Cyrilovite from Cyrilov, western Moravia, Czech Republic; new data on the type material

Cyrilovit z Cyrilova, západní Morava; nová data z typového materiálu (Czech summary)

(3 text-figs, 4 tabs)

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The type material of cyrilovite from Cyrilov was studied by the means of electron microprobe, X-ray powder diffraction and IR spectroscopy. Cyrilovite is close to the end-member composition $NaFe^{3+}_3 (OH)_4 (PO_4)_2 . 2 H_2O$ with minor concentrations of Al (0.00–0.10 *apfu*) and Ca (0.01–0.05 *apfu*), insignificant substitutions $Fe^{3+} \rightarrow Al$ and $CaFe^{2+} \rightarrow NaFe^{3+}$ are indicated. The lattice parameters – $a = 7.3227(3)$, $c = 19.332(1)$ Å, $V = 1036.61(8)$ Å^3 and density $D_{calc} = 3.112$ g.cm^{-3} of cyrilovite calculated from powder data are very close to those obtained from the single crystal refinement – $a = 7.3255(4)$, $c = 19.328(2)$ Å, $V = 1037.2(2)$ Å^3 and $D_{calc} = 3.106$ g.cm^{-3}, respectively, (Cooper et al. 2000) and to cyrilovite from other localities. The infrared absorption spectrum is very close to that of cyrilovite from Cyrilov published by Tarte et al. (1984). Two structurally nonequivalent water molecules and hydroxyls may be present in the crystal structure of cyrilovite. The number of (PO_4) vibrations may also indicate that two structurally nonequivalent (PO_4) groups are present in the crystal structure but not detected by the crystal structure analysis, possibly indicating lower symmetry.

Key words: cyrilovite, electron microprobe, X-ray powder study, IR spectroscopy, Cyrilov, Moravia.

Introduction

Cyrilovite $NaFe^{3+}_3 (OH)_4 (PO_4)_2 . 2 H_2O$ was described as a new mineral species from phosphate-bearing pegmatite at Cyrilov, western Moravia by Novotný – Staněk (1953); the authors, however, did not provide a correct chemical formula. It was recognized by Lindberg – Pecora (1954) for avelinoite, the mineral species identical with cyrilovite (Strunz 1956), which was found in pegmatite from Sapucaia, Minas Gerais, Brazil. Some new data about cyrilovite from Cyrilov also were presented by Strunz (1956), Lindberg (1957), Staněk (1971) and Tarte et al. (1984); crystal structure of cyrilovite from Cyrilov was refined only recently by Cooper et al. (2000). New data on the cyrilovite type material are presented in this paper.

Cyrilovite is known only from several localities in granitic pegmatites e. g., Sapucaia and Boa Vista, Minas Gerais, Brazil; Vermilhas, Portugal; Hagendorf, Bavaria, Germany (Lindberg – Pecora 1954, Cassedanne – Cassedanne 1978, Coreia Neves 1966, Strunz et al. 1976), and in oxidation zone of some ore deposits e. g., Kokpatas, Uzbekistan; Iron Monarch, Australia; Rochefort-en-Terre, Arz Valley, Morbihan, France; Bosa, Sardinia, Italy (Kasymov 1966, Pilkington et al. 1979, Fontan et al. 1981, Cozzupoli et al. 1987). Cyrilovite originated by low-temperature hydrothermal alteration of primary Fe, Mn-phosphates in granitic pegmatites, or from low temperature aqueous solutions in oxidation zone of ore deposits. It is associated with Fe-hydroxides (e. g., goethite) and secondary phosphates (e. g., rockbridgeite-frondelite, apatite, dufrenite, wavellite). High activity of Na and highly oxidized conditions are required in both environments (Cooper et al. 2000).

Locality and paragenesis

The phosphate-bearing pegmatite from Cyrilov is located in the Bory pegmatite district, western Moravia, Czech Republic (Novák et al. 1992). It comprises: (i) numerous, less evolved, symmetrically zoned pegmatite dikes with abundant schorl, biotite, muscovite, andalusite and sekaninaite (chiefly located at the Hatě district in the vicinity of Dolní Bory), (ii) more evolved phosphate-bearing pegmatites (Cyrilov, Dolní Bory - dike Oldřich, Horní Bory, Víděň and Rousměrov) and (iii) the most evolved lepidolite-subtype pegmatites (Dobrá Voda, Dolní Bory - dike No. 21, Laštovičky).

