# Herderite from Mogok, Myanmar, and comparison with hydroxyl-herderite from Ehrenfriedersdorf, Germany

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#### **ABSTRACT**

The crystal structures of herderite, CaBePO<sub>4</sub>[ $F_{0.75}(OH)_{0.25}$ ], a = 9.7446(4), b = 7.6769(3), c =4.7633(2) Å,  $\beta = 90.667(1)^{\circ}$ , V = 356.31(4) Å<sup>3</sup>,  $P2_1/a$ , and Z = 4, from the Mogok Stone Tract, Myanmar, and hydroxyl-herderite, CaBePO<sub>4</sub>[(OH)<sub>0.52</sub>F<sub>0.48</sub>], a = 9.7615(4), b = 7.6680(3), c = 4.7853(2)Å,  $\beta = 90.184(1)^\circ$ , V = 358.18(4) Å<sup>3</sup>,  $P2_1/a$ , and Z = 4, from the Sauberg mine, Morgenröther Zug, Ehrenfriedersdorf, Germany, have been refined to  $R_1$  indices of 1.7%, in both cases, using MoK $\alpha$ single-crystal X-ray diffraction data. Herderite from Mogok is associated with complex rare-element beryl-type granite pegmatite. Most herderite-series minerals are the product of late-stage processes in granite pegmatite evolution, typically of an exsolved fluid upon crystallization, and significant F-enrichment in these fluids appears to result in the domination of herderite over hydroxyl-herderite. Herderite is now documented from Mogok, Myanmar; Yichung, China; Brazil; and probably Erongo, Namibia. The pegmatites at Ehrenfriedersdorf, Germany have produced samples at the boundary between herderite and hydroxyl-herderite, if not possibly herderite as well.

**Keywords:** Herderite, hydroxyl-herderite, crystal structure, Mogok, Myanmar, Ehrenfriedersdorf, Germany

#### INTRODUCTION

Herderite is ideally CaBePO<sub>4</sub>(F) and an end-member in a solid-solution series with hydroxyl-herderite, CaBePO<sub>4</sub>(OH). However, chemical verification of herderite as a species (e.g., F-dominant samples) and the conditions of its origin have always been a problem. Herderite was initially described by Haidinger (1828) from samples associated with the Sn-bearing pegmatites at Ehrenfriedersdorf in the Erzgebirge of Germany. As Grew pointed out in his review of Be minerals (Grew 2002), neither in Haidinger (1828) nor in studies since (e.g., Leavens et al. 1978; Dunn et al. 1979; King and Foord 1994) has the complete chemical composition of the sample been accurately measured; likewise F was not demonstrated as being dominant in this sample. Samples from Ehrenfriedersdorf were unavailable for study by the late-twentieth century authors noted above, and the sole analysis indicating F > OH was a measurement of only F by microprobe (and comparison with theoretical F content; Dunn and Wight 1976) on a gem of unknown provenance from Brazil. Consequently, there has been speculation (e.g., Gaines et al. 1997) that herderite, as a species in nature, distinct from hydroxyl-herderite, does not exist. With the status of herderite in doubt, paragenesis becomes somewhat of a conundrum. Hydroxyl-herderite occurs in granite pegmatites, greisens, and miarolitic cavities associated with granites and granitic pegmatites (cf. Grew 2002; Černý 2002). Fluorine-rich species in pegmatitic and miarolitic environments require some mechanism to dewater the magma or fluid and thus enhance F content.

The first author had the opportunity to visit Mogok, Myanmar, in December 1998. His fascination with the wide diversity of

minerals found in the region of the Mogok Stone Tract has led to further visits to Myanmar, slow acquisition of a Mogok collection, and an investigation of the minerals and their parageneses from this famous source of rubies and other gems. Among the samples from the Mogok area acquired over the intervening years for the American Museum of Natural History (AMNH) mineral collection was a pale blue-green single crystal. XRD examination of a fragment from the crystal identified it as herderite. When a preliminary microprobe analysis of this crystal pointed to a true F-dominant herderite (>8 wt% F), we decided that further chemical and crystallographic study was needed to substantiate the specimen and the species.

