

CARROLLITE AND RELATED MINERALS OF THE LINNAEITE GROUP: SOLID SOLUTIONS AND NOMENCLATURE IN THE LIGHT OF NEW DATA FROM THE SIEGERLAND DISTRICT, GERMANY

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ABSTRACT

Carrollite, ideally CuCo_2S_4 , is a major component of "rejuvenation stage" mineralization in the Siegerland ore province, Germany. Carrollite from seven representative deposits has been analyzed. Combining this dataset with published data on carrollite and related minerals, the existence of a solid-solution series in the Cu–Co–Ni–S system, and the mechanisms of substitution responsible, are established. The characteristic feature of carrollite at Siegerland is a pronounced incorporation of Ni in the structure, reaching a maximum of 30.20 wt.% in the Eiserner Spies deposit, corresponding to 1.55 Ni atoms per formula unit (Me_3S_4). Concentrations of Ni display an ideal negative correlation with Co and Cu. Nickel thus apparently substitutes for both Co and Cu over the entire range of compositions represented by our dataset. This solid-solution series extends from end-member carrollite, CuCo_2S_4 , toward what we consider as cuprian siegenite, itself a member of an extensive solid-solution between linnaeite and polydymite. Our data show no evidence for solid-solution series between carrollite and fletcherite. The substitution scheme for the observed linear correlation trends relating Ni, Co and Cu involves at least two different mechanisms of substitution, each operating independently. Two alternative models are proposed: (1) substitution of Ni^{3+} for Co^{3+} into octahedral sites and of Ni^{2+} for Cu^{2+} into tetrahedral sites, and (2) substitution of Ni^{3+} for Co^{3+} into octahedral sites combined with the coupled substitution $\text{Cu}^{2+} + \text{Co}^{3+} \rightarrow \text{Ni}^{3+} + \text{Ni}^{2+}$. Our data suggest that the substitution of Ni^{3+} for Co^{3+} is dominant. Nickelian carrollite contains $\text{Ni}/(\text{Ni} + \text{Co} + \text{Cu}) > 0.05$, up to a maximum halfway along the $\text{CuCo}_2\text{S}_4 - \text{CoNi}_2\text{S}_4$ join at $\text{Cu}_{0.5}\text{Co}_{1.5}\text{Ni}_{1.0}$. Phases with $\text{Ni} > 1.0$ and $\text{Cu} < 0.5$ should be termed cuprian siegenite, so long as $\text{Cu}/(\text{Cu} + \text{Ni} + \text{Co})$ exceeds 0.05.

Keywords: carrollite, siegenite, micro-analytical data, solid solution, nomenclature, Siegerland, Germany.

TELLURIDES FROM THE PALEOPROTEROZOIC OMAI GOLD DEPOSIT, GUIANA SHIELD

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ABSTRACT

The Omai deposit, a Paleoproterozoic gold-bearing vein system, is located in the Barama–Mazaruni Supergroup, a typical greenstone belt in north-central Guyana. The deposit is hosted mainly by a shallow-level quartz-monzodioritic stock (Omai stock), quartz-feldspar porphyry and rhyolite dykes, and, to a lesser extent, by adjacent mafic volcanic and sedimentary rocks. In the Omai stock, the gold-bearing quartz veins show an uniform pattern, generally striking 030° and dipping at 20–30°. The veins hosted by the quartz–feldspar porphyries and rhyolites are stockworks. The metallic minerals (<2% of the vein volume) have a complex mineralogy consisting of various sulfides, associated with native elements, tungstates, tellurides, and sulphosalts. Telluride minerals are petzite, calaverite, hessite, tellurobismuthite, altaite, melonite, coloradoite, and volynskite. The metal inventory is defined by the Au–Ag–Te–W–Bi–Pb–Zn–Cu–Hg–Mo assemblage, characteristic of both epi- and mesothermal deposits. The gangue includes mainly quartz, with minor amounts of ankerite, calcite, albite, white mica, chlorite, and epidote. The conditions of ore formation at Omai are estimated from the stability of telluride – sulfide – oxide – silicate assemblages. The bulk of ore minerals and gangue precipitated at low temperatures (220° to 170°C), whereas solutions are characterized by relatively narrow ranges in S_2 (10^{-17} to 10^{-11}), Te_2 (10^{-14} to $10^{-10.5}$) and O_2 (10^{-43} to 10^{-36}) fugacities and a weakly acidic pH (between 4 and 5.4). Sulfides were probably transported as sulfide [$Au(HS)_2^-$ and $Au(HS)^0$ or $HAu(HS)_2^0$] or thiosulphate complexes, whereas Te was transported as aqueous H_2TeO_3 , Te_2^{2-} , and HTe^- complexes.

