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Mountkeithite, a new pyroaurite-related mineral with an expanded interlayer containing exchangeable $MgSO_4$

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ABSTRACT. Mountkeithite $[(Mg,Ni)_9(Fe^{3+},Cr,Al)_3(OH)_{24}]^{3+}[(CO_3,SO_4)_{1.5}(Mg,Ni)_2(SO_4)_2(H_2O)_{11}]^{3-}$ occurs with pyrite, magnetite, magnesite, hexahydrite, and morenosite in a low-temperature hydrothermal vein system in the Mount Keith disseminated nickel sulphide deposit, Western Australia. Electron-microprobe analyses gave the following average composition: MgO 31.7, NiO 6.1, CuO 0.1, Cr_2O_3 6.1, Fe_2O_3 8.3, Al_2O_3 2.6, SO_3 14.7; microanalysis for C and H gave CO_2 3.9, H_2O 30.6; total 104.1. The X-ray powder diffraction pattern has the following strongest lines: 11.30 (10), 5.63 (8), 4.63 (2), 3.765 (6), 2.645 (4), 2.545 (4), 1.554 (5), and 1.505 (3); these can be indexed on a hexagonal cell with $a = 10.698$ Å, $c = 22.545$ Å.

Mountkeithite occurs as friable aggregates and rosettes composed of soft, pearly to translucent, pale pink to white flakes with perfect basal cleavage. Optical properties are uniaxial negative with $\varepsilon = 1.51$ and $\omega = 1.52$; weakly dichroic from colourless to palest pink. Specific gravity is 2.12 (calc. 1.95). Mountkeithite is soluble, with effervescence, in dilute HCl. Mountkeithite changes to a pyroaurite-like phase, with a 7.8 Å basal spacing when immersed in water, but can be changed back to an 11.3 Å phase by immersion in 1M $MgSO_4$; mountkeithite also collapses to a 7.8 Å basal spacing under vacuum. Phases similar to mountkeithite have been synthesized by immersing grains of stichtite in 1M $MgSO_4$ at 20, 50, 105, and 200 °C.

The structure of mountkeithite is interpreted as consisting of positively charged brucite-like hydroxyl layers, containing both divalent and trivalent cations, separated by about 7 Å-thick, negatively charged interlayers containing carbonate and sulphate anions, magnesium sulphate, and water. Carrboydite, motukoreaite, and hydrohonessite are minerals with similarly large basal spacings of 10–11 Å, and are also believed to have pyroaurite-related structures with expanded interlayers.

MOUNTKEITHITE was first recognized as a possible new mineral by Peter Bridge, of the Western Australian Government Chemical Laboratories, in November 1973. His unpublished laboratory Report 19739/73 described a specimen (MDC 5196) from the Mount Keith nickel deposit, that consisted of a low-temperature hydrothermal vein in a slightly bleached, pale-green serpentinite. Minerals in the vein included crystals of flattened pyrite, euhedra of magnetite, magnesite, morenosite, and a 'white flaky mineral altering from stichtite'. Preliminary X-ray diffraction photographs indicated that the white flaky mineral had an 11 Å basal spacing.

In 1976, MDC 5196 was examined by the authors as part of a continuing study of nickel sulphide deposits in Western Australia, and the 11 Å basal spacing was confirmed. Other samples from Mount Keith (previously collected by Dr R. E. T. Hill) were studied and also found to contain pale-pink and white flaky grains with 11 Å basal spacings. X-ray diffraction patterns were similar to those given by a number of other, recently discovered phases from the oxidation zones of some Western Australian nickel deposits. These included carrboydite (Nickel and Clarke, 1976) from the Carr Boyd mine, and an unnamed nickel 'hydroxide' from Kambalda, now characterized as hydrohonessite (Nickel and Wildman, 1981).

The presence of stichtite, a Mg-Cr member of the pyroaurite group, in the serpentinite and its apparent alteration to a pale-pink or white phase in the vein suggested that low-temperature hydrothermal solutions could effect changes to pyroaurite-group minerals that would convert them to phases with 11 Å basal spacings. The structural relationships between the 11 Å phases and the 7.8 Å pyroaurite-group minerals was, at the time, poorly understood (e.g. Allman *et al.*, 1968; Nickel and Clarke, 1976), and a study of the Mount Keith material was undertaken in the hope that it would provide some clues as to the nature of this relationship.

