CRYSTAL STRUCTURE DETERMINATIONS OF FELSŐBÁNYAITE AND BASALUMINITE, AL4(SO4) (OH)10.4H2O

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ABSTRACT

Single crystal X-ray structural data (T - 300 K) are reported for felsőbányaite and basaluminite, Al₄ (SO₄) (OH)_{10.4}H₂O. The X-ray work confirmed the identity of these two mineral species within limits of error. Structure parameters for felsőbányaite: a = 13.026(1) Å; b = 10.015(1) Å; c = 11.115(1) Å; $\beta = 104.34(1)^{\circ}$; Z = 4; space group P2₁-C₂²; 234 variable parameters were refined to R = 0.101, R_w = 0.092; 1044 X-ray reflections with Fo > 1\sigma Fo and sin $\nu/\lambda \le 60^{\circ}$. The atomic arrangement exhibits a typical sheet structure, in which the individual AlO₆ octahedra are combined via common O atom corners and edges to Al₈O₂₂ layers. These layers are interconnected by hydrogen bridges to SO₄ tetrahedra and water molecules, forming the three dimensional arrangement. Basaluminite is a microcrystalline variety of felsőbányaite.

Keywords: Felsőbányaite; Basaluminite; Sulphate minerals; Chemical analyses; Crystal structure; Crystal chemistry.

INTRODUCTION

The very rare mineral felsőbányaite was discovered in the middle of the last century in Felsőbánya, Hungary (= Baia Sprie, Rumania) in the oxidation zone of the main lodge in the eastern part of Bányahegy. It forms spherulites on baryte and antimonite crystals up to 5 mm diameter, built by radially arranged platelets. For the first time this mineral was mentioned by KENNGOTT (1853), a detailed description was given by HAIDINGER (1854) together with a chemical analyses performed by K. v. HAUER, yielding the formula $AI_4(SO_4)$ (OH)₁₀.5H₂O. Optical data and heating experimens were reported by KRENNER (1928), chemical analyses and X-ray powder data by KOCH and SARUDI (1964). Single crystal X-ray experiments on felsőbányaite with determination of lattice constants and space group extinction were performed by PERTLIK (1993).

The first description of two distinct minerals, hydrobasaluminite, $Al_4(SO_4)$ (OH)₁₀.36H₂O, and basaluminite $Al_4(SO_4)$ (OH)₁₀.5H₂O, from the Lodge pit of the Irchester Ironstone Company near Wellingborough is given by BANNISTER and HOLLINGWORTH (1948). Chemical analyses and X-ray powder data for these two minerals together with the conditions of formation were reported by HOLLINGWORTH and BANNISTER (1950). Further reports about basaluminite from different occurrences, especially weathering zones, were published by SPENCER (1949, 1957), FOMINYKH

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(1965), TIEN (1968), SREBODOL'SKIY (1968, 1970), BALL (1969), BRYDON and SINGH (1969), SUNDERMANN (1969), MITCHELL (1970) and WIESER (1974). These reports include chemical analyses, X-ray powder data, differential thermal and thermogravic analyses as well as optical data for the visible and IR part of the spectrum.

The unit cell parameters for basaluminite from indexed X-ray powder patterns were derived by CLAYTON (1980). Refined unit cell parameters and indexed powder patterns of basaluminite from Sussex, England, taken with Guinier-Hägg-type focussing camera using monochromatized CuK α_1 radiation were reported by FARKAS (1980). These calculations were performed with the program system TREOR (cf. WERNER, 1964, 1969).

"Expressis verbis" the indentity of the two minerals felsőbányaite and basaluminite was presumed by PAPP and WEISZBURG (1989) and WEISZBURG and PAPP (1990). In the opinion of these authors basaluminite doesn't represent a distinct mineral species but only a microcrystalline variety of felsőbányaite. One of the aims of the present investigations was therefore to confirm or to refuse this statement.

EXPERIMENTAL

For the present investigations crystals of basaluminite from Sussex, England, were provided by the Swedish Museum of Natural History, crystals of felsőbányaite from the type locality by the Institute for Mineralogy and Crystallography, University of Vienna.

The Al:S proportion in basaluminite was determined by semiquantitative EDXanalyses. Further elements detected during these experiments are Na, K, Mg, Ca, Fe and P in wt% < 1.0. A compilation of chemical analyses data of basaluminite (different occurences) is given in Table 1.

