

# MINERALOGICAL RESEARCHES IN “PEȘTERA CU LILIECI DIN SATUL PEȘTERA”, BRAN-RUCĂR PASSAGE

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*Abstract.* By means of X-rays diffractometric analyses on powders, we evidence an association of minerals in “Peștera cu Lilieci din satul Peștera”, made up of *hydroxylapatite* (carbonate-hydroxylapatite), *brushite*, *taranakite*, *calcite*, *quartz*, *illite* and *birmessite*.

## 1. INTRODUCTION

Peștera cu Lilieci (Bat Cave), also known locally as Peștera Mare (Great Cave) from Satul Peștera (Peștera Village) or Peștera Bădichii (Bădichii Cave), is dug in a Kimmeridgian Tithonian limestone massif, regarded as ostiolite in the mass of the Moieciu conglomerates with a Vraconian-Cenomanian age (SANDULESCU *et al.*, 1972).

The cave is situated on the right slope of the Valea cu Calea brook, a tributary of the Râul Turcului (râu = river), Bran–Rucăr Passage, at only 200–250 m from the church of the Peștera village (Fig. 1).

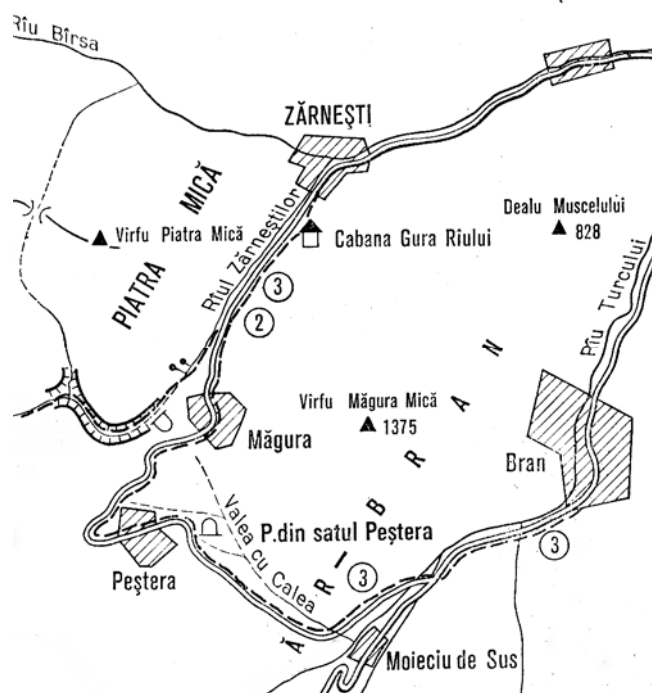


Fig. 1. – Geographic position of the “Peștera cu Lilieci din satul Peștera”.

This is a small cave, the total length of the galleries being less than 162 m. Although the first researches were made in Peștera cu Lilieci in 1957, when NICOLAESCU-PLOȘOR made some archeological investigations, the first mapping of the cave was done in 1972 by CONSTANTINESCU, (Fig. 2).

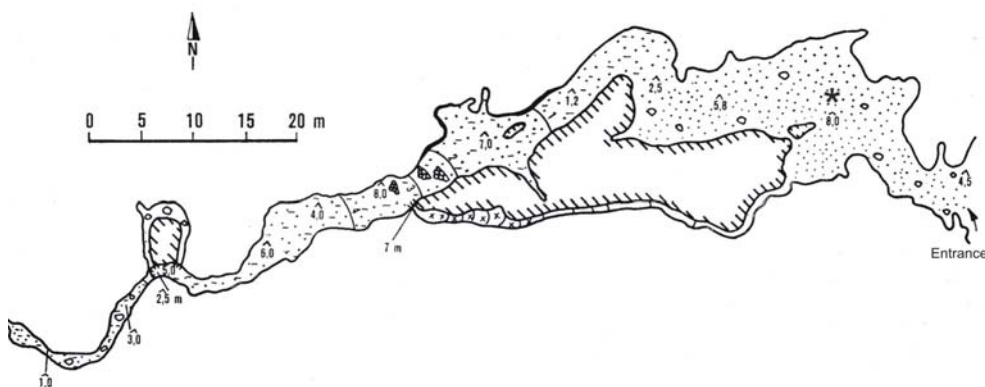


Fig. 2. – Peștera cu Lilieci din satul Peștera\* – sampling sites.