## EXPERIMENTAL METHODS

The Mogok herderite crystal examined here (AMNH 108370) was part of a group of specimens acquired without a specific location. However, other crystals have been identified from Sakhan Gyi (22° 53.95' N, 96° 24.43' E), a town and mining area well known for pegmatite minerals (e.g., elbaite, beryl, topaz, scheelite, cassiterite, danburite, amblygonite, and fluorite) and marble-hosted spinels that is located toward the western limit of the Mogok Stone Tract, and Pan Daw village (22° 57.62′ N, 96° 24.98′ E), near Kyauk Sin, a source of amazonite, topaz, aquamarine, cassiterite, and fluorite, in addition to the granite mineral suite (for maps see Chhibber 1934; Kammerling et al. 1994; Themelis 2007). Additional finds include diggings on Loi-Sau Mountain (ca. 22° 59.7′ N 96° 38.3′ E) near Pantahoe village, about 16 km northeast of Mogok, from a pegmatite pocket with pink elbaite; this pegmatite also yielded jeremejevite [Al<sub>6</sub>(BO<sub>3</sub>)F<sub>3</sub>] (Kyaw Thu, personal communication). Pazun-seik (22° 57.72′ N, 96° 24.27′ E) located near Nam-peik where herderite has been recovered from open-pit mining, has also yielded beryl, cassiterite, microcline, topaz, fluorite, spessartine, and rhodochrosite as well as ruby and sapphire. All sources are associated with late pegmatites intruding the Kabaing granite (~16 Ma; Bertrand and Rangin 2003; Themelis 2007; Thu 2007) and adjacent marble units. Fragments from the base of the 2 cm long pale-green prism {011} (AMNH108370) were used in this work. In addition, fragments from a herderite specimen from the type locality in an Sn mine associated with greisen and late Variscan granites at Ehrenfriedersdorf, Sachsen, Germany, were provided

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by Rainer Thomas (who has been doing Raman spectroscopy on herderite samples) to compare with the Mogok crystal. The fragments studied are from sample no. 20517 from the Mining Academy, Freiberg, Germany, collected at the Sauberg mine, Morgenröther Zug, Ehrenfriedersdorf. This sample is not a portion of the holotype, which resides at the Werner-Sammlung der Bergakademie Freiberg (Massanek et al. 1999) and may be considered a topotype (Embrey and Hey 1970).

#### **Electron-microprobe analysis**

Chemical compositions were determined on a Cameca SX100 electron microprobe at AMNH, operating at 15 kV and 10 nA using natural and synthetic standards for major and minor constituent elements (including O but not Be, see Table 1). To account for the ZAF effects of Be, the analytical results were evaluated for the amount of Be that should occur in the stoichiometric structure and the raw microprobe data were recalculated using Armstrong's offline CITZAF program (Armstrong 1991). The results of CITZAF show a conspicuous decrease in F compared to the results without accounting for the presence of Be. Compositions were then recalculated as oxides from the elements because of relatively large errors in analysis of O (typically  $\pm \sim 2$  wt%). H<sub>2</sub>O was added to fill O5, the site occupied by (OH,F); the results are given in Table 1.

#### X-ray diffraction

A small fragment of each sample was selected for X-ray diffraction measurements on the basis of optical clarity and freedom from inclusions. The crystals were attached to tapered glass fibers and mounted on a BRUKER P4 four-circle diffractometer at the University of Manitoba equipped with monochromatic Mo $K\alpha$  X-radiation and an APEX 4K CCD area detector. The intensities of ~6300 reflections were collected to ~60.00 °20 using 15 s per 0.2° frame with a crystal-to-detector distance of 4 cm. Empirical absorption (SADABS, Sheldrick 1998), Lorentz, polarization, and background corrections were applied, and the data were reduced to structure factors, resulting in ~1050 unique reflections. The unit-cell parameters were obtained by least-squares refinement from the positions of ~1200 reflections with  $I > 10\sigma I$ , and are given in Table 2. A CIF file is on deposit.

The crystal structures were refined with the Bruker SHELXTL Version 5.1 system of programs (Sheldrick 1997). Scattering curves for neutral atoms were taken from the *International Tables for Crystallography* (Wilson 1992). Site occupancies were fixed to unity for all atomic sites except O5-F [assuming ideal composition CaBePO<sub>4</sub>(F,OH)], with the O5-F site occupancy constrained as OH + F = 1.0. The structures were refined to  $R_1$  indices of ~3%, with anisotropic-displacement parameters for all sites. The positional parameters of the H atom were refined with the constraint that the O3-H distance is equal to 0.98 Å. Atom positions and anisotropic-displacement parameters are given in Table 2 and selected interatomic distances in Table 3.

