

OKIEITE, $\text{Mg}_3[\text{V}_{10}\text{O}_{28}]\cdot 28\text{H}_2\text{O}$, A NEW DECAVANADATE MINERAL FROM THE BURRO MINE, SLICK ROCK MINING DISTRICT, SAN MIGUEL COUNTY, COLORADO, USA

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ABSTRACT

Okieite, $\text{Mg}_3[\text{V}_{10}\text{O}_{28}]\cdot 28\text{H}_2\text{O}$, is a new decavanadate mineral from the Burro mine, Slick Rock district, San Miguel County, Colorado, USA (type locality); the mineral is also found at the Hummer mine, Paradox Valley, Montrose County, also in Colorado. The mineral is rare; it occurs with dickthomsssenite on montroseite- and corvusite-bearing sandstone. Crystals of okieite from the Burro mine are equant to prismatic, commonly appearing like curving columns (up to about 3 mm in length) and often exhibiting rounded faces. The streak of okieite is light orange yellow, and the luster is vitreous. The Mohs hardness is *ca.* 1½, the tenacity is brittle, the fracture is curved or conchoidal, there is no cleavage, and the measured density is 2.20(2) g/cm³. Okieite is biaxial (–), with $\alpha = 1.720(3)$, $\beta = 1.745(3)$, $\gamma = 1.765(3)$ (white light); $2V = 84(2)^\circ$ with strong $r < v$ dispersion. The optical orientation is $X \wedge a = 37^\circ$, $Y \wedge c = 28^\circ$, $Z \wedge b = 31^\circ$. No pleochroism is observed in okieite. The empirical formula from electron-probe microanalysis (calculated on the basis of V = 10 and O = 56 *apfu* as indicated by the structure) is $\text{Mg}_{2.86}[\text{H}_{0.28}\text{V}_{10}\text{O}_{28}]\cdot 28\text{H}_2\text{O}$. Okieite is triclinic, $P\bar{1}$, with a 10.55660(19), b 10.7566(2), c 21.3555(15) Å, α 90.015(6), β 97.795(7), γ 104.337(7)°, and V 2326.30(19) Å³, as determined by single-crystal X-ray diffractometry. The strongest four diffraction lines in the powder diffractograms are [d in Å(hkl): 9.71(100)($\bar{1}01, 0\bar{1}1, 011, 101$); 8.32(19)($\bar{1}10$); 11.04(17)(002); and 6.42(12)(110, $\bar{1}\bar{1}1, 1\bar{1}2$). The atomic arrangement of okieite [$R_1 = 0.0352$ for 11,327 $I > 2\sigma I$ reflections] consists of a $\{\text{V}_{10}\text{O}_{28}\}^{6-}$ (decavanadate) structural unit and a $\{[\text{Mg}(\text{H}_2\text{O})_6]_3\cdot 10\text{H}_2\text{O}\}^{6+}$ interstitial complex. Only hydrogen bonding links the structural unit with the components of the interstitial complex. Okieite is isostructural with synthetic $\text{Mg}_3[\text{V}_{10}\text{O}_{28}]\cdot 28\text{H}_2\text{O}$. The name okieite is for Craig (“Okie”) Howell of Naturita, Colorado.

Keywords: okieite, new mineral, decavanadate, crystal structure, Burro mine, Hummer mine, Colorado, USA.

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INTRODUCTION AND OCCURRENCE

Vanadium and uranium are relatively rare elements in the Earth's crust, occurring in abundances of approximately 100 and 2 ppm, respectively; thus, extraction of these resources must take place where geologic processes greatly concentrate the elements. One rich source of both uranium and vanadium is the Colorado Plateau, and particularly the Uravan Mineral Belt, an arcuate 120 km long mineralization belt mostly in southwest Colorado but extending into southeast Utah. The deposits occur as roll-front deposits in the sandstone of the Salt Wash member of the Morrison Formation (Carter & Gualtieri 1965, Shawe 2011), where groundwater solutions rich in U and V encountered locally reducing conditions created by decaying organic matter derived from logs and carbonized plant material that once grew in Jurassic forests. The unique geochemical conditions in the Uravan Mineral Belt have added significantly to our knowledge of vanadium crystal chemistry. In the past decade, deposits of the Colorado Plateau have been an unusually rich source of new vanadium minerals, with 30 minerals containing essential vanadium being described from the Uravan belt and adjacent areas. Here we describe another new Colorado Plateau (Uravan Mineral Belt) vanadium mineral, okieite.

Okieite was found underground at the Burro mine, Slick Rock district, San Miguel County, Colorado, USA (38.04507, -108.88972). The Burro mine is the type locality for metamunirite (Evans 1991), burritoite $[\text{Ca}_2(\text{NH}_4)_2(\text{V}_{10}\text{O}_{28}) \cdot 15\text{H}_2\text{O}]$; Kampf *et al.* 2017], ammoniozippeite $[(\text{NH}_4)_2(\text{UO}_2)_2(\text{SO}_4)\text{O}_2 \cdot \text{H}_2\text{O}]$; Kampf *et al.* 2018a], ammoniomathesiusite $[(\text{NH}_4)_5(\text{UO}_2)_4(\text{SO}_4)_4(\text{VO}_5) \cdot 4\text{H}_2\text{O}]$; Kampf *et al.* 2018b], ammoniolasallite $[(\text{NH}_4)_2\text{Mg}_2(\text{V}_{10}\text{O}_{28}) \cdot 20\text{H}_2\text{O}]$; Kampf *et al.* 2019a], uroxite $[(\text{UO}_2)_2(\text{C}_2\text{O}_4)(\text{OH})_2(\text{H}_2\text{O})_2 \cdot \text{H}_2\text{O}]$; Kampf *et al.* 2019b], metauroxite $[(\text{UO}_2)_2(\text{C}_2\text{O}_4)(\text{OH})_2(\text{H}_2\text{O})_2]$; Kampf *et al.* 2019b], and caseyite $[(\text{V}^{5+}_2\text{O}_2)\text{Al}_{7.5}(\text{OH})_{15}(\text{H}_2\text{O})_{13}]_2[\text{H}_2\text{V}^{5+}_9\text{O}_{28}]_2[\text{V}^{5+}_{10}\text{O}_{28}]_2 \cdot 90\text{H}_2\text{O}]$; Kampf *et al.* 2020]. The mine is near the southern end of the Uravan Mineral Belt. The specimens of the new mineral were collected by one



Fig. 1. Okieite crystals from the Burro mine; field of view 0.84 mm.

of the authors (PMA). The mineral is rare; it occurs with dickthomssenite $(\text{MgV}_2\text{O}_6 \cdot 7\text{H}_2\text{O})$ on montroseite- and corvusite-bearing sandstone. Besides the minerals noted above for which the mine is the type locality, other secondary minerals confirmed from the mine include barnesite, gypsum, hewettite, magnesio-pascoite $[\text{Ca}_2\text{Mg}(\text{V}^{5+}_{10}\text{O}_{28}) \cdot 16\text{H}_2\text{O}]$, metarossite, natrojarosite, natrozippeite, navajoite, pascoite, rossite, schindlerite $[(\text{NH}_4)_4\text{Na}_2(\text{V}_{10}\text{O}_{28}) \cdot 10\text{H}_2\text{O}]$, sherwoodite, and wernerbaurite $[(\text{NH}_4)_2\text{Ca}_2(\text{V}_{10}\text{O}_{28}) \cdot 16\text{H}_2\text{O}]$.