Felsőbányaite was analized using classical chemical analytical methods for the main elements, by neutron activation for the trace elements and organic microprobe analytical method for hydrogen. The results of these analyses are compiled in Table 2 together with results from the literature.

Crystals of basaluminite and felsőbányaite, suitable for X-ray work, were checked by classical film methods. For unit cell parameters from the literature and from the present measurements for the two title compounds cf. Table 3.

The structure of basaluminite was solved by direct method strategy, using single crystal X-ray intensity data corrected for Lorentz- and polarization effects in usual ways. The parameters were refined by least squares method. These structural data, determined for basaluminite, were used as initial parameters for the structure determination of felsőbányaite based on X-ray intensity data measured directly on a felsőbányaite single crystal. This calculation resulted in a somewhat lower R value and smaller standard deviations than in the case of basaluminite. Especially for felsőbányaite: 1044 data with Fo > 1 σ Fo, and 2 $\upsilon \le 60^{\circ}$. The R values (R and Rw) are 0.101 and 0.092, w = $[\sigma(Fo)]^{-2}$. The data were measured on a Stoe AED 2 four circle diffractometer with monochromatized MoK radiation. The atomic parameters determined for felsőbányaite and some relevant interatomic distances are given in Table 4 and Table 5, respectively. Solution and refinement of the two structures were performed with the program system She1x1-76 (SHELDRICK, 1976) using neutral scattering functions (IBERS and HAMILTON, 1974).

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	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
A1203	39.70	43.0	41.3	43.8	44.8	44.08	43.77	47.48	44.49	44.75
SO3	20.06	15.6	14.2	17.0	17.0	16.72	15.27	17.81	16.86	18.10
H ₂ O	39.94	38.7	39.7	32.8	34.2	38.58	36.27	34.24	31.56	35.60
Fe ₂ O ₃	-	0.3	0.2	1.0	1.0	0.071	0.78	-	0.45	-
CaO	-	-	-	2.4	1.0	- ·	-	0.25	0.44	0.20
MgO	_	-	-	0.1	0.1	-	-	0.25	-	-
K ₂ O	-	-	-	0.1	0.1	-		-	-	_
Na ₂ O	-	-	_	trace	trace	-	-	-	-	-
P ₂ O ₅	-	trace	1.0	_	-	0.021	0.25	-	-	-
SiO ₂	-	2.4	3.6	1.4	1.4	0.37	2.90	0.15	6.20	

Chemical analyses of basaluminite (in wt%) from the literature without any corrections or recalculations

(1) HOLLINGWORTH and BANNISTER (1950), analyzed by J. L. Lassaigne

(2) & (3) HOLLINGWORTH and BANNISTER (1950)

(4) & (5) TIEN (1968)

(6) & (7) SUNDERMANN and BECK (1969)

(8) SREBODOL'SKIY (1968, 1970)

(9) WIESER (1974)

(10) CLAYTON (1980)

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TABLE 2

(1)(2a) (2b) (3) (4) 45.63 37.27 44.70 48.5(5) 47.63 Al₂O₃ SO3 16.47 14.50 17.39 15.9(5) 18.70 37.27 H₂O 31.53 37.82 34.1(5) 33.66 Fe₂O₃^x ---1.49 0.7(2) ------Sb₂O₅^x -_ 0.6(2)_ -SiO₂ 15.13 < 0.5 _ ----_

Chemical analyses of felsőbányaite in (wt %). E.s.d.'s in parentheses.

x Arbitrary given for trevalent iron and pentavalent antimony

(1) HAIDINGER (1854): Analyst K. V. HAUER

(2a,b) KOCH and SARUDI (1964). Results before and after substracting impurities

(3) This work. Trace elements in (ppm):

Na 380; Sc 0.2; La 8; V 515; Mn 265; Zn 700.

