

THE CRYSTAL STRUCTURE OF BLATTERITE, $\text{Sb}^{5+}_3(\text{Mn}^{3+}, \text{Fe}^{3+})_9(\text{Mn}^{2+}, \text{Mg})_{35}(\text{BO}_3)_{16}\text{O}_{32}$,
AND STRUCTURAL HIERARCHY IN THE Mn^{3+} -BEARING ZIGZAG BORATES

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ABSTRACT

The crystal structure of blatterite, $\text{Sb}^{5+}_3(\text{Mn}^{3+}, \text{Fe}^{3+})_9(\text{Mn}^{2+}, \text{Mg})_{35}(\text{BO}_3)_{16}\text{O}_{32}$, orthorhombic, a 37.654(8), b 12.615(3), c 6.2472(8) Å, V 2968(1) Å³, space group $Pn\bar{m}$, $Z = 2$, $D(\text{calc.}) = 4.25 \text{ g.cm}^{-3}$, has been solved by direct methods and refined to an R index of 4.3% based on 2713 observed reflections measured with $\text{MoK}\alpha$ X-radiation. The crystal used for the collection of the X-ray intensity data was analyzed by electron microprobe; the resulting composition agrees with that derived from the crystal-structure analysis. This has led to a major revision of the chemical formula. There are 31 crystallographically distinct cations in blatterite: two Sb -sites occupied by octahedrally coordinated Sb^{5+} , five Mn -sites occupied by octahedrally coordinated Mn^{3+} , nine M -sites occupied by octahedrally coordinated $(\text{Mn}^{2+}, \text{Mg})$, eight B -sites occupied by triangularly coordinated B, and seven X -sites occupied by $(\text{Mn}^{2+}, \text{Q})$; five of the X sites are octahedrally coordinated, and two of the X sites are tetrahedrally coordinated. Blatterite is a member of the (3 Å) wallpaper-borate structures in which $[\text{MO}_4]$ chains of edge-sharing octahedra extend along the c axis and are cross-linked by (BO_3) groups. Many of the topological features of these structures can be idealized as colorings of the regular net 3^6 . There is a group of Mn^{3+} -bearing structures (fredrikssonite, orthopinakiolite, takéuchiite, blatterite, pinakiolite) that we designate as the *zigzag borates*. As indicated earlier by Takéuchi *et al.* (1978), these can be described as having three important structural motifs: F walls, C walls and S columns. The crystal chemistry of this group is discussed, revised chemical formulae are given for all members of the group, and an explanation is given for the extensive positional disorder associated with the X cations of the C walls in the Mn^{3+} -bearing structures.

Keywords: blatterite, crystal-structure solution, borate, zigzag borates, cation disorder, orthopinakiolite, fredrikssonite, takéuchiite.

THE CRYSTAL STRUCTURE OF TREMBATHITE, $(\text{Mg}_{1.55}\text{Fe}_{1.43}\text{Mn}_{0.02})\text{B}_7\text{O}_{13}\text{Cl}$, A MINERAL OF THE BORACITE GROUP: AN EXAMPLE OF THE INSERTION OF A CLUSTER INTO A THREE-DIMENSIONAL NET

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ABSTRACT

The crystal structure of trembathite, $(\text{Mg}_{1.55}\text{Fe}_{1.43}\text{Mn}_{0.02})\text{B}_7\text{O}_{13}\text{Cl}$, a 8.588(2), c 21.050(6) Å, V 1344.5(1) Å³, was refined in the space group $R3c$ to an R index of 3.2% based on 1472 observed reflections measured with $\text{MoK}\alpha$ X-radiation. Trembathite is a mineral of the boracite group. In contrast to the cubic boracite structure, it contains only three borate tetrahedra connected *via* one oxygen atom. The borate clusters are connected to each other by single borate tetrahedra. We designate the single borate tetrahedra as *topologically active* and the condensed $[\text{B}_4\text{O}]$ or $[\text{B}_3\text{O}]$ groups as *topologically passive*. Connecting the centers of the topologically active borate tetrahedra results in a three-dimensional net in which the nodes are isostructural to the anion positions of the ReO_3 structure-type. The anion position around the central cations in the ReO_3 structure-type form chains of corner-sharing octahedra, which we name as *cs*-chains. The same topology with four condensed octahedra $[\text{M}_4(\text{O},\text{OH})_4]$ ($M = \text{Fe}, \text{Ge}, \text{Mo}, \text{Al}, \text{Ge}$) surrounded by six TO_4 tetrahedra ($T = \text{As}, \text{P}, \text{Ge}$) occurs in the structures of pharmacosiderite, $\text{K}[(\text{FeOH})_4(\text{AsO}_4)_3](\text{H}_2\text{O})_6$. In both cases, the structure-building principle involves the insertion of clusters of polyhedra into the *cs*-chains.

Keywords: trembathite, crystal structure, electron-microprobe analysis, structure topology.