Analysis of separated grains of mountkeithite showed that sulphate was an important constituent, and the presence of morenosite and hexahydrite in the vein further suggested that a sulphate-rich environment was necessary for formation of the mineral. Experiments, undertaken in early 1977, demonstrated that $MgSO_4$ and water could be incorporated into the interlayer of stichtite at temperatures ranging from 20 to 200 °C. The low temperature required for alteration and the fact that sulphate was involved in the alteration suggested that other 11 Å phases formed in the sulphate-rich oxidation zone may bear a similar 'expanded interlayer' relationship to an appropriate member of the pyroaurite group (e.g. the relationship between takovite and carrboydite).

The name and proposed structure of mountkeithite were first submitted to the IMA Commission on New Minerals and New Mineral Names in April 1977, and accepted, in a slightly modified form, in July 1980.

The name of the mineral is for the locality, Mount Keith, Western Australia. Specimen material is housed in the collection of the CSIRO, Division of Mineralogy, Floreat Park, Western Australia, and the collection of the Government Chemical Laboratories, Perth, Western Australia. A sample of mountkeithite will be lodged with the British Museum (Natural History). There is no additional material for distribution.

Occurrence. The Mount Keith nickel prospect (Burt and Sheppy, 1975) is situated approximately 400 km NNW of Kalgoorlie, Western Australia. It is a large, low-grade deposit containing about 300 million tonnes of disseminated ore of average grade 0.6% Ni. Sulphides, dominantly pentlandite with lesser pyrrhotine, occur within the interstices of an aggregate of coarse olivine grains, now completely replaced by complex intergrowths of silicate, hydroxycarbonate, and carbonate minerals, including lizardite, antigorite, chrysotile, chlorite, talc, brucite, pyroaurite, stichtite, iowaite, and magnesite.

The deposit has not been mined, although samples for metallurgical testing were recovered from an exploration shaft sunk near the centre of the mineralized zone. Fresh ore, from near the base of the shaft, consists of a dark-green to black serpentinite with interstitial grains of sulphide and chromite. Bright-pink or purple whorl-like aggregates of stichtite are abundant and appear to enclose and replace chromite. Samples from within the supergene weathering zone are generally bleached, and the serpentinite varies in colour from dark green to a pale lime-green. Much of the ore is traversed by a system of open fractures or veins, the surfaces of which are commonly coated by slickensided, lime-green serpentine. Vein minerals include flattened pyrite crystals, up to 10 mm in diameter, euhedral magnetite in both cubes and octahedra, crystalline magnesite, hexahydrite, morenosite, and mountkeithite. Mountkeithite is intergrown with pyrite and contains numerous inclusions of small magnetite granules.

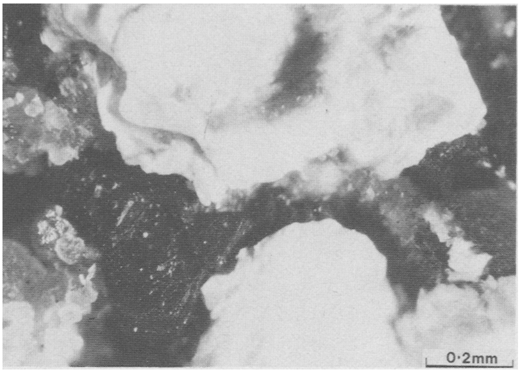

FIG. 1. Typical flake of mountkeithite (white), intergrown with crystalline magnetite (black) and magnesite (translucent grey).

Physical and optical properties. Mountkeithite is palest pink to white, pearly to translucent, and occurs as scales, rosettes, and whorl-like aggregates of similar habit to associated stichtite or chromiferous pyroaurite (fig. 1). Individual grains are flaky, measuring 0.5 to 1.0 μm in thickness and up to 1 mm in diameter. Flakes, in cross-section, are length-slow, and the mineral is uniaxial negative or biaxial negative with a low 2V. In transmitted light, the mineral is palest pink and non-pleochroic to weakly pleochroic. Refractive indices are variable about $\omega = 1.52$, $\varepsilon = 1.51$. A Gladstone–Dale calculation gives a mean refractive index = 1.54.