The new mineral forms from the oxidation of montroseite-corvusite assemblages in a moist environment. Under ambient temperatures and generally oxidizing near-surface conditions, water reacts with

TABLE 1. ANALYTICAL DATA IN FOR OKIEITE

Constituent	Mean	Range	S.D.	Standard	Normalized
MgO (wt.%)	10.00	9.46–10.52	0.44	diopside	7.52
V ₂ O ₅	78.99	77.45–80.04	1.14	V metal	59.38
H ₂ O*					33.10
Total					100.00

* Based upon the crystal structure with V = 10 and O = 56 *apfu*.

The empirical formula (based on 56 O *apfu*) is $\text{Mg}_{2.86}(\text{H}_{0.28}\text{V}^{5+}_{10}\text{O}_{28}) \cdot 28\text{H}_2\text{O}$.

The endmember formula is $\text{Mg}_3(\text{V}_{10}\text{O}_{28}) \cdot 28\text{H}_2\text{O}$, which requires MgO 7.88, V₂O₅ 59.25, H₂O 32.87, total 100.00 wt.%.

TABLE 2. OBSERVED AND CALCULATED POWDER X-RAY DIFFRACTION DATA (d IN Å) FOR OKIEITE

l_{obs}	d_{obs}	d_{calc}	l_{calc}	$h\ k\ l$	l_{obs}	d_{obs}	d_{calc}	l_{calc}	$h\ k\ l$	l_{obs}	d_{obs}	d_{calc}	l_{calc}	$h\ k\ l$
		21.1450	2	0 0 1			3.2631	2	3 0 1			2.5131	1	$\bar{1}$ 4 3
17	11.04	10.5725	50	0 0 2			3.1793	1	$\bar{2}$ $\bar{1}$ 5			2.4965	1	$\bar{4}$ 0 3
		10.4151	1	0 1 0	9	3.150	3.1571	3	2 2 1	1	2.481	2.4897	1	$\bar{4}$ 2 3
		9.6768	31	$\bar{1}$ 0 1			3.1527	5	2 $\bar{2}$ 4			2.4814	1	$\bar{4}$ 1 4
100	9.71	9.4766	26	0 $\bar{1}$ 1			3.1269	1	$\bar{1}$ $\bar{2}$ 5			2.4742	1	1 0 8
		9.2153	22	0 1 1			3.1166	3	$\bar{2}$ 2 5			2.4709	1	0 $\bar{4}$ 3
		8.6726	22	1 0 1			3.0832	2	$\bar{2}$ $\bar{2}$ 3			2.3692	1	0 $\bar{4}$ 4
19	8.32	8.3831	16	1 1 0			3.0718	1	0 3 3	4	2.354	2.3578	2	1 1 8
		8.0378	2	$\bar{1}$ 1 1			3.0647	2	$\bar{3}$ 2 3			2.3494	1	0 0 9
		6.8680	1	$\bar{1}$ 1 2			3.0584	1	$\bar{1}$ $\bar{3}$ 1			2.3028	1	$\bar{1}$ $\bar{4}$ 3
		6.4944	3	1 1 0			3.0429	1	2 0 5			2.2125	1	1 $\bar{1}$ 9
12	6.42	6.4127	3	$\bar{1}$ $\bar{1}$ 1	7	3.024	3.0207	1	0 0 7			2.1575	1	1 2 8
		6.3055	9	1 $\bar{1}$ 2			3.0126	1	$\bar{1}$ 0 7			2.1491	1	$\bar{1}$ 5 0
		6.0221	2	1 1 1			2.9968	2	$\bar{1}$ $\bar{3}$ 2	3	2.1343	2.1395	1	1 $\bar{5}$ 1
		5.9352	1	0 $\bar{1}$ 3			2.9151	2	3 1 1			2.1360	1	$\bar{1}$ $\bar{4}$ 5
4	5.67	5.6438	6	$\bar{1}$ 1 3	3	2.897	2.9034	2	2 $\bar{3}$ 3			2.1145	1	0 0 10
		5.4389	1	1 0 3			2.8742	1	0 1 7			2.1087	1	1 $\bar{5}$ 2
		5.1885	2	$\bar{1}$ 2 0			2.8549	3	$\bar{2}$ 3 4			2.1068	1	$\bar{5}$ 1 1
2	5.137	5.0984	1	0 $\bar{2}$ 1	6	2.831	2.8392	2	$\bar{3}$ 0 5			2.1045	1	$\bar{5}$ 1 2
		5.0844	2	$\bar{2}$ 1 1			2.8314	1	2 2 3			2.0870	1	$\bar{5}$ 2 1
		4.8071	1	2 $\bar{1}$ 1			2.8219	1	$\bar{2}$ 2 6	7	2.0872	2.0834	2	$\bar{5}$ 2 2
1	4.797	4.7752	1	2 0 1			2.8153	1	3 $\bar{2}$ 3			2.0802	1	0 $\bar{5}$ 1
		4.5303	1	1 1 3			2.7918	1	2 1 5			2.0659	1	0 5 1
2	4.440	4.4158	1	$\bar{2}$ 0 3			2.7896	1	1 0 7			2.0599	1	$\bar{5}$ 2 3
		4.3445	1	$\bar{1}$ $\bar{1}$ 4			2.7354	1	$\bar{1}$ $\bar{3}$ 4			2.0581	2	0 1 10
2	4.304	4.2535	1	$\bar{1}$ 2 3	2	2.727	2.7270	1	$\bar{2}$ $\bar{2}$ 5	4	2.0516	2.0518	1	$\bar{1}$ 5 3
		4.2290	1	0 0 5			2.6891	1	$\bar{1}$ 4 0			2.0428	2	$\bar{5}$ 0 2
		4.1885	1	$\bar{2}$ $\bar{1}$ 1			2.6793	1	$\bar{3}$ 3 3			2.0404	1	$\bar{5}$ 1 4
3	4.130	4.1630	1	2 1 0			2.6497	1	$\bar{1}$ 0 8			2.0254	1	5 0 0
		4.1073	2	1 $\bar{2}$ 3			2.6431	1	0 0 8			2.0233	1	$\bar{5}$ 0 3
		4.0726	1	1 2 1			2.6389	1	$\bar{4}$ 1 1	1	2.0116	2.0142	2	1 0 10
4	3.922	3.9881	2	2 1 1			2.6347	1	3 1 3			2.0090	1	1 $\bar{1}$ 10
		3.8996	3	2 $\bar{1}$ 3	10	2.621	2.6286	1	$\bar{3}$ 0 6			1.9864	1	$\bar{5}$ 3 2
		3.7846	2	2 $\bar{2}$ 2			2.6105	1	$\bar{2}$ $\bar{1}$ 7			1.9811	1	1 2 9
2	3.763	3.7726	1	$\bar{1}$ 2 4			2.6038	2	0 4 0	5	1.9625	1.9648	1	$\bar{5}$ 3 3
		3.7498	1	$\bar{2}$ 2 3			2.5943	1	$\bar{2}$ 4 0			1.9617	1	0 $\bar{5}$ 4
		3.7134	1	2 1 2			2.5881	1	2 $\bar{2}$ 6			1.9475	1	1 1 10
		3.5118	1	1 $\bar{3}$ 1			2.5733	1	0 4 1	1	1.9238	1.9390	1	$\bar{1}$ 0 11
2	3.503	3.4956	2	$\bar{2}$ 0 5			2.5678	1	$\bar{4}$ 1 3			1.9223	1	0 0 11
		3.4762	1	$\bar{3}$ 1 0			2.5636	1	2 $\bar{4}$ 1					
		3.4483	1	$\bar{1}$ $\bar{2}$ 4	4	2.551	2.5598	1	$\bar{4}$ 2 1					
		3.3335	1	0 $\bar{3}$ 2			2.5564	1	$\bar{4}$ 0 1					
5	3.331	3.3031	1	0 1 6			2.5442	1	$\bar{4}$ 0 2					
							2.5406	2	0 1 8					

* Unit-cell parameters refined from powder data: $a = 10.571(8)$, $b = 10.750(8)$, $c = 21.350(7)$ Å, $\alpha = 90.06(3)$, $\beta = 97.87(2)$, $\gamma = 104.36(2)^\circ$, and $V = 2327(3)$ Å³.