DESCRIPTION OF SCHOENFLIESITE, $\text{MgSn}(\text{OH})_6$, AND ROXBYITE, $\text{Cu}_{1.72}\text{S}$, FROM A 1375 BC SHIPWRECK, AND RIETVELD NEUTRON-DIFFRACTION REFINEMENT OF SYNTHETIC SCHOENFLIESITE, WICKMANITE, $\text{MnSn}(\text{OH})_6$, AND BURITITE, $\text{CaSn}(\text{OH})_6$

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ABSTRACT

Schoenfliesite, $\text{MgSn}(\text{OH})_6$, and roxybite, $\text{Cu}_{1.72}\text{S}$, have been identified as products of corrosion of a 1375 BC bronze harpoon head from a shipwreck in the eastern Mediterranean Sea. The atomic structures of isostructural, synthetic schoenfliesite, burtite [$\text{CaSn}(\text{OH})_6$] and wickmanite [$\text{MnSn}(\text{OH})_6$] have been studied by neutron powder diffraction at 4 K. The results show that the hydrogen atoms are disordered over two positions in these cubic minerals. The bond lengths in the $\text{Sn}(\text{OH})_6$ octahedra and polyhedron volumes for the three compounds are similar, and the octahedra exhibit a systematic change in angle variance. The bond lengths in the $M(\text{OH})_6$ octahedra and polyhedron volumes change systematically with the increase in radius of the M cation.

Keywords: schoenfliesite, roxybite, wickmanite, burtite, crystal structure, hydrogen bonding, hydrogen disorder, shipwreck, eastern Mediterranean Sea.

BERNALITE FROM THE CLARA MINE, GERMANY, AND THE INCORPORATION OF TUNGSTEN IN MINERALS CONTAINING FERRIC IRON

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ABSTRACT

The very rare mineral bernalite is a ferric iron hydroxide with a structure related to that of perovskite. The present description of only the second known occurrence focusses on two samples from the Clara mine, central Black Forest, Germany. X-ray powder diffraction, scanning electron microscopy, and optical microscopy are used to characterize the mineral. The bernalite is polysynthetically twinned, with the twinning seen both microscopically, in the case of apparently “single” crystals, and macroscopically, in crystallographically oriented intergrowths of crystals. The samples investigated contain small amounts of tungsten and trace amounts of silicon. Crystal-chemical considerations suggest that W⁶⁺ ions in bernalite and in other ferric iron minerals substitute for Fe³⁺ ions.

Keywords: bernalite, occurrence, twinning, crystal chemistry, tungsten, ferric iron, Clara mine, Germany.

RAMAN AND INFRARED SPECTRA OF PHASE E: A PLAUSIBLE HYDROUS PHASE IN THE MANTLE

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ABSTRACT

Micro-Raman and micro-infrared spectra of the silicate and principal OH-stretching regions have been obtained from a well-characterized crystal of phase E ($\text{Mg}_{2.17}\text{Si}_{1.01}\text{H}_{3.62}\text{O}_6$) containing approximately 18 wt.% H_2O . The number of observed infrared and Raman bands exceeds the number predicted by factor-group analysis for a crystal of phase E with space group $R3m$. This finding suggests that some long-range order may exist in phase E, or that it possesses a superstructure that lowers the overall symmetry of the crystal. Although phase E contains layers of brucite-type units linked by SiO_4 tetrahedra and MgO_6 octahedra, the lower frequencies and the broadness of the OH-stretching bands of phase E (in comparison to brucite) indicate that the hydrogen bonding is stronger than that in brucite. There is a close similarity between the Raman spectra of phase E and forsterite below 1000 cm^{-1} . This may be explained by assuming that the vibrations of the isolated SiO_4 tetrahedra dominate the Raman spectrum of phase E below 1000 cm^{-1} . Tentative assignments of the observed bands are made by comparison with computed spectral modes and the corresponding spectra of brucite and forsterite.

Keywords: hydrous magnesium silicate, phase E, micro-Fourier-transform infrared spectroscopy, micro-Raman spectroscopy, mantle.

DISTINCTION OF JAROSITE-GROUP COMPOUNDS BY RAMAN SPECTROSCOPY

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ABSTRACT

Raman spectra (200–1300 cm⁻¹) were measured for synthesized jarosite-group compounds [$M\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6$, $M^+ = \text{K}^+, \text{NH}_4^+, \text{Na}^+, \text{Ag}^+$, and $\frac{1}{2}\text{Pb}^{2+}$]. The Raman spectra of jarosite-group compounds are characterized by a tendency for the wavenumbers assigned to two vibrational modes of SO_4^{2-} , $\nu_1(\text{SO}_4^{2-})$ and $\nu_3(\text{SO}_4^{2-})$, and three vibrational modes of Fe–O bonds to decrease with increase in the c unit-cell parameter. The wavenumbers assigned to the $\nu_2(\text{SO}_4^{2-})$ and $\nu_4(\text{SO}_4^{2-})$ vibrational modes are independent of the value of c . For plumbojarosite, the peaks corresponding to the $\text{nn}_1(\text{SO}_4^{2-})$ and $\text{nn}_3(\text{SO}_4^{2-})$ vibrational modes are broad owing to two overlapping peaks assigned to two types of sulfate groups, SO_4^{2-} ions adjacent and not adjacent to Pb^{2+} ions. Raman spectra can serve to identify the specific type of jarosite-group compound in poorly crystalline or low-concentration geochemical samples.

Keywords: jarosite-group compounds, Raman spectra, monovalent cations, vibrational modes, sulfate, unit-cell parameters.