The mineral samples were collected from the section of a cliff, opened probably during the archeological digs, situated in the great hall, 25/15 m, at approximately 20 m from the entrance measuring 5/4.5 m.

The analyses evidenced an association of phosphates made up of *hydroxylapatite* (*carbonate-hydroxylapatite*), *brushite* and *taranakite* beside *calcite*, *quartz*, *illite* (polytype 2 M1) and, very probably, *birnessite*. A number of amorphous iron sesquioxides probably give the red color of the *terra rossa* type deposit and also give the allochromatic color of the crust-like deposits of hydroxylapatite.

## 2. ANALYTIC METHODS

The analytic method used was that of the X-rays diffractometry on powders.

The radiation (Cu,  $K\alpha$ ,  $\lambda = 1.54056 \text{ \AA}$ ) was monochromatized using a graphite bent monochromator. The scanning speed was 0.01, or 0.02° ( $2\theta$ ) per second for 4, respectively 2.4 seconds per step.

## 3. RESULTS AND DISCUSSIONS

**Hydroxylapatite** (*carbonate-hydroxyl-apatite*, ?),  $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ , is made of crust-like, crystalline aggregates, with a lamellar shape and an orange or crème coloration.

Both the occurrence pattern (the relationships with the other minerals in the association) and the textural aspects point to a hydroxylapatite resulting from precipitation processes.

The interreticular distances measured and computed for the main diffractometric reflexes for the two representative hydroxylapatite samples from Peștera cu Lilieci, as well as the Miller indexes established for the hypothesis of the hexagonal symmetry, mineral spatial group  $P6_3/m$ , are given in Table 1.

The  $a$  parameters of the cells from the two samples, given under the table, are very close to the similar parameters of some synthetic hydroxylapatite (MORGAN *et al.*, 2000), while the  $c$  parameters are slightly lower, a fact allowing us to suppose the presence of carbonate-hydroxylapatite instead of hydroxylapatite.

Table 1

Diffractometric data from two representative samples of hydroxylapatite from Peștera cu Lilieci din satul Peștera\*

Crt. no.	Sample PPL 3 B			Sample PPL 5 B			(hkl)
	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	
1	8.1437	8.1520	6	8.0844	8.1606	6	(100)
2	–	–	–	5.2173	5.2501	3	(101)
3	3.8746	3.8811	6	3.8770	3.8833	5	(111)
4	3.4219	3.4304	60	3.4220	3.4289	65	(002)
5	3.0716	3.0812	13	3.0743	3.0844	11	(210)
6	2.8080	2.8107	100	2.8045	2.8130	100	(211)
7	2.7634	2.7722	74	2.7635	2.7724	82	(112)
8	2.7156	2.7173	21	–	–	–	(300)
9	2.6176	2.6246	18	2.6166	2.6251	24	(202)
10	–	–	–	2.5337	2.5285	17	(301)
11	2.2965	2.2923	3	–	–	–	(212)
12	2.2674	2.2610	18	2.2615	2.2634	15	(130)
13	2.0517	2.0570	5	2.0580	2.0566	6	(113)
14	1.9972	1.9945	9	–	–	–	(203)
15	1.9392	1.9406	22	1.9380	1.9417	27	(222)
16	–	–	–	1.8864	1.8889	12	(132)
17	1.8735	1.8702	10	–	–	–	(230)
18	1.8347	1.8364	30	1.8373	1.8365	29	(213)
19	1.8046	1.8044	15	1.8099	1.8060	15	(321)
20	1.7758	1.7789	4	–	–	–	(410)
21	1.7473	1.7497	8	1.7485	1.7500	13	(303)
22	1.7182	1.7152	14	1.7184	1.7144	20	(004)
23	1.6435	1.6420	5	1.6464	1.6432	4	(322)

