization of nesquehonite and then sulphates.

The first mineral phase, calcite, creates steep-sided rhombohedra and sphaerulites that form at normal temperature, and not necessary in hot springs (*cf.* Steinen at al. 1987). In Holocene and recent travertines of Siva Brada sphaerulitic fabric belongs to widespread phenomena. The next mineral phase, aragonite, forms needles and sphaerulites. It has also been found in travertines of Siva Brada in the shape of small sphaerulites and needle cement, the latter in the stage of calcitization. Aragonite crystals have been observed mainly in pores, where they form bunches of loosely packed needles growing radially from the substrate, while sphaerulites are dispersed in matrix.

The third mineral phase, nesquehonite, is unstable and, at 25°C and 1 atm. CO_2 pressure, it is 24 times as soluble as calcite (Langmuir 1965). If it were present in travertines, CO_2-rich waters flowing through the pore system would have dissolved it. Probably, this is the cause why nesquehonite has not yet been found in travertines of Siva Brada.

Halite and sulphates are the end-products of evaporation of a solution and their occurrence in travertines is not expected. Although they could precipitate from standing water body under favourable conditions, their high solubility makes their presence impossible, in particular, when they are exposed to meteoric circumstances.

It is worthy of notice that all the crystals show wavy extinction. This phenomenon implies that crystals are polycrystalline because deformation of crystal lattices is out of the question. In relation to calcite, wavy extinction is caused by the fact, that subcrystals that form thin sheets are not parallel to each other (Phot. 4). In turn, aragonite is built up of thin needles, difficult to measure even under the SEM (Phot. 2), whose c-axes are not parallel to each other (Phot. 12). Sulphates occur as efflorescences with a fabric approximating that of a sphaerulite, and as sphaerulites. In the author's opinion, if crystal lattice is not distorted, wavy extinction proves the high rate of crystallization, just as in the case of sphaerulites (*cf.* Folk et al. 1985; Steinen et al. 1987; Kostecka 1993).

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WSTĘPNE UWAGI O WYTRĄCANIU MINERAŁÓW Z NATURALNYCH WÓD ŹRÓDLANYCH NASYCONYCH CO_2 (SIVA BRADA, SŁOWACJA)

Streszczenie

Próbki wody mineralnej z „żywego" krateru na wzgórzu Siva Brada (Słowacja) (Rys. 1), zostały poddane prostym eksperymentom odparowania w celu znalezienia asocjacji mineralnych. Pierwszy eksperyment przeprowadzono w warunkach nieco podwyższonej temperatury (30°C w dzień, 17°C w nocy), drugi — w temperaturze pokojowej (21,5—23°); trzeci eksperyment — również w temperaturze pokojowej (24,5—26°), lecz podczas wyjątkowo gorącego lata 1994. Otrzymany osad został poddany badaniom w mikroskopie petrograficznym i skaningowym, badaniom rentgenowskim, analizie w podczerwieni i EDS (energy dispersive spectroscopy).

W wyniku pierwszego eksperymentu wytrąciły się następujące minerały: kalcyt, aragonit, halit, tenardyt, pikromeryt, heksahydryt, konyait (Rys. 2) oraz arkanit (Rys. 3) i nesquehonit (Fot. 1). W drugim eksperymencie nie stwierdzono heksahydrytu i konyaitu (Rys. 4), w trzecim natomiast nie znaleziono heksahydrytu (Rys. 5). Arkanit (Rys. 3) został wykryty metodą analizy w podczerwieni.

W każdym przypadku pierwszym precypitatem był kalcyt w postaci romboedrów i sferulitów (Fot. 4 i 5). Jako drugi krystalizował aragonit, tworząc polikrystaliczne igiełki i sferulity (Fot. 2, 6, 7 i 12). Trzecim z kolei minerałem był nesquehonit w formie konkrecji i rozet o radialnie zorientowanych kryształach (Fot. 1, 3 i 9). Prawdopodobnie następnym minerałem jest halit pojawiający się w postaci kryształów dendrytycznych lub szkieletowych (Fot. 10), rzadziej kubicznych. Jednak kolejność krystalizacji halitu i minerałów siarczanowych nie była badana. Siarczany występują głównie jako wykwity odznaczające się falistym wygaszaniem światła (Fot. 11) oraz sferulity (Fot. 8).