Specific gravity was determined by adjusting the density of bromoform diluted by alcohol until a flake of mountkeithite remained suspended. This gave a value of 2.12. Calculated SG, based on two structural formula units (i.e. 48(OH) per unit cell) is 1.95.

X-ray data. Single crystal X-ray diffraction studies could not be done due to the small size of mountkeithite crystals, and composite nature of even the smallest flakes. Debye–Scherrer and Gandolfi X-ray powder diffraction patterns were moderately good, but some lines were broadened or diffuse. The strongest lines are given in Table I. Diffraction patterns of flakes, composed of sub-oriented polycrystalline aggregates, distinguish a family of strong $00l$ reflections (11.30, 5.63, 3.765, and 2.82) and also suggest that 4.63 and 3.03 might belong to a family of hk0 reflections (see fig. 3).

Possible hexagonal parameters from the X-ray data are a = 5.3, 9.3, 10.7, 14.1 Å, etc. and c = 11.3, 22.6, 33.9, 45.2 Å, etc. The first six lines of the diffraction pattern can be indexed on a hexagonal cell with a = 9.3 Å and c = 11.3 Å. However, the strong but often diffuse doublet of 2.645 and 2.545 cannot be indexed using these parameters. Good

TABLE I. *X-ray powder diffraction data for mountkeithite*

hkl	$d_{obs.}$	$d_{calc.}$	$I_{obs.}$
002	11.30	11.25	10
004	5.63	5.63	8
200	4.63	4.63	2
006	3.765	3.758	6
107	3.03	3.042	<1
008	2.82	2.818	<1
207	2.645	2.644	4
311, 305	2.545	2.552, 2.547	4
314	2.34	2.338	1b
405	2.06	2.060	2b
414, 318	1.90	1.903, 1.899	<1
504	1.76	1.760	<1
2.1.13	1.554	1.554	5
0.0.15, 4.1.10	1.505	1.503, 1.505	3

Calculated d spacings based on a hexagonal cell with $a = 10.698$ Å, $c = 22.545$ Å. Intensities are estimated.

coincidence between observed and calculated parameters (Table I) can be obtained by taking $a = 10.698$ Å and $c = 22.545$ Å. This corresponds to taking a as the diagonal of four 'brucite-like' subcells (each with $a \sim 3.1$ Å) and c as twice the spacing of the strong basal reflection.

Electron-diffraction photographs of flakes of mountkeithite are indistinguishable from stichtite and indicate a hexagonal array of sub-cells in the basal plane.

Composition. Numerous, semi-quantitative electron-microprobe analyses were made on small, hand-picked flakes of mountkeithite mounted, without polishing, on a zinc disc. Major elements varied within the following ranges; Mg 15.6–23.7, Ni 1.7–7.9, Cu 0–0.5, Cr 2.2–6.0, Fe 3.3–8.3, Al 0.1–5.4, S 4.6–7.8. Minor elements included Si, Ca, Na, K, and Cl; these were less than 0.5% and generally only at trace levels. Aluminium shows considerable variation at a relatively constant Cr : Fe ratio, which corresponds to the Cr : Fe ratio of unaltered or partially altered iron-rich stichtite or chromiferous pyroaurite in the bleached serpentinite. The most aluminous mountkeithites are white in colour, whereas pale-pink shades are typical of low-aluminium varieties. Higher nickel values are generally found in the pink varieties and higher copper values in the white varieties. A plot of mountkeithite analyses, in terms of Al, Cr, and Fe, is shown in fig. 2.

Electron-microprobe analyses, using elemental standards for Ni, Cu, Cr, and Al, pyrite for Fe and S, and MgO for Mg, of flakes of pink mountkeithite separated from sample number MDC 5196 gave average weight per cent values (20 spot analyses) of: Mg 19.1±2.4, Ni 4.8±1.1, Cu 0.1±0.1, Cr 4.2±0.9, Fe 5.8±1.2, Al 1.4±0.8, S = 5.9±1.0. Analysis of several micrograms of the separated mountkeithite (made by R. D. MacDonald using volatilization and thermal conductivity measurement techniques) gave C = 1.06±0.13 and H = 3.42±0.06. Calculated oxide weight per cent values are: MgO 31.7, NiO 6.1, CuO 0.1, Cr_2O_3 6.1, Fe_2O_3 8.3, Al_2O_3 2.6, SO_3 14.7, CO_2 3.9, H_2O 30.6; total 104.1.

