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PARAGENESIS AND ORIGIN OF SECONDARY BERYLLOPHOSPHATES: BERYLLONITE AND HYDROXYLHERDERITE FROM THE BEP GRANITIC PEGMATITE, SOUTHEASTERN MANITOBA, CANADA

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ABSTRACT

Rare occurrences of phosphates with beryllium are limited to granitic pegmatites and, exceptionally, to rare-element granites. Primary crystallization is extremely scarce; most of these phosphates form as late subsolidus phases. Paragenetic relationships of these minerals and conditions of their origin are poorly understood. Beryllonite, hydroxylherderite and carbonate-fluorapatite form thin coatings in fissures of beryl in the Archean petalite-subtype BEP pegmatite in southeastern Manitoba. The composition of beryllonite corresponds to its ideal formula, and hydroxylherderite contains only traces of fluorine. The B-type carbonate-fluorapatite shows considerably reduced P_2O_5 content, "excess" fluorine, and an apparently high CO_2 content (estimated at ~5 to ~7 wt.%). Beryllonite was the first phosphate to precipitate, followed and in part replaced by hydroxylherderite; carbonate-fluorapatite rims, penetrates and replaces both preceding phases. Low a(F) is indicated during the formation of beryllonite and hydroxylherderite, but it must have increased to stabilize carbonate-fluorapatite. The process probably involved F released during the $(OH)F_{-1}$ exchange in the associated montebrasite.

Keywords: beryllonite, hydroxylherderite, carbonate-fluorapatite, beryl, alteration, BEP granitic pegmatite, Manitoba, Canada.

SOMMAIRE

Les exemples de phosphates contenant le béryllium sont rares, et limités aux pegmatites granitiques et, de façon exceptionnelle, aux granites enrichis en éléments rares. Une cristallisation primaire semble tout-à-fait inhabituelle. Au contraire, la plupart de ces phosphates se forment tardivement, au dessous du solidus. Les relations paragénétiques de ces minéraux et les conditions de leur origine ne sont pas bien documentées. Béryllonite, hydroxylherdérite et carbonate-fluorapatite se présentent en liserés étroits le long de fissures dans le béryl de la pegmatite granitique de BEP, de sous-type à pétalite et d'âge archéen, dans le sud-est du Manitoba. La composition de la béryllonite correspond à sa formule idéale, et la hydroxylherdérite contient des traces de fluor seulement. La carbonate-fluorapatite, de type B, fait preuve d'une teneur en P_2O_5 considérablement réduite, un "excédent" en fluor, et une teneur en CO_2 qui semble élevée (entre environ 5 et 7% en poids). La béryllonite, formée d'abord, a ensuite été partiellement remplacée par la hydroxylherdérite; une bordure de carbonate-fluorapatite recoupe et remplace les deux phases déjà formées. Une faible valeur de a(F) est indiquée au cours de la formation de la béryllonite et de l'hydroxylherdérite, mais ce paramètre a dû passer à une valeur plus élevée quand la carbonate-fluorapatite est apparue. Le processus implique probablement le fluor libéré pendant l'échange $(OH)F_{-1}$ dans la montebrasite associée.

(Traduit par la Rédaction)

Mots-clés: béryllonite, hydroxylherdérite, carbonate-fluorapatite, béryl, altération, pegmatite granitique de BEP, Manitoba, Canada.

INTRODUCTION

Beryllium-bearing phosphates are very rare constituents of granitic pegmatites (and related granites), restricted to the moderately to highly fractionated members of the peraluminous LCT family (*cf.* Černý 1991 for classification criteria). These phosphates only rarely form as primary phases associated with rock-forming feldspars and quartz, or in albite-dominated units. Most of the occurrences of Be-phosphates are related to late stages of consolidation of the parent pegmatite bodies, either as replacements of early Be-bearing

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phases such as beryl, or in miarolitic cavities. Many of the minerals concerned (phosphates of beryllium and beryllophosphates of a great variety of cations) are extremely rare, known only from their type localities. To date, the overall number of localities seems also to be quite restricted (*cf.* Černý 2002).

The early literature contains numerous physical and structural data, and crystal-chemical relationships are largely well understood (Hawthorne & Huminicki 2000). However, detailed observations and interpretation of paragenetic relationships such as those of Charoy (1999) or Nysten & Gustafsson (1993) are very scarce. Thus any occurrence of Be-bearing phosphates deserves attention in this respect. We report here on an assemblage of beryllophosphates from southeastern Manitoba, and on the probable conditions of its evolution.