The Cyrilov pegmatite was mined for feldspars and quartz at the end of 19th century. Its internal structure may be inferred only by analogy with other pegmatite dikes in the Bory district and from fragments found in old dumps. The pegmatite dike, about 8 m thick and about 200 m long, penetrates migmatites and granulites. It is very likely symmetrically zoned consisting inwards of granitic unit, graphite unit, blocky K-feldspar zone, quartz core and albite-rich unit. Typical subordinate minerals represent muscovite, biotite, schorl and primary Fe, Mn-phosphates; commonly rare accessory minerals include apatite, andalusite, garnet, ferrocolumbite, wolframite and “wolframoxiolite”.

Primary Fe, Mn-phosphates (zwieselite, graftonite, triphylite; Čech et al. 1964) were hydrothermally altered and replaced by secondary phosphate-minerals: het-

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erosite, frondelite, lipscombite, mitridatite, phosphosiderite, strengite, leucophosphite and cyrilovite (Staněk 1971). The latter occurs as one of the latest minerals of this sequence in small open vugs originated by hydrothermal dissolution of primary phosphates. Cyrilovite is commonly associated with frondelite, mitridatite, phosphosiderite, strengite and abundant undetermined Fe, Mn-oxides and hydroxides.

Experimental

X-ray powder diffraction pattern of cyrilovite was obtained from the hand-picked sample using the diffractometer Philips X'pert System in the range 3–73° 2θ Cu*K*α (step scanning 0.03°/20 s, graphite secondary monochomator). To minimaze a complicated shape of background due to classic glass sample holder, the studied sample was placed on the surface of flat silicon wafer from alcoholic suspension. Position and intensities of reflections were calculated using Pearson VII profil shape function by ZDS program package (Ondruš 1995).

Several cyrilovite crystals were analyzed by electron microprobe using a CAMECA SX-50 operating in the wavelength-dispersion mode at the University of Manitoba, Winnipeg. Beam voltage for all elements was 15 kV and a spot diameter of 10 mm. The beam current was 20 nA, counting times of peak and background determinations for all elements were 20 s. The following standards were used for *K*α X-ray lines: Na, P, Fe – maricite, Al – kyanite, Ca – diopside. In addition Mg, K, Ti and Mn were sought but not detected. The analytical data were reduced and corrected using the f(rZ) method (Pouchou – Pichoir 1985).

The infrared absorption spectrum of cyrilovite (mineral phase dispersed in KBr disk) was measured with the FTIR Nicolet 740 spectrophotometer type apparatus in the region 4000–400 cm^{-1}.

The SEM photographs were performed on the electron scanning microscope TESLA BS 320.

The type material (holotype) of cyrilovite with the number A 6107 deposited in the collection of the Department of Mineralogy and Petrography, Moravian Museum, Brno was used for the laboratory study.

Cyrilovite

Description

Cyrilovite forms fine crystalline crusts commonly on frondelite consisting of small tabular crystals. Two rather different morphological types of crystals both closely associated were recognized on hand specimen: (i) abundant, perfectly developed, yellow crystals, commonly less than 0.1 mm in size with a strong vitreous luster; (ii) sporadical, less perfectly developed, brown crystals, up to 1 mm in size, but commonly about 0.2 mm, with less intensive vitreous luster. These large tabular crystals are parallel to (001) with dominant dipyramidal planes (114) and commonly subordinate (001) and (021) planes; other observed forms – (110), (113) and (5.5.12) (Strunz 1956) are commonly developed as narrow facets (Fig. 1).

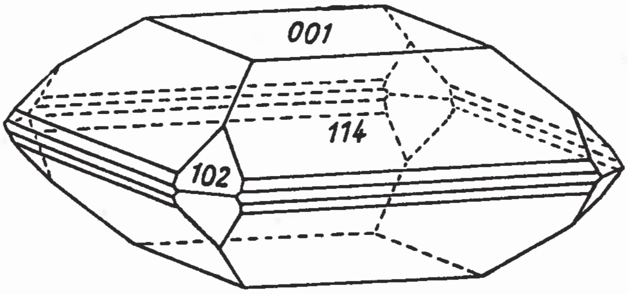

Fig. 1. Crystal of cyrilovite from Cyrilov (Strunz 1956).