For determination of cell contents, the weight per cent analysis was calculated to atomic proportions and H apportioned between OH^- and H_2O to achieve a charge balance between (Mg^{2+} + Ni^{2+} + Cu^{2+} + Cr^{3+} + Fe^{3+} + Al^{3+}) and (SO_4^{2-} + CO_3^{2-} + OH^-), assuming Fe to be trivalent, S to occur as sulphate, and C as carbonate. For a hexagonal cell with $a = 10.698$ Å and $c = 22.545$ Å, and using the measured density of 2.12, the contents are:

$$Mg_{21.52}Ni_{2.24}Cu_{0.04}Cr_{2.21}Fe_{2.85}Al_{1.42}(OH)_{52.14}(CO_3)_{2.42}(SO_4)_{5.04}(H_2O)_{20.40}.$$

The grouping of elements to give a structural formula is somewhat arbitrary, but is based on the knowledge that magnesium sulphate can be reversibly introduced into the interlayer portion of a pyroaurite-type structure, consisting of positively charged, brucite-like hydroxyl layers and negatively charged interlayers. During the resulting expansion

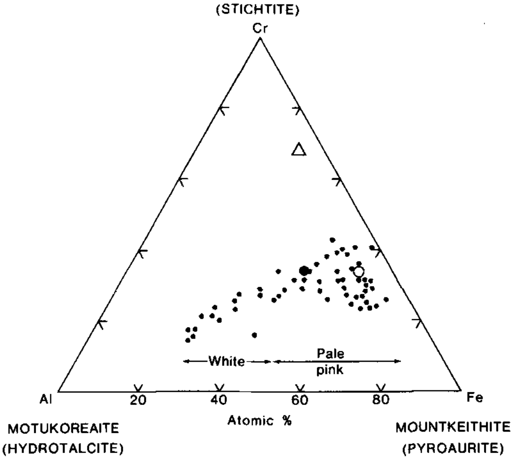

FIG. 2. Range of compositions of analysed mountkeithite. Small dots are semiquantitative spot analyses of unpolished grains of mountkeithite; large black dot is average analysis of mountkeithite from MDC 5196; open circle is chromian pyroaurite from bleached serpentinite host, MDC 5196; large triangle is the average composition (chemical analysis) of stichtite from Mt. Keith, determined by bulking stichtite mineral separates from drill hole MKD 32.

or contraction it appears that the hydroxyl layers largely remain passive and compositionally unaffected, whilst metal sulphate and H_2O addition, and sulphate- and carbonate-ion exchange occur easily and rapidly within the interlayer.

The structural formula has been calculated for an hydroxyl layer with 24(OH), which is the number of close-packed (OH) in a brucite-like hydroxyl layer with hexagonal sub-cell dimensions $a = 10.7$ Å, $c \sim 4.7$ Å. All trivalent metals, and sufficient divalent metals* to give a total of 12, are allocated to the octahedral sites of the hydroxyl layer; residual divalent metals are allocated to the interlayer. The positive charge of the hydroxyl layer, due to trivalent metals, is balanced in the interlayer by taking CO_3^{2-} together with sufficient SO_4^{2-}; residual SO_4^{2-} is taken with the excess divalent cations as metal sulphate. All H_2O is allocated to the interlayer.

The resulting structural formula is:

hydroxyl layer

$$[(Mg_{8.15}Ni_{0.85}Cu_{0.02})_{\Sigma 9.02}(Fe^{3+}_{1.31}Cr_{1.02}Al_{0.65})_{\Sigma 2.98}(OH)_{24}]^{2.98+}$$

interlayer

$$[(CO_3)_{1.11}(SO_4)_{0.38}(Mg_{1.76}Ni_{0.18})_{\Sigma 1.94}(SO_4)_{1.94}(H_2O)_{9.39}]^{2.98-}.$$

The general formula for mountkeithite can be expressed:

$$[Mg_{12-x}(Fe^{3+},Cr)_x(OH)_{24}]^{x+}[(CO_3,SO_4)_{0.5x}\,n(Mg,Ni)SO_4 . mH_2O]^{x-}.$$

Possible substitutions include Ni and Cu (for Mg) in the hydroxyl layer, Al (for Fe or Cr) in the hydroxyl layer (tending to motukoreaite), Cl (for CO_3 or SO_4) in the interlayer, and Cu (for Mg or Ni) in the interlayer.