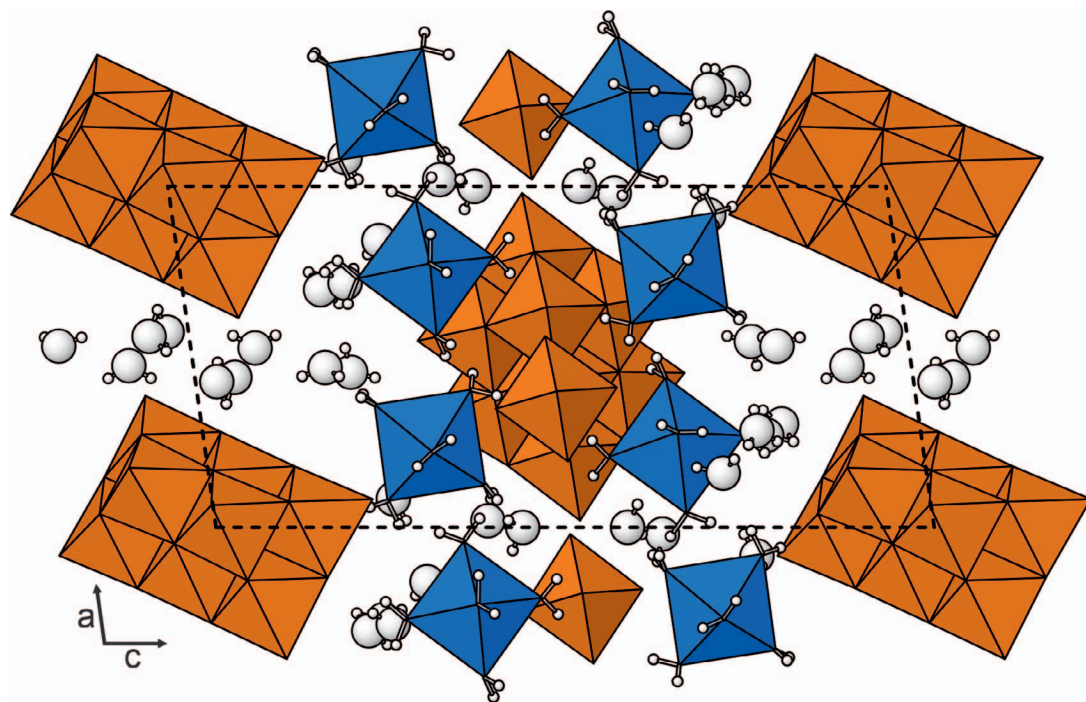


FIG. 2. Crystal structure of okieite viewed down [010]. VO_6 octahedra are orange, $\text{Mg}(\text{H}_2\text{O})_6$ octahedra are blue, O atoms of uncoordinated H_2O groups are large white balls, and H atoms are small white balls. The unit cell outline is shown by dashed lines.

pyrite to form aqueous solutions with relatively low pH. The various secondary vanadate phases that form depend upon prevailing Eh-pH conditions and the presence of other cations [e.g., $(\text{NH}_4)^+$, Na^+ , Ca^{2+} , Mg^{2+} , Mn^{2+} , Pb^{2+} , Al^{3+}]. Okieite was also confirmed on a specimen collected by one of the authors (JM) underground at the Hummer mine, Paradox Valley, Montrose County, Colorado (38.22556, -108.75028), about 23 km NE of the Burro mine. The Hummer mine is the type locality for hummerite (Weeks *et al.* 1951). The geology of this occurrence is similar to that of the Burro mine. At the Hummer mine, okieite occurs with gypsum on montroseite- and corvusite-bearing sandstone. All of the data used for the characterization of the species were obtained from the Burro mine samples. The Hummer mine is considered a cotype locality.

The name okieite ($^{\circ}\text{OU ki: aɪt/}$) is for Craig (“Okie”) Howell of Naturita, Colorado (b. 1963), who works for Nuvemco LLC, a company involved in the acquisition, exploration, development, and mining of uranium and vanadium properties in the southwest USA. Mr. Howell was particularly instrumental in the discovery of several new mineral species at the Burro, Blue Streak, and Pickett Corral mines, including

ammoniolasallite, ammoniomathiesiusite, ammoniozippeite, bicapite ($\text{KNa}_2\text{Mg}_2[\text{H}_2\text{PV}^{5+}_{12}\text{O}_{40}(\text{V}^{5+}\text{O})_2] \cdot 25\text{H}_2\text{O}$; Kampf *et al.* 2019c), bluestreakite [$\text{K}_4\text{Mg}_2(\text{V}^{4+}_2\text{V}^{5+}_8\text{O}_{28}) \cdot 14\text{H}_2\text{O}$; Kampf *et al.* 2015], and burroite [$\text{Ca}_2(\text{NH}_4)_2(\text{V}_{10}\text{O}_{28}) \cdot 15\text{H}_2\text{O}$], as well as okieite, described herein.

Three cotype specimens of okieite are deposited in the collections of the Natural History Museum of Los Angeles County, Los Angeles, California, USA, with catalogue numbers 66784 and 66785 for the Burro mine specimens and 66786 for the Hummer mine sample.

APPEARANCE AND PHYSICAL PROPERTIES

Crystals of okieite from the Burro mine are equant to prismatic, commonly appearing as curving columns (up to about 3 mm in length) and often exhibiting rounded faces (Fig. 1). Crystals from the Hummer mine form drusy crusts of equant crystals only about 0.2 mm in diameter. The color of okieite varies from bright red to red orange to yellow orange. The streak is light orange yellow, and the luster is vitreous. The mineral is nonfluorescent in ultraviolet light. The Mohs hardness is *ca.* 1½ based on scratch tests. The

TABLE 3. DATA COLLECTION AND STRUCTURE REFINEMENT DETAILS FOR OKIEITE

Diffractometer	Rigaku R-Axis Rapid II
X-ray radiation/power	MoK α ($\lambda = 0.71075$ Å)/ 50 kV, 40 mA
Temperature	293(2) K
Structural Formula	{[Mg(H ₂ O) ₆] ₃ ·10(H ₂ O)} {V ⁵⁺ ₁₀ O ₂₈ }
Space group	<i>P</i> 1̄
Unit cell dimensions	<i>a</i> = 10.55660(19) Å <i>b</i> = 10.7566(2) Å <i>c</i> = 21.3555(15) Å $\alpha = 90.015(6)^\circ$ $\beta = 97.795(7)^\circ$ $\gamma = 104.337(7)^\circ$
<i>V</i>	2326.30(19) Å ³
<i>Z</i>	2
Density (for above formula)	2.191 g/cm ³
Absorption coefficient	2.093 mm ⁻¹
<i>F</i> (000)	1540
Crystal size	260 × 230 × 200 mm
θ range	3.14 to 29.99°
Index ranges	$-14 \leq h \leq 14$, $-15 \leq k \leq 15$, $-29 \leq l \leq 29$
Reflections collected/unique	53566/13509; <i>R</i> _{int} = 0.034
Reflections with <i>I</i> > 2 σ <i>I</i>	11327
Completeness to $\theta = 29.99^\circ$	99.6%
Refinement method	Full-matrix least-squares on <i>F</i> ²
Parameters/restraints	810/88
GoF	1.112
Final <i>R</i> indices [<i>I</i> > 2 σ <i>I</i>]	<i>R</i> ₁ = 0.0352, <i>wR</i> ₂ = 0.0887
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0431, <i>wR</i> ₂ = 0.0932
Largest diff. peak/hole	+0.65/−0.68 e Å ⁻³