The SEM study confirmed two distinct morphological types of the cyrilovite crystals: (i) tabular to short columnar crystals with dominant dipyramidal and prismatic planes and basis, and apparently better development of crystal planes (Fig. 2a), corresponding to small yellow crystals; (ii) tabular crystals with dominant dipyramidal planes, minor basis and locally with sporadical prismatic planes (Fig. 2b). Morphology of large tabular cyrilovite crystals with dominant dipyramidal planes is very similar to that in some granitic pegmatites (Lindberg – Pecora 1954, Strunz et al. 1976) and at the local-

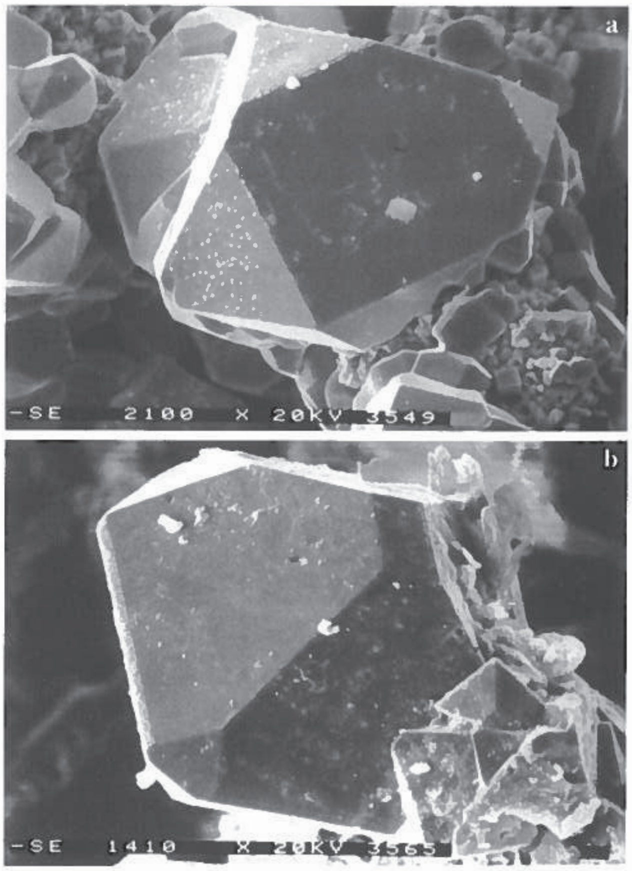

Fig. 2. SEM photographs of cyrilovite from Cyrilov.
a) short columnar yellow crystal with dominant prismatic planes, dipyramidal planes and basis. Scale bar 260 μm
b) tabular brown crystals with dominant dipyramidal planes and minor basis. Scale bar 380 μm.