(4) Calculated for the formula AI4(SO4)(OH)10.4H2O

TABLE 3

Unit cell parameters for basaluminite and felsőbányaite, respectively. E.s.d. 's in parentheses. (1) and (3) recalculated from the literature by a 3x3 matrix (101/010/00-1)

	(1)	(2)	(3)	(4)
a (Å)	12.950(3)	12.954(5)	12.94(7)	13.026(1)
b (Å)	10.011(3)	10.004(6)	10.02(7)	10.015(1)
c (Å)	11.086 (3)	11.064(9)	11.23(6)	11.115(1)
β(°)	104.085(7)	104.1(1)	104.08(3)	104.34(1)
Z	4 .	4	_	4
space group		P21		P21

Basaluminite from

(1) Oxford Clay at Crook Hill, Dorset; powder patterns (CLAYTON, 1980)

(2) Sussex, England (collection of the Swedish Museum of Natural History); single crystal data and powder patterns (FARKAS, 1980)

Felsőbányaite from

(3) Felsőbánya, Hungary (=Baia Sprie in Rumania); powder patterns (WEISZBURG and PAPP, 1990)

(4) occurrence as (3), single crystal data (PERTLIK, 1993)

E.s.d.'s in parentheses. Denotation of the atoms according to Parkas (1980).					
Atom	x	Y	Z		
Al(1)	0.5813(8)	0.155(1)	0.6371(9)		
AI(2)	0.7053(7)	0.422(1)	0.7072(9)		
AI(3)	0.6906(7)	0.913(2)	0.7111(9)		
A1(4)	0.8157(9)	0.169(2)	0.7942(9)		
AI(5)	0.8177(7)	0.663(1)	0.7732(9)		
A1(6)	0.4659(7)	0.417(1)	0.5998(8)		
A1(7)	0.9238(8)	0.923(1)	0.8589(9)		
A1(8)	0.0432(9)	0.668(2)	0.9091(9)		
/ ((0)	0.0.024(5)	,			
O(11)	0.528(2)	0.086(3)	0.484(2)		
O(12)	0.600(2)	0.334(3)	0.551(2)		
O(13)	0.717(2)	0.079(3)	0.643(2)		
O(14)	0.691(2)	0.240(3)	0.797(2)		
O(15)	0.567(3)	0.003(4)	0.720(3)		
O(16)	0.469(2)	0.243(4)	0.672(3)		
O(22)	0.739(2)	0.577(3)	0.634(2)		
O(23)	0.818(2)	0.324(3)	0.686(2)		
O(24)	0.782(2)	0.508(3)	0.863(2)		
O(26)	0.571(2)	0.489(3)	0.728(2)		
O(31)	0.623(2)	0.851(3)	0.545(2)		
O(33)	0.839(2)	0.838(3)	0.722(2)		
O(34)	0.781(3)	-0.005(5)	0.846(4)		
O(35)	0.685(2)	0.756(3)	0.784(2)		
O(43)	0.930(2)	0.079(3)	0.758(3)		
O(44)	0.881(3)	0.238(4)	0.938(3)		
Q(53)	0.949(2)	0.609(3)	0.765(2)		
Q(54)	0.887(2)	0.750(3)	0.921(2)		
O(66)	0.364(2)	0.487(3)	0.682(2)		
O(73)	0.042(2)	0.840(3)	0.846(2)		
O(74)	0.975(2)	0.008(3)	0.005(2)		
O(83)	0.163(2)	0.604(3)	0.865(2)		
		0.0(0)()	0.122(10)		
S(1)	0.9042(7)	-0.060(1)	0.4236(8)		
Os(11)	0.853(4)	0.061(5)	0.474(4)		
Os(12)	0.897(2)	-0.180(3)	0.499(2)		
Os(13)	0.019(2)	-0.061(3)	0.407(3)		
Os(14)	0.833(2)	- 0.089(4)	0.297(3)		
5(2)	0.2887(8)	0.063(2)	0.9244(9)		
S(2)	0.3887(8)	0.000(2)	0.9244(2)		
Os(21)	0.295(3)	0.010(.7)	0.036(4)		
Os(22)	0.457(3)	0.062(4)	0.885(3)		
Os(23)	0.437(3)	0.172(4)	0.826(3)		
OS(24)	0.337(3)	0,172(4)	0.020(3)		
Ow(1)	0.871(2)	- 0.294(3)	0.152(3)		
Ow(2)	0.327(3)	0.425(5)	0.969(3)		
Ow(3)	0.214(4)	0.097(5)	0.587(4)		
Ow(4)	0.851(2)	0.332(3)	0.366(3)		
Ow(5)	0.648(2)	0.280(3)	0.335(3)		
Ow(6)	0.650(2)	0.226(3)	0.021(2)		

Atomic parameters for felsőbányaite. The isotopic displacement factors in the range from 0.16 to 1.20 are not given. {defined as $\exp \left(-8\pi^2 u^2 \sin^2\theta/\lambda^2\right) [nm^2]$ } E.s.d. 's in parentheses. Denotation of the atoms according to Farkas (1980).