Crt. no.	Sample PPL 3 B			Sample PPL 5 B			(hkl)
	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	
24	–	–	–	1.5737	1.5705	19	(330)
25	1.4993	1.4986	4	1.5009	1.4985	5	(124)
26	1.4713	1.4725	6	–	–	–	(502)
27	–	–	–	1.4482	1.4504	5	(304)
28	1.4499	1.4477	5	1.4482	1.4484	5	(323)
29	1.4320	1.4319	7	–	–	–	(511)

$$a = 9.413(5) \text{ Å}$$

$$c = 6.861(5) \text{ Å}$$

$$V = 526.5(5) \text{ Å}^3$$

$$a = 9.423(8) \text{ Å}$$

$$c = 6.858(6) \text{ Å}$$

$$V = 527.3(8) \text{ Å}^3$$

\*Radiation Cu  $K\alpha$  filtered with Ni ( $\lambda = 1.54056 \text{ Å}$ ),  $2\theta = 10\text{--}86^\circ$ . Number of refining cycles: 3; 3.

**Brushite**,  $\text{CaH}(\text{PO}_4) \cdot 2 \text{H}_2\text{O}$ , appears as shining white, powdery sediments in the guano deposit from the cave, associated with the hydroxylapatite.

The interreticular distances measured and computed, the relative intensities and the Miller indexes recorded for two representative brushite samples are given in Table 2. The parameters of the elemental cells of the two samples, computed by refining by the method of the smallest squares, based on 37 reflexes belonging to the brushite in the samples.

The reference parameters of the samples are relatively close to those computed for the stoichiometric brushite by BEEVERS (1958) or CURRY & JONES (1971), [ $a = 5.812(2) \text{ Å}$ ,  $b = 15.180(3) \text{ Å}$ ,  $c = 6.239(2) \text{ Å}$ ,  $\beta = 116.43(3)^\circ$ ], a fact allowing us to suppose the brushite from Peștera cu Lilieci din satul Peștera has a chemical composition close the stoichiometric one.

Table 2

Diphractometric data from two representative samples of brushite from Peștera cu Lilieci din satul Peștera

Crt. no.	Sample PPL 3 A			Sample PPL 3 B			(hkl)
	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	
1	7.5858	7.5788	100	7.5610	7.5735	100	(020)
2	3.7755	3.7894	1	3.7769	3.7867	14	(040)
3	3.6003	3.6038	1	–	–	–	(130)
4	3.0352	3.0424	22	3.0439	3.0391	14	(–1.4.1)
5	2.9140	2.9223	10	2.9341	2.9352	16	(121)
6	2.6138	2.6190	10	2.6176	2.6191	18	(150)
7	2.6138	2.6201	10	2.6176	2.6206	18	(022)

Crt. no.	Sample PPL 3 A			Sample PPL 3 B			(hkl)
	d <sub>meas.</sub> (Å)	d <sub>calc.</sub> (Å)	I / I <sub>0</sub>	d <sub>meas.</sub> (Å)	d <sub>calc.</sub> (Å)	I / I <sub>0</sub>	
8	2.5935	2.6002	4	2.5997	2.6060	8	(200)
9	2.5232	2.5263	2	–	–	–	(060)
10	2.4254	2.4301	3	2.4382	2.4370	5	(141)
11	2.4144	2.4182	2	–	–	–	(-2.2.2)
12	2.2622	2.2640	2	2.2674	2.2620	6	(-1.6.1)
13	2.1667	2.1680	4	2.1687	2.1680	4	(-1.5.2)
14	2.1419	2.1440	3	2.1419	2.1467	5	(240)
15	2.1132	2.1165	1	2.1124	2.1136	6	(-2.4.2)
16	2.0947	2.0966	1	2.0958	2.0948	6	(-2.5.1)
17	2.0785	2.0812	1	2.0958	2.0924	6	(112)
18	2.0161	2.0178	1	–	–	–	(211)
19	1.9984	1.9990	3	1.9972	2.0000	3	(170)
20	1.9984	1.9959	3	1.9747	1.9718	3	(-2.1.3)
21	1.8953	1.8947	3	–	–	–	(080)
22	1.8736	1.8733	3	1.8735	1.8765	10	(062)
23	1.8514	1.8513	1	–	–	–	(-2.3.3)
24	1.8105	1.8119	4	1.8128	1.8132	10	(260)
25	1.7946	1.7953	2	1.7946	1.7931	5	(-2.6.2)
26	1.7766	1.7762	1	1.7758	1.7748	4	(-1.8.1)
27	1.7766	1.7755	1	1.7758	1.7751	4	(-1.7.2)
28	1.7456	1.7467	1	1.7514	1.7537	3	(033)
29	1.7081	1.7085	1	–	–	–	(-3.4.1)
30	1.6583	1.6598	1	1.6581	1.6570	3	(-3.2.3)
31	–	–	–	1.6435	1.6428	2	(330)
32	1.5324	1.5313	1	1.5319	1.5318	6	(280)
33	1.5221	1.5212	1	–	–	–	(-2.8.2)
34	1.5086	1.5079	1	1.5113	1.5117	2	(172)
35	1.4828	1.4832	1	–	–	–	(271)
36	1.4541	1.4522	1	1.4559	1.4576	5	(321)
37	1.4336	1.4328	1	1.4380	1.4394	2	(143)