Table 1. X-ray powder diffraction pattern of cyrilovite from Cyrilov

h	k	l	d_{obs}	I/I_o	d_{calc}	h	k	l	d_{obs}	I/I_o	d_{calc}
			7.74	40b	*	3	2	3	1.9373	13	1.9370
			7.23	32b	*	2	2	7	1.8888	4	1.8888
			5.97	4	*	3	2	4	1.8724	3	1.8724
1	0	2	5.84	32	5.84	1	0	10	1.8690	11	1.8691
1	1	0	5.18	32	5.18	2	0	9	1.8524	26	1.8527
1	0	3	4.839	96	4.838	4	0	0	1.8307	57	1.8307
0	0	4			4.833	4	0	1	1.8227	20	1.8225
1	1	2	4.567	13	4.564	2	1	9	1.7959	9	1.7961
1	1	3	4.038	28	4.036	4	1	1	1.7688	5	1.7686
1	0	4			4.034	4	0	3	1.7609	8	1.7610
2	0	0	3.663	9	3.661	4	1	2	1.7468	7	1.7468
2	0	1	3.600	66	3.597	3	3	0	1.7257	14	1.7260
1	1	4	3.537	1	3.533	3	3	1	1.7194	17	1.7192
			3.455	7	*	3	2	6	1.7179	14	1.7183
			3.401	11	*	3	0	8			1.7173
			3.326	11	*	4	0	4	1.7119	51	1.7120
2	1	0	3.273	10	3.275	2	1	10	1.6649	21	1.6648
2	1	1	3.230	62	3.229	1	1	11			1.6642
2	0	3	3.185	100	3.183	4	0	5	1.6545	13	1.6546
2	1	2	3.102	73	3.102	3	2	7	1.6364	11	1.6362
1	1	5			3.098	4	2	1	1.6316	22	1.6316
1	0	6	2.950	14	2.949	4	2	2	1.6143	13	1.6144
2	1	3	2.920	34	2.919	4	1	5			1.6139
2	0	4			2.918	0	0	12	1.6107	30	1.6110
1	1	6	2.736	4	2.736	4	2	3	1.5870	43	1.5870
2	1	4	2.711	7	2.711	4	1	6	1.5550	23	1.5554
2	0	5	2.659	88	2.658	3	2	8			1.5548
1	0	7	2.584	11	2.584	2	2	10	1.5487	57	1.5490
2	2	1	2.567	9	2.566	4	2	5	1.5078	31	1.5078
2	2	2	2.501	48	2.501	3	1	10	1.4839	7	1.4840
2	1	5			2.499	4	2	6	1.4593	29	1.4597
2	0	6	2.419	13	2.419	4	0	8			1.4592
0	0	8			2.416	5	0	2	1.4482	18	1.4480
2	2	3	2.402	3	2.402	2	1	12	1.4451	11	1.4455
1	0	8	2.295	36	2.295	5	1	0	1.4361	9	1.4361
3	0	3	2.282	8	2.283	4	1	8	1.4313	10	1.4311
2	2	4			2.282	4	3	3	1.4280	12	1.4281
3	1	2	2.252	11	2.252	4	2	7	1.4084	16	1.4085
2	0	7	2.205	50	2.205	5	1	3	1.4017	13	1.4017
3	1	3	2.179	48	2.179	3	2	10	1.4001	5	1.4003
3	0	4			2.179	2	0	13	1.3774	9	1.3778
2	2	5	2.154	16	2.151	4	3	5	1.3690	2	1.3696
2	1	7	2.111	14	2.111	2	1	13	1.3537	14	1.3540
3	1	4	2.088	3	2.088	5	2	2	1.3464	16	1.3465
3	0	5	2.064	14	2.064	3	2	11	1.3288	5	1.3290
1	0	9	2.060	9	2.061	4	2	9	1.3021	9	1.3022
2	2	6	2.018	60	2.018	4	4	0	1.2943	21	1.2945
3	2	2	1.9868	34	1.9876	4	4	1	1.2918	17	1.2916
3	1	5			1.9866						

Diffractometer Philips X'pert System. step scanning 0.03°/20 s (3–73° 2θ Cu*K*α).

Table 2. Physical properties of cyrilovite.

locality	Cyrilov			Sapucaia	Bosa
source	a	b	c	d	e
a Å	7.32	7.3227	7.3255	7.334	7.313
c Å	19.4	19.332	19.328	19.368	19.315
V Å^3	1039.5	1036.61	1037.2	1041.8	1033.0
$D_{meas.}$ g.cm^{-3}	3.085			3.08	3.096
$D_{calc.}$ g.cm^{-3}		3.112	3.106	3.09	3.114
ε	1.777			1.769	1.775
ω	1.805			1.803	1.802

a) Novotný – Staněk (1953); b) this work; c) Cooper et al. (2000); d) Lindberg – Pecora (1954), Lindberg (1957); e) Cozzupoli et al. (1987).

ity Bosa, Sardinia, Italy (Cozzupoli et al. 1987); cyrilovite from oxidation zone of ore deposits commonly forms spherulitic aggregates (Kasymov 1966, Pilkington et al. 1979, Fontan et al. 1981).

Physical and optical properties

Cyrilovite is pale yellow to pale green in thin sections, commonly transparent, nonpleochroic and uniaxial negative, ω = 1.805, ε = 1.777 (Novotný – Staněk 1953); however, we typically found biaxial crystals with small 2V. No difference was observed in optical properties between both brown and yellow crystals. It seems very likely that the different color on hand specimen results from the size of crystals. Streak of cyrilovite is pale yellow, hardness – about 4, cleavage none and fracture conchoidal.