$R_{\text{int}} = \Sigma |F_o^2 - F_o^2(\text{mean})| / \Sigma [F_o^2]$. GoF = $S = \{\Sigma [w(F_o^2 - F_c^2)^2] / (n - p)\}^{1/2}$. $R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$. $wR_2 = \{\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]\}^{1/2}$; $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ where *a* is 0.047, *b* is 0.8894 and *P* is $[2F_c^2 + \text{Max}(F_o^2, 0)]/3$.

tenacity is brittle, the fracture is curved or conchoidal, and no cleavage was observed. The density measured by flotation in methylene iodide – toluene is 2.20(2) g/cm³; the calculated density is 2.186 g/cm³ for the empirical formula and 2.291 g/cm³ for the ideal formula. Okieite is biaxial (–), with $\alpha = 1.720(3)$, $\beta = 1.745(3)$, $\gamma = 1.765(3)$, determined in white light. The $2V$ is 84(2)°, measured directly on a spindle stage, and the calculated $2V$ is 82.5°. Dispersion is strong, $r < v$. The optical orientation is $X \wedge a = 37^\circ$, $Y \wedge c = 28^\circ$, $Z \wedge b = 31^\circ$. No pleochroism is observed. The Gladstone–Dale compatibility, $1 - (K_p/K_c)$, is –0.032 for the

empirical formula, in the range of excellent compatibility (Mandarino 1981). At room temperature okieite is rapidly soluble in H₂O, and the mineral dehydrates readily in air at low relative humidity.

CHEMICAL COMPOSITION

Electron probe microanalyses (EPMA; six points on four crystals) were obtained at the University of Utah using a Cameca SX-50 electron microprobe with four wavelength-dispersive spectrometers and Probe for EPMA software. Analytical conditions were 15 kV accelerating voltage, 10 nA beam current, and 2–5 μ m beam diameter. Counting times were 30 s on peak and 30 s on background for each element. Time-dependent intensity corrections were applied to Mg and V. Raw X-ray intensities were corrected for matrix effects with a $\phi\rho(z)$ algorithm (Pouchou & Pichoir 1991).

During vacuum deposition of the conductive carbon coat required for the EPMA, okieite was subjected to an ultimate vacuum of 2×10^{-5} torr for approximately 5 min. This clearly resulted in the loss of much of the weakly held H₂O; no further loss was detected during the EPMA. Dehydration resulted in significant deterioration of the sample surface, making analyses particularly challenging. The very large H₂O loss resulted in much higher concentrations for the remaining constituents than are to be expected for the fully hydrated phase; therefore, the other analyzed constituents were normalized to provide a total of 100% when combined with the calculated H₂O content. Analytical data are given in Table 1. The empirical formula (based on *V* = 10 and *O* = 56 *apfu* as indicated by the structure) is Mg_{2.86}[H_{0.28}V⁵⁺₁₀O₂₈]·28H₂O. The ideal structural formula is {[Mg(H₂O)₆]₃·10H₂O}{V₁₀O₂₈}, which requires MgO 7.88, V₂O₅ 59.25, H₂O 32.87, total 100.00 wt.%.

CRYSTAL STRUCTURE EXPERIMENTAL

The X-ray powder diffraction (PXRD) studies and the single-crystal structure study were carried out using a Rigaku R-Axis Rapid II curved imaging plate microdiffractometer with monochromatized MoK α radiation. For the PXRD, a Gandolfi-like motion on the ϕ and ω axes was used to randomize the sample. Observed *d* values and intensities were derived by profile fitting using JADE 2010 software. Data, including unit-cell parameters refined from the powder data using JADE 2010 with whole pattern fitting, are given in Table 2.

The Rigaku CrystalClear software package was used for processing the single-crystal structure data, including the application of an empirical absorption correction using the multi-scan method with ABSCOR (Higashi 2001). The structure was solved by direct

TABLE 4. ATOM COORDINATES AND DISPLACEMENT PARAMETERS ($\text{\AA}^2$) FOR OKIEITE

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>U</i> _{eq}
Mg1	0.36917(7)	0.64193(7)	0.11023(3)	0.02735(15)
Mg2	0.23862(7)	0.85844(7)	0.31292(3)	0.02656(15)
Mg3	0.75933(8)	0.18451(8)	0.34306(4)	0.03254(17)
V1	0.01520(3)	0.24888(3)	0.02261(2)	0.02130(7)
V2	0.12572(3)	0.14832(3)	0.91377(2)	0.02208(7)
V3	0.75514(3)	0.08895(3)	0.06674(2)	0.02428(8)
V4	0.02095(4)	0.11761(3)	0.15205(2)	0.02500(8)
V5	0.14332(3)	0.01474(3)	0.04581(2)	0.01895(7)
V6	0.34355(3)	0.37877(3)	0.39573(2)	0.02093(7)
V7	0.42276(3)	0.38689(3)	0.60670(2)	0.02050(7)
V8	0.17482(3)	0.35069(4)	0.50661(2)	0.02517(8)
V9	0.36215(4)	0.17641(3)	0.49823(2)	0.02397(8)
V10	0.39873(3)	0.59412(3)	0.50324(2)	0.01773(7)
O1	0.06522(16)	0.39142(14)	0.99779(8)	0.0330(3)
O2	0.17525(17)	0.29183(15)	0.89061(8)	0.0347(4)
O3	0.61415(15)	0.11541(17)	0.07257(8)	0.0355(4)
O4	0.08283(18)	0.16550(17)	0.22359(7)	0.0395(4)
O5	0.27990(13)	0.97670(14)	0.02565(7)	0.0246(3)
O6	0.17888(14)	0.06880(14)	0.12153(7)	0.0258(3)
O7	0.85274(15)	0.13681(14)	0.14553(7)	0.0268(3)
O8	0.84431(14)	0.24162(14)	0.03357(7)	0.0245(3)
O9	0.07565(14)	0.26637(13)	0.10694(7)	0.0260(3)
O10	0.27240(14)	0.08471(14)	0.91462(7)	0.0259(3)
O11	0.03930(15)	0.06061(14)	0.84063(6)	0.0264(3)
O12	0.16701(13)	0.17884(12)	0.00731(6)	0.0205(3)
O13	0.04829(13)	0.84284(13)	0.06119(6)	0.0204(3)
O14	0.95164(12)	0.04522(12)	0.04757(6)	0.0194(3)
O15	0.31475(16)	0.37403(16)	0.31963(7)	0.0315(3)
O16	0.54684(15)	0.60777(15)	0.31724(7)	0.0298(3)
O17	0.01984(15)	0.33261(17)	0.50823(8)	0.0368(4)
O18	0.35370(17)	0.02477(15)	0.49628(8)	0.0350(4)
O19	0.23501(13)	0.54519(14)	0.50620(7)	0.0237(3)
O20	0.56074(14)	0.24441(13)	0.49583(6)	0.0230(3)
O21	0.19312(14)	0.18510(14)	0.50294(7)	0.0286(3)
O22	0.18109(14)	0.36292(14)	0.41829(7)	0.0256(3)
O23	0.34768(14)	0.21188(13)	0.41261(7)	0.0256(3)
O24	0.24547(14)	0.37078(14)	0.59206(7)	0.0250(3)
O25	0.41054(14)	0.21767(13)	0.58570(6)	0.0238(3)
O26	0.54356(13)	0.42496(13)	0.40829(6)	0.0201(3)
O27	0.39081(13)	0.56665(12)	0.41289(6)	0.0189(3)
O28	0.39932(13)	0.39639(12)	0.50111(6)	0.0192(3)
OW1	0.20778(18)	0.70822(19)	0.12965(9)	0.0399(4)
H1A	0.165(3)	0.747(3)	0.1072(12)	0.048
H1B	0.189(3)	0.716(3)	0.1640(10)	0.048
OW2	0.28382(17)	0.59360(17)	0.01863(9)	0.0376(4)
H2A	0.234(3)	0.528(2)	0.0120(14)	0.045
H2B	0.248(3)	0.647(2)	0.0018(13)	0.045
OW3	0.45945(17)	0.82211(17)	0.08233(9)	0.0391(4)
H3A	0.535(2)	0.848(3)	0.0788(14)	0.047
H3B	0.420(3)	0.874(3)	0.0680(14)	0.047
OW4	0.46756(17)	0.70250(18)	0.20060(8)	0.0361(4)
H4A	0.467(3)	0.656(2)	0.2292(12)	0.043
H4B	0.542(2)	0.751(2)	0.2053(13)	0.043
OW5	0.28875(17)	0.46719(16)	0.14409(9)	0.0399(4)