THE PARENT PEGMATITE

The BEP granitic pegmatite is located in the Bird River Greenstone Belt of the Bird River Subprovince of the Archean Superior Province of the Canadian Shield. The pegmatite is a member of the Bernic Lake pegmatite group, which comprises the most highly evolved pegmatite population in the Winnipeg River – Cat Lake pegmatite field (Černý *et al.* 1981). The BEP pegmatite, an irregular subvertical dike with numerous xenoliths of brecciated wallrock amphibolite, is closely associated with the underlying BCSE pegmatite, a subhorizontal dike apparently subparallel to the shallow-dipping Buck, Pegli and Tanco pegmatites to the north (Fig. 80 in Černý *et al.* 1981, Figs. 1 to 3 in Černý & Lenton 1995).

The BEP pegmatite, not exceeding 1.5 m in thickness, contains a fine- to medium-grained assemblage of albite + quartz + magnesian schorl (+ biotite + fluorapatite + muscovite + beryl) along the contacts with amphibolite wallrock and xenoliths, which are replaced by magnesian biotite and rare holmquistite to a depth of a few millimeters. The interior of the pegmatite consists of quartz, blocky amblygonite–montebrasite (4.6 to 6.6 wt.% F), coarse crystals of beryl, minor K-feldspar, and locally also a pseudomorphic intergrowth of spodumene + quartz after petalite (also known as squi; *e.g.*, Černý *et al.* 1998). Accessory minerals are represented by manganoan fluorapatite, disordered manganocolumbite, cassiterite and altered triphylite–lithiophilite. Beryl, fluorapatite, cassiterite and manganocolumbite are particularly associated with "cleavelandite" in marginal parts of the centrally located pods of quartz core.

Products of hydrothermal alteration include a white mica, secondary montebrasite (0.8 to 1.5 wt.% F) developed at the expense of the primary blocky amblygonite–montebrasite, fluorapatite rimming and penetrating the amblygonite–montebrasite, and the assemblage dominated by the beryllophosphates. This assemblage forms a thin, pink to rusty film not exceeding 0.5 mm in thickness, in fissures of large crystals of beryl.

EXPERIMENTAL METHODS

The beryllophosphates were analyzed with a Cameca SX–50 electron microprobe operating in the wavelength-dispersion mode, with an accelerating voltage of 15 kV, a specimen current of 20 nA, and a beam diameter of 10 μm. The following standards were used: albite (Na$K\alpha$), andalusite (Al$K\alpha$), fluorapatite (Ca$K\alpha$, P$K\alpha$, F$K\alpha$), olivine (Mg$K\alpha$), spessartine (Mn$K\alpha$), fayalite (Fe$K\alpha$), $SrTiO_3$ (Sr$L\alpha$), and tugtupite (Cl). Beryl was analyzed with an accelerating voltage of 15 kV, a specimen current of 20 nA, a beam diameter of 5 μm, and the following standards in addition to, and instead of, those quoted above: quartz (Si$K\alpha$), Eifel sanidine (K$K\alpha$), andalusite (Al$K\alpha$), anorthite (Ca$K\alpha$), synthetic Rb analogue of leucite (Rb$L\alpha$) and pollucite (Cs$L\alpha$). The data were reduced and corrected by the *PAP* method of Pouchou & Pichoir (1984, 1985). Beryllonite and carbonate-fluorapatite gave satisfactory results, but hydroxylherderite vaporized under the electron beam; however, the results still led to an unambiguous herderite-type stoichiometry.

X-ray powder-diffraction data were collected by standard methods using a Philips PW 1710 instrument, in some cases with CaF_2 [a = 5.465397(4) Å] as an internal standard. Unit-cell dimensions of hydroxylherderite were refined from powder-diffraction data using the modified least-squares CELREF program of Appleman & Evans (1973). The indices of refraction were measured using the Becke line method in sodium light (λ = 589 nm) with an accuracy estimated at ±0.002.