Keywords: Omai gold deposit, Guiana Shield, tellurides, Te_2 , S_2 and O_2 fugacities, pH, electron-microprobe analyses.

THE GUIBARÉ AND FÉTÉ KOLÉ GOLD-BEARING TOURMALINE–QUARTZ VEINS IN
THE BIRIMIAN GREENSTONE BELTS OF BURKINA FASO

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ABSTRACT

The Guibaré and Fété Kolé deposits in Burkina Faso consist of Birimian gold-bearing quartz and tourmaline veins in metamorphosed volcano-sedimentary formations of an Early Proterozoic greenstone belt. Tourmaline compositions for the two prospects vary according to occurrence: dravite in the Guibaré deposit, hosted by metabasic to meta-ultrabasic rocks, in contrast to schorl in the Fété Kolé deposit, hosted by metasedimentary and metavolcanic rocks. The major control on tourmaline composition thus seems to be the $Mg/(Mg + Fe)$ value of the host rock rather than the composition of the hydrothermal fluid. In both deposits, the gold is not associated with sulfides, but rather is concentrated within tourmaline layers, typically at the tips of the tourmaline crystals and as small grains filling fractures in tourmaline. Mössbauer and infrared spectroscopic data show the presence of ferric iron coupled to deprotonation in these tourmalines, indicating a relatively oxidizing environment for the Au deposits. A drop of fluid pressure and deprotonation could have induced physicochemical changes that caused deposition of the gold.

Keywords: tourmaline, gold, electron-microprobe data, Mössbauer data, infrared data, Birimian, Burkina Faso.

PYRITE AND VIVIANITE INTERVALS IN THE BOTTOM SEDIMENTS OF EUTROPHIC BAPTISTE LAKE, ALBERTA

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ABSTRACT

The highly eutrophic status of Baptiste Lake, Alberta, may be related to altered groundwater flow resulting from land-clearing activities since colonial settlement approximately 80 yr B.P. Sections from the top 42 cm of sediment cores from Baptiste Lake, which represent 150 yr B.P. to the present, were analyzed for forms and concentrations of iron and phosphorus. The 30–42 cm interval (90–150 yr B.P.) is marked by intensive formation of vivianite; concentrations of potentially bioavailable orthophosphate ion in some sections exceed 1 wt.%. This probably reflects a period when inflowing groundwater contained high concentrations of iron and phosphorus, which precipitated at the sediment surface under oxic conditions that likely prevailed at the time. In contrast, the top 25 cm (80 yr B.P. to the present) show sedimentary formation of pyrite consistent with anoxic conditions at the sediment–water interface, the deposition of organically enriched sediments, and severe eutrophication. The waters of Baptiste Lake have become more eutrophic since colonial settlement.

Keywords : pyrite, vivianite, lake sediments, phosphorus, groundwater, trophic level, Baptiste Lake, Alberta.

MINERALOGY OF THE ARCHEAN BARITE DEPOSIT OF GHATTIHOSAHALLI,
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ABSTRACT