CONTRASTING PATTERNS OF ⁶Al ORDER IN SYNTHETIC PARGASITE AND Co-SUBSTITUTED PARGASITE

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ABSTRACT

Infrared spectroscopy in the principal OH-stretching region and Rietveld refinement indicate that ⁶Al is randomly distributed over the *M*(2) and *M*(3) sites in pargasite, whereas ⁶Al is strongly ordered at *M*(2) in Co-substituted pargasite. The introduction of Co results in an overall expansion of the structure. However, comparison with unit-cell data of previously reported synthetic pargasite and ferropargasite reveals a marked decrease of the *b* dimension in Co-substituted pargasite; this is a direct result of the different patterns of order of ⁶Al in the amphiboles being compared. The degrees of ⁶Al disorder observed in synthetic pargasite and Co-substituted pargasite are compatible with the range of disorder observed in analogous natural amphiboles, and are strongly related to the mean radius of the octahedrally coordinated divalent cations.

Keywords: pargasite, synthesis, Rietveld refinement, infrared spectroscopy, cobalt, order–disorder.

SYNTHETIC FLUORO-AMPHIBOLES: SITE PREFERENCES OF Al, Ga, Sc AND INDUCTIVE EFFECTS ON MEAN BOND-LENGTHS OF OCTAHEDRA

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ABSTRACT

The crystal structure and site populations of four synthetic amphiboles of nominal M^{3+} -substituted fluoro-magnesiokatophorite ($\text{NaCaNaMg}_4M^{3+}\text{Si}_7\text{AlO}_{22}\text{F}_2$, $M^{3+} = \text{Al, Ga, Sc}$) and Ga-substituted fluoropargasite ($\text{NaCa}_2\text{Mg}_4\text{GaSi}_6\text{Al}_2\text{O}_{22}\text{F}_2$) compositions have been refined to R indices of 1–3% using intensity data collected with MoK α X-radiation. The crystals used in the collection of the X-ray data were subsequently analyzed with an electron microprobe. Both techniques confirm significant deviations from the expected stoichiometry. Octahedrally coordinated trivalent cations are totally ordered at the $M(2)$ site. In the Ga-bearing crystals, Ga and Al occur in both octahedral and tetrahedral coordination (*i.e.*, there is Ga–Al disorder between the M and T sites); in Ga-bearing fluoro-magnesiokatophorite, tetrahedrally coordinated Ga and Al are completely ordered at the $T(1)$ site, whereas in Ga-bearing fluoropargasite, Ga is partly disordered over $T(1)$ and $T(2)$, and Al is ordered at $T(1)$. Therefore, only the fraction of Ga occurring in octahedral coordination in amphiboles should be used to calculate partition coefficients to be compared to those of the other $^{61}M^{3+}$ lithophile elements of interest in geochemical studies. In the M^{3+} -substituted fluoro-magnesiokatophorite crystals, ^ANa is ordered at the $A(m)$ site, whereas in Ga-substituted fluoropargasite, ^ANa and ^ACa are disordered between $A(m)$ and $A(2)$, in accord with the model of Hawthorne *et al.* (1996). Examination of the available data on synthetic fluoro-amphiboles shows that there are significant and systematic variations in $\langle \text{Mg–O,F} \rangle$ both at $M(1)$ and $M(3)$ as a function of bulk composition. These variations can be rationalized as structural strain in response to misfit between the strip of octahedra and the chain of tetrahedra.

Keywords: amphibole, fluoro-magnesiokatophorite, fluoropargasite, synthesis, crystal structure, cation order.

THE ROLE OF Ti IN HYDROGEN-DEFICIENT AMPHIBOLES: SODIC–CALCIC AND SODIC AMPHIBOLES FROM COYOTE PEAK, CALIFORNIA

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ABSTRACT

The crystal structures of eleven sodic–calcic and sodic amphiboles from lithic-wacke inclusions in the alkali ultramafic diatreme at Coyote Peak, Humboldt County, California, have been refined to *R* values of 1–2% using single-crystal MoK α X-ray data. The crystals used in the collection of the intensity data were subsequently analyzed by electron- and ion-microprobe techniques. They were analyzed for H and Li, and the unit formulae were calculated on the basis of 24(O,OH,F). Site populations were assigned from the results of site-scattering refinement and stereochemical analysis, taking into account the unit formula determined for each crystal. These amphiboles range in composition from fluororichterite and fluorian eckermannite through titanian fluorian potassic-richterite, titanian fluorian richterite and titanian fluororichterite to titanian oxygenian arfvedsonite; Ti contents range from 0.12 to 0.77 *apfu*, and (OH + F) contents range from 2.00 to 0.72 *apfu*. Where Ti \leq 0.13 *apfu*, (OH + F) \approx 2.0 *apfu*. Where Ti > 0.13 *apfu*, Ti varies linearly with the amount of O²⁻ at the O(3) site [= 2 – (OH + F)], with a slope of 0.52. Thus Ti is incorporated into these amphiboles *via* the substitution Ti⁴⁺ + 2 O²⁻ \rightarrow (Mg,Fe²⁺) + 2 (OH)⁻. Variation in *M*(1)–O(3), *M*(3)–O(3) and *M*(1)–*M*(1) distances as a function of Ti content of the amphibole indicates that all Ti in excess of 0.13 *apfu* occurs at the *M*(1) site, where it is associated with a short *M*(1)–O(3) distance; Ti up to 0.13 *apfu* occurs at the *M*(2) site. The presence of O²⁻ at the O(3) site in these amphiboles induces a range of values of both the O(5)–O(6)–O(7) angle, and the difference between the *M*(4)–O(5) and *M*(4)–O(6) bond-lengths not found in normal [O(3) = OH,F] amphiboles. Some features of the formula derived from electron-microprobe compositions can be used as diagnostic indicators of the presence of extensive ⁶Li and O²⁻ substitutions in monoclinic amphiboles.