TABLE 5

Selected interatomic distances for felsőbányaite (Å) (E.s.d. 's in parentheses), <mean values>

Al(1) - O(11) O(12) O(13) O(14) O(15) O(16)	= 1.81(3) = 2.08(3) = 1.91(2) = 2.16(2) = 1.81(4) = 1.83(3)	Al(2) - O(12) O(14) O(22) O(23) O(24) O(26)	= 2.13(2) = 2.11(3) = 1.85(3) = 1.83(3) = 1.97(3) = 1.94(2) $\overline{<1.97>}$
Al(3) - O(13) O(15) O(31) O(33) O(34) O(35)	= 1.90(3) = 1.87(3) = 1.94(3) = 2.05(2) = 1.86(4) = 1.78(3) $\leq 1.90 >$	$\begin{array}{r} AI(4) - & O(13) \\ O(14) \\ O(23) \\ O(34) \\ O(43) \\ O(44) \end{array}$	= 2.06(3) = 1.79(2) = 1.97(3) = 1.93(5) = 1.87(3) = 1.76(4) $\overline{<1.90>}$
Al(5) - O(22) O(24) O(33) O(35) O(53) O(54)	= 1.85(3) = 1.97(3) = 1.88(3) = 1.99(2) = 1.82(2) = 1.88(3) $\overline{<1.90>}$	$\begin{array}{l} Al(4) - & O(11) \\ O(12) \\ O(16) \\ O(26) \\ O(31) \\ O(66) \end{array}$	= 1.93(3) = 2.11(2) = 1.93(4) = 1.87(3) = 1.85(2) = 1.94(2) $\overline{<1.94>}$
Al(7) – O(33) O(34) O(43) O(54) O(73) O(74)	= 1.85(3) = 1.97(4) = 1.94(3) = 1.97(3) = 1.79(3) = 1.81(3)	$\begin{array}{l} Al(8) - & O(44) \\ O(53) \\ O(54) \\ O(73) \\ O(74) \\ O(83) \end{array}$	= 1.88(4) = 1.86(3) = 2.23(3) = 1.86(3) = 1.91(3) = 1.86(2)
S(1) - Os(11) Os(12) Os(13) Os(14)	$\overline{<1.89>} = 1.55(5) = 1.48(3) = 1.56(5) = 1.51(3) = 1.51(3)$	S(2) - Os(21) Os(22) Os(23) Os(24)	$\overline{<1.93>}$ = 1.35(5) = 1.40(5) = 1.48(3) = 1.53(4) $\overline{<1.44>}$

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The isotropic displacement factors for the single atoms are more or less terms for correcting extinction and absorption effects but without any physical significance. The reason for the inaccuracy of structure determination of felsőbányaite and basaluminite is, that the X-ray beam generates a rapid dehydration of the crystals and the structures begin to collaps and change during the measurements. This behaviour is typical for other hydrated sulphate minerals also and explains the lack of high quality X-ray powder data and structure determinations for most of these sulphate minerals.

DISCUSSION

In the felsőbányaite (=basaluminite) structure each of the eight crystallographically different AI atoms are coordinated to six O atom neighbours, forming slight distorted AlO₆ octahedra. The relatively high inaccuracy of the single Al-O distances and the resulting standard deviations are consequences of the insufficient quality of the investigated crystal caused by decomposition during X-ray experiments (cf. "Experimental", this article). The mean Al-O values within a range from 1.89 Å to 1.97 Å are comparable to those measured e.g. in the Al(OH)₃ polymorphs with structures built up by AlO₆ octahedra, too. The following values were determined: 1.94 Å in nordstrandite (BOSMANS, 1970), 1.90 Å and 1.91 Å in gibbsite (SAALFELD and WEDDE, 1974), and 1.90 Å (2x) in bayerite (ZIGAN et al. 1978).