The mineralogy of the mid-Archean barite deposit in the Ghattihosahalli supracrustal belt in central Karnataka, India, has been comprehensively investigated. The deposit occurs as thin seams closely associated with chromiferous quartzites located in the upper part of a stratigraphic sequence consisting of mafic and ultramafic rocks in the lower portion and predominantly siliceous to argillaceous sediments in the middle. The belt has undergone medium-pressure amphibolite-facies metamorphism. The predominant variety of barite is coarse grained and contains only minor amounts of quartz, pyrite and scarce graphite. In contrast, the fine-grained impure barite, which constitutes a smaller proportion of the deposit, contains a spectrum of Ba–Cr minerals. These include (1) barian-chromian mica showing a significant solid-solution toward the barian end-member $\text{Ba}(\text{Mg}, \text{Fe}^{2+})\text{Al}[\text{AlSi}_3\text{O}_{10}](\text{OH})_2$, (2) K,Ba-feldspar covering the entire solid-solution from K-feldspar to celsian, with a compositional gap between $\text{Or}_{62}\text{Cn}_{30}\text{Ab}_{08}$ and $\text{Or}_{88}\text{Cn}_{08}\text{Ab}_{04}$, (3) chromian dravite (3–19 wt.% Cr_2O_3), (4) epidote containing up to 80 mol.% of the $\text{Cr}^{3+}\text{Al}_2$ end-member, (5) uvarovitic garnet ($\text{Uva}_{61}\text{Grs}_{34} - \text{Uva}_{41}\text{Grs}_{52}$), (6) rutile and titanite with up to 2.8 and 1.6 wt.% Cr_2O_3 , respectively. Mineral compositions vary considerably from one seam to the other; within the limits of individual samples, the variations are largely controlled by the bulk composition of the rock. The stratiform nature and lithological association, together with the mineralogical and chemical characteristics, suggest a mode of formation through submarine volcanic exhalation and precipitation, with a minor interplay of clastic sedimentation.

Keywords: barite deposit, Archean, K,Ba-feldspar, Ba,Cr-bearing minerals, Ghattihosahalli, India.

OCCURRENCE OF EARLY CARBONIFEROUS HIGH-Zr RHYOLITES, COBEQUID HIGHLANDS, NOVA SCOTIA: TEMPERATURE EFFECT OF A CONTEMPORANEOUS MAFIC MAGMA[§]

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ABSTRACT

The Fountain Lake Group of the Cobequid Highlands is a bimodal rhyolite–basalt volcanic unit that was extruded during the earliest Carboniferous along the southern margin of the Maritimes Basin in northern Nova Scotia. In the eastern Cobequid Highlands, up to 4 km of felsic lava flows and pyroclastic deposits are overlain by 1.5 km of continental tholeiitic basalt. A distinctive high-Zr rhyolite (>900 ppm Zr) occurs as ignimbrites near the top of the felsic volcanic sequence, as dykes cutting the main extrusive rhyolite succession (but not younger units), and as linear intrusive bodies along the northern margin of the Wentworth pluton. In the western Cobequid Highlands, the volcanic succession is less than 1 km thick and comprises rhyolite with lesser basalt. Rhyolites have abundances of trace elements (*i.e.*, Zr, Nb, Y, Ga and Rb) typical of A-type, within-plate granitic magma. Older rhyolites have low ϵ_{Nd} (< +1), low Ga/Al and high Th/La, similar to early phases of coeval granite plutons in the region. Low-Zr rhyolites in the west show either a progressive increase in Zr with fractionation or no systematic pattern, whereas those in the east show a slight decrease. Younger high-Zr rhyolites show a rapid increase in Zr with fractionation, and Zr is almost entirely in the groundmass. These high-Zr rhyolites are only slightly enriched in other high field-strength elements and REE compared with the low-Zr rhyolites. Their Nd isotopic composition is close to that of contemporaneous large gabbroic plutons, suggesting that the felsic magma may in part be derived from fractionation of mafic magma. The high Zr content is the result of high-temperature suppression of zircon crystallization by ambient mafic magmas correlative with the thick continental tholeiite

basalts.

Keywords: rhyolite, Carboniferous, Nova Scotia, zirconium.