MINERALOGY

Beryl

Short columnar to stumpy crystals of milky, greyish or pale greenish beryl attain up to 45 × 25 cm in size. They are commonly slightly fractured and displaced, healed by veinlets of quartz and albite. Thin fissures subparallel to {0001} show lustrous overgrowths of shallow hillocks, defined mainly by extensive basal pinacoid faces flanked by stepped slopes of alternating basal and pyramidal facets (Fig. 1). Electron-microprobe and atomic-absorption spectroscopic analyses of massive beryl indicate moderate enrichment in alkali metals and minor contents of other components (in wt.%): Li_2O 0.34 to 0.48, Na_2O 0.47 to 1.85, K_2O 0 to 0.02, Rb_2O 0.02 to 0.11, Cs_2O 0.23 to 1.02, CaO 0.01 to 0.19, MnO 0 to 0.02, total Fe as FeO 0 to 0.11, MgO below the detection limit. Electron-microprobe analysis of the beryl in the hillocks shows only a moderate enrichment in some of the alkali metals, up to 1.95 wt.% Na_2O and 1.38 wt.% Cs_2O.

Beryllonite

Beryllonite (ideal composition: $NaBePO_4$) is a subordinate component of the phosphate assemblage under study. It forms subhedral grains with rectangular outline, up to 0.5 × 0.3 mm in size (Fig. 2A), but it is mostly strongly corroded and replaced by hydroxylherderite and carbonate-fluorapatite (Fig. 2B)**.** Beryllonite proved to be stable under the analytical conditions employed. Coupled with a theoretical Be content, the data yielded satisfactory empirical formulas with negligible contents of non-essential elements (Table 1). The identity of the mineral was confirmed by the presence of up to eight of its strongest X-ray powder-diffraction peaks in hydroxylherderite-dominant samples (*cf.* Mrose 1952, Lahti 1981), and by its optical properties: α 1.553, β 1.558, γ 1.560, $\gamma - \alpha$ 0.017, and medium to large (+) 2*V* (*cf.* Winchell & Winchell 1951, Lahti 1981).

Hydroxylherderite

Hydroxylherderite [ideal composition: $CaBe(PO_4)(OH)$] is the most abundant mineral of the assemblage examined, in the form of lath-shaped crystals up to 0.5 × 0.1 mm in size (Fig. 3A), but largely as fine-grained aggregates (Fig. 2B). Colloform textures also are locally observed (Fig. 3B). Hydroxylherderite became unstable under the electron beam, and the analytical wt.% totals were invariably low by 3 to as much as 5%. Nevertheless, the results of the analyses, coupled with an assumed amount of Be equivalent to 1 *apfu* and (OH + F) = 1 *apfu*, gave satisfactory empirical formulas. The proportion of F is negligible, at or below the detection limit considering the analytical conditions employed. The same results were obtained with a higher voltage (30 kV) and longer counting time (30 s); attempts to apply these conditions resulted mainly in faster breakdown of the mineral. The extent of substitution of Na, Mg, Sr, Fe and Mn for Ca is negligible, as is the content of Al (Table 1). Thus the BEP mineral has evidently the composition of a virtually pure end-member hydroxylherderite, with no perceptible solid-solution toward either väyrynenite (manganoan end-member) or bergslagite (arsenate analogue).

The unit-cell dimensions *a* 9.787(4), *b* 7.662(3), *c* 4.803(2) Å, β 90.05(5)° closely correspond to those quoted in the literature as virtually constant for the herderite series, not influenced by the (F,OH) substitution (Lager & Gibbs 1974, Leavens *et al.* 1978). How-

