BURROITE, Ca₂(NH₄)₂(V₁₀O₂₈)·15H₂O, A NEW DECAVANADATE MINERAL FROM THE BURRO MINE, SAN MIGUEL COUNTY, COLORADO

ANTHONY R. KAMPF[§]

Mineral Sciences Department, Natural History Museum of Los Angeles County, 900 Exposition Boulevard, Los Angeles, California 90007, U.S.A.

BARBARA P. NASH

Department of Geology and Geophysics, University of Utah, Salt Lake City, Utah 84112, U.S.A.

JOHN M. HUGHES

Department of Geology, University of Vermont, Burlington, Vermont 05405, U.S.A. Department of Geology and Environmental Earth Science, Miami University, Oxford, Ohio 45056, U.S.A.

JOE MARTY

5199 E. Silver Oak Rd., Salt Lake City, Utah 84108, U.S.A.

Abstract

Burroite, $Ca_2(NH_4)_2(V_{10}O_{28}) \cdot 15H_2O$, is a new mineral species (IMA2016-079) discovered in the Burro mine, Slick Rock district, San Miguel County, Colorado, U.S.A. $(38^{\circ}2'42''N \ 108^{\circ}53'23''W)$. The mineral is found as orange-yellow, somewhat flattened prisms up to 2 mm in length occurring on a montroseite- and corvusite-bearing sandstone. Burroite has yellow streak, vitreous luster, brittle tenacity, a Mohs hardness of $1\frac{1}{2}$ -2, good cleavage on {001}, and irregular fracture. The measured density is 2.43(2) g·cm⁻³. The partially determined optical properties are $\alpha = 1.764(3)$, $\beta = n.d.$, $\gamma > 1.81$, orientation $X \approx \mathbf{a}$, *Y* probably $\approx \mathbf{c}^*$. Electron probe microanalysis gave the empirical formula (based on 43 O *apful*) [Ca_{1.88}(NH₄)_{1.82}Na_{0.18}]_{23.88}(H_{0.23}V⁵⁺₁₀O₂₈)·15H₂O. Burroite is triclinic, space group *P*I, with *a* 8.779(2), *b* 10.311(2), *c* 12.060(2) Å, $\alpha 96.740(4)^{\circ}$, $\beta 107.388(5)^{\circ}$, and $\gamma 114.439(6)^{\circ}$, and V = 911.2(3) Å³. The strongest four lines in the diffraction pattern are [*d* in Å(*I*)(*hkl*)]: 11.06(100)(001), 9.02(46)(010), 8.10(21)(0Ī1), and 7.71(94)(TI0,TI01). The atomic arrangement of burroite is the [V₁₀O₂₈]⁶⁻ decavanadate group; charge balance in the structure is maintained by the [Ca₂(NH₄)₂·15H₂O]⁶⁺ interstitial complex. Linkage between the structural unit and the components of the interstitial complex is principally by hydrogen bonding. The mineral is named for the Burro mine in which it was found.

Keywords: burroite, new mineral species, decavanadate, crystal structure, Burro mine, San Miguel County, Colorado, USA.

INTRODUCTION

The Uravan Mineral Belt of the Colorado Plateau, which spans the Colorado–Utah border, has been a rich source of uranium and vanadium ores. The ores are contained in roll-front deposits in the sandstone of the Salt Wash member of the Jurassic Morrison Formation (Carter & Gualtieri 1965, Shawe 2011). In these deposits, U^{6+} and V^{4+} were transported as dissolved species in weakly alkaline, moderately reducing CO₂-rich groundwater, and, upon encountering more reducing conditions by contacting carbonaceous material and H₂S (Evans & Garrels 1958, Hostetler & Garrels 1962), were deposited as uraninite, $U^{4+}O_2$, and

[§] Corresponding author e-mail address: akampf@nhm.org

montroseite, V³⁺O(OH). Subsequent exposure of those minerals to near-surface solutions, which had reacted with sulfide minerals and become more acidic, resulted in the crystallization of numerous secondary phases.

Thirteen of the 15 previously known decavanadate minerals (see Kampf *et al.* 2017) were first described from deposits in the Uravan Mineral Belt. Those compounds contain the $[V_{10}O_{28}]^{6-}$ decavanadate cluster, or its structural variants, as the structural unit; the structural variants are protonated decavanadate units $([H_xV_{10}O_{28}]^{(6-x)-})$ and the mixed-valence decavanadate group $([(V^{4+}_xV^{5+}_{10-x})O_{28}]^{(6+x)-})$.