Stability. Mountkeithite is readily soluble in dilute HCl with effervescence and evolution of gas, presumed to be CO_2. In water, the mineral changes to a pyroaurite-like structure, but can equally rapidly be changed back to a phase with an 11.3 Å basal spacing by immersion in 1M $MgSO_4$ solution. In one experiment, a large flake of natural mountkeithite was separated from MDC 5196 and mounted, using epoxy resin, on a glass fibre. X-ray diffraction photographs, taken using a Debye-Scherrer camera (fig. 3) showed that the basal spacing collapsed to 7.8 Å when the flake was immersed in water for 1 day, but increased again to 11.3 Å after standing in 1M $MgSO_4$. A similar flake of mountkeithite, placed in a Gandolfi camera attached to a vacuum pump, collapsed to 7.8 Å under vacuum (fig. 4), but returned to 11.3 Å when air was admitted. The mineral decomposes by dehydration above 80 °C, and collapses progressively to a poorly diffracting material with a 6 Å basal spacing at 200 °C.

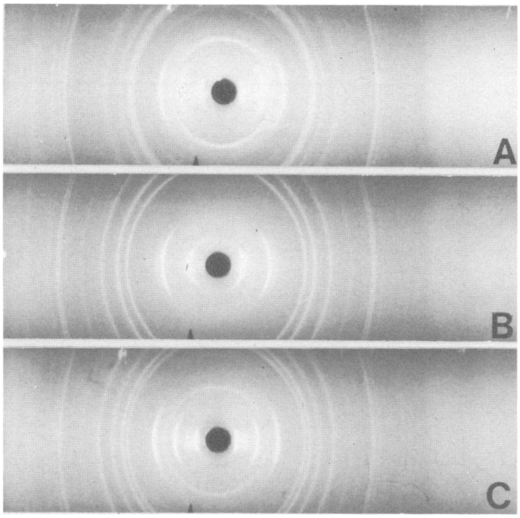

FIG. 3. Debye-Scherrer photographs of a flake of mountkeithite from MDC 5196. A. Mountkeithite; B. After 2 hours' immersion in water; C. After 3 hours' immersion in 1M $MgSO_4$.

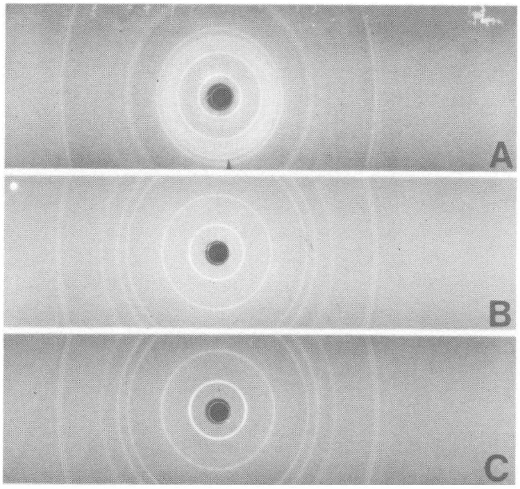

FIG. 4. Effect of water immersion and vacuum treatment on X-ray diffraction photographs of mountkeithite. A. Debye-Scherrer photograph of mountkeithite; B. Gandolfi photograph of flake of mountkeithite after immersion in water; C. X-ray diffraction photograph of a flake of mountkeithite in an evacuated Gandolfi camera.

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* The ratio of Mg:Ni has been preserved during the allocation of divalent metals between the hydroxyl layer and interlayer. Comparison of analyses of chromiferous stichtite and mountkeithite suggests that more Ni than Mg may have entered the interlayer.