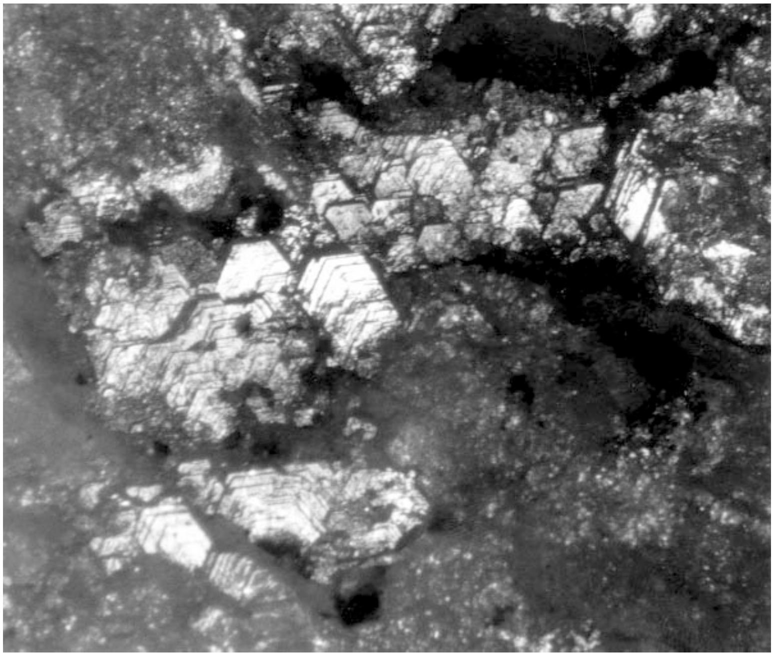

FIG. 1. Lustrous hexagonal growth hillocks on the basal parting plane of beryl, in part etched dull (mottled grey) and in part coated by a layer of beryllonite + hydroxylherderite + carbonate-fluorapatite (black); the shorter edge of the photograph is 4 mm long.

ever, optical properties do vary with F/(OH): our sample of hydroxylherderite shows α 1.614, β 1.634, γ 1.643, $\gamma - \alpha$ 0.029, and large (–) $2V$. These data match those of end-member hydroxylherderite, as established by Leavens *et al.* (1978).

Carbonate-fluorapatite

Carbonate-fluorapatite constitutes extremely fine-grained aggregates that have replaced both Be-phosphates, but mainly the hydroxylherderite (Figs. 2A, B, 3A). Carbonate-fluorapatite also fills interstices in colloform, botryoidal hydroxylherderite (Fig. 3B). The electron-microprobe analysis of carbonate-fluorapatite is not only inevitably incomplete, but the components that cannot be monitored (CO_2, H_2O), and the variability of substitution mechanisms involving them, are such that a meaningful calculation of empirical formulas is impossible. However, the data strongly suggest a Cl-poor, Si- and S-free carbonate-fluorapatite of type B (*e.g.,* Hofmann & Klee 1995), with $(CO_3)^{2-}$ substituting for $(PO_4)^{3-}$ groups, and a corresponding decrease in cation site-occupancy and charge. The P_2O_5 contents are lower by 2 to 8 wt.%, and those of CaO, by 2 to 5 wt.%, than expected in carbonate-free fluorapatite (Table 2); also, the fluorine content is locally higher than ideal, as found in carbonate-fluorapatite that incorporates $(CO_3F)^{3-}$ groups (Vignoles *et al.* 1982). Up to seven of the strongest diffraction-maxima of apatite were identified in X-ray powder-diffraction patterns of hydroxylherderite, all of them perceptibly broadened, as expected from the extensive compositional range. Optical properties could not be examined in detail because of the very fine-grained nature of the mineral; however, the low approximate maximal and minimal indices of refraction, 1.625 to 1.630 and 1.618 to 1.624, respectively, and the relatively high birefringence, 0.005 to 0.007, correspond to those of "francolite", "dahllite" and other forms of carbonate-fluorapatite (Winchell & Winchell 1951).

GENETIC CONSIDERATIONS

Textural relations of the minerals examined are largely unambiguous. All phases reside within fissures in large crystals of beryl. Phosphate-free surfaces of