TABLE 4. CONTINUED.

	x/a	y/b	z/c	U_{eq}
H5A	0.224(2)	0.406(3)	0.1318(14)	0.048
H5B	0.339(2)	0.425(3)	0.1629(13)	0.048
OW6	0.52598(18)	0.5673(2)	0.08965(10)	0.0422(4)
H6A	0.562(3)	0.595(3)	0.0614(11)	0.051
H6B	0.563(3)	0.574(3)	0.1208(10)	0.051
OW7	0.07736(16)	0.79591(17)	0.24393(8)	0.0357(4)
H7A	0.010(2)	0.756(3)	0.2553(13)	0.043
H7B	0.060(3)	0.842(3)	0.2181(12)	0.043
OW8	0.26707(19)	0.67583(17)	0.31124(9)	0.0406(4)
H8A	0.215(3)	0.615(3)	0.2902(12)	0.049
H8B	0.295(3)	0.645(3)	0.3418(11)	0.049
OW9	0.22118(19)	0.04346(16)	0.31647(9)	0.0394(4)
H9A	0.257(3)	0.100(3)	0.3443(11)	0.047
H9B	0.179(3)	0.073(3)	0.2926(12)	0.047
OW10	0.6398(2)	0.09791(19)	0.75502(10)	0.0460(5)
H10A	0.619(3)	0.035(2)	0.7723(15)	0.055
H10B	0.610(3)	0.152(2)	0.7691(15)	0.055
OW11	0.59587(17)	0.08254(18)	0.61767(8)	0.0359(4)
H11A	0.613(3)	0.072(3)	0.5832(11)	0.043
H11B	0.549(3)	0.130(3)	0.6124(13)	0.043
OW12	0.1235(2)	0.8094(2)	0.38315(10)	0.0474(5)
H12A	0.084(3)	0.737(2)	0.3885(16)	0.057
H12B	0.081(3)	0.855(3)	0.3968(15)	0.057
OW13	0.55962(19)	0.1639(2)	0.34468(10)	0.0496(5)
H13A	0.536(3)	0.216(2)	0.3626(15)	0.059
H13B	0.503(3)	0.093(2)	0.3530(16)	0.059
OW14	0.2789(2)	0.01421(19)	0.66148(10)	0.0535(5)
H14A	0.280(3)	0.035(3)	0.6960(11)	0.064
H14B	0.300(3)	0.081(3)	0.6409(14)	0.064
OW15	0.79383(19)	0.1759(2)	0.43987(8)	0.0428(4)
H15A	0.848(3)	0.223(3)	0.4643(13)	0.051
H15B	0.745(3)	0.128(3)	0.4593(14)	0.051
OW16	0.7741(3)	0.3783(2)	0.34979(11)	0.0571(6)
H16A	0.859(2)	0.406(4)	0.3562(17)	0.068
H16B	0.747(3)	0.423(3)	0.3709(16)	0.068
OW17	0.7308(2)	0.1843(2)	0.24619(9)	0.0527(5)
H17A	0.781(3)	0.199(3)	0.2216(14)	0.063
H17B	0.656(2)	0.162(3)	0.2276(15)	0.063
OW18	0.0407(2)	0.7953(3)	0.65790(12)	0.0722(8)
H18A	0.015(4)	0.764(4)	0.6894(13)	0.087
H18B	-0.026(3)	0.755(4)	0.6312(14)	0.087
OW19	0.1592(2)	0.3222(2)	0.71687(10)	0.0497(5)
H19A	0.199(3)	0.390(3)	0.7349(14)	0.060
H19B	0.176(3)	0.324(3)	0.6811(11)	0.060
OW20	0.4550(2)	0.3586(2)	0.21812(10)	0.0508(5)
H20A	0.525(3)	0.400(3)	0.2151(16)	0.061
H20B	0.439(3)	0.360(3)	0.2508(12)	0.061
OW21	0.2840(2)	0.1457(2)	0.77235(9)	0.0451(4)
H21A	0.252(3)	0.105(3)	0.7983(13)	0.054
H21B	0.242(3)	0.193(3)	0.7578(14)	0.054
OW22	0.4686(3)	0.1171(2)	0.18337(11)	0.0596(6)
H22A	0.443(4)	0.171(3)	0.2012(15)	0.072
H22B	0.443(4)	0.105(4)	0.1498(11)	0.072
OW23	0.0195(3)	0.5360(2)	0.37978(15)	0.0771(8)

TABLE 4. CONTINUED.