INTERMEDIATE SCAPOLITE: ^{29}Si MAS AND ^{27}Al SATRAS NMR SPECTROSCOPY AND RIETVELD STRUCTURE-REFINEMENT

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ABSTRACT

The crystal structures of five intermediate members of the marialite (Ma) – meionite (Me) solid-solution series, with compositions ranging from Me_{15} to Me_{65} ($3.66 \leq \text{Al} \leq 4.53 \text{ apfu}$), were examined using NMR spectroscopy and the Rietveld method of structure refinement (XRD data). ^{27}Al SATRAS and ^{29}Si MAS NMR spectra indicate that Al enters the $T(1)$ site only where Al exceeds 4 apfu , in agreement with the structure refinement. High-resolution ^{29}Si spectra demonstrate a well-ordered structure for those samples of scapolite with 4 apfu Al; however, Al–Si disorder is introduced and Al–O–Al bonds are formed as the Al content becomes greater or lesser than 4 apfu . ^{27}Al spectra show that there is a difference in symmetry of the $T(2)$ and $T(3)$ sites that can be related to the adjacent alkali site containing either Ca or Na and the anion site containing Cl or CO_3 . For the five scapolite samples, X-ray powder-diffraction data were analyzed in both space groups $I4/m$ and $P4_2/n$, to find the most adequate model of the structure. Average interatomic distances for the tetrahedral sites indicate a difference between $T(2)$ and $T(3)$ for all the samples, except PAM–4. The number of weak reflections violating body-centered symmetry is constant for all the samples, except for PAM–4, which does not contain odd reflections at all. Space group $I4/m$ thus is indicated for sample PAM–4, and space group $P4_2/n$ is indicated for samples TANZ, PAM–5, MAD, and MIN. There is a linear increase in the unit-cell dimension a from 12.04 to 12.12 Å, although c remains almost constant. The increase is closely related to the flexibility of $T\text{--O--}T$ angles parallel to a .

Keywords: scapolite, marialite, meionite, ^{27}Al SATRAS NMR, ^{29}Si MAS NMR, Rietveld refinement, X-ray diffraction.

CARBONATE GROUPS IN DAVYNE: STRUCTURAL AND CRYSTAL-CHEMICAL CONSIDERATIONS

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ABSTRACT

We have refined the crystal structure of a specimen of davyne from Mt. Vesuvius, Italy, characterized by the presence of a significant amount of carbonate. The crystals have $P6_3$ symmetry, with cell parameters a 12.6916(9), c 5.3333(5) Å. The chemical formula, obtained through electron-microprobe analysis, is $(\text{Na}_{4.58}\text{K}_{0.67}\text{Ca}_{2.56})(\text{Si}_{6.06}\text{Al}_{5.94}\text{O}_{23.94})\text{Cl}_{2.35}(\text{CO}_3)_{0.88}(\text{SO}_4)_{0.19}$. According to its structural and chemical features, it may be classified as carbonate-bearing davyne, as it represents an intermediate phase between davyne *sensu stricto* and a hypothetical CO_3 end-member. The carbonate groups are located inside the large channel as in cancrinite *sensu stricto*; their position is influenced by the presence of a limited number of SO_4 groups. In fact, the triplets of oxygen atoms of the carbonate groups may alternatively be involved in the tetrahedral coordination of SO_4 . Because of the short C–C contacts, a deviation from stoichiometry may be expected. This fact, as well as a deficiency of cations, was observed in the structural refinement and confirmed by the electron- microprobe data.

Keywords: CO_3 -bearing davyne, cancrinite-group, feldspathoid, structure refinement.