X-ray powder diffraction data

The powder diffraction pattern of cyrilovite (Table 1) agrees very well with the data calculated (Lazy Pulverix, Yvon et al. 1977) from the crystal structure of this mineral (Cooper et al. 2000), which is used for indexing of measured reflexions. A certain problem is presence of six reflections (in Table 1 marked *), which were not possible to index. They probably belong to admixture of other mineral phase (not identified in the ICDD 1–47 set); however, no other phase was found by electron microprobe study.

The lattice parameters of cyrilovite – *a* = 7.3227(3), *c* = 19.332(1) Å, *V* = 1036.61(8) Å^3 - calculated by the least squares refinement program compiled by Burnham (1962) agree very well with results of the single crystal refinement – *a* = 7.3255(4), *c* = 19.328(2) Å, *V* = 1037.2(2) Å^3 (Cooper et al. 2000). The calculated density for cyrilovite based on the empirical formula with Z = 4 D_{calc} = 3.112 g.cm^{-3} is close to the measured D_{meas} = 3.085 g.cm^{-3} (Novotný – Staněk 1953) and to the data from the single crystal study D_{calc} = 3.106 g.cm^{-3} (Cooper et al. 2000). Review of physical properties of near-end-member cyrilovite from several localities is given in Table 2.

Chemical composition

Electron microprobe study did not find any apparent difference in chemical composition of both morphological types of cyrilovite. It is homogeneous, commonly very close to the end-member composition (Table 3). Large brown crystals are locally slightly enriched in Ca and Al relative to the small ones. Minor concentrations of Al (0.00–0.10 *apfu*) and Ca (0.01–0.05 *apfu*) indicate insignificant substitution $Fe^{3+} \rightarrow Al$ and $CaFe^{2+} \rightarrow NaFe^{3+}$ (Cooper et al. 2000). Comparing the published chemical analyses (Lindberg 1957, Tarte et al. 1984, Cozzupoli et al. 1987), all cyrilovite analyses are very close to the end-member composition with up to ~ 5 vol. % of the wardite component in the single analyzed spots except the locality Arz Valley, Morbihan, France (Fontan et al. 1981), where aluminian cyrilovite with 23 vol. % of wardite component was found.

Table 3. Chemical composition of cyrilovite from Cyrilov.

	1	2	1a	2a	2b	3	4
P_2O_5	28.67(0.22)	28.92(0.27)	28.91	29.28	29.40	33.96	29.96
Al_2O_3	0.11(0.05)	0.28(0.44)	0.12	1.06	0.02	n. d.	0.00
Fe_2O_3	48.60(0.35)	48.55(0.59)	49.29	47.64	49.35	50.89	47.90
CaO	0.25(0.10)	0.41(0.06)	0.08	0.37	0.53	n. d.	0.58
Na_2O	6.24(0.11)	6.10(0.06)	6.46	6.13	6.15	n. d.	6.66
H_2O *	14.64	14.72	14.80	14.87	14.93	15.14	14.94
total	98.51	98.85	99.66	99.35	100.38	99.99	100.04
P	1.989	1.993	1.984	2.000	1.999		2.036
Al	0.011	0.027	0.011	0.101	0.002		–
Fe^{3+}	2.996	2.974	3.006	2.892	2.983		2.894
Ca	0.022	0.036	0.007	0.032	0.046		0.050
Na	0.991	0.963	1.015	0.959	0.958		1.037
OH	4.000	4.000	4.000	4.000	4.000		4.000
H_2O	2.000	2.000	2.000	2.000	2.000		2.000
catsum	6.009	5.992	6.023	5.983	5.988		6.017

* H_2O calculated from stoichiometry, OH = 4, H_2O = 2; n. d. – not determined.

1 – yellow crystals (8 spots); 2 – brown crystals (8 spots); 1a – lowest CaO; 2a – highest Al_2O_3, 2b – highest CaO; 3 – original cyrilovite analysis (Novotný – Staněk 1953); Tarte et al. (1984).

Infrared absorption spectrum

The infrared absorption spectrum of the studied mineral (Fig. 3) is very close to the spectrum published for cyrilovite from Cyrilov by Tarte et al. (1984); infrared spectrum of cyrilovite from Bosa, Sardinia (Cozzupoli et al. 1987) is considerably more complex, but also very similar.