TABLE II. *Basal spacings of some related minerals with brucite-like hydroxyl layers*

Mineral	*c*Å	OH layer	Interlayer	Expanded interlayer
Brucite	4.7	4.7	—	—
Pyroaurite	7.8	4.7	3.1	—
Mountkeithite	11.3	4.7	—	6.6

Relationships between the *c* parameter of brucite, pyroaurite, and mountkeithite are shown in Table II. Similar relationships are believed to exist for other pyroaurite-related minerals with expanded interlayers.

Synthesis. The mineral assemblage associated with mountkeithite, together with the vein-like nature of the occurrence, indicate crystallization in a magnesium- and sulphate-rich, low-temperature hydrothermal environment. Experiments were conducted to test the assumption that mountkeithite could form from stichtite or chromiferous pyroaurite in aqueous solutions containing magnesium sulphate.

Stichtite, from samples of Mount Keith diamond drill core, was separated by hand picking, and was purified to give a >95%-pure concentrate in the +300 to −100 mesh range. A series of experimental runs was conducted over 55 days at 2,000 psi and at temperatures of 50, 105, and 200 °C by sealing about 20-mg samples of stichtite in gold capsules with 0.2 ml of 1M $MgSO_4$; a similar group of experimental runs contained stichtite and pure water.

X-ray diffraction photographs, taken after opening the capsules and allowing them to air dry, contained lines consistent with a combination of an 11 Å-phase (fig. 5) and hexahydrite; stichtite was not altered in the runs which contained water only. After standing in water for one day (to dissolve the hexahydrite) the 11 Å-phase reverted to a pyroaurite-type structure with a 7.8 Å basal spacing, presumably stichtite. Treatment of this 'reacted' stichtite (and in later experiments 'fresh' stichtite) with 1M $MgSO_4$ at room temperature for 1 day or longer was sufficient to cause reaction to the 11 Å-phase.

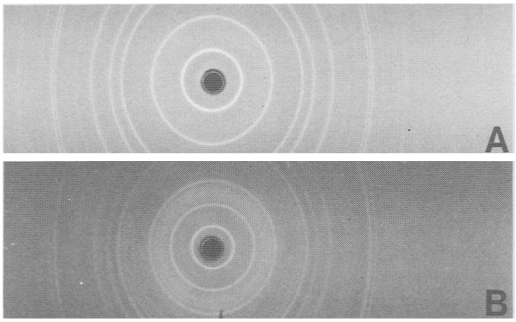

FIG. 5. X-ray diffraction photograph of 11 Å-phase synthesized from stichtite. A. Stichtite; B. 11 Å-phase.

Experiments conducting using 1M solutions of Na_2SO_4, $MgCl_2$ and $Mg(NO_3)_2$ at room temperature and also at 200 °C and 2000 psi for 14 days did not cause a change from stichtite to the 11 Å-phase.

Compositions of the water-soluble and acid-soluble portions of the synthetic 11 Å-phase are compared with the stichtite, used as starting material, in Table III. Analyses have been corrected for minor insoluble silicates and oxides, and allowance has been made for the small percentage of $MgSO_4$ that remained as a surface coating on grains.

The ratio of trivalent metals, Cr:Fe:Al, is the same in both the stichtite and the 11 Å-phase, but absolute values of the metals in the 11 Å-phase are lower, due to dilution. Calculations based on maintaining constant $\Sigma(Cr_2O_3 + Fe_2O_3 + Al_2O_3)$ suggest that 100 g stichtite + 10 g MgO + 21.2 g SO_3 + 24.8 g $H_2O \rightarrow$ 156 g 11 Å-phase. The added components have the formula $Mg(SO_4)_{1.06} \cdot 5.6H_2O$, which is close to hexahydrite and similar to epsomite in composition.