THOMASCLARKITE-(Y), A NEW SODIUM – RARE-EARTH-ELEMENT BICARBONATE MINERAL SPECIES FROM MONT SAINT-HILAIRE, QUEBEC

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ABSTRACT

Thomasclarkite-(Y), ideally $\text{Na}(\text{Y},\text{REE})(\text{HCO}_3)(\text{OH})_3 \cdot 4\text{H}_2\text{O}$, is a newly identified mineral species from Mont Saint-Hilaire, Quebec. It occurs as individual, well-formed, blocky, white-to-yellow crystals up to 2 mm in length. Associated minerals include aegirine, albite, analcime, ancylite-(Ce), astrophyllite, calcite, catapleiite, dawsonite, elpidite, epididymite, franconite, gaidonnayite, galena, genthelvite, microcline, molybdenite, natrolite, petersenite-(Ce), polythionite, pyrochlore, rhodochrosite, rutile, sérandite, siderite, sphalerite, zircon and an unidentified NaREE carbonate. The transparent to translucent mineral has a vitreous luster and a white streak. It is soft (Mohs hardness 2–3) and brittle, with an uneven fracture, a perfect {010} cleavage and good {101} parting. Merohedral twinning on {101} is ubiquitous. Thomasclarkite-(Y) is pseudo-uniaxial negative, $\omega = 1.540(4)$ and $\epsilon = 1.40(2)$. It is monoclinic, space group $P2$, a 4.566(1), b 13.018(6), c 4.566(2) Å, β 90.15(3)°, V 270.2(2) Å³ and $Z = 1$. The strongest X-ray powder-diffraction lines [d in Å(hkl)] are 12.97(10)(010), 6.52(3)(020), 4.57(3)(100, 001), 4.32(5)(110, 011), 3.223(3)(101, 101), 3.133(5)(111, 111), 2.593(3)(131, 131), 2.035(3)(201, 102), 2.016 (4)(230, 032) and 1.844(3)(231, 132). The infrared spectrum is given. Two electron-microprobe analyses and a thermogravimetric analysis gave Na_2O 6.72 (6.20–7.24), CaO 0.33 (0.35–0.32), Y_2O_3 14.52 (14.97–14.07), La_2O_3 8.63 (8.54–8.72), Ce_2O_3 17.11 (18.44–15.79), Pr_2O_3 1.92 (1.94–1.90), Nd_2O_3 5.63 (5.60–5.66), Sm_2O_3 1.22 (1.27–1.17), Gd_2O_3 1.22 (1.13–1.31), Dy_2O_3 2.04 (2.08–2.00), Er_2O_3 2.20 (2.20–2.20), Yb_2O_3 1.32 (1.45–1.19), ThO_2 0.54 (0.48–0.60), F 0.17 (0.35–0.00), CO_2 12.00 and H_2O 26.60, $\text{O} = \text{F} - 0.07$, total 102.10 wt.%. The empirical formula, based on 10 anions and one atom of C, is $(\text{Na}_{0.80}\text{Ce}_{0.18}\text{Ca}_{0.02})_{\Sigma 1.00}(\text{Y}_{0.48}\text{La}_{0.20}\text{Ce}_{0.21}\text{Pr}_{0.04}\text{Nd}_{0.12}\text{Sm}_{0.03}\text{Gd}_{0.03}\text{Dy}_{0.04}\text{Er}_{0.04}\text{Yb}_{0.03})_{\Sigma 1.22}(\text{HCO}_3)[\text{O}_{6.97}\text{F}_{0.03}]\text{H}_{9.93}$. $D_{\text{calc.}} = 2.34 \text{ g/cm}^3$ and $D_{\text{meas.}} = 2.30(2) \text{ g/cm}^3$. The structure has been refined to $R = 0.055$ using a twinned crystal. The structure is layered on {010}. One layer consists of $[\text{HCO}_3]^-$ groups oriented perpendicular to (010) and H-bonded to a H_2O layer. A second layer consists of $(\text{Y}\phi_8)$ polyhedra and $(\text{Na}\phi_6)$ polyhedra which are H-bonded to the H_2O layer. In other bicarbonate minerals, the H-bonding is between the CO_3 groups forming chains, whereas in thomasclarkite-(Y), the H-bonding is directed toward the H_2O groups.

Keywords: thomasclarkite-(Y), new mineral species, bicarbonate, crystal structure, twinning, Mont Saint-Hilaire, Quebec.

WILUITE, $\text{Ca}_{19}(\text{Al,Mg,Fe,Ti})_{13}(\text{B,Al,G})_5\text{Si}_{18}\text{O}_{68}(\text{O,OH})_{10}$, A NEW MINERAL SPECIES
ISOSTRUCTURAL WITH VESUVIANITE, FROM THE SAKHA REPUBLIC, RUSSIAN
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ABSTRACT