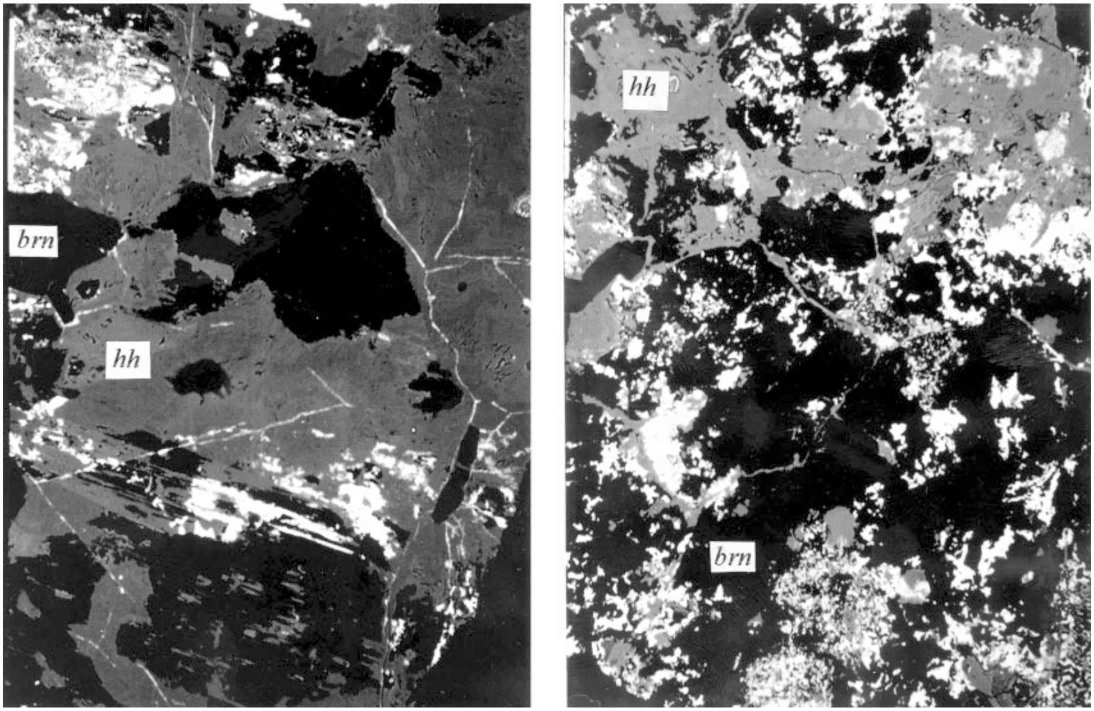

FIG. 2. Beryllonite from the BEP pegmatite in back-scattered-electron images. (A) Subrectangular grains of beryllonite (black, in part plucked; *brn*) embedded in and replaced by hydroxylherderite (dark grey, *hh*); both minerals are invaded by carbonate-fluorapatite (white). The replacement of beryllonite proceeds in part parallel to its excellent cleavage (bottom). The scale bar is 100 μm long. (B) Irregular, corroded grains of beryllonite (black, *brn*) are surrounded by hydroxylherderite (grey, *hh*). Both minerals are replaced by carbonate-fluorapatite (white). The scale bar is 100 μm long.

fractures in beryl, subparallel to {0001}, show an overgrowth of lustrous hillocks of flat, pyramidal shape, terminated by the basal pinacoid. However, the phosphate-coated beryl has an undulating matte surface. This feature suggests that the hillocky regeneration of the beryl surfaces preceded the formation of the phosphates, probably in an unrelated early event, which was followed by an *in situ* reaction between the phosphate-bearing solution and the beryl substrate. Among the phosphates, beryllonite is the early phase, and is enveloped, strongly corroded and replaced by hydroxylherderite. Both minerals are rimmed and penetrated by carbonate-fluorapatite.

Replacement of beryl by late beryllophosphates is possibly the most common style of occurrence of these minerals (*cf.* Černý 2002). Many individual occurrences and assemblages of beryllophosphates are very rare to unique, but the beryllonite – hydroxylherderite – carbonate-fluorapatite sequence encountered in the BEP pegmatite has several complete or partial analogues at widely dispersed localities, with variable contents of F in hydroxylherderite and diverse compositions of apatite (*e.g.*, Newry, Maine: Palache & Shannon 1928; Viitaniemi, Finland: Volborth 1954, Lahti 1981; Meldon, England: von Knorring & Condliffe 1984; Norrö, Sweden: Nysten & Gustafsson 1993; Beauvoir, France: Charoy 1999). The general sequence beryllonite – hurlbutite – F-bearing hydroxylherderite – fluorapatite (to carbonate-fluorapatite) is suggestive of increasing $f(F_2)$ and slightly decreasing $a(P_2O_5)$ in late fluids affecting beryl. Such an interpretation is supported by Burt (1975, Fig. 4; see also Fig. 7 in Charoy 1999), although the system explored by Burt did not involve OH. The presence of carbonate-fluorapatite in the BEP assemblage, instead of fluorapatite, should not be surprising, as $a(CO_2)$ strongly increases in low-temperature stages of consolidation of granitic pegmatites (*e.g.*, London 1986, 1992). Late carbonate-enriched apatite is common in evolved rare-element granitic pegmatites in general, and in those of the Bernic Lake group of southeastern Manitoba in particular (Černý & Lenton 1995, Černý *et al.* 1998).