The presence of absorption bands characteristic for vibrations of (PO_4) groups, water molecules and hydroxyls was proved, which is consistent with the chemical composition of cyrilovite. The tentative assignment of the absorption bands observed (Table 4) is based on detailed studies of the vibrations of these groups (e. g., Farmer 1974, Liu et al. 1998, Sejkora – Čejka 1999).

Sharp bands at 3544 and 3486 cm^{-1} were assigned to the stretching vibrations n (OH) of relatively free independent hydroxyls, i. e. of hydroxyls bonded with very weak H bonds, or, more probably not participating in the H-bonding network. Bands and shoulders in the region 3450–2847 cm^{-1} (with relatively strong shoulders at 3420 and 3246 cm^{-1}) are related to the stretching vibrations n O-H of hydrogen bonded water molecules and other hydroxyls. However, it is difficult to infer from the character of the infrared spectrum of cyrilovite how much of these groups participate in H-bonded network. Two absorption bands at 1634 and 1616 cm^{-1} were assigned to the bending vibrations d H_2O. This indicates that two structurally nonequivalent water molecules and hydroxyls may be present in the crystal structure of cyrilovite.

Absorption bands at 1160, 1122, 1111, 1061, 1045, 1020 and 999 cm^{-1} were assigned to the triply degenerate antisymmetric stretching vibration n_3 (PO_4). Absorption bands at 959 and 948 cm^{-1} were assigned to the symmetric stretching vibration n_1 (PO_4), which became infrared active. Absorption bands at 636, 614, 596 and 571 cm^{-1} may be connected with the triply degenerate bending vibration n_4 (PO_4) and those at 494 and 451 cm^{-1} to the infrared activated doubly degenerate bending vibration n_2 (PO_4).

Infrared - activation of n_1 (PO_4) and n_2 (PO_4) and the splitting of doubly degenerate n_2 (PO_4) and triply degenerate n_3 and n_4 (PO_4) indicate the T_d symmetry lowering of the (PO_4) tetrahedra in the structure. The number of (PO_4) vibrations may also indicate that two structurally

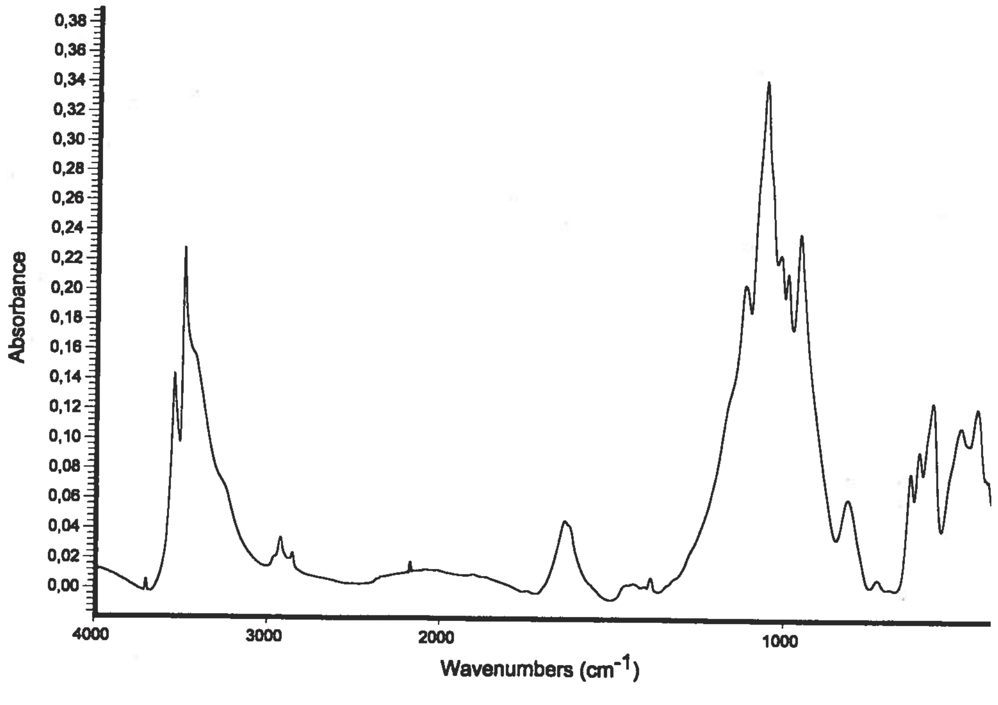

Fig. 3. The infrared absorption spectrum of cyrilovite from Cyrilov.