	x/a	y/b	z/c	U_{eq}		
H23A	0.100(3)	0.513(4)	0.3921(19)	0.093		
H23B	-0.004(4)	0.502(4)	0.4182(15)	0.093		
OW24	0.0811(3)	0.5347(3)	0.86385(12)	0.0760(8)		
H24A	0.041(4)	0.476(3)	0.881(2)	0.091		
H24B	0.091(4)	0.601(3)	0.8812(19)	0.091		
OW25	-0.0002(3)	0.9785(3)	0.42692(14)	0.0726(7)		
H25A	-0.067(3)	0.930(3)	0.4086(19)	0.087		
H25B	0.020(4)	1.042(3)	0.4084(19)	0.087		
OW26	0.0838(3)	0.4634(3)	0.25242(14)	0.0729(7)		
H26A	0.020(3)	0.454(4)	0.266(2)	0.087		
H26B	0.114(4)	0.404(3)	0.247(2)	0.087		
OW27*	0.4274(12)	0.3205(9)	0.0298(11)	0.042(3)		
H27A*	0.3624(19)	0.281(3)	0.0386(16)	0.050		
H27B*	0.483(2)	0.285(3)	0.0347(16)	0.050		
OW28*	0.4400(11)	0.3127(16)	0.0557(19)	0.064(4)		
OW29 [§]	0.7297(10)	0.4546(10)	0.2151(6)	0.068(3)		
H29A [§]	0.657(3)	0.439(4)	0.2153(18)	0.082		
H29B [§]	0.748(4)	0.457(4)	0.1791(16)	0.082		
OW30 [§]	0.6966(10)	0.4921(11)	0.1922(6)	0.056(3)		
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mg1	0.0249(4)	0.0242(3)	0.0301(4)	0.0040(3)	-0.0027(3)	0.0043(3)
Mg2	0.0271(4)	0.0256(4)	0.0266(3)	0.0012(3)	-0.0014(3)	0.0084(3)
Mg3	0.0317(4)	0.0331(4)	0.0345(4)	-0.0064(3)	0.0080(3)	0.0095(3)
V1	0.02224(16)	0.01562(15)	0.02573(16)	0.00174(12)	0.00294(13)	0.00437(12)
V2	0.02300(16)	0.01963(16)	0.02435(16)	0.00620(12)	0.00645(13)	0.00503(12)
V3	0.02096(16)	0.02608(17)	0.02807(17)	0.00368(13)	0.00632(13)	0.00860(13)
V4	0.02915(18)	0.02639(18)	0.01974(16)	-0.00028(13)	0.00011(13)	0.00926(14)
V5	0.01570(15)	0.01813(15)	0.02184(16)	0.00239(12)	-0.00074(12)	0.00384(11)
V6	0.02310(16)	0.02027(16)	0.01790(15)	-0.00090(12)	-0.00155(12)	0.00494(12)
V7	0.02183(16)	0.02296(16)	0.01703(15)	0.00331(12)	0.00272(12)	0.00622(12)
V8	0.01758(16)	0.02826(18)	0.02792(18)	0.00272(14)	0.00145(13)	0.00339(13)
V9	0.02657(17)	0.01774(16)	0.02603(17)	0.00190(12)	0.00100(13)	0.00400(13)
V10	0.01847(15)	0.01831(15)	0.01775(14)	0.00048(11)	0.00155(11)	0.00762(12)
O1	0.0355(9)	0.0189(7)	0.0438(9)	0.0041(6)	0.0060(7)	0.0049(6)
O2	0.0396(9)	0.0250(8)	0.0407(9)	0.0121(7)	0.0137(7)	0.0061(7)
O3	0.0266(8)	0.0408(9)	0.0438(9)	0.0048(7)	0.0096(7)	0.0144(7)
O4	0.0499(10)	0.0438(10)	0.0249(8)	-0.0049(7)	-0.0065(7)	0.0185(8)
O5	0.0182(6)	0.0260(7)	0.0296(7)	0.0031(6)	0.0009(5)	0.0065(5)
O6	0.0234(7)	0.0277(7)	0.0248(7)	-0.0009(6)	-0.0032(5)	0.0071(6)
O7	0.0292(8)	0.0308(8)	0.0240(7)	0.0014(6)	0.0070(6)	0.0124(6)
O8	0.0233(7)	0.0222(7)	0.0297(7)	0.0038(5)	0.0038(6)	0.0090(5)
O9	0.0265(7)	0.0214(7)	0.0278(7)	-0.0037(6)	-0.0009(6)	0.0043(6)
O10	0.0218(7)	0.0258(7)	0.0312(7)	0.0066(6)	0.0083(6)	0.0055(6)
O11	0.0318(8)	0.0281(7)	0.0207(7)	0.0059(5)	0.0045(6)	0.0097(6)
O12	0.0170(6)	0.0177(6)	0.0250(7)	0.0019(5)	0.0016(5)	0.0017(5)
O13	0.0199(6)	0.0187(6)	0.0225(6)	0.0046(5)	0.0010(5)	0.0058(5)
O14	0.0190(6)	0.0173(6)	0.0216(6)	0.0029(5)	0.0026(5)	0.0042(5)
O15	0.0376(9)	0.0345(8)	0.0206(7)	-0.0019(6)	-0.0022(6)	0.0089(7)
O16	0.0336(8)	0.0364(8)	0.0210(7)	0.0059(6)	0.0038(6)	0.0115(7)
O17	0.0203(7)	0.0440(10)	0.0443(10)	0.0051(8)	0.0034(7)	0.0055(7)
O18	0.0429(9)	0.0211(7)	0.0389(9)	0.0025(6)	0.0021(7)	0.0062(7)
O19	0.0204(7)	0.0262(7)	0.0267(7)	0.0020(6)	0.0033(5)	0.0100(5)

TABLE 4. CONTINUED.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O20	0.0253(7)	0.0210(7)	0.0246(7)	0.0001(5)	0.0021(5)	0.0098(5)
O21	0.0252(7)	0.0241(7)	0.0334(8)	0.0036(6)	0.0020(6)	0.0018(6)
O22	0.0214(7)	0.0277(7)	0.0254(7)	0.0010(6)	−0.0030(5)	0.0053(6)
O23	0.0307(8)	0.0200(7)	0.0240(7)	−0.0018(5)	−0.0022(6)	0.0056(6)
O24	0.0220(7)	0.0302(8)	0.0240(7)	0.0048(6)	0.0060(5)	0.0072(6)
O25	0.0255(7)	0.0220(7)	0.0241(7)	0.0057(5)	0.0035(5)	0.0063(5)
O26	0.0231(7)	0.0218(7)	0.0170(6)	0.0008(5)	0.0028(5)	0.0089(5)
O27	0.0194(6)	0.0195(6)	0.0178(6)	0.0008(5)	−0.0006(5)	0.0069(5)
O28	0.0196(6)	0.0189(6)	0.0192(6)	0.0014(5)	0.0020(5)	0.0054(5)
OW1	0.0388(10)	0.0515(11)	0.0358(9)	0.0092(8)	0.0022(8)	0.0247(8)
OW2	0.0386(10)	0.0265(8)	0.0417(9)	0.0035(7)	−0.0127(7)	0.0067(7)
OW3	0.0262(8)	0.0298(9)	0.0563(11)	0.0148(8)	−0.0017(8)	0.0013(7)
OW4	0.0361(9)	0.0406(10)	0.0272(8)	0.0077(7)	−0.0035(7)	0.0056(7)
OW5	0.0301(9)	0.0288(9)	0.0537(11)	0.0117(8)	−0.0058(8)	0.0003(7)
OW6	0.0334(10)	0.0482(11)	0.0459(11)	0.0048(9)	0.0060(8)	0.0120(8)
OW7	0.0292(8)	0.0355(9)	0.0383(9)	0.0101(7)	−0.0058(7)	0.0061(7)
OW8	0.0493(11)	0.0305(9)	0.0409(10)	0.0030(7)	−0.0113(8)	0.0171(8)
OW9	0.0515(11)	0.0274(8)	0.0369(9)	−0.0057(7)	−0.0154(8)	0.0170(8)
OW10	0.0572(12)	0.0337(10)	0.0514(11)	0.0033(8)	0.0246(9)	0.0103(9)
OW11	0.0347(9)	0.0439(10)	0.0303(8)	−0.0013(7)	−0.0055(7)	0.0175(7)
OW12	0.0546(12)	0.0413(11)	0.0479(11)	0.0061(9)	0.0211(9)	0.0075(9)
OW13	0.0363(10)	0.0667(14)	0.0507(12)	−0.0083(10)	0.0111(8)	0.0196(9)
OW14	0.0801(15)	0.0369(10)	0.0445(11)	−0.0081(9)	0.0192(11)	0.0111(10)
OW15	0.0405(10)	0.0451(11)	0.0345(9)	−0.0047(8)	0.0037(8)	−0.0042(8)
OW16	0.0855(16)	0.0381(11)	0.0540(12)	−0.0049(9)	0.0246(12)	0.0194(11)
OW17	0.0464(11)	0.0846(16)	0.0352(10)	−0.0068(10)	0.0108(8)	0.0288(11)
OW18	0.0336(11)	0.113(2)	0.0645(15)	−0.0420(15)	0.0121(10)	0.0062(12)
OW19	0.0398(11)	0.0597(13)	0.0457(11)	0.0065(10)	0.0084(9)	0.0038(9)
OW20	0.0569(13)	0.0624(14)	0.0416(11)	0.0103(10)	0.0130(10)	0.0275(11)
OW21	0.0522(12)	0.0448(11)	0.0399(10)	0.0007(8)	0.0184(9)	0.0088(9)
OW22	0.0721(16)	0.0527(14)	0.0539(13)	0.0081(11)	0.0131(12)	0.0130(11)
OW23	0.0578(15)	0.0532(14)	0.121(2)	0.0188(15)	0.0057(15)	0.0183(12)
OW24	0.121(2)	0.0603(16)	0.0573(15)	−0.0081(12)	0.0145(15)	0.0422(17)
OW25	0.0684(17)	0.0734(18)	0.0837(19)	0.0088(14)	0.0209(14)	0.0267(14)
OW26	0.0660(17)	0.0661(17)	0.0862(19)	−0.0230(14)	−0.0075(14)	0.0257(14)
OW27	0.028(3)	0.040(3)	0.052(6)	−0.010(3)	0.005(3)	−0.002(2)
OW28	0.040(3)	0.062(4)	0.087(12)	−0.032(6)	−0.001(5)	0.014(3)
OW29	0.059(4)	0.068(4)	0.078(5)	0.009(4)	0.006(3)	0.018(3)
OW30	0.049(4)	0.059(5)	0.060(5)	0.003(3)	0.008(3)	0.009(3)