**TABLE 1.** Chemical composition (wt%)\* and unit formulae (apfu) for herderite crystals

	108370	20517	121024†
P <sub>2</sub> O <sub>5</sub>	43.16	43.98	42.93
SiO <sub>2</sub>	0.05	0.01	0.00
$Al_2O_3$	0.02	0.01	-
FeO	0.01	0.01	-
MgO	0.00	0.00	-
BeO	(15.30)	(15.54)	(15.24)
CaO	34.45	34.99	34.59
$Na_2O$	0.29	0.03	-
F	8.70	5.71	3.39
H <sub>2</sub> O	(1.39)	(2.90)	(3.87)
O = F	-3.66	-2.40	-1.43
Sum	99.70	100.76	98.60
Р	0.994	0.997	0.994
Si	0.001	0.000	0.000
Al	0.001	0.000	-
Fe	0.000	0.000	-
Mg	0.000	0.000	-
Be	1	1	1
Ca	1.004	1.004	1.014
Na	0.015	0.005	-
F	0.748	0.483	0.293
OH	0.252	0.517	0.707

<sup>\*</sup> Values in parentheses are calculated from stoichiometry.

### CHEMICAL COMPOSITION

The analyzed herderite specimens are very close to ideal CaBePO<sub>4</sub>(F,OH). Crystal 108370 (Mogok) has F dominant and hence is herderite, and crystal 20517 (Ehrenfriedersdorf, Sachsen, Germany) has almost equal contents of F and (OH). However, in general, the results of Leavens et al. (1978) show that samples from the series with OH dominant over F are much more common. The results from site refinement are not much different from the microprobe (see Table 2), but we accept the results from the microprobe analyses for the purpose of a chemical formula for consistency with most data in the literature. Thus both herderite and hydroxyl-herderite occur in nature despite the suggestion by Gaines et al. (1997) that this might not be the case. Sodium is the main additional constituent and only in the Mogok sample. The most likely paragenetic reason for "high" Na may derive from the sodic nature of the pegmatites, an interpretation made from observing many sodic minerals (including sodic moonstone) at this occurrence, rather than from field-based geochemistry, which is lacking. All analyses record minor excess Ca and minor insufficient P, which do not seem to correlate with other minor components such as Na, which might be expected to substitute for Ca. Thus, the excess Na may be due to nano-inclusions of a solid or fluid which were not visible on the surfaces of the samples or by binocular microscopy.

## **CRYSTAL STRUCTURE**

Three structure refinements can now be compared to examine the effect of OH-F substitution in the herderite—hydroxyl-herderite solid solution. A structure refinement of hydroxyl-herderite was done by Lager and Gibbs (1974) on a Smithsonian sample (NMNH 121024) from the Golconda Mine, Minas Gerais, Brazil, and the description of the structure is adequate not to require repetition here. However, this refinement does not represent end-member hydroxyl-herderite; it contains 3.39 wt% F (0.293 F apfu) according to Leavens et al. (1978).

**TABLE 2.** Miscellaneous information for structure refinement of herderite crystals

108370	20547
100570	20517
9.7446(4)	9.7615(4)
7.6769(3)	7.6680(3)
4.7633(2)	4.7853(2)
90.667(1)	90.184(1)
356.31(4)	358.18(4)
4	4
$P2_1/a$	P2₁/a
80	200
6207	6360
3377	3530
1044	1050
1.5	1.5
1.7	1.7
4.9	5.1
	9.7446(4) 7.6769(3) 4.7633(2) 90.667(1) 356.31(4) 4 P2 <sub>1</sub> /a 80 6207 3377 1044 1.5 1.7

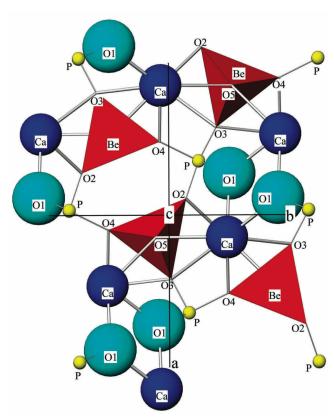
<sup>\*</sup> From Lager and Gibbs (1974).

<sup>†</sup> Analysis from Leavens et al. (1978), stoichiometric estimates added.