TABLE 1. REPRESENTATIVE CHEMICAL COMPOSITIONS OF BERYLLONITE AND HYDROXYLHERDERITE FROM THE BEP PEGMATITE

	beryllonite			hydroxylherderite		
	U1-73	U1-74	U1-86	H1-11	H1-71	H1-81
P_2O_5	56.27	55.51	55.70	41.29	41.34	41.69
Al_2O_3	0.16	0.08	0.04	0.03	0.06	0.10
FeO	0.05	0.12	0.05	0.27	0.33	0.20
MnO	0.01	0.02	0.01	0.25	0.35	0.34
MgO	-	0.01	-	-	0.04	0.02
CaO	0.12	0.38	0.85	32.83	32.64	32.59
SrO	0.12	0.05	0.05	0.03	0.08	0.05
BeO^{**}	19.83	19.56	19.63	14.55	14.57	14.69
Na_2O	23.81	23.19	23.29	0.26	0.33	0.38
F	-	-	-	0.03	0.08	0.02
Cl	-	-	-	0.08	0.07	0.04
H_2O^{**}	-	-	-	5.21	5.19	5.27
-O=F	-	-	-	-0.01	-0.03	-0.01
-O=Cl	-	-	-	-0.02	-0.02	-0.01
total	100.37	98.92	99.62	94.80	95.03	95.37
			atoms per $1P^{\dagger}$			
P	1.000	1.000	1.000	1.000	1.000	1.000
Al	0.004	0.002	0.001	0.001	0.002	0.003
Fe^{2+}	0.001	0.002	0.001	0.006	0.008	0.005
Mn	0.000	0.000	0.000	0.006	0.008	0.008
Mg	-	0.000	-	-	0.002	0.001
Ca	0.003	0.009	0.019	1.006	0.999	0.989
Sr	0.001	0.001	0.001	0.000	0.001	0.001
Be	1.000	1.000	1.000	1.000	1.000	1.000
Na	0.969	0.957	0.958	0.014	0.018	0.021
F	-	-	-	0.003	0.007	0.002
Cl	-	-	-	0.004	0.003	0.002
OH	-	-	-	0.993	0.989	0.996
O	3.996	3.993	4.001	4.028	4.031	4.020
total cat.	2.978	2.971	2.980	3.035	3.039	3.028
total an.	3.996	3.993	4.001	5.028	5.030	5.020

*all Fe as FeO; **calculated; - = below detection limit; † assuming Be = 1 for beryllonite, Be = 1 and (F + OH) = 1 for hydroxylherderite.

The late *relative* increase in $a(F_2)$ sufficient to stabilize the carbonate-fluorapatite could have been enhanced by the low-temperature $(OH)F_{-1}$ exchange in adjacent amblygonite–montebrasite. Pseudomorphic replacement in blocky amblygonite–montebrasite by anion exchange is a generally widespread subsolidus process in granitic pegmatites (*cf.* Černá *et al.* 1972, and references therein), commonly accompanied by precipitation of fluorapatite (*cf.* Loh & Wise 1976 for relevant experimental studies). This (OH)-metasomatism strongly affects the primary amblygonite–montebrasite in the BEP pegmatite. Mobilization of phosphorus during the apatite-generating alteration of amblygonite–montebrasite and decomposition of nearby triphylite–lithiophilite could have provided P for the secondary phosphates.

The BEP locality represents one of the few granitic pegmatites capable of generating secondary Be-bearing phosphates. In the vast majority of cases, (i) beryl is not found in close proximity of rock-forming phosphates (such as amblygonite–montebrasite or triphylite–

TABLE 2. REPRESENTATIVE CHEMICAL COMPOSITIONS OF CARBONATE-FLUORAPATITE FROM THE BEP PEGMATITE

	A3-13	A1-43	A2-23	A1-84	FAP
P_2O_5	37.67	37.58	36.78	34.34	42.22
Al_2O_3	0.15	0.22	0.72	0.11	
FeO^{*}	0.65	0.47	1.65	0.14	
MnO	0.10	0.44	0.07	0.05	
MgO	0.21	0.00	0.00	0.05	
CaO	50.86	51.17	52.53	53.30	55.60
SrO	0.44	0.02	0.20	0.22	
Na_2O	0.94	0.25	0.37	0.28	
F	3.91	3.35	3.89	5.19	3.67
Cl	0.23	0.34	0.11	0.02	
-O=F	-1.65	-1.41	-1.64	-2.19	-1.59
-O=Cl	-0.05	-0.08	-0.02	-0.00	
total**	93.46	92.35	94.66	91.51	100.00

FAP = $Ca_5(PO_4)_3F$; * all Fe as FeO; ** remainder to 100% = H_2O and CO_2; Si and S not detected.