To the decavanadate minerals referenced above, we can add the new mineral burroite, $Ca_2(NH_4)_2$ ($V_{10}O_{28}$)·15H₂O; the mineral name is derived from the Burro mine, Slick Rock district, San Miguel County, Colorado, U.S.A., where the mineral was discovered. Burroite was approved by the Commission on New Minerals, Nomenclature, and Classification of the International Mineralogical Association (IMA2016–079). One holotype and one cotype specimen of burroite, both micromounts split from the single specimen that contains the new phase, are deposited in the collections of the Natural History Museum of Los Angeles County, Los Angeles, California, U.S.A., with catalogue numbers 66299 and 66300, respectively.

OCCURRENCE

Burroite was found underground at the Burro mine, Slick Rock district, San Miguel County, Colorado, U.S.A. (38°2'42″N 108°53'23″W). The Burro mine is near the southern end of the Uravan Mineral Belt, in which uranium and vanadium minerals occur together in bedded or roll-front deposits in the sandstone of the Salt Wash member of the Jurassic Morrison Formation (Carter & Gualtieri 1965, Shawe, 2011). The sample of the new mineral was collected by one of the authors (JM) on May 27, 2014.

Mining in the northern portion of the Slick Rock district of the Uravan Mineral Belt dates back to about 1900, when the area was known as the McIntyre district. That district was combined with the Dolores district to the south in the late 1940s to form the Slick Rock district. The McIntyre district was one of the first areas of the Colorado Plateau region to be recognized for its deposits rich in uranium, vanadium, and radium. Mining for the elements was sporadic throughout the first half of the 20th century; uranium production in the mid-1940s provided the material for the first atomic bombs.

There is no Burro mine mentioned in early mining reports within the area originally known as the McIntyre district; apparently, no Burro mine was established in the area prior to the 1950s. In December 1955, the Union Carbide Nuclear Corporation (later to become the Union Carbide Corporation) began sinking three two-compartment, cribbed shafts of the Burro No. 3, 5, and 7 mines. Mining began in the first quarter of 1957 at the Burro Mines complex, which consisted of the Burro lode claims (1 through 10), located north and immediately adjacent to the U.S. Department of Energy's C-SR-13 uranium lease tract, and the Jack lode claims (1 through 5), located north of the Burro claims. Union Carbide continued to operate the mines at least until 1970, the last year for which Atomic Energy Commission (AEC) production records were reported. Through 1970, the Burro Mines complex produced 361,430 tonnes of ore at a U grade of 0.25%, yielding 903,960 kg of U₃O₈, and a V grade of 1.5%, yielding 5,510,990 kg of V₂O₅. Subsequently, Umetco Minerals Corporation continued mining until 1983, when depressed uranium prices caused the cessation of mining in the district.

The foregoing history is largely taken from Shawe (2011), from Davis & Sim (2013), from AEC-PED-1 (Mine Operation Data Report, January 1, 1959, Production Evaluation Division, Grand Junction Operations Office, U.S. Atomic Energy Commission, page 158), and from Ed Cotter (*pers. commun.*).

The extensive underground workings of the Burro mines are interconnected and they are now simply referred to collectively as the Burro mine. In recent years, all but one of the portals have been sealed and some exterior mine areas have been reclaimed; the one unsealed portal is securely gated. A portion of the Burro mine is leased from the U.S. Department of Energy by Gold Eagle Mining Inc. of Montrose, Colorado, under the ownership of Don Coram, who granted us access on several occasions. Our collecting of post-mining secondary minerals has been limited to a relatively small portion of the underground workings.

Burroite is very rare; it has thus far been found on only two specimens. It occurs on montroseite- and corvusite-bearing sandstone in an apparently NH_4 -rich secondary assemblage that also contains the NH_4 bearing decavanadates schindlerite and wernerbaurite. Other secondary minerals found in the mine include barnesite, gypsum, hewettite, magnesiopascoite, metamunirite (Evans 1991), metarossite, navajoite, pascoite, rossite, sherwoodite, and other potentially new minerals, currently under study.

The new mineral forms from the oxidation of montroseite-corvusite assemblages in a moist environment. Mining operations have exposed both unoxidized and oxidized phases. Under ambient temperatures and generally oxidizing near-surface conditions, water reacts with pyrite to form aqueous



FIG. 1. Burroite (yellow orange) with schindlerite (darker orange); FOV 3.5 mm across.

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.*, NH₄⁺, Na⁺, Ca²⁺, Mn²⁺, Pb²⁺). The NH₄⁺ presumably derives from organic matter in the deposit.