<sup>&</sup>lt;sup>1</sup> Deposit item AM-08-050, CIF. Deposit items are available two ways: For a paper copy contact the Business Office of the Mineralogical Society of America (see inside front cover of recent issue) for price information. For an electronic copy visit the MSA web site at http://www.minsocam.org, go to the American Mineralogist Contents, find the table of contents for the specific volume/issue wanted, and then click on the deposit link there.

	Х	у	Z	<i>U</i> <sub>11</sub>	Occup.	$U_{22}$	$U_{33}$	$U_{23}$	U <sub>13</sub>	<i>U</i> <sub>12</sub>	$U_{\rm eq}$
					10	08370					
Ca	0.33014(2)	0.11216(3)	0.99591(5)	0.00938(14)	1	0.01037(14)	0.00985(13)	-0.00072(7)	0.00069(8)	-0.00046(7)	0.00986(10)
Р	0.08160(3)	0.27075(4)	0.47351(6)	0.00602(15)	1	0.00686(15)	0.00773(15)	-0.00007(9)	0.00021(10)	-0.00013(9)	0.00687(10)
Be	0.33895(16)	0.4141(2)	0.5397(3)	0.0087(6)	1	0.0095(6)	0.0090(6)	0.0004(5)	0.0003(5)	0.0006(5)	0.0091(3)
01	0.04005(9)	0.39814(11)	0.24585(18)	0.0098(4)	1	0.0090(4)	0.0105(4)	0.0022(3)	-0.0009(3)	0.0004(3)	0.00978(18)
02	0.45729(9)	0.28281(11)	0.65111(18)	0.0074(4)	1	0.0106(4)	0.0107(4)	0.0013(3)	0.0021(3)	0.0022(3)	0.00954(17)
O3	0.19339(9)	0.34517(12)	0.67592(17)	0.0066(4)	1	0.0124(4)	0.0093(4)	-0.0007(3)	-0.0005(3)	-0.0023(3)	0.00944(17)
04	0.14551(9)	0.10755(11)	0.33633(19)	0.0119(4)	1	0.0073(4)	0.0119(4)	-0.0003(3)	0.0032(3)	0.0013(3)	0.01034(18)
O5F	0.32911(8)	0.40918(10)	0.21090(16)	0.0126(4)	0.776(4) F	0.0151(4)	0.0090(4)	-0.0004(3)	-0.0012(3)	0.0012(3)	0.0122(3)
O5	0.32911(8)	0.40918(10)	0.21090(16)	0.0126(4)	0.224 O	0.0151(4)	0.0090(4)	-0.0004(3)	-0.0012(3)	0.0012(3)	0.0122(3)
					2	0517					
Ca	0.33054(3)	0.11215(3)	0.99672(5)	0.00899(14)	1	0.00969(14)	0.00946(14)	-0.00093(8)	0.00065(9)	-0.00064(8)	0.00938(10)
Р	0.08141(3)	0.27112(4)	0.47175(6)	0.00598(16)	1	0.00669(16)	0.00747(16)	0.00001(10)	0.00013(11)	-0.00018(10)	0.00671(10)
Be	0.33905(17)	0.4143(2)	0.5364(4)	0.0078(7)	1	0.0099(7)	0.0093(7)	0.0009(5)	0.0002(5)	0.0003(5)	0.0090(3)
01	0.03997(10)	0.39849(12)	0.2455(2)	0.0094(4)	1	0.0089(4)	0.0104(4)	0.0024(3)	-0.0007(3)	0.0006(3)	0.00957(19)
O2	0.45738(9)	0.28281(13)	0.65139(19)	0.0071(4)	1	0.0104(4)	0.0101(4)	0.0015(3)	0.0017(3)	0.0020(3)	0.00917(19)
O3	0.19302(9)	0.34533(13)	0.67079(19)	0.0065(4)	1	0.0120(4)	0.0096(4)	-0.0010(3)	-0.0006(3)	-0.0022(3)	0.00936(19)
04	0.14412(10)	0.10715(12)	0.3332(2)	0.0113(4)	1	0.0069(4)	0.0112(4)	-0.0003(3)	0.0030(3)	0.0011(3)	0.00978(19)
O5F	0.33181(9)	0.41132(12)	0.20637(19)	0.0125(5)	0.515(4) F	0.0138(5)	0.0094(4)	-0.0007(3)	-0.0013(3)	0.0012(3)	0.0119(3)
O5	0.33181(9)	0.41132(12)	0.20637(19)	0.0125(5)	0.485 O	0.0138(5)	0.0094(4)	-0.0007(3)	-0.0013(3)	0.0012(3)	0.0119(3)

TABLE 3. Final atom positions, occupancy, and displacement parameters (Å) for herderite crystals



**FIGURE 1.** The crystal structure of herderite projected down the **c**-axis, showing the orientation of the O2-O4 edge of the BeO<sub>4</sub> polyhedron, which increases (rather than decreases) with increasing F content (at O5).