Sferulity kalcytowe mają gładkie powierzchnie zewnętrzne, są zbudowane z gęsto upakowanych krystalitów, a przy nikolach skrzyżowanych można w nich obserwować jednoosiowy pseudokrzyż z wygaszania (Fot. 5). Sferulity aragonitowe są zbudowane z dość luźno upakowanych igiełek (Fot. 6 i 7), mają żółtawe zabarwienie a pseudokrzyż z wygaszania jest znacznie mniej wyraźny niż w przypadku sferulitów kalcytowych. Ponadto sferulity aragonitowe nie tworzą idealnych form kulistych, a ich powierzchnie zewnętrzne są najeżone ostro zakończonymi krystalitami (Fot. 6).

Sferulity siarczanowe powstałe w trakcie pierwszego eksperymentu tworzą formy kuliste pojedyncze (Fot. 8), bliźniacze lub wieloosobnikowe. W świetle przechodzącym odznaczają się miodowobrunatnym zabarwieniem, a przy nikolach skrzyżowanych słabo reagują na światło spolaryzowane; pseudokrzyż jest widoczny jedynie w przypadku cienkich przekrojów. Badania skaningowe wykazują, że wspomniany typ sferulitów składa się z krystalitów wewnętrznie pustych (Fot. 8), których zarysy w przekroju prostopadłym do ich dłuższej osi przypominają plaster miodu. Prawdopodobnie obecność pustek jest przyczyną silnego rozproszenia światła, co uwidoczniło się ciemnymi barwami osobników i słabą reakcją na światło spolaryzowane.

Minerały siarczanowe nie były przedmiotem szczegółowych badań mikroskopowych, zostały zidentyfikowane rentgenograficznie (Rys. 2, 4 i 5) i w podczerwieni (Rys. 3).

W wodzie mineralnej ze wzgórza Siva Brada stosunek Mg/Ca wynosi 0,6 (Franko et al. 1975); ta wystarczająco niska wartość umożliwia precypitację kalcytu. W miarę krystalizacji kalcytu wartość stosunku Mg/Ca odpowiednio rośnie, co z kolei hamuje dalszy rozwój kalcytu, lecz umożliwia krystalizację aragonitu (por. Lippmann 1973). Po wykorzystaniu wszystkich jonów Ca z roztworu krystalizuje nesquehonit ($MgCO_3 \cdot 3H_2O$), a następnie halit i siarczany.

Wszystkie wytrącone z wody mineralnej precypitaty wykazują faliste wygaszanie światła. Zdaniem autorki, w przypadku niezdeformowanej sieci przestrzennej minerału (a deformacje należy tu wykluczyć), ściemnianie faliste świadczy o bardzo szybkiej krystalizacji danej fazy mineralnej (por. Folk et al. 1985; Steinen et al. 1987; Kostecka 1993).

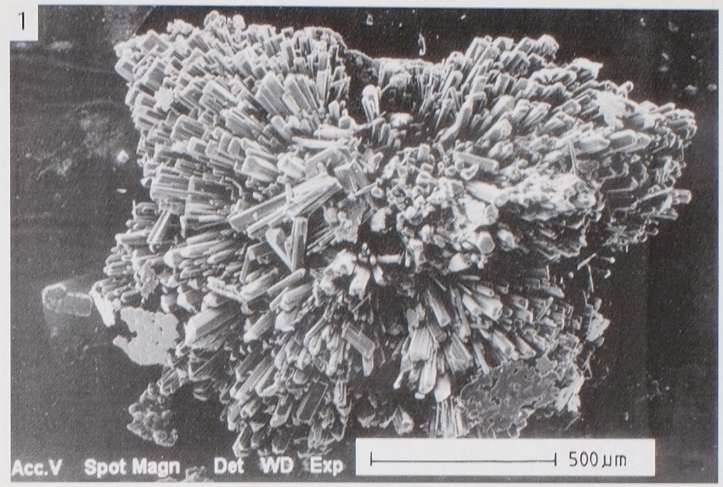
OBJAŚNIENIA DO RYSUNKÓW

Rys. 1. Lokalizacja badanego obszaru na mapie Słowacji: 1 — Siva Brada; 2 — granica państwa.
Rys. 2. Dyfraktogram rentgenowski minerałów z próbki I: H — halit; C — kalcyt; A — aragonit; T — tenardyt; P — pikromeryt; H — heksahydryt; K — konyait.
Rys. 3. Widmo absorpcyjne w podczerwieni niektórych minerałów z próbki I.
Rys. 4. Dyfraktogram rentgenowski minerałów z próbki II: N — nesquehonit; H — halit; P — pikromeryt; T — tenardyt; A — aragonite; C — kalcyt.
Rys. 5. Dyfraktogram rentgenowski minerałów z próbki III: H — halit; N — nesquehonit; K — konyait; P — pikromeryt; T — tenardyt; A — aragonit; C — kalcyt.