THE MOBILITY OF ZIRCONIUM AND IDENTIFICATION OF SECONDARY Zr-BEARING PHASES IN BAUXITE FROM POÇOS DE CALDAS, MINAS GERAIS, BRAZIL: A MASS-BALANCE AND X-RAY ABSORPTION SPECTROSCOPIC STUDY

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ABSTRACT

The geochemistry and mineralogy of Zr have been studied in a bauxitic profile derived from a nepheline microsyenite at Poços de Caldas, Minas Gerais, southeastern Brazil. The Zr in the parent rock is hosted by the sorosilicate hainite (5–6% ZrO₂) and a Ca–Na amphibole (0.1–0.2% ZrO₂). Comparison of the variations in Zr, Ti, Nb and Th contents as function of the apparent density of different zones in the top few meters of the saprolite shows Th to be the least mobile element of the profile. Therefore, it was chosen as an invariant constituent in order to evaluate the mobility of other elements, especially the Zr. Mass-balance calculations demonstrate the leaching of Zr all along the profile, with a rate of removal of up to 40% at the weathering front. In the residual bauxitic materials, Zr is essentially localized in ferruginous products such as goethite-rich plasma filling the pores, and granules within the gibbsite-dominant matrix. Its mineralogical expression appears to be within polyhedra with an average of 6.6 O atoms, with average distances Zr–O of 2.16 Å, probably in adsorption on the surface of goethite in the pores and in granules of the gibbsite-rich matrix.

Keywords: bauxite, thorium, mass balance, zirconium, mobilization, X-ray absorption spectroscopy, baddeleyite, Poços de Caldas, Brazil.

HYDROTHERMAL REE-RICH EUDIALYTE FROM THE PILANESBERG COMPLEX, SOUTH AFRICA

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ABSTRACT

The Pilanesberg Complex, in South Africa, one of the world's largest alkaline complexes, contains large resources of Zr and the rare-earth elements (*REE*). Eudialyte is the main carrier of Zr, and contains appreciable concentrations of *REE*. It is particularly abundant in the green nepheline syenite (20% by volume). It forms complexly zoned poikilitic domains that enclose aegirine, albite, microcline, nepheline, zircon, pectolite, sodalite, and an unidentified Na–Zr silicate. These domains mantle corroded silicates and replace outer zones of microcline euhedra. The eudialyte is partially replaced by fergusonite-(Y) and britholite. Compositionally, the eudialyte at Pilanesberg is unusual; it contains the highest concentration of Nb (up to 3.8 wt% Nb₂O₅) and the lowest concentrations of Na (<11.4 wt% Na₂O) and Fe (<0.4 wt% FeO) reported in the literature. In addition, it has one of the highest contents of *REE* (up to 7.6 wt% *REE*₂O₃, mainly Ce and La and traces of Sm and Nd) and Mn (up to 7.5 wt%) reported in the literature. On the basis of 78 anions, its formula is (Na_{12.30}REE_{1.39}Ca_{0.89}K_{0.20})_{14.78}(Ca_{5.42}Mn_{0.58})₆(Mn_{2.96}Fe_{0.04})₃(Zr_{2.66}Nb_{0.28}Hf_{0.06})₃(Nb_{0.66}Si_{0.28}Ta_{0.06})₁Si_{25.64}O₇₄(OH_{1.01}Cl_{0.92}F_{0.07})₂•2H₂O. On the basis of textural relationships, the Pilanesberg eudialyte is interpreted to be hydrothermal. It seems to have formed from an orthomagmatic Na–Nb–*REE*–Cl–F-bearing hydrothermal fluid that exsolved from an agpaitic syenitic magma. In this system, zirconium was probably remobilized from magmatic zircon as a F-complex (*e.g.*, ZrF₆²⁻), and *REE* were introduced as Cl- and F-complexes. The incorporation of the *REE* in eudialyte probably occurred in response to the reduced Cl-, and F-activities that accompanied precipitation of sodalite and the unknown (F-bearing) Na–Zr silicate.

Keywords: eudialyte, nepheline syenite, Pilanesberg, alkaline complex, hydrothermal, rare-earth elements, South Africa.