TABLE III. *Chemical analyses of synthetic 11Å-phase*

		Synthetic 11 Å-phase		
	Stichtite (untreated)	Total	Water-soluble	Acid-soluble
MgO	38.9%	31.3%	5.2%	26.1%
Fe_2O_3	6.1	3.9	—	3.9
Cr_2O_3	16.1	10.3	—	10.3
Al_2O_3	0.9	0.6	—	0.6
SO_3	nil	13.6	8.1	5.5
H_2O	—	>11.5	11.5	—
Atomic proportions (Basis, $\Sigma R = 12$)				
Mg	9.10		1.83	9.21
Fe	0.72			0.69
Cr	2.00 } 2.90			1.93 } 2.79
Al	0.18			0.17
SO_4^{2-}	—		1.44	0.98
H_2O	—		14.64	—

Structural formula

hydroxyl layer $[Mg_{9.21}(Fe^{3+}_{0.69}Cr_{1.93}Al_{0.17})_{\Sigma 2.79}(OH)_{24}]^{2.79+}$

interlayer $[(CO_3)_{0.80}(SO_4)_{0.59}(MgSO_4)_{1.83}$
$H_2O_{14.64+x}]^{2.78-}$

* Note CO_3 value determined by difference; pyroaurite interlayer water (x) not determined.

Water-soluble components (Table III) have the formula $Mg(SO_4)_{0.79}.8H_2O$; the lower sulphate value suggests that some irreversible exchange of sulphate for carbonate may have occurred.

Related minerals. The most closely related mineral to mountkeithite appears to be motukoreaite $(Na_{0.73}K_{0.07})_{\Sigma 0.80}(Mg_{18.13}Mn_{0.32}Zn_{0.21})_{\Sigma 18.66}Al_{11.15}(CO_3)_{6.22}(SO_4)_{3.97}(OH)_{51.19}.27.20\ H_2O$ (Rodgers *et al.*, 1977). Brindley (1979) recast the formula of motukoreaite, to facilitate comparison with members of the pyroaurite group, by normalizing the composition to three octahedral cations. This gives $(Na_{0.07}K_{0.01})[Mg_{1.82}Mn_{0.03}Zn_{0.02}Al_{1.12}(OH)_{5.15}]^{1.95+}(CO_3)_{0.63}(SO_4)_{0.40}.2.74H_2O$, which has a much higher positive charge on the hydroxyl layer than most pyroaurite-related minerals, due to both a higher $R^{3+}:R^{2+}$ ratio and a deficiency of hydroxyl ions. Brindley discusses the possibility that vacant hydroxyl positions may be filled by water molecules, with no effect on layer charge, or by sulphate ions, which will satisfy the charge deficit but supply more oxygens than can be accommodated in the hydroxyl layer. He believes the latter alternative is unlikely due to the ease of exchange of sulphate ions by carbonate ions, and favours incorporation of sulphate ions and water in the interlayer.

The method of formula calculation (suggested above) based on essentially 'stoichiometric' hydroxyl layers and compositionally variable interlayers would seem to be a better basis for comparison of minerals with expanded pyroaurite-related structures. Motukoreaite has been recalculated according to the scheme outlined above:

hydroxyl layer

$$[(Mg^{2+}_{6.15}Zn^{2+}_{0.10}Mn^{2+}_{0.15}Na^{+}_{0.34}K^{+}_{0.03}Al^{3+}_{5.23})_{\Sigma 12.00}(OH)^{-}_{24}]^{4.86+}$$

interlayer

$$[(CO_3)^{2-}_{2.43}Mg^{2+}_{2.35}(SO_4)^{2-}_{1.87}(CO_3)^{2-}_{0.48}(H_2O)_{12.73}]^{4.86-}$$

This suggests that, like mountkeithite, the composition of motukoreaite could be explained in terms of a water-rich, metal sulphate (carbonate)-containing interlayer.

A similar structural relationship has been reported for koenenite, $[Mg_7Al_4(OH)_{22}]^{4+}$ $[Na_4Mg_2Cl_{12}]^{4-}$, and its pyroaurite-related natural alteration product (similar to chloromanasseite) by Allman *et al.* (1968). Koenenite consists of brucite-like OH layers alternating with Cl double layers along a common *c* axis. Its natural alteration product has a pyroaurite-like structure with a single $MgCl_2/H_2O$ layer.

The new mineral hydrohonessite (Nickel and Wildman, 1981) also has an 11 Å basal spacing and comes from the sulphate-rich environment associated with the weathering of nickel-sulphide ores at Kambalda. It is probable that it too is a pyroaurite-related mineral with an expanded, water-rich, metal sulphate-containing interlayer.

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