OBJAŚNIENIA DO FOTOGRAFII

Fot. 1. Konkrecja nesquehonitu z doskonale wykształconymi kryształami. Mikrofotografia skaningowa.
Fot. 2. Igły aragonitowe rozgałęziające się miotełkowo. Mikrofotografia skaningowa.
Fot. 3. Doskonale wykształcone kryształy nesquehonitu. Mikrofotografia skaningowa.
Fot. 4. Romboedr kalcytowy zbudowany z cienkich płytek, ułożonych nierównolegle względem siebie. Mikrofotografia skaningowa.
Fot. 5. Sferulity i hemisferulity kalcytowe. Zauważ gładkie powierzchnie zewnętrzne i dobrze widoczny jednoosiowy pseudokrzyż z wygaszania. Nikole skrzyżowane.
Fot. 6. „Bukiet kwiatów". Niedoskonały sferulit aragonitowy. Mikrofotografia skaningowa.

Fot. 7. „Tratwa kalcytowa" obrośnięta aragonitowymi sferulitami. Zewnętrzne powierzchnie sferulitów najeżone ostro zakończonymi krystalitami. Światło spolaryzowane.
Fot. 8. Sferulit aragonitowy o budowie plastra miodu. Krystality wewnątrz puste. Mikrofotografia skaningowa.
Fot. 9. Rozety nesquehonitu. Nikole skrzyżowane.
Fot. 10. Wykwity tenardytu (1) i szkieletowe kryształy halitu analizowane metodą EDS. Mikrofotografia skaningowa.
Fot. 11. Wykwity siarczanów. Ściemnianie faliste dobrze widoczne. Nikole skrzyżowane.
Fot. 12. „Tratwy" aragonitowe. Zauważ faliste ściemnianie kryształów. Nikole skrzyżowane.



Phot. 1. Concretion of nesquehonite with perfectly developed crystals. SEM micrograph

Phot. 2. Aragonite needles with broom-like excrescences. SEM micrograph

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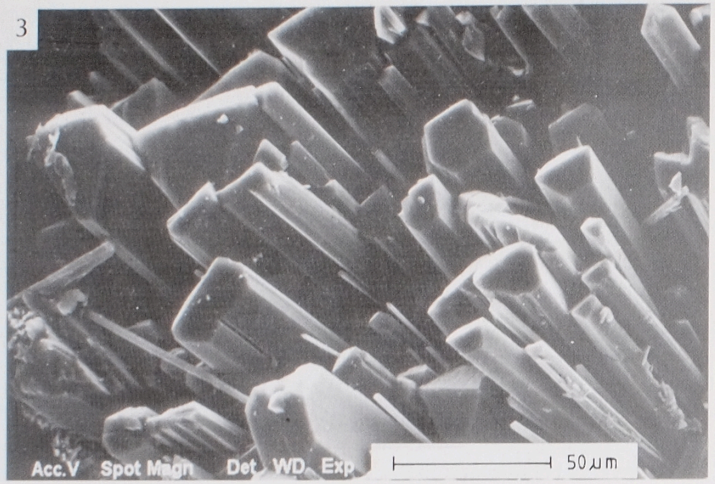
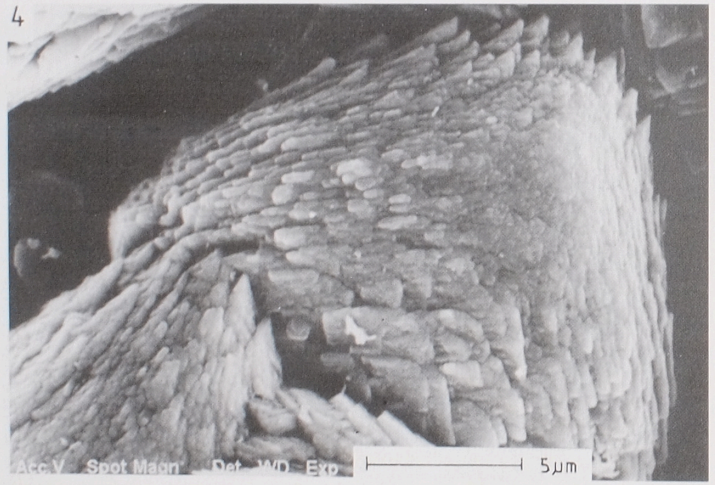


Phot. 3. Perfectly developed crystals of nesquehonite. SEM micrograph



Phot. 4. Calcite rhombohedron built up of thin plates that are not parallel to each other. SEM micrograph