NIOBIAN CALCOTANTITE AND PLUMBOAN-STANNOAN CESSTIBTANTITE FROM
THE ISLAND OF UTÖ, STOCKHOLM ARCHIPELAGO, SWEDEN

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ABSTRACT

Niobian calciotantite and plumboan-stannoan cesstibtantite are associated with albite, K-feldspar, quartz and pink Ca-enriched tourmaline in the pollucite-bearing Grundberg outcrop of the northern Nyköpinggruvan pegmatite on the island of Utö, Stockholm Archipelago, south-central Sweden. Cesstibtantite forms overgrowths on subhedral, compositionally zoned grains of niobian calciotantite; both phases are locally intergrown with skeletal manganocolumbite, cassiterite and an unknown Ta-rich oxide phase. The maximum Nb content of the (Na,Pb)-poor calciotantite is as high as 19.90 wt.% Nb₂O₅ (1.263 *apfu* Nb, 31.6% of the *B*-site population). The (Pb,Sn,Nb)-enriched cesstibtantite averages at (Pb_{0.35}Sb_{0.25}Sn_{0.10}Ca_{0.12}Na_{0.12}) (Ta_{1.40}Nb_{0.55}W_{0.05}) (O_{5.70}[OH]_{0.30}) ([OH]_{0.66}Cs_{0.30}). Low-temperature hydrothermal fluids triggered a unique two-stage alteration of cesstibtantite, leaving the other minerals intact. Diffuse-cation exchange spreads from microfractures in cesstibtantite. Gradual loss of Cs, Pb, Sb and Sn is compensated by progressive introduction of Ca, Na and Sr, and minor F substitutes for OH, but *B*-site cations are not affected. The resulting compositions attains (Ca_{0.32}Na_{0.25}Sb_{0.25}Sn_{0.03}Pb_{0.20}Sr_{0.02}) (Ta_{1.40}Nb_{0.55}W_{0.05}) (O_{5.85}[OH]_{0.15}) ([OH]_{0.55}O_{0.10}F_{0.10}Cs_{0.15}). Extremely thin rims of the microfractures, 1 to 4 μm across, display much enhanced cation-exchange of the above type, combined with substantial fluorination, which generates secondary cesian microlite (Ca_{0.80}Na_{0.40}Sb_{0.15}Sr_{0.05}Pb_{0.05}Sn_{0.02}) (Ta_{1.40}Nb_{0.55}W_{0.05}) (O_{5.90}[OH]_{0.10}) ([OH]_{0.36}O_{0.15}F_{0.30}Cs_{0.10}).

Keywords: calciotantite, cesstibtantite, microlite, electron-microprobe analysis, tantalum, niobium, cesium, granitic pegmatite, Utö Island, Sweden.

STRONTIOMELANE, $\text{SrMn}^{4+}_6\text{Mn}^{3+}_2\text{O}_{16}$, A NEW MINERAL SPECIES OF THE CRYPTOMELANE GROUP FROM ST. MARCEL – PRABORNA, AOSTA VALLEY, ITALY

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ABSTRACT

Strontiomelane is a new mineral species from the Praborna mine near Saint-Marcel, Aosta Valley, Italian Alps. It occurs as small anhedral inclusions (about 100–200 μm) and veinlets associated with braunite, cryptomelane, hollandite, quartz, fluorapatite, titanite, jadeite and strontio piemontite. The new species is opaque, black with a submetallic luster and a strong grey-white pleochroism; in plane-polarized light in oil immersion, it presents a medium anisotropy with grey-blue to white tints. Strontiomelane, whose ideal formula is $\text{SrMn}^{4+}_6\text{Mn}^{3+}_2\text{O}_{16}$, crystallizes in space group $P2_1/n$ (14) with a 10.00(1), b 5.758(7), c 9.88(1) Å, β 90.64(6)° and $Z = 2$. The strongest lines in the X-ray powder pattern [d in Å(I)(hkl)] are: 3.15(100)($\bar{1}03$), 3.13(80)(103), 2.409(80)(122), 2.170(60)(320) and 1.556(50)($\bar{2}25$). The calculated density is 4.66(1) $\text{g}\cdot\text{cm}^{-3}$. Strontiomelane is the Sr end-member of

the cryptomelane group. The proposed name is derived from the chemical element strontium and the color black, *melas*, - *anos* in Greek.

Keywords: strontiomelane, strontium, manganese oxide, cryptomelane group, Praborna, Alps, Italy.