* OW27 and OW28, separated by 0.56(2) Å, correspond to a split H₂O site, but share the same H atom sites. The linked refined occupancy is 0.50(6) for each.

§ OW29 and OW30, separated by 0.740(6) Å, are a split H₂O site, but share the same H atom sites. Their linked refined occupancies are 0.53(3) and 0.47(3), respectively.

methods using SIR2011 (Burla *et al.* 2012). SHELXL-2013 (Sheldrick 2015) was used to refine the structure. All non-hydrogen atoms were refined with anisotropic displacement parameters. Difference Fourier syntheses located all H atom positions. The H sites were refined with soft restraints of 0.82(3) Å on the O–H distances and 1.30(3) Å on the H–H distances and with the U_{eq} of each H set to 1.2 times that of the donor O atom. Data collection and refinement details are given in

Table 3, atom coordinates and displacement parameters in Table 4, and selected bond distances and bond-valence sums in Table 5.

ATOMIC ARRANGEMENT OF OKIEITE

The structure of okieite (Fig. 2) consists of two distinct parts, a structural unit and an interstitial complex, as suggested by Schindler & Hawthorne

TABLE 5. SELECTED BOND DISTANCES (Å) AND BOND VALENCES (BV)* (vu) IN OKIEITE

Bond	Distance	BV	Bond	Distance	BV	Bond	Distance	BV
Mg1–OW5	2.0380(18)	0.38	Mg2–OW10	2.044(2)	0.37	Mg3–OW17	2.050(2)	0.37
–OW2	2.0490(18)	0.37	–OW12	2.045(2)	0.37	–OW16	2.055(2)	0.36
–OW3	2.0639(18)	0.36	–OW9	2.0459(18)	0.37	–OW15	2.056(2)	0.36
–OW4	2.0852(18)	0.34	–OW8	2.0602(18)	0.36	–OW13	2.070(2)	0.35
–OW1	2.0919(19)	0.34	–OW7	2.0672(18)	0.35	–OW18	2.071(2)	0.35
–OW6	2.105(2)	0.33	–OW11	2.0977(18)	0.33	–OW14	2.074(2)	0.35
Mean, Σ	2.072	2.11	Mean, Σ	2.06	2.16	Mean, Σ	2.063	2.15
V1–O1	1.6081(15)	1.64	V2–O2	1.6036(16)	1.65	V3–O3	1.6036(15)	1.65
–O9	1.8207(15)	0.95	–O11	1.8299(15)	0.92	–O7	1.8505(15)	0.88
–O8	1.8331(14)	0.92	–O10	1.8393(15)	0.90	–O10	1.8696(15)	0.83
–O13	1.9929(14)	0.61	–O12	1.9952(14)	0.60	–O8	1.8710(15)	0.83
–O12	1.9956(14)	0.60	–O13	2.0056(14)	0.59	–O5	2.0495(15)	0.52
–O14	2.2142(13)	0.34	–O14	2.2416(13)	0.32	–O14	2.3249(13)	0.26
Mean, Σ	1.9108	5.05	Mean, Σ	1.9192	4.99	Mean, Σ	1.9282	4.98
V4–O4	1.6087(16)	1.63	V5–O6	1.6795(14)	1.36	V6–O15	1.6106(15)	1.63
–O7	1.8248(15)	0.94	–O5	1.7005(14)	1.29	–O22	1.8116(15)	0.97
–O9	1.8752(15)	0.82	–O12	1.9216(13)	0.73	–O23	1.8410(14)	0.90
–O11	1.8781(15)	0.82	–O13	1.9246(14)	0.72	–O27	1.9794(13)	0.63
–O6	2.0514(15)	0.52	–O14	2.1294(13)	0.43	–O26	2.0257(14)	0.56
–O14	2.3226(13)	0.26	–O14	2.1338(13)	0.42	–O28	2.2432(13)	0.32
Mean, Σ	1.9268	4.99	Mean, Σ	1.9149	4.95	Mean, Σ	1.9186	4.99
V7–O16	1.6107(14)	1.62	V8–O17	1.6042(16)	1.65	V9–O18	1.6116(16)	1.62
–O24	1.8196(14)	0.95	–O21	1.8414(16)	0.90	–O21	1.8259(15)	0.93
–O25	1.8435(14)	0.89	–O24	1.8666(14)	0.84	–O23	1.8617(14)	0.85
–O26	1.9999(14)	0.60	–O22	1.8996(15)	0.77	–O25	1.8909(14)	0.79
–O27	2.0086(14)	0.58	–O19	2.0308(15)	0.55	–O20	2.0500(15)	0.52
–O28	2.2400(13)	0.32	–O28	2.3175(13)	0.26	–O28	2.3001(13)	0.27
Mean, Σ	1.9204	4.96	Mean, Σ	1.9267	4.97	Mean, Σ	1.9234	4.99
V10–O20	1.6822(14)	1.35						
–O19	1.6874(14)	1.33						
–O26	1.9320(13)	0.71						
–O27	1.9394(13)	0.70						
–O28	2.1236(13)	0.43						
–O28	2.1289(13)	0.43						
Mean, Σ	1.9156	4.95						

* Bond valence parameters are from Gagné & Hawthorne (2015).