PHYSICAL AND OPTICAL PROPERTIES

Burroite is orange-yellow, and the crystals form as short, somewhat flattened prisms to 2 mm in length; the faces of burroite crystals typically have rounded, craterlike depressions (Figs. 1 and 2). The mineral is nonfluorescent. Crystals are transparent to translucent, display a vitreous luster, and exhibit brittle tenacity. Burroite displays a good cleavage on {001}, irregular fracture, and a yellow streak. The mineral has a Mohs hardness of about 1¹/₂-2, based upon scratch tests. No parting was observed. The density of burroite measured by flotation in a mixture of methylene iodide and toluene is 2.43(2) g cm⁻³; the density calculated using the empirical formula is 2.442 g·cm⁻³ and using the ideal formula is 2.449 g·cm⁻³. At room temperature, burroite is very slowly soluble in H₂O and rapidly soluble (within seconds) in dilute HCl.



FIG. 2. Crystal drawing of burroite; clinographic projection in non-standard orientation, **a** vertical.

The measurement of optical properties of burroite was problematic for several reasons. The crystals were of poor quality and, in particular, have a very high mosaicity that made optical determinations, including extinction observations, very difficult. In addition, the unfavorable orientation of the optical indicatrix made conoscopic observation and the determination of β impossible. Finally, the decomposition of crystals in

Constituent	Mean	Range	S.D.	Normalized	Standard
(NH ₄) ₂ O	4.11	3.82-4.37	0.23	3.54	syn. Cr ₂ N [§]
Na ₂ O	0.49	0.36-0.70	0.12	0.42	albite
CaO	9.14	9.02-9.29	0.08	7.88	diopside
V_2O_5	78.74	77.56-80.59	1.06	67.84	V metal
H ₂ O	7.52*			20.32 [†]	
Total	100.00			100.00	

TABLE 1. ANALYTICAL DATA IN (wt.%) FOR BURROITE

* By difference

[†] Based upon the crystal structure with V = 10 and O = 43 apfu.

§ Secondary standards: GaN and HfN

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

$I_{\rm obs}$	dobs		d_{calc}	<i>I</i> calc	hkl	lobs	$d_{\rm obs}$		d_{calc}	<i>I</i> calc	hkl
100	11.06		11.0572	100	001	6	2.870		2.8839	5	103
46	9.02		9.0188	49	010				2.8045	3	232
21	8.10		8.1895	23	011	16	2 778	5	2.7834	7	320
01	7 71	5	7.8019	52	110	10	2.110	J	2.7643	4	004
34	1.11	l	7.6413	85	101				2.7537	2	114
17	7.50		7.3789	20	100	6	2 705	5	2.7163	2	024
13	6.92		6.9417	11	111	0	2.705	J	2.6908	3	133
8	6.143		6.1973	8	011	8	2.604		2.6099	8	211
12	5.912		5.9273	11	110				2.5471	2	303
			5.5689	2	102				2.4735	3	113
13	5.447		5.4325	13	012			(2.4596	2	300
			5.1239	2	111	13	2.454	1	2.4552	6	233
7	4 694	Ş	4.7197	4	110			(2.4404	3	241
'	1.001	l	4.6509	5	021			(2.3921	4	034
7	4.530		4.5191	6	121	5	2.379	1	2.3784	2	124
7	4 190	Ş	4.1938	4	112			(2.3598	3	220
'	4.100	l	4.1777	3	210	5	2 311	Ş	2.3139	2	333
7	3 947	Ş	3.9615	3	103	0	2.011	l	2.3057	3	134
'	0.047	l	3.9010	7	220				2.2666	2	311
			3.8871	2	221				2.1722	4	342
11	3.832		3.8206	9	202				2.1432	2	221
4	3.701		3.6871	7	113	8	2.1256		2.1306	8	310
9	3.489		3.5006	10	221	10	2 0750	5	2.0776	7	035
			3.4037	2	130	15	2.0750	l	2.0741	7	334
٥	3 334	5	3.3443	2	023	3	2.0047		2.0128	3	343
9	0.004	l	3.3229	6	203	3	1.9815		1.9825	3	135
11	2 1 1 5	5	3.1290	4	031				1.9526	2	311
	5.115	J	3.1220	6	013				1.9293	2	$\overline{3}$ $\overline{2}$ 1
11	2 010	5	3.0490	5	123				1.8563	2	443
	3.010)	3.0063	8	030	7	1 0 0 0 7	5	1.8305	4	145
		(2.9637	4	222	/	1.0207	5	1.8261	3	320
14	2.950	{	2.9536	6	213	4	4 7000	ſ	1.7363	2	523
			2.9337	3	223	4	1.7308	1	1.7354	2	444

* Only calculated lines with l > 1 are included.

index of refraction liquids >1.8 made the measurement of γ impossible. For these reasons the optical data are not complete.