UVAROVITE IN PODIFORM CHROMITITE: THE MOA–BARACOA OPHIOLITIC MASSIF, CUBA

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ABSTRACT

The chromitite pods of the Moa–Baracoa massif, in the eastern ophiolitic belt of Cuba, contains pre-existing gabbro sills. This association is affected by two processes of hydrothermal alteration. The chromitites and the hosting dunites and harzburgites are affected first by regional serpentinization; a second alteration, represented by chloritization accompanied with formation of ferrian chromite, is mainly located in the pods and their immediate vicinity. The altered chromitite pods and enclosed gabbro sills are cross cut by millimeter-wide veins. The vein filling consists of a sequence of clinocllore, uvarovite, chromian clinocllore, rutile, titanite and calcite. Uvarovite also occurs in the vicinity of veins. Uvarovite is concentrically zoned, covering compositions in the uvarovite–grossular solid solution series between Uva₁₇ and Uva₆₃; the andradite component is very low. These compositions suggest a complete miscibility along the grossular–uvarovite join at relatively low temperature. On the basis of the mineral sequence and mineral chemistry (major and trace elements), the uvarovite crystals, as well as the vein assemblage, formed by a low-temperature leaching, Ca probably from the gabbro sills, and Cr and Al from the chromite during the formation of ferrian chromite. Cr and Al would have been mobile only at the scale of a pod during this process.

Keywords: uvarovite, zoning, hydrothermal, chromitite, ophiolite, Moa–Baracoa massif, Cuba.

STRUCTURAL STATE OF K-FELDSPAR IN SOME HERCYNIAN GRANITES FROM IBERIA: A REVIEW OF DATA AND CONTROLLING FACTORS

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ABSTRACT

Several hundred concentrates of K-feldspar from Hercynian granites of central Portugal (western Iberian Massif) and associated pegmatites and aplites were studied using X-ray powder-diffraction methods, in order to assess their structural state and make inferences concerning petrogenesis. Indicators of structural state include the diffraction pattern in the 2θ region $29\text{--}31^\circ$, obliquity, the ordering index Δ_{SM} and Al–Si distribution derived from unit-cell parameters. In about 96% of the concentrates from granites, the structural state of K-feldspar assumes a range of values, instead of a single end-member value of fully ordered or disordered state; pegmatites and aplites show less variability in structural state than the associated granites. In the great majority of samples from granites, more than 84% of Al is accumulated in the $T1$ site of the K-feldspar. The dataset as a whole, along with some case studies, indicates that the cooling rate of the plutons was a determining factor in controlling the ordering process of K-feldspar; rapid unroofing of the “older” granites enhanced the cooling rate, thus retarding ordering.

Keywords: K-feldspar, structural state, granites, thermal history, Iberian Massif, Portugal.

HIGH-TEMPERATURE X-RAY INVESTIGATION OF SANIDINE – ANALBITE
CRYSTALLINE SOLUTIONS: THERMAL EXPANSION, PHASE TRANSITIONS, AND
VOLUMES OF MIXING

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ABSTRACT

Unit-cell parameters and volumes have been measured for an eleven-member sanidine – analbite series from room temperature to 950°C. Slopes dV/dT and, in turn, coefficients of thermal expansion, vary systematically with composition, as sodic members expand to a greater degree than potassic ones. Volumes of K–Na mixing (V_{ex}) reach maximum values in the sodic part of the compositional range at all temperatures, but decrease in magnitude by about 20% from room to elevated temperature, as the distribution of V_{ex} values also becomes increasingly symmetrical with respect to composition. Transformation from triclinic to monoclinic symmetry occurs above room temperature for the five most sodic samples. Transformation temperature is related linearly to the mole fraction of potassium in the feldspar.

Keywords: volumes of mixing, phase transitions, temperature, alkali feldspars, sanidine, analbite.

CRYSTAL STRUCTURES OF NEAR-END-MEMBER PHLOGOPITE AT HIGH TEMPERATURES AND HEAT-TREATED Fe-RICH PHLOGOPITE: THE INFLUENCE OF THE O,OH,F SITE