$a = 5.803(3) \text{ \AA}$   
 $b = 15.159(5) \text{ \AA}$   
 $c = 6.235(2) \text{ \AA}$   
 $\beta = 116.39(2)^\circ$

$a = 5.802(4) \text{ \AA}$   
 $b = 15.157(9) \text{ \AA}$   
 $c = 6.246(5) \text{ \AA}$   
 $\beta = 116.07(4)^\circ$

\*Radiation Fe K $\alpha$  filtered with Ni ( $\lambda = 1.93735 \text{ \AA}$ ),  $2\theta = 10\text{--}90^\circ$ . Number of refining cycles: 10; 4.

**Taranakite**,  $K_2Al_6(PO_4)_6(OH)_2 \cdot 18 H_2O$ , was found as white, chalk-like veins, crossing the *terra rossa* clay in the immediate vicinity of the hydroxylapatite crusts. The mineral is directly associated only with  $\alpha$ -quartz and seems to be formed on the illite from the *terra rossa*.

The interreticular distances measured and computed, the relative intensities and the Miller indexes recorded for a representative sample of taranakite are given in Table 3. 34 reflexes are conferred to the taranakite.

After three cycles of refining by the method of the smallest squares, we obtained:  $a = 8.690$  (4) Å,  $c = 95.8$  (1) Å,  $V = 6268$  (8) Å<sup>3</sup>,  $a : c = 0.091:1$ . The set of parameters recorded by us is very close to the data given by FIORE & LAVIANO (1991) for a sample of taranakite from an Apulian cave [PU 71/6:  $a = 8.660$  (7) Å,  $c = 95.8$  (1) Å].

Table 3

Diphractometric data from one representative sample of taranakite from Peștera cu Lilieci din satul Peștera

Crt. no.	Sample PPL 6 B			(hkl)	Crt. no.	Sample PPL 6 B			(hkl)
	$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$			$d_{\text{meas.}}(\text{Å})$	$d_{\text{calc.}}(\text{Å})$	$I / I_0$	
1	15.7992	15.9745	100	(006)	18	2.8175	2.8137	52	(125)
2	7.9125	7.9872	31	(0.0.12)	19	2.7878	2.7850	12	(217)
3	7.4340	7.4347	60	(012)	20	2.7338	2.7269	23	(2.1.10)
4	5.9002	5.9192	43	(1.0.10)	21	2.6237	2.6268	39	(1.2.14)
5	5.0359	5.0643	7	(0.1.14)	22	2.5695	2.5695	11	(2.1.16)
6	4.6556	4.6869	11	(1.0.16)	23	2.5400	2.5321	5	(0.2.28)
7	4.3124	4.3054	33	(113)	24	2.4748	2.4782	6	(306)
8	4.1882	4.1927	5	(116)	25	2.3942	2.3915	34	(0.1.38)
9	4.0133	4.0231	18	(119)	26	2.3438	2.3435	11	(2.0.32)
10	3.7424	3.7514	30	(202)	27	2.2618	2.2561	8	(0.2.34)
11	3.5846	3.5900	58	(208)	28	2.1739	2.1675	9	(223)
12	3.3552	3.3665	28	(1.1.18)	29	2.1507	2.1527	10	(226)
13	3.2937	3.2976	36	(2.0.14)	30	2.1207	2.1243	14	(3.0.24)
14	3.1776	3.1864	24	(0.2.16)	31	2.0891	2.0853	12	(312)
15	3.1363	3.1470	82	(1.1.21)	32	2.0638	2.0635	26	(137)
16	2.9532	2.9403	22	(1.1.24)	33	2.0410	2.0395	7	(1.3.10)
17	2.8394	2.8395	43	(122)	34	1.9932	1.9966	6	(3.1.14)