The structures of these three Al(OH)₃ modifications are typical layer structures built up by AlO₆ octahedra arranged in sheets, and with a ratio Al:O = 1:3. Three common oxygen edges are characteristic of each octahedron. The ratio Al:O in the comparable AlO₆ octahedra sheets of the title compound is 8:22. It follows from this ratio, that two of the 22 oxygen atoms of the complex Al₈O₂₂ layer are bonded to one Al atom only. The bond valence calculations for the oxygen atoms, bonded to Al atoms (cf. Table 6) confirm, that the atoms O(66) and O(83) in the notation by FARKAS (1980) with v₁ values < 0.6 are O atoms of water molecules. An Al₈O₂₂ layer of this type is (to the best knowledge of the authors) unique in crystal chemistry. The mean Al-Al-distances in the layers of the Al(OH)₃ polymorphs differ slightly from 2.89 Å (bayerite) to 2.92 Å (gibbsite). Felsőbányaite shows mean values from 2.82 Å to 3.13 Å.

TABLE 6

Atom	Vi Vi	Atom	Vi
O(11)	1.121	O(33)	1.457
O(12)	0.877	O(34)	1.460
O(13)	1.338	O(35)	1.106
O(14)	1.229	O(43)	1.011
O(15)	1.204	O(44)	1.284
O(16)	1.086	O(53)	1.201
O(22)	1.168	O(54)	0.748
O(23)	1.038	O(66)	0.458
O(24)	0.844	O(73)	1.255
O(26)	1.011	O(74)	1.148
O(31)	1.042	O(83)	0.568

Bond valences (V_i) for the aluminium but not sulfur bonded oxygen atoms in felsőbányaite. The bond valence parameter R_i for Al-O in equation V_i = exp {(R_i -d_i)/0.37] is 1.651 (cf. BRESE and O'KEEFFE, 1991)

The connection of the AlO_6 -octahedra – distorted hexagonal nets – determined in bayerite, gibbsite, and nordstrandite together with the individual identity periods are drawn in projections onto more or less the best plane of these layers (cf. *Fig. 1*). *Fig. 2* represents the arrangement of the AlO_6 octahedra in felsőbányaite, which is quite different from the distorted hexagonal nets. The structures of boehmite (CRISTOPH et al., 1979) and diaspore (HILL, 1979) both with formula AlO(OH) are not discussed here because all the individual octahedra are interconnected by more than three common O atom edges.





а

b



Fig. 1. The three Al(OH)₃ polymorphs in a simplified graph showing the interconnection of the AlO₆ octahredra and the individual identity periods. a) bayerite; b) gibbsite; c) nordstrandite.

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Fig. 2. The arrangement of AIO₆ octahedra via common oxygen corners and edges to Al₈O₂₂ layers. A projection onto (100).



Fig. 3. The structure of felsőbányaite in a mixed technique projected parallel to [010] showing the Al₈O₂₂ layers as well as the SO₄ tetrahedra and H₂O molecules (of which the O atoms are drawn as single balls).

Whereas in the Al(OH)₃ polymorphs the layers are interconnected by hydrogen atoms with corresponding hydrogen bridges between adjacent layers, the motiv of layer interconnection in felsőbányaite or basaluminite is very complex. H₂O molecules and SO₄ groups are located between the individual layers, which are only bonded to each other by H atoms of hydrogen bridges. No clear system of bridges could be established, because it was not possible to determine the H atom positions by structure calculations. The Al₈O₂₂-sheets, the arrangement of the isolated SO₄ tetrahedra and the O atoms of the H₂O molecules are visualized in a projection onto (010) in *Fig. 3*.

CONCLUSION

The identity of felsőbányaite and basaluminite as already assumed by PAPP and WEISZBURG (1989) and WEISZBURG and PAPP (1990) has been established. The identity of the two minerals based on the similarities in their morphology, physical properties (including optical and IR spectroscopical data) as well as the same chemical composition has been confirmed by the present single crystal structure determination and comparison on the structural data. Since the description of the mineral felsőbányaite given by KRENNER (1928) is sufficient for the mineral samples has also been established, the name basaluminite is to be discredited (or used only as a synonym for microcristalline felsőbányaite). The original name felsőbányaite is to be regarded as correct for the basic aluminium sulfate mineral of composition $Al_4(SO_4)(OH)_{10}.4H_2O$.

ACKNOWLEDGEMENT

One of the authors (F. L.) thank Dr. P.-E. WERNER, Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, for discussion and many helpful comments.

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Manuscript received 26 June, 1997