(2001) for such hydrated minerals with a polyanion. The structural unit is the $\{V_{10}O_{28}\}^{6-}$ decavanadate cluster, which is also found in numerous other new Urvan Mineral Belt minerals. Three Mg atom sites in the interstitial complex are each octahedrally coordinated by six H_2O groups and do not share any bonds with the decavanadate group or with other $Mg(H_2O)_6$ octahedra. The interstitial complex also includes 12 isolated H_2O sites, four of which occur in pairs of approximately half-occupied sites. The formula of the interstitial unit in okieite can be expressed structurally as $\{[Mg(H_2O)_6]_3 \cdot 10(H_2O)\}^{6+}$.

Bonding between the $\{V_{10}O_{28}\}^{6-}$ cluster and the $\{[Mg(H_2O)_6]_3 \cdot 10H_2O\}^{6+}$ interstitial complex is weak, as it is in many hydrated decavanadate structures. The linkage between the two structural components is *via* hydrogen bonding between the hydrogen atoms of the interstitial complex and the outermost oxygen atoms of the decavanadate group; the weak bonding in three dimensions is reflected in the low hardness, conchoidal fracture, and lack of cleavage of okieite.

Synthetic decavanadate compounds have been investigated extensively for their material properties. As with many of the decavanadate minerals, the synthetic analog of okieite has previously been characterized; the mineral

corresponds to $\text{Mg}_3\text{V}_{10}\text{O}_{28}\cdot 28\text{H}_2\text{O}$ as synthesized by Iida & Ozeki (2004).

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REFERENCES

- BURLA, M.C., CALIANDRO, R., CAMALLI, M., CARROZZINI, B., CASCARANO, G.L., GIACOVAZZO, C., MALLAMO, M., MAZZONE, A., POLIDORI, G., & SPAGNA, R. (2012) *SIR2011*: a new package for crystal structure determination and refinement. *Journal of Applied Crystallography* **45**, 357–361.
- CARTER, W.D. & GUALTIERI, J.L. (1965) Geology and uranium–vanadium deposits of the La Sal quadrangle, San Juan County, Utah, and Montrose County, Colorado. *United States Geological Survey Professional Paper* **508**.
- EVANS, H.T., JR. (1991) Metamunirite, a new anhydrous sodium metavanadate from San Miguel County, Colorado. *Mineralogical Magazine* **55**, 509–513.
- GAGNÉ, O.C. & HAWTHORNE, F.C. (2015) Comprehensive derivation of bond-valence parameters for ion pairs involving oxygen. *Acta Crystallographica* **B71**, 562–578.
- HIGASHI, T. (2001) *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- IIDA, A. & OZEKI, T. (2004) $\text{Mg}_2\text{Na}_2\text{V}_{10}\text{O}_{28}\cdot 20\text{H}_2\text{O}$ and $\text{Mg}_3\text{V}_{10}\text{O}_{28}\cdot 28\text{H}_2\text{O}$. *Acta Crystallographica* **C60**, i43–i46.
- KAMPF, A.R., HUGHES, J.M., MARTY, J., NASH, B.P., CHEN, Y.-S., & STEELE, I.M. (2015) Bluestreakite, $\text{K}_4\text{Mg}_2(\text{V}^{4+}_2\text{V}^{5+}_8\text{O}_{28})\cdot 14\text{H}_2\text{O}$, a new mixed-valence decavanadate mineral from the Blue Streak mine, Montrose County, Colorado: Crystal structure and descriptive mineralogy. *Canadian Mineralogist* **53**, 1007–1018.
- KAMPF, A.R., NASH, B.P., HUGHES, J.M., & MARTY, J. (2017) Burroite, $\text{Ca}_2(\text{NH}_4)_2(\text{V}_{10}\text{O}_{28})\cdot 15\text{H}_2\text{O}$, a new decavanadate mineral from the Burro mine, San Miguel County, Colorado. *Canadian Mineralogist* **55**, 473–481.
- KAMPF, A.R., PLÁŠIL, J., OLDS, T.A., NASH, B.P., & MARTY, J. (2018a) Ammoniozippelite, a new uranyl sulfate mineral from the Blue Lizard mine, San Juan County, Utah, and the Burro mine, San Miguel County, Colorado, USA. *Canadian Mineralogist* **56**, 235–245.
- KAMPF, A.R., NASH, B.P., ADAMS, P.M., MARTY, J., & HUGHES, J.M. (2018b) Ammoniolasalite, $(\text{NH}_4)_2\text{Mg}_2(\text{H}_2\text{O})_{26}[\text{V}_{10}\text{O}_{28}]$, a new decavanadate species from the Burro Mine, Slick Rock District, Colorado. *Canadian Mineralogist* **56**, 859–870.
- KAMPF, A.R., PLÁŠIL, J., OLDS, T.A., NASH, B.P., & MARTY, J. (2019a) Ammoniomathiesiusite, a new uranyl sulfate–vanadate mineral from the Burro mine, San Miguel County, Colorado, USA. *Mineralogical Magazine* **83**, 115–121.
- KAMPF, A.R., PLÁŠIL, L., NASH, B.P., NĚMEC, I., & MARTY, J. (2019b) Uroxite and metauroxite, the first two uranyl–oxalate minerals. *Mineralogical Magazine* **84**, DOI: 10.1180/mgm.2019.57 (in press).
- KAMPF, A.R., HUGHES, J.M., NASH, B.P., & MARTY, J. (2019c) Bicapite, $\text{KNa}_2\text{Mg}_2(\text{H}_2\text{PV}^{5+}_{14}\text{O}_{42})\cdot 25\text{H}_2\text{O}$, a new mineral with a bicapped Keggin anion from the Pickett Corral mine, Montrose County, Colorado, USA. *American Mineralogist* **103**, 1851–1856.
- KAMPF, A.R., COOPER, M.A., HUGHES, J.M., NASH, B.P., MARTY, J., & HAWTHORNE, F.C. (2020) Caseyite, a new mineral containing a variant of the flat- Al_{13} polyoxometalate cation. *American Mineralogist* **104**, DOI: 10.2138/am-2019-7181 (in press).
- MANDARINO, J.A. (1981) The Gladstone–Dale relationship. IV. The compatibility concept and its application. *Canadian Mineralogist* **19**, 441–450.
- POUCHOU, J.-L. & PICHOR, F. (1991) Quantitative analysis of homogeneous or stratified microvolumes applying the model “PAP”. In *Electron Probe Quantitation* (K.F.J. Heinrich & D.E. Newbury, eds.). Plenum Press, New York, United States (31–75).
- SCHINDLER, M. & HAWTHORNE, F.C. (2001) A bond-valence approach to the structure, chemistry, and paragenesis of hydroxyl-hydrated oxysalt minerals. I. Theory. *Canadian Mineralogist* **39**, 1225–1242.
- SHAW, D.R. (2011) Uranium–vanadium deposits of the Slick Rock district, Colorado. *United States Geological Survey Professional Paper* **576-F**.
- SHELDRIK, G.M. (2015) Crystal Structure refinement with *SHELX*. *Acta Crystallographica* **C71**, 3–8.
- WEEKS, A.D., CISNEY, E.A., & SHERWOOD, A.M. (1951) Hummerite and montroseite, two vanadium minerals from Montrose County, Colorado. Abstracts of papers presented at the thirty-first annual meeting of the Mineralogical Society of America at Washington, D.C. *American Mineralogist* **36**, 310–328.

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