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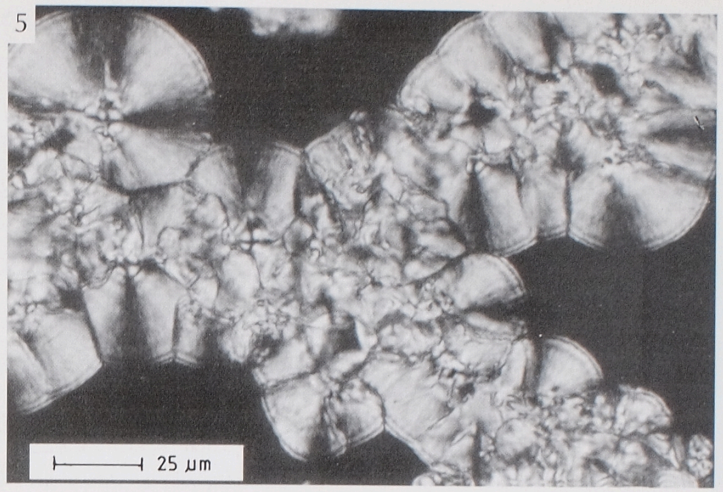

Phot. 5. Calcite sphaerulites and hemisphaerulites. Note smooth external surfaces of sphaerulites and well developed uniaxial pseudocross. Crossed polars

Phot. 6. „Bunch of flowers". Imperfect aragonite sphaerulite. SEM micrograph

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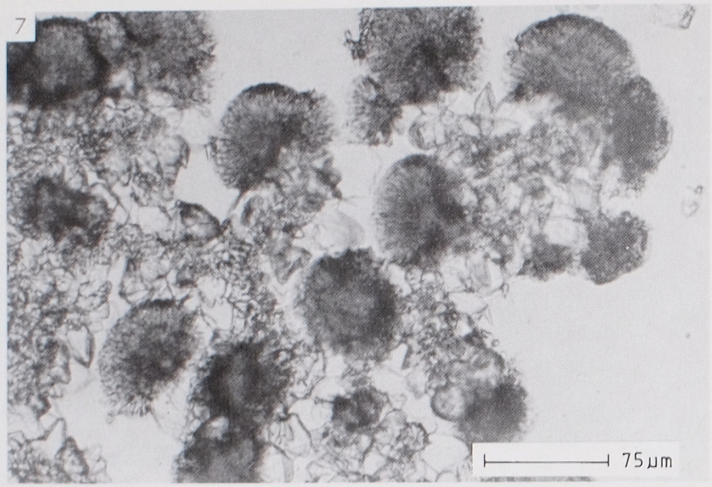
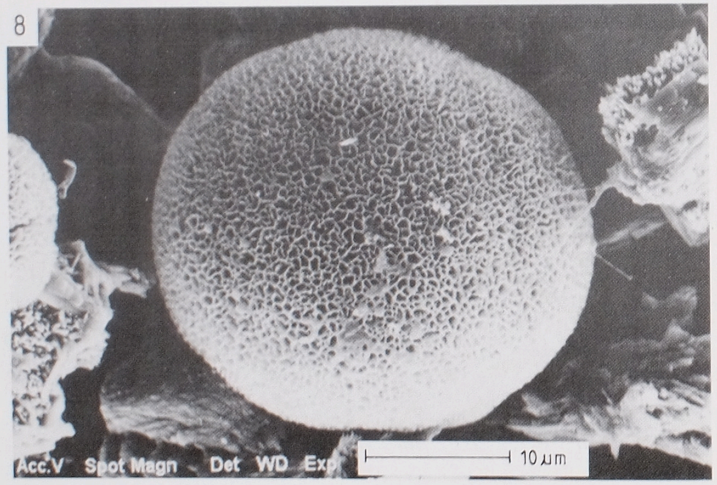


Phot. 7. Calcite raft with aragonite sphaerulites. Note spicular external surfaces of sphaerulites. Plane light



Phot. 8. Sulphate sphaerulite with honeycomb crystal fabric. Crystals are empty inside. SEM micrograph