Wiluite, $\text{Ca}_{19}(\text{Al,Mg,Fe,Ti})_{13}(\text{B,Al,G})_5\text{Si}_{18}\text{O}_{68}(\text{OH,F,O})_{10}$, tetragonal, a 15.752(1), c 11.717(1) Å, V 2907.3(3) Å³, space group $P4/nnc$, $Z = 2$, is a new mineral species of the vesuvianite group from the Wilui River, Yakutia, Russia. It occurs as holosymmetric crystals with forms {100} and {101} dominant plus minor {110} and {001}, up to $3 \times 3 \times 5$ cm, slightly elongate along [001]. No twinning was observed. Wiluite is dark green with a colorless streak, a vitreous luster, and it does not fluoresce under long- or short-wave ultraviolet light. It has a Mohs hardness of 6, is brittle with an irregular fracture, and has poor cleavage on {100}. The measured density is 3.36(3) g/cm³, and the calculated density is 3.358 g/cm³. In transmitted light, wiluite is colorless and nonpleochroic; it is uniaxial positive with $\omega = 1.721(2)$ and $\epsilon = 1.725(2)$. The strongest ten reflections in the X-ray powder diffraction pattern [$d(\text{in } \text{Å})(I)(hkl)$] are 2.776(100)(342), 2.617(61)(252), 2.491(61)(260), 2.592(43)(224), 1.66(26)(346), 1.640(23)(292), 2.121(20)(135), 3.52(18)(240), 1.987(15)(561), 2.929(14)(004), and 2.912(14)(233). An analysis of wiluite gave SiO₂ 36.11, Al₂O₃ 12.03, TiO₂ 0.80, MgO 6.48, FeO 1.21, Fe₂O₃ 2.28, CaO 35.54, Ce₂O₃ 0.18, B₂O₃ 3.06, F 0.50, H₂O 0.61, O≡ F -0.24, sum = 99.20 wt.%. The concentration of FeO was determined by wet chemistry, and that of H₂O was determined with a LECO induction furnace; the concentration of the other elements was determined by electron-microprobe analysis. The corresponding unit formula, based on 19(Ca + Ce), is $(\text{Ca}_{18.97}\text{Ce}_{0.03})_{\Sigma 19}(\text{Al}_{6.53}\text{Ti}_{0.30}\text{Fe}_{0.86}^{3+}\text{Mg}_{4.81}\text{Fe}_{0.50}^{2+})_{\Sigma 13}(\text{B}_{2.63}\text{Al}_{0.53}\text{G}_{1.84})_{\Sigma 5}\text{Si}_{17.99}\text{O}_{68}[(\text{OH})_{2.03}\text{O}_{7.97}]_{\Sigma 10}$. Wiluite is isostructural with vesuvianite, and contains essential B [$B > 2.5$ apfu (atoms per formula unit)]; vesuvianite is uniaxial (-), wiluite is uniaxial (+). It occurs with grossular and serpentine minerals in a serpentinite. The name is for the locality, and has been in use as a varietal name for “vesuvianite” from this locality for over 200 years.

REFINEMENT OF THE CRYSTAL STRUCTURE OF TIENSHANITE: SHORT-RANGE-ORDER CONSTRAINTS ON CHEMICAL COMPOSITION

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ABSTRACT

The crystal structure of tienshanite, hexagonal, a 16.785(2), c 10.454(1) Å, V 2550.7(6) Å³, $P6/m$, has been refined to an R index of 2.2% for 2353 observed (5σ) reflections measured with MoK α X-radiation. The crystal used in the collection of the X-ray intensity data was subsequently analyzed using an electron microprobe in wavelength-dispersion mode. Site populations were assigned on the basis of the refined site-scattering values, the formula unit calculated from the electron-microprobe data, and the mean bond-lengths. There is significant positional disorder at the Ti site, which is occupied by Ti^{4+} and Nb^{5+} . The refined structure results in a revised chemical formula for tienshanite: $K Na_3 (Na, K, G)_6 (Ca, Y, RE)_2 Ba_6 (Mn^{2+}, Fe^{2+}, Zn, Ti)_6 (Ti, Nb)_6 Si_{36} B_{12} O_{114} [O_{5.5}(OH, F)_{3.5}] F_2$. Formulae can be written for the Ti^{4+} - and Nb^{5+} -bearing end-members. However, local bond-valence requirements impose certain compositional constraints associated with local order involving (Ti^{4+}, Nb^{5+}) and [(OH, F), O]. Thus tienshanite compositions can extend from $Ti^{4+}_6 \rightarrow Ti^{4+}_3 Nb^{5+}_3$.

Keywords: tienshanite, crystal structure, chemical formula, electron-microprobe analysis.

THE CRYSTAL STRUCTURE OF VUONNEMITE, $\text{Na}_{11}\text{Ti}^{4+}\text{Nb}_2(\text{Si}_2\text{O}_7)_2(\text{PO}_4)_2\text{O}_3(\text{F},\text{OH})$, A PHOSPHATE-BEARING SOROSILICATE OF THE LOMONOSOVITE GROUP

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ABSTRACT

The crystal structure of vuonnemite, $\text{Na}_{11}\text{Ti}^{4+}\text{Nb}_2(\text{Si}_2\text{O}_7)_2(\text{PO}_4)_2\text{O}_3(\text{F},\text{OH})$ from Ilímaussaq, Greenland, a 5.4984(6), b 7.161(1), c 14.450(2) Å, α 92.60(1), β 95.30(1), γ 90.60(1)°, V 565.9(1) Å³, and from the Kola Peninsula, Russia, a 5.4970(4), b 7.1630(6), c 14.437(2) Å, α 92.55(1), β 95.30(1), γ 90.61(1)°, V 565.4(1) Å³, has been solved by direct methods in the space group $P\bar{1}$ ($Z = 1$) to a residual R value of ~2%. Vuonnemite is a phosphate-bearing sorosilicate. The structure is layered along [001], and consists of (1) a layer of dominantly edge- and face-sharing coordination polyhedra for Na and P atoms, (2) a layer with corner-linked Si_2O_7 dimers and NbO_6 octahedra, reinforced with $\text{Na}\phi_8$ hexagonal bipyramids *via* edge-sharing, and (3) a closest-packed layer of NaO_6 and TiO_6 octahedra. For all samples of vuonnemite analyzed, the (Mn + Ca) content matches the Ti content in excess of 1.0 Ti *apfu*. The substitution $(\text{Mn} + \text{Ca})^{2+} + \text{Ti}^{4+} \rightleftharpoons \text{Nb}^{5+} + \text{Na}^+$ operates, whereby Ti in excess of 1 *apfu* is disordered onto the Nb site, and (Mn + Ca) populates the Na(2) and Na(3) sites. The compositional range for vuonnemite is now extended from near end-member compositions of $(\text{Mn} + \text{Ca})_0\text{Na}_{11}\text{Ti}_1\text{Nb}_2(\text{Si}_2\text{O}_7)_2(\text{PO}_4)_2\text{O}_3(\text{F},\text{OH})$ to $(\text{Mn} + \text{Ca})_{0.33}\text{Na}_{10.67}\text{Ti}_{1.33}\text{Nb}_{1.67}(\text{Si}_2\text{O}_7)_2(\text{PO}_4)_2\text{O}_3(\text{F},\text{OH})$. Correlation of mineral chemistry, crystal structure and unit-cell parameters allow prediction of many of the structural features of epistolite, the only member of the lomonosovite group for which a successful structure analysis has not been done.