The optical sign of burroite was not determined. For burroite, $\alpha = 1.764(3)$, $\beta = n.d.$, $\gamma > 1.81$ (white light); the average *n* calculated using the Gladstone-Dale relationship is 1.816. Dispersion could not be observed, and 2*V* could not be measured or calculated. No pleochroism was observed, and orientation was determined as $X \approx \mathbf{a}$; *Y* probably $\approx \mathbf{c}^*$.

CHEMICAL COMPOSITION

Analyses (eight points on four crystals) were performed 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 keV accelerating voltage, 10 nA beam current, and a beam diameter of 10 μ m. Counting times were 30 s on peak and 30 s on background for each element. There was no observed damage from the electron beam. Background positions for NK α were selected to accommodate the proximity of the first order VL1 line. A time-dependent intensity correction was applied to Na, Ca, and V; the N intensity was time independent. Raw X-ray intensities were corrected for matrix effects with a $\phi p(z)$ algorithm (Pouchou & Pichoir 1991).

Partial dehydration of the very water-rich crystals occurred when they were subjected to an ultimate vacuum of 2×10^{-5} torr for approximately 5 min during vacuum deposition of the conductive carbon coat. Although there could have been additional loss of H₂O from the sample immediately upon exposure to the high vacuum of the microprobe chamber (~10⁻⁶ torr), continued loss of H₂O from the sample evidently did not occur, as there were no systematic increases in concentration of constituents measured during the analytical session.

The H₂O loss results in higher concentrations for the remaining constituents than are to be expected for the fully hydrated phase. Because insufficient material is available for a direct determination of H₂O, it was calculated based upon the structure determination. The analyzed constituents were then normalized to provide a total of 100% when combined with the calculated H₂O content. Analytical data are given in Table 1. For those data, the empirical formula (based on 43 O *apfu*) is $[Ca_{1.88}(NH_4)_{1.82}Na_{0.18}]_{\Sigma_3.88}(H_{0.23}V^{5+}_{10}O_{28}) \cdot 15H_2O$. The simplified structural formula is $Ca_2(NH_4)_2$ ($V_{10}O_{28}$) · 15H₂O, which requires (NH₄)₂O 3.88, CaO 8.35, V₂O₅ 67.67, H₂O 20.11, total 100.00 wt.%.

CRYSTAL STRUCTURE: EXPERIMENTAL

All crystals examined exhibit very high mosaicity, manifested as generally broad and diffuse reflections; nevertheless, we were successful in integrating the reflections and solving and refining the structure, but H-atom positions and the finest details of the burroite atomic arrangement await better crystals. Singlecrystal X-ray studies were carried out using a Rigaku R-Axis Rapid II curved imaging plate microdiffractometer with monochromatized MoKa radiation; Xray powder diffraction data were recorded using the same diffractometer and radiation. A Gandolfi-like motion on the ϕ and ω axes was used to randomize the single-crystal sample for the powder measurements. Observed d values and intensities were derived by profile fitting using JADE 2010 software (Materials Data, Inc.); data (in Å) are given in Table 2.

The single-crystal structure data were processed using the Rigaku CrystalClear software package and included the application of numerical and empirical absorption corrections, the latter with a multi-scan approach using ABSCOR (Higashi 2001). The struc-

Diffractometer	Rigaku R-Axis Rapid II	
X-ray radiation/power	Mo <i>K</i> a ($\lambda = 0.71075$ Å)/50 kV	/, 40 mA
Temperature	298(2) K	
Structural Formula	Ca ₂ (NH ₄) ₂ (V ₁₀ O ₂₈)·15H ₂ O	
Space group	P1	
Unit cell dimensions	<i>a</i> = 8.779(2) Å	$\alpha = 96.740(4)^{\circ}$
	b = 10.311(2) Å	$\beta = 107.388(5)^{\circ}$
	c = 12.060(2) Å	$\gamma = 114.439(6)^{\circ}$
V	911.2(3) Å ³	,
Ζ	1	
Density (for above formula)	2.449 g⋅cm ⁻³	
Absorption coefficient	2.858 mm ⁻¹	
F(000)	666	
Crystal size	150 $ imes$ 100 $ imes$ 30 μ m	
θ range	2.26 to 25.05°	
Index ranges	-10 < h < 10, -11 < k < 12	., −14 < <i>l</i> < 14
Reflections collected/unique	11666/3196; $R_{\rm int} = 0.057$	
Reflections with $F_0 > 4\sigma(F)$	2711	
Completeness to $\theta = 25.05^{\circ}$	99.00%	
Min./max. transmission	0.674/0.919	
Refinement method	Full-matrix least-squares on	F ²
Restraints/parameters	0/262	
GoF	1.197	
Final R indices $[F > 4\sigma(F)]$	$R_1 = 0.0946, wR_2 = 0.2607$	
R indices (all data)	$R_1 = 0.1073$. $wR_2 = 0.2737$	
Largest diff. peak/hole	+0.68/-0.57 e A ⁻³	