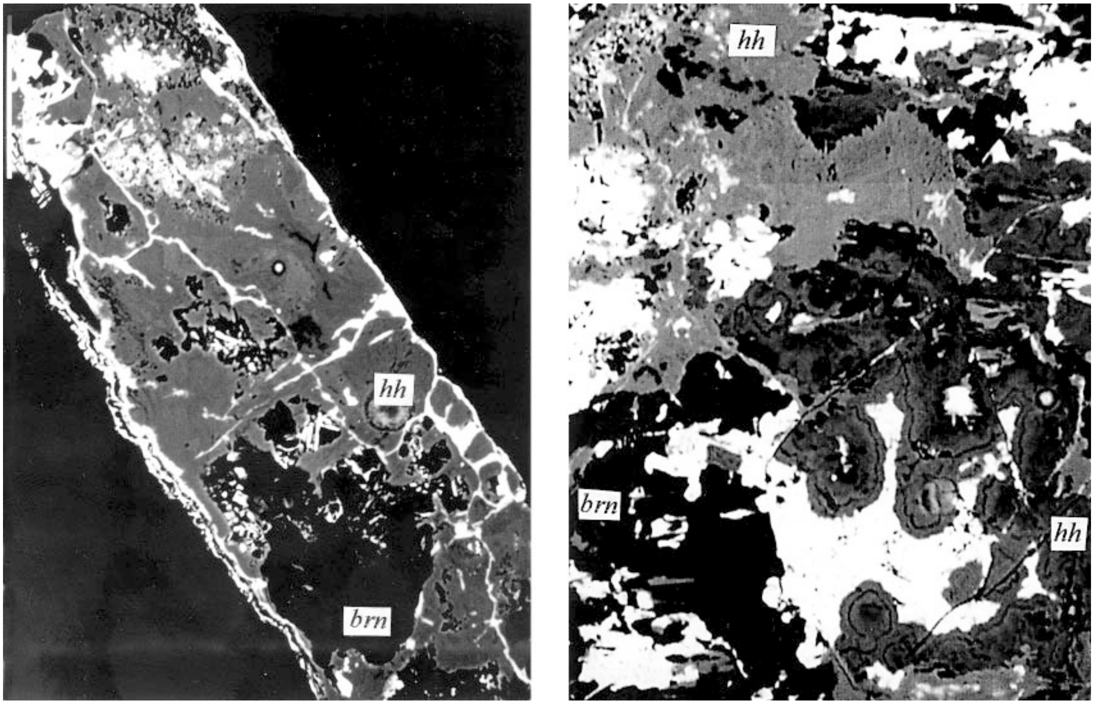

FIG. 3. Hydroxylherderite from the BEP pegmatite in back-scattered-electron images. (A) A lath-shaped crystal of hydroxylherderite (grey, *hh*) with an inclusion of severely corroded beryllonite (black, *brn*), rimmed, veined and replaced by carbonate-fluorapatite (white). The scale bar is 50 μm long. (B) A relict grain of beryllonite (black, *brn*) with hydroxylherderite (grey, *hh*) replaced by carbonate-fluorapatite (white) in the top part; colloform, somewhat darker hydroxylherderite seems to line a cavity, filled later by carbonate-fluorapatite (lower central part). The vertical side of the image is 200 μm long.

lithiophilite), (ii) except at very low temperatures, the mobilized Be and P are generally not transported over long distances from their altered sources (secondary silicates of Be and phosphates are largely pseudomorphic after the primary phases, or confined to their close vicinity), (iii) beryl is quite resistant to alteration at low temperatures and moderate pH (*e.g.*, Černý 1968), (iv) the behavior of Be is very sensitive to fluctuations in pH (as summarized in Černý 2002), and (v) the individual phosphates with beryllium probably only form over very narrow ranges of pH (Harvey & Meier 1989, Kampf *et al.* 1992).

Thus it is not surprising that Be-bearing phosphates are quite scarce on global scale. However, over and above the rarely realized genetic conditions, yet another factor contributes to the very low number of occurrences known to date: at many localities, these phases are quite inconspicuous in their appearance in hand specimens. All patches of pale-colored "stain" and extremely fine-grained "smears" on beryl should be closely examined!

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