Keywords: vuonnemite, phosphosilicate, crystal structure, lomonosovite group, Ilímaussaq, Greenland, Kola Peninsula, Russia.

GRUMIPLUCITE, A NEW MERCURY – BISMUTH SULFOSALT SPECIES FROM THE LEVIGLIANI MINE, APUAN ALPS, TUSCANY, ITALY

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ABSTRACT

Grumiplucite, ideally HgBi_2S_4 , is a new mercury – bismuth sulfosalt species. It occurs as submillimeter prismatic, grey-black metallic crystals, together with mercury, cinnabar and mercurian sphalerite, in cavities of quartz – carbonate veins in the small mercury deposit of Levigliani, Apuan Alps, Tuscany, Italy. Crystals are elongate according to [010] axis and ubiquitously twinned on (001). Grumiplucite has a monoclinic symmetry, space group $C2/m$, with unit-cell dimensions a 14.164(5), b 4.053(1), c 13.967(3) Å, β 118.28(3)°, V 706.1(6) Å³, $Z = 4$. The strongest five reflections of the X-ray powder-diffraction pattern [d in Å(I)] are: 3.05(s), 2.914(ms), 2.865(ms), 3.86(m), and 3.55(m). Grumiplucite is the natural analogue of the synthetic compound HgBi_2S_4 . In reflected light, it is creamy white (parallel polars) with low birefractance; under crossed polars, the mineral is distinctly anisotropic (bluish grey to yellowish grey). The calculated density is 7.02 g/cm³. It formed at a low temperature in a low- $f(\text{S}_2)$ environment.

Keywords: grumiplucite, new mineral species, mercury – bismuth sulfosalt, Levigliani mine, Apuan Alps, Italy.

AN UNUSUAL ASSEMBLAGE OF HIGH-Ti OXIDES AND FERROAN CLINOCHLORE
ALONG ZONES OF BRITTLE DEFORMATION IN THE VOURINOS (RODIANI)
OPHIOLITE COMPLEX, GREECE

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ABSTRACT

Lenticular bodies of fine-grained light-green chlorite are found at the contact between chromite ore and host serpentinite in the Motsali area of the Vourinos (Rodiani) ophiolite complex, northern Greece. An unusual assemblage of pseudobrookite, the ferrous-iron analogue of pseudobrookite, perovskite, titanite and rutile is present within these bodies. Examination by optical microscopy, electron microprobe, infrared spectroscopy, X-ray fluorescence spectroscopy and thermal analysis showed that the original Fe–Ti oxides (pseudobrookite and possibly titanian hematite) and metamorphic clinochlore were converted to an aggregate of fine-grained chlorite with a similar composition, ferroan clinochlore and the ferrous-iron analogue of pseudobrookite. The ferrous-iron analogue of pseudobrookite and perovskite may be associated with rodingite formation, whereas rutile and pseudobrookite, which indicate advanced oxidation of pre-existing Fe–Ti oxides, formed at an earlier stage. The absence of titanian hematite and the formation of the ferrous-iron analogue of pseudobrookite are due to the removal of Fe, which was consumed by the formation of ferroan clinochlore under reducing conditions. The occupancy of the octahedral sites in ferroan clinochlore is controlled by the $^{IV}AlSi_{-1}$ and the $^{VI}Al(Fe + Mg)_{-1}$ substitutions, expressed by the total R^{2+} content, *i.e.*, it is represented by the exchange vector $Si^{VI}AlG^{IV}Al_{-1}R^{2+}_{-2}$, where **G** represents a vacancy. Chlorite geothermometry indicates that the association ferrous-iron analogue of pseudobrookite + perovskite + ferroan clinochlore formed at 220–250°C. This inference is consistent with the absence of mixed-layer chlorite – smectite and

STABILITY AND COMPOSITION OF K–Ti SILICATES, K–Ba PHOSPHATE AND K–Mg FLUORIDE AT 0.85–2.6 GPa: IMPLICATIONS FOR THE GENESIS OF POTASSIC ALKALINE MAGMAS