$$a = 8.690(4) \text{ Å}$$

$$c = 95.84(10) \text{ Å}$$

\*Radiation Cu  $K\alpha$  monocromatized ( $\lambda = 1.54056 \text{ Å}$ ),  $2\theta = 5-46^\circ$ . Number of refining cycles: 4; 5; 3.

**Calcite**,  $\text{CaCO}_3$ , appears frequently as white or yellowish-white crusts, still untouched by the phosphatic solutions resulting from the guano deposit, or, also as crusts, at the base of the hydroxyl-apatite (carbonate-hydroxyl-apatite) deposits).

The parameters of the elemental cells of some calcite samples, computed by refining by the method of the smallest squares of the Rx diffraction data, based on 18 main reflexes from the  $2\theta$  range between  $25$  and  $90^\circ$  (Fe  $K\alpha$ ,  $\lambda = 1.93735 \text{ \AA}$ ) (Table 4), are the following:

(1) sample PPL 1 B:  $a = 4.975$  (2)  $\text{\AA}$ ,  $c = 17.022$  (8),  $V = 364.9$  (3)  $\text{\AA}^3$  ( $n = 5$ );

(2) sample PPL 9 A:  $a = 4.978$  (1)  $\text{\AA}$ ,  $c = 17.029$  (6),  $V = 364.6$  (3)  $\text{\AA}^3$  ( $n = 4$ );

In all samples, the computed parameters are slightly lower than those given by EFFENBERGER *et al.* (1981) for the synthetic calcite [ $a = 4.9896$  (2)  $\text{\AA}$ ,  $c = 17.0610$  (11)  $\text{\AA}$ ], indicating, probably, the presence of magnesium calcite in Peștera cu Lileci.

Table 4

Diphractometric data from two representative samples of calcite from Peștera cu Lileci din Satul Peștera

Crt. no.	Sample PPL 4 A			Sample PPL 9 A			(hkl)
	$d_{\text{meas.}}(\text{\AA})$	$d_{\text{calc.}}(\text{\AA})$	$I / I_0$	$d_{\text{meas.}}(\text{\AA})$	$d_{\text{calc.}}(\text{\AA})$	$I / I_0$	
1	3.8439	3.8434	5	3.8454	3.8468	12	(012)
2	3.0228	3.0270	100	3.0280	3.0295	100	(104)
3	2.8316	2.8362	1	2.8371	2.8382	2	(006)
4	2.4811	2.4872	10	2.4873	2.4895	17	(110)
5	2.2735	2.2778	11	2.2779	2.2799	22	(1.1.-3)
6	2.0857	2.0881	7	2.0871	2.0900	18	(202)
7	1.9216	1.9217	4	1.9218	1.9234	6	(024)
8	1.9051	1.9073	9	1.9076	1.9087	12	(018)
9	1.8682	1.8700	23	1.8697	1.8715	18	(1.1.-6)
10	1.6222	1.6208	3	1.6223	1.6223	11	(1.2.-1)
11	1.5998	1.5992	16	1.6004	1.6007	10	(122)
12	1.5812	1.5827	1	1.5828	1.5839	1	(1.0.10)
13	1.5216	1.5207	3	1.5223	1.5220	6	(1.2.-4)
14	1.5147	1.5135	3	1.5150	1.5147	2	(208)
15	1.5055	1.5052	2	1.5068	1.5064	2	(1.1.-9)
16	1.4698	1.4688	1	1.4704	1.4701	2	(2.1.-5)
17	1.4372	1.4360	8	1.4384	1.4373	6	(300)
18	1.4190	1.4181	2	1.4200	1.4191	2	(0.0.12)

\*Radiation Fe  $K\alpha$  filtered with Mn ( $\lambda = 1.93735 \text{ \AA}$ ),  $2\theta = 20-90^\circ$ . Number of refining cycles: 5; 3; 6; 4.