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ABSTRACT

The crystal structure of end-member phlogopite-1M from White Well, Australia, was determined by refinement using single-crystal X-ray data to 600°C ($R = 0.069$, $wR = 0.096$ at 600°C). Cell parameters were refined at 20, 150, and thereafter at 50°C intervals to 600°C. The rate of expansion of the metric unit-cell dimensions is linear. The expansivity of the c dimension is $1.81 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$, and 1.40 and $1.34 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$ for the a and b dimensions, respectively (compared to fluorophlogopite in a previous study: 24° to 600°C, $\alpha_a = 0.86$, $\alpha_b = 0.75$, and $\alpha_c = 1.81 \times 10^{-5} \text{ }^\circ\text{C}^{-1}$). The OH-rich octahedra ($M1$, $M2$) expand without changing shape significantly at elevated temperatures, in contrast to F-rich octahedra in fluorophlogopite, which become elongate approximately along the c axis. The interlayer site becomes elongate in phlogopite with increasing temperature relative to fluorophlogopite, although the site shows general flattening with increasing temperature. We also refined the room-temperature structure of a sample of Fe-rich phlogopite-1M sample from Silver Crater, near Bancroft, Ontario, and the same phlogopite after heat treatment at 904°C for 24 hours (untreated: $R = 0.039$, $wR = 0.043$; heat-treated: $R = 0.039$, $wR = 0.047$). In contrast to earlier studies, there was no change in Fe site occupancy from the octahedra to the tetrahedra. The octahedra ($M1$, $M2$) and the interlayer site are flattened in both the heated and unheated samples, but the heated sample shows significant flattening in all these sites over the unheated sample.

Keywords: phlogopite, heat-treated phlogopite, crystal structure.

THE CRYSTAL STRUCTURE OF CHOLOALITE

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ABSTRACT

The crystal structure of choloalite, $(\text{Cu}_{2.79}\text{Sb}_{0.21})_{\Sigma 3.00}(\text{Pb}_{2.70}\text{Ca}_{0.30})_{\Sigma 3.00}\text{Te}_{6.00}\text{O}_{18.00}\text{Cl}_{0.92}$, a 12.520(4) Å, V 1963(2) Å³, space group $P4_132$, $Z = 4$, has been solved by Patterson and direct methods, and refined to an R index of 5.3% based on 956 unique reflections measured using $\text{MoK}\alpha$ radiation on an automated four-circle diffractometer. The structure consists of distorted TeO_6 octahedra, $\text{Cu}\phi_5$ square pyramids (where $\phi = \text{O}$ and Cl), $\text{Pb}(1)\text{O}_9$ triaugmented trigonal prisms, and $\text{Pb}(2)\text{O}_{12}$ icosahedra. The $\text{Pb}(1)\text{O}_9$ polyhedra polymerize to form a three-dimensional network, as do the $\text{Cu}\phi_5$ square pyramids and $\text{Pb}(2)\text{O}_{12}$ polyhedra. The two networks fit together in three-dimensional space, leaving voids that are filled by the TeO_6 octahedra. It is likely that the ideal formula of choloalite is $\text{CuPbTe}^{4+}_2\text{O}_6$, with CuO_4 square planes as opposed to $\text{Cu}\phi_5$ square pyramids.

Keywords: choloalite, crystal structure, tellurite, lead, copper.

BORATE MINERALS. II. A HIERARCHY OF STRUCTURES BASED UPON THE BORATE FUNDAMENTAL BUILDING BLOCK

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ABSTRACT

A hierarchical structural classification is developed for borate minerals, based on the linkage of (BO₃) triangles and (BO₄) tetrahedra to form *FBBs* (fundamental building blocks) that polymerize to form the structural unit, a tightly bonded anionic polyhedral array whose excess charge is balanced by the presence of large low-valence interstitial cations. Thirty-one minerals, with nineteen distinct structure-types, contain isolated borate polyhedra. Twenty-seven minerals, with twenty-five distinct structure-types, contain finite clusters of borate polyhedra. Ten minerals, with ten distinct structure-types, contain chains of borate polyhedra. Fifteen minerals, with thirteen distinct structure-types, contain sheets of borate polyhedra. Fifteen minerals, with thirteen distinct structure-types, contain frameworks of borate polyhedra. It is only the close-packed structures of the isolated-polyhedra class that show significant isotypism.

Keywords: borate, minerals, crystal structures, structural hierarchy.