With the results presented here, we now have structures that span from 29 to 75% of the herderite end-member. Consistent with difference in the ionic radius of the only varying constituents, 1.30 Å for F and 1.34 for OH in threefold coordination (Shannon 1976), the effect of increasing herderite content is generally a contraction of the structure, with a decrease in a, c, and V (Table 2), but an increase in b. The increase in b reflects some small rotations among polyhedra in the structure and an extension of

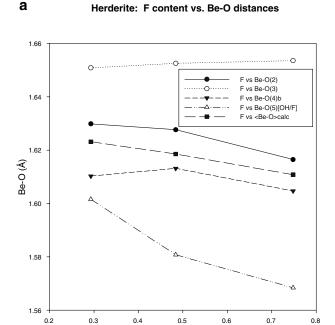
the O2-O4 edge of the Be polyhedron, aligned approximately parallel to the b axis (Fig. 1). With increasing F content, the bonds from Be and Ca to the O5 anion (=F/OH) shorten, with Be-O5 showing the greatest change (Table 4; Fig. 2). Phosphorus is not bonded to O5, and the  $PO_4$  tetrahedron shows no change in the average bond length, <P-O>.

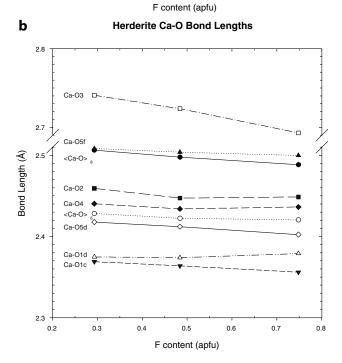
Individual M-O5 bonds decrease noticeably with increasing F in the structure. For Ca-O bonding, the longest bonds, Ca-O3 at ≥2.7 Å, show the greatest change (a decrease) between structures with greater F content, and only Ca-O1d appears to remain constant or, perhaps, increases slightly (Fig. 2b).

## PARAGENESIS AND HERDERITE ABUNDANCE

As described by Grew (2002), Černý (2002), and Leavens et al. (1978), granite pegmatites and associated miarolitic cavities and greisens are the primary sources of hydroxyl-herderite and, by analogy, that should be the case for herderite as well. The Mogok occurrences are areas of gem-bearing granite pegmatites yielding muscovite, tourmaline (elbaitic), topaz, beryl, and fluorapatite, which signify evolved liquids rich in the components H<sub>2</sub>O, Be, F, P, and B.

In granitic gem pegmatites with miarolitic cavities and sometimes peripheral greisens, the magmatic source of Be, P, F, and  $H_2O$  is clear, but the mechanism(s) for enrichment of F > OHand crystallization conditions for herderite need to be addressed. Generally, F enrichment in an exsolved magmatic fluid requires either extreme melt evolution dominated by fractional crystallization (Webster et al. 2004; Thomas et al. 2005), or an F-enriched magmatic source since the (fluid/melt) partition coefficient for F increases strongly with the F activity (content) of magmatic systems. One mechanism for generating such magmas involves dewatering of the melt, and low pressure can permit exsolution of a water-rich fluid or vapor, enhancing the F content in the residual melt. High temperature can stabilize F-rich minerals over OH-rich phases, as is well demonstrated for micas and amphiboles (e.g., Munoz 1984; Gilbert et al. 1982; Petersen et al. 1982). The greisen and miarolitic cavities at Ehrenfriedersdorf suggest relatively shallow conditions, which are appropriate for enrichment of the F component. Moreover, that pegmatite has been interpreted as derived from a late-stage, F- and P-rich, alu-





**FIGURE 2.** Variation of selected interatomic distances as a function of F content: (a) Be-O distances; (b) Ca-O distances.

minosilicate liquid (Webster et al. 1998), which is consistent with an exsolved fluid also rich in P and F. Finally, in the context of Mogok, in particular, magma or fluid interaction with carbonate minerals that forms skarn may be a mechanism for raising Ca activity to saturate the late fluid with respect to both herderite and fluorite. Free crystals of herderite from most localities, including Mogok, indicate crystallization in open cavities, and hence, from an exsolved fluid. The pegmatites at Sakhan Gyi and Pan Daw are complex, of the rare-element beryl type (Themelis 2007; Thu 2007), which are rich in F, B, and Be, and some crystallizations