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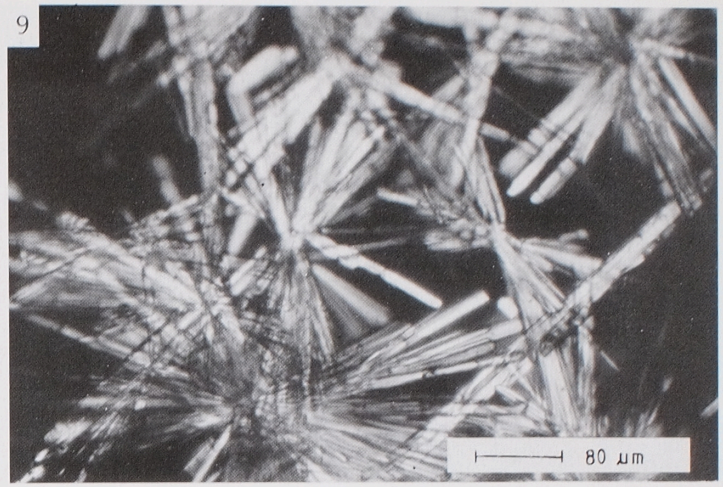
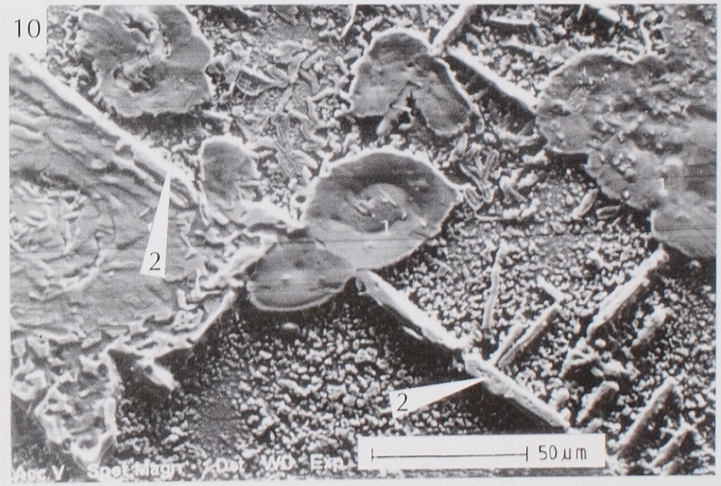


Phot. 9. Rosettes of nesquehonite. Crossed polars



Phot. 10. Efflorescences of thenardite (1), and skeletal crystals of halite (2), analysed with EDS. SEM micrograph

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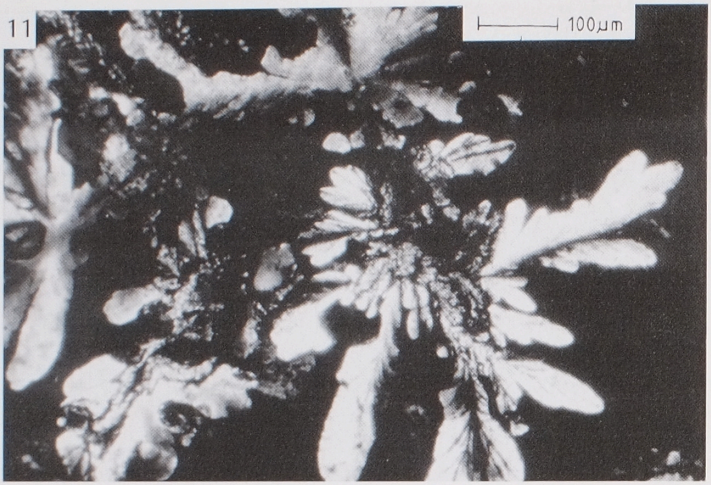
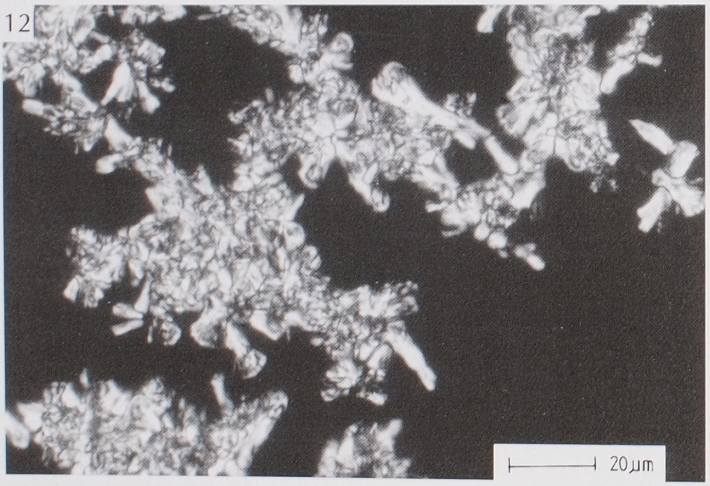


Phot. 11. Efflorescences of sulphates, wavy extinction well visible. Crossed polars



Phot. 12. Aragonite rafts. Note wavy extinction of crystals. Crossed polars