Table 4. Infrared spectra of cyrilovite from Cyrilov.

Cyrilov (this paper) [cm^{-1}]			*1 [cm^{-1}]	*2 [cm^{-1}]	Tentative assignment
3544	m	sr	3550	3640	stretching n O-H in (OH) groups
3486	s	sr	3495	3485	
3420	m	sh		3400	stretching n O-H in H_2O molecules
3246	mw	sh	3260	3260	
2971	vw	sh		2960	
2930	w		2950	2920	n O-H or overtones/combination bands
2847	w			2855	
1634	m		1630	1632	bending d H_2O
1616	m	sh	1550	1600	
1160	mw	sh			
1122	s		1117	1119	
1111	s	sh	1106	1108	
1061	vs		1060	1061	triply degenerate antisymmetric
1045	s	sh		1044	stretching vibration n_3 $(PO_4)^{3-}$
1020	ms	sh	1022	1016	
999	ms	sr	996	996	
959	sr	sr	961	960	symmetric stretching n_1 $(PO_4)^{3-}$
948	m	sh			
817	mw		820	815	libration modes H_2O or
730	vw		728	725	vibration of M-O bonds
636	mw	sr	635	636	
614	mw	sr	610	607	triply degenerate bending
596	mw	sh	585	590	vibration n_4 $(PO_4)^{3-}$
571	m		574	573	
494	mw		502	492	doubly degenerate
451	mw			450	bending vibration n_2 $(PO_4)^{3-}$

FTIR spectrophotometer Nicolet 740, KBr tablet. *1 cyrilovite, Cyrilov (Tarte et al. 1984), 2* cyrilovite, Bosa, Sardinia (Cozzupoli et al. 1987), some weak bands have been ommited.
Intensity and character of absorption bands: vs – very strong, s – strong, ms – medium strong, m – medium, mw – medium weak, w – weak, vw – very weak; sh – shoulder, sr – sharp.

nonequivalent (PO_4) groups are present in the crystal structure of cyrilovite. These two groups were not detected in the present crystal structure analysis of cyrilovite but their presence may indicate lower symmetry that is also in agreement with the observed biaxial optics.

Absorption bands near 817 and 730 cm^{-1} may be assigned to molecular water liberation modes or vibrations of M-O bonds.

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Cyrilovit z Cyrilova, západní Morava; nová data z typového materiálu

Typový materiál cyrilovitu z Cyrilova byl studován pomocí elektronového mikroanalyzátoru, práškové rentgenové difrakce a IČ spektroskopie. Byly zjištěny dva morfologicky poněkud odlišné typy, které se liší barvou a tvarem a velikostí krystalů. Chemické složení cyrilovitu obou typů se blíží koncovému členu $NaFe^{3+}_3 (OH)_4 (PO_4)_2 . 2 H_2O$, nízké koncentrace Al (0,00–0,10 *apfu*) a Ca (0,01–0,05 *apfu*) naznačují nevýrazné substituce $Fe^{3+} \rightarrow Al$ a $CaFe^{2+} \rightarrow NaFe^{3+}$. Mřížkové parametry - $a = 7.3227(3)$, $c = 19.332(1)$ Å, $V = 1036.61(8)$ Å^3 a hustota $D_{calc} = 3.112$ g.cm^{-3} počítaná z práškových dat jsou velmi blízké datům získaným ze studia monokrystalu - $a = 7.3255(4)$, $c = 19.328(2)$ Å, $V = 1037.2(2)$ Å^3 a $D_{calc} = 3.106$ g.cm^{-3}, (Cooper et al. 2000) a také cyrilovitu z jiných lokalit. IČ spektrum studovaného cyrilovitu je velmi podobné cyrilovitu z Cyrilova, studovanému Tartem et al. (1984). Dvě strukturně neekvivalentní molekuly H_2O a skupiny OH mohou být přítomny ve struktuře cyrilovitu. Počet (PO_4) vibrací může naznačovat přítomnost dvou strukturně neekvivalentních (PO_4) skupin, které nebyly zjištěny strukturní analýzou. Mohou být vysvětleny nižší symetrií cyrilovitu indikovanou také opticky.