**Quartz** resulted from the decomposition reaction of illite under the phosphatic solutions from the guano deposit with the formation of taranakite. It appears as milky-white, crystalline aggregates, with filamentous extensions, situated nearby the taranakite veins at their edges. The parameters of the elemental cell, computed after four cycles of refining on a distinct sample analysed starting from 19 reflexes conferred to the quartz (Table 5), are:  $a = 4.912$  (3) Å,  $c = 5.381$  (6) Å,  $V = 112.4$  (1) Å<sup>3</sup>. They are closed to those computed by WILL *et al* (1988) for the stoichiometric  $\alpha$ -quartz [ $a = 4.91239$ (4) Å,  $c = 5.40385$ (7) Å and  $V = 112.933$  Å<sup>3</sup>], the parameter being practically identical.

Table 5

Diphractometric data from one representative sample of  $\alpha$ -quartz from Peștera cu Lilieci din satul Peștera

Crt. no.	Sample PPL 6 C			(hkl)
	$d_{\text{meas.}}$ (Å)	$d_{\text{calc.}}$ (Å)	$I / I_0$	
1	4.2480	4.2562	19	(100)
2	3.3388	3.3434	100	(101)
3	2.4567	2.4573	7	(110)
4	2.1264	2.1281	5	(200)
5	1.8171	1.8178	14	(1.1.-2)
6	1.6709	1.6717	3	(202)
7	1.6564	1.6586	7	(103)
8	1.6082	1.6087	4	(120)
9	1.5412	1.5418	12	(2.1.-1)
10	1.4536	1.4546	5	(1.1.-3)
11	1.3820	1.3822	5	(1.2.-2)
12	1.3740	1.3748	10	(023)
13	1.3740	1.3722	10	(301)
14	1.2890	1.2875	5	(014)
15	1.2299	1.2287	3	(220)
16	1.1999	1.1998	4	(2.1.-3)
17	1.1840	1.1837	7	(114)
18	1.1132	1.1184	4	(2.2.-2)
19	1.1132	1.1145	4	(033)

$$a = 4.915(2) \text{ \AA}$$

$$c = 5.402(2) \text{ \AA}$$

\*Radiation Cu  $K\alpha$  monochromatized ( $\lambda = 1.54056$  Å),  $2\theta = 20-87^\circ$ . Number of refining cycles: 3; 3; 3.



**Illite** is the dominant mineral in the mass of *terra rossa* clay from the base of the phosphatic deposit.

The recorded diffractograms, without a treatment with glycol, allowed us to identify the monoclinic polytype (2M1) of the mineral.

The parameters of the elemental cell, computed on a representative sample, are:  $a = 5.196$  (7) Å,  $b = 9.00$  (1) Å,  $c = 19.96$  (3) Å,  $\beta = 95.10$  (22)°.

**Birnessite** may be only guessed in the association of minerals near the guano deposit from Peștera cu Lilieci, Peștera village.

A phase, having as the main diffractometric reflexes the lines from 7.17 Å, 3.52 Å, 3.17 Å, 2.45 Å and 1.67 Å (in the decreasing order of the intensities) is residual in many diffractograms after the extraction of the lines belonging to other phases.

In the absence of other analytic methods, we can only guess that the birnessite represents the local recrystallization which also led to the forming of the iron sesquioxides jellies in the mass of *terra rossa* clay.

#### 4. CONCLUSIONS

In "Catalogul Sistematic al Peșterilor din România" (Systematic Catalogue of the Romanian Caves) (GORAN, 1982), Peștera cu Lilieci din satul Peștera or Peștera cu Lilieci, with its length of 162 m, seems a small cave but, however, is only on the 6th place among 350 caves inventoried up to the present in Bran-Rucăr Passage.

The mineralogical analyses made on the samples collected from this cave provide new scientific data not only about this cave, already known after all, but also on the entire endokarst from this region.

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