**TABLE 4.** Selected interatomic distances (Å) in herderite crystals

	108370	20517
P-O1	1.512(1)	1.512(1)
P-O2a	1.541(1)	1.543(1)
P-O3	1.555(1)	1.553(1)
P-O4	1.547(1)	1.548(1)
<p-o></p-o>	1.539	1.539
Be-O2	1.616(2)	1.628(2)
Be-O3	1.654(2)	1.653(2)
Be-O4b	1.605(2)	1.613(2)
Be-O5	1.569(2)	1.581(2)
<be-o></be-o>	1.611	1.619
02-04	2.688	2.678
Ca-O1c	2.356(1)	2.364(1)
Ca-O1d	2.379(1)	2.374(1)
Ca-O2	2.449(1)	2.447(1)
Ca-O3e	2.590(1)	2.602(1)
Ca-O3	2.590(1)	2.602(1)
Ca-O4	2.436(1)	2.434(1)
Ca-O5d	2.402(1)	2.412(1)
Ca-O5f	2.500(1)	2.504(1)
$<$ Ca $-$ O $>_6$	2.420	2.422
<ca-o><sub>8</sub></ca-o>	2.488	2.498

Notes: a = x - 1/2, -y + 1/2, z; b = -x + 1/2, y + 1/2, -z + 1; c = x + 1/2, -y + 1/2, -z + 1; -z + 1/2, -z + 1/2,

are associated with contacts to adjacent marble-skarn. Sourcing of elbaite, topaz, beryl, amblygonite, jeremejevite, and fluorite from the herderite localities support this interpretation. Fluorite is found abundantly in the low-temperature assemblages in botryoidal habit around the pegmatites and forms large crystals in some pockets (Kyaw Thu, personal communication), which also argues for an F-rich fluid.

Specific estimates of *P-T* conditions of the pockets or skarns in the Mogok pegmatites have not been made, but based on the Ehrenfriedersdorf pegmatite, which formed by liquid-liquid immiscibility (initiating at 600–700 °C; Thomas et al. 2006; Webster et al. 1998), and rare-element beryl types (e.g., Thomas 1994; Sirbescu and Nabelek 2003), something below the crystallization conditions of 400-600 °C, 1-2 kbar would be expected (the strong enrichments in fluxing and volatile components stabilize melts to such low temperatures). Descriptions of specimens of herderite from Ehrenfriedersdorf (Jahn 1998) indicate growth on quartz or gilbertite (botryoidal muscovite) with coexisting fluorite ± cassiterite, apatite, and wolframite, which would suggest formation in the hydrothermal or secondary zone of greisens and lower temperature, perhaps 250–400 °C (Hösel et al. 1994; Kumann and Leeder 1994). We can only suggest these constraints would be similar for the Mogok occurrences; greater specificity requires an investigation more detailed and field-oriented than is possible here.

Recently Huang et al. (2002) reported herderite with up to 7.25 wt% F (~65 mol% end-member herderite) from the Yichun topaz-lepidolite granite (which contains primary magmatic apatite and amblygonite), Jiangxi Province, China; they interpreted herderite as a low-temperature (~250 °C) secondary phase. The F-rich pegmatite at Davib-Ost farm, Erongo, Namibia yielded a pale-blue herderite phase associated with schorl, muscovite, and orthoclase (see Cairncross and Bahmann 2006). Positions of key bands in the Raman spectra of samples from Davib-Ost farm, which shift between hydroxyl-herderite and herderite, suggest

that the samples are more F-rich than those from Ehrenfriedersdorf, thus indicating that the phase is herderite (Rainer Thomas, personal communication). The samples presented above, combined with the herderite from Mogok, Myanmar described here, and the sole sample from Brazil (Dunn and Wight 1976) yield four occurrences of herderite. The sample studied here from the Sauberg mine, Ehrenfriedersdorf, is essentially on the boundary between herderite and hydroxyl-herderite, so it is possible both species are present within the deposit. It is still not known whether the holotype is or is not herderite. In any case, herderite is not common compared to hydroxyl-herderite, and its presence likely indicates uncommon F-enrichment in late-stage/evolved fluids from Be-rich granite pegmatites.

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