HYDROGEN-ATOM POSITIONS IN *P4/nnc* VESUVIANITEGEORGE A. LAGER¹

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ABSTRACT

The crystal structure of a large crystal ($11.0 \times 7.8 \times 4.7$ mm) of gem-quality Fe-bearing vesuvianite [a 15.553 (11), c 11.785(16) Å, V 2837(5) Å³; ϵ 1.713(5), ω 1.710(5), $2V$ 0°(5); D 3.43 mg m⁻³;

3.68 wt% FeO] from Kenya has been refined at 298 K by single-crystal neutron-diffraction methods to a weighted R value of 3.98% for 1448 unique Bragg reflections. No violations of $P4/nnc$ space-group symmetry were observed on examination of the crystal by time-of-flight neutron methods. The protons are associated with oxygen atoms O(11) [H(1) 0.4592(7), 0.4814(9), 0.3083(8)] and O(10) [H(2) 0.25, 0.25, 0.280(10)], as predicted by bond-valence calculations. Difference-Fourier maps show a double minimum between adjacent O(10) atoms along c , indicating a statistical distribution of H(2) between two sites. The partial occupancy of the H(1) site (85%) is consistent with the substitution of fluorine for oxygen at the O(11) site. Although hydrogrossular and vesuvianite have similar structures and occur in many of the same environments, the neutron-diffraction results rule out the possibility of a hydrogarnet-type substitution [$(O_4H_4)^{4-} = (SiO_4)^{4-}$] in this specimen. Its infrared spectrum (6500–7500 cm^{-1}) most closely resembles that of intermediate-fluorine, boron-poor vesuvianite.

Keywords: vesuvianite, crystal-structure refinement, neutron diffraction, hydrogen atoms.

THE CRYSTAL STRUCTURE OF DARAPIOSITE AND A COMPARISON WITH Li- AND Zn-BEARING MINERALS OF THE MILARITE GROUP

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ABSTRACT

By using material from a second occurrence of darapiosite in the Dara-i-Pioz alkaline massif, in northern Tajikistan, we have achieved a better characterization of this rare species belonging to the milarite group: a 10.262(2), c 14.307(1) Å, V 1305.0(3) Å³, space group $P6/mcc$, $(\text{Mn}_{1.54}\text{Zr}_{0.30}\text{Y}_{0.23}\text{Mg}_{0.03})_{\Sigma 2.10}(\text{Na}_{1.22}\text{K}_{0.36}\text{G}_{0.42})_{\Sigma 2.00}\text{K}_{1.00}(\text{Li}_{1.53}\text{Zn}_{1.15}\text{Fe}^{2+}_{0.31})_{\Sigma 2.99}(\text{Si}_{11.98}\text{O}_{30.00})$, $Z = 2$, $\rho_{\text{meas}} = 2.93(3)$ g/cm³. The crystal structure has been refined to $R = 0.032$ from single-crystal X-ray data. Darapiosite is a double-ring silicate and is close to dusmatovite in chemical composition; Li dominates at the $T2$ site, which contains also Zn and Fe, and Na is the major cation at the B site. The presence of Li at $T2$ for the Li-bearing members of the milarite group is correlated with the dimensions of the cell parameters and of the coordination polyhedra. The occurrence of Zn at $T2$ further modifies relations between this site and other parts of the structure.

Keywords: darapiosite, crystal structure, milarite group, crystal chemistry.

REDETERMINATION OF THE CRYSTAL STRUCTURE OF HANAWALTITE

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ABSTRACT

The crystal structure of hanawaltite, $\text{Hg}^+\text{Hg}^{2+}\text{O}_3\text{Cl}_2$, a 11.796(4), b 13.890(6), c 6.471(2) Å, V 1060.2(6) Å³, space group $Pbma$, $Z = 4$, has been refined to an R index of 0.049 on the basis of 662 merged, observed reflections. The crystal structure has two distinct layers; a $\text{Hg}^+\text{Cl-O}$ layer consisting of ribbons of $[\text{Hg-Hg}]^{2+}$ dimers resembling the configuration in the calomel structure and a $\text{Hg}^{2+}\text{Hg}^+\text{Cl-O}$ layer with alternating mercurous and mercuric oxychloride chains.

Keywords: hanawaltite, crystal structure, mercurous, mercuric, oxychloride.