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ABSTRACT

The stability and compositions of K–Ti silicate ($K_2TiSi_3O_9$) and similar compounds, K–Ba phosphate [$K_6Mg_3Ba_2Ca(PO_4)_6$], and K–Mg fluoride [$(K,Na)MgF_3$], were determined between 0.85 and 2.57 GPa at 900–1400°C. Starting compositions of $K_4Ti_2Si_7O_{20}$ and $K_4Ti_7Si_{10}O_{36}$, equivalent to the K–Ti silicates stable near the solidus of natural lamproites at higher pressures, produced assemblages consisting of only $K_2TiSi_3O_9$ + rutile \pm SiO_2 . The phase $K_2TiSi_3O_9$ is the Ti analogue of the cyclosilicate wadeite ($K_2ZrSi_3O_9$). The K–Ba phosphate found in this study is of similar composition to that found in experimental studies of lamproites at high pressure, but has not been reported to occur naturally. The phase $KMgF_3$ is similar to that found in experiments in the system potassian richterite – phlogopite – F at 2 GPa, and is the K-analogue of the fluoroperovskite-group mineral neighborite. The relationships of the low-pressure K–Ti silicate formed in this study to those stable at high pressure (4–12 GPa) are discussed. The compounds found in this work appear to be stable in various forms in the lower crust and lithospheric upper mantle and can act as sources for the large-ion lithophile (K,Ba), and first- and second-period transition elements (Ti, Zr, *etc.*) characteristic of potassic alkaline magmas formed from metasomatized sources in the mantle.

Keywords: K–Ti silicate, K–Ba phosphate, fluoroperovskite-group mineral, wadeite, lamproite, upper mantle.

DISTRIBUTION OF GOLD IN TIN-RICH SAMPLES FROM THE CORVO OREBODY, PORTUGAL

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ABSTRACT

The mineralogical distribution of gold in samples from the Corvo orebody, in the Iberian Pyrite Belt of Portugal, was determined in a detailed study of a few hand samples of RT (“rubané tin”) ore. In one sample, 42% of the gold occurs invisibly in pyrite I, pyrite II, and arsenopyrite, with the balance assumed to be carried by cassiterite, which makes up 56.5 wt.% of the sample. Extrapolation of these results to a sample of high-grade tin concentrate (68.99 wt.% Sn) suggests that cassiterite in the concentrate contains about 149 ppb Au, representing 88% of the total gold in the concentrate. These results have implications on the metallogeny of the Neves – Corvo volcanogenic massive sulfide deposit as well as on the metallurgical treatment of the ores.

Keywords: Corvo, Neves – Corvo, volcanogenic massive sulfide, Iberian Pyrite Belt, invisible gold, ore mineralogy, pyrite, arsenopyrite, cassiterite, secondary-ion mass spectrometry (SIMS), image analyses, metallogeny, metallurgy, Portugal.

THE NATURE OF INVISIBLE GOLD IN SULFIDES FROM THE XIANGXI Au–Sb–W ORE DEPOSIT IN NORTHWESTERN HUNAN, PEOPLE’S REPUBLIC OF CHINA

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ABSTRACT

Pyrite from the red-slate-hosted Proterozoic Xiangxi Au–Sb–W deposit in northwestern Hunan Province, China, was studied to determine the nature of the invisible gold that it contains. TEM measurements, confirmed by EDS, reveal that some of the submicrometer gold occurs mainly as 0.07–0.15 μm spheres on growth surfaces of the pyrite and in microfissures in stibnite. Application of the Lamb – Mössbauer effect to results of analyses by ¹⁹⁷Au Mössbauer spectroscopy indicates that 89% of the gold in pyrite crystals occurs in the metallic state, and that 11% is chemically bound. Considerations of crystal chemistry and updated experimental and analytical data on As–Sb sulfide systems, particularly pyrite – arsenopyrite – löllingite, indicate that chemically bound gold may occur in octahedra, comprised most likely of AuAs₂, likely of AuAsS, or less likely of AuS₂, in As-bearing pyrite and arsenopyrite. The valence, structure, and coordination of chemically bound gold in the octahedra are probably intermediate between those in pyrite and in aurostibite.

Keywords: invisible gold, Au-bearing octahedra, sulfides, transmission electron microscopy, ¹⁹⁷Au Mössbauer spectroscopy, Xiangxi, People’s Republic of China.

GLOSSARY OF GEOLOGICAL LOCALITIES IN THE FORMER AUSTRO-HUNGARIAN EMPIRE, NOW IN ROMANIA

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ABSTRACT

This paper gives the Romanian equivalents of geologically relevant historical German and Hungarian locality names in Romania. Finding a locality in eastern Europe, usually within the borders of the former Austro-Hungarian Empire, has in general proved to be a challenge. This listing of equivalents is intended to help mineral collectors and museum curators.

Keywords: Austro-Hungarian Empire, Romania, Transylvania, Banat, type localities.

the observed small size of the chlorite crystals.

Keywords: perovskite, clinocllore, ferrous-iron analogue of pseudobrookite, pseudobrookite, rutile, geothermometer, rodingite, ophiolite, Vourinos (Rodiani), Greece.

Keywords: wiluite, new mineral species, electron-microprobe analysis, X-ray powder-diffraction data, vesuvianite, boron mineral, Sakha Republic, Russian Federation.

Keywords: amphibole, crystal-structure refinement, electron-microprobe analysis, secondary-ion mass spectrometry, hydrogen, titanium, Coyote Peak, California.