TABLE 3. DATA COLLECTION AND STRUCTURE REFINEMENT DETAILS FOR BURROITE

* $R_{\text{int}} = \Sigma |F_o^2 - F_o^2(\text{mean})| \Sigma [F_o^2]$. GoF = $S = \{\Sigma [w(F_o^2 - F_o^2)^2]/(n-p)\}^{1/2}$. $R_1 = \Sigma ||F_o| - |F_c||/\Sigma |F_o|$. $wR_2 = \{\Sigma [w(F_o^2 - F_o^2)^2]/\Sigma [w(F_o^2)^2]\}^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where *a* is 0.1098, *b* is 1.8098 and *P* is $[2F_c^2 + \text{Max}(F_o^2, 0)]/3$

ture was solved by direct methods as implemented in SIR2011 (Burla *et al.* 2012). SHELXL-2013 (Sheldrick 2015) was used for the refinement of the structure. All non-hydrogen atoms were refined with anisotropic displacement parameters; because of the marginal quality of the data, it was not possible to differentiate N and O based upon site scattering, and H atom sites could not be unambiguously located in the difference-Fourier maps.

Data collection and refinement details are given in Table 3, atom coordinates and anisotropic displacement parameters in Table 4, and selected bond distances and bond-valence sums in Table 5. A CIF file that also contains observed and calculated structure factors has been deposited and is available from the Depository of Unpublished Data on the MAC website [document Burroite CM55_10.3749/canmin.1700004].

ATOMIC ARRANGEMENT OF BURROITE

The atomic arrangement of burroite (Fig. 3) consists of two distinct parts, a structural unit and an interstitial unit, as suggested by Schindler &

Hawthorne (2001) for such hydrated minerals with a polyanion. The structural unit is the $[V_{10}O_{28}]^{6-}$ decavanadate cluster that has been found in numerous new Colorado Plateau minerals as summarized in Kampf *et al.* (2017), who list the existing and new minerals that form with $[V_{10}O_{28}]^{6-}$ decavanadate clusters, including protonated $[H_xV_{10}O_{28}]^{(6-x)-}$ decavanadate clusters and $[(V^{4+}_xV^{5+}_{10-x})O_{28}]^{(6+x)-}$ mixed-valence decavanadate clusters.

The many minerals that contain the $[V_{10}O_{28}]^{6-}$ decavanadate cluster are similar in that they possess the same structural unit, but differ in the interstitial units that link those $[V_{10}O_{28}]^{6-}$ groups; the compositions of those interstitial units are given in Kampf *et al.* (2017). In burroite, the interstitial unit has the ideal formula $[Ca_2(NH_4)_2 \cdot 15H_2O]^{6+}$, which balances the charge on the structural unit. Each Ca atom in the interstitial unit, depicted in Figure 3, is bonded to eight oxygen atoms, seven of which belong to H₂O groups (Table 5); the eighth oxygen atom is a member of the decavanadate group. Two of these symmetrically equivalent Ca atoms link to form a Ca₂–(H₂O)₁₂ dimer, with the Ca polyhedra in a *trans* configuration

	x/a	y/b	Z/C	$U_{ m eq}$	U^{11}	U^{P2}	U ³³	U ^{P3}	U^{13}	U^{12}
Ca	0.6060(2)	0.9002(2)	0.40626(18)	0.0630(6)	0.0610(11)	0.0728(13)	0.0708(12)	0.0251(10)	0.0323(9)	0.0396(10)
۷1	0.18926(19)	0.01872(18)	0.98853(14)	0.0566(5)	0.0535(9)	0.0679(11)	0.0620(10)	0.0236(8)	0.0283(7)	0.0352(8)
V2	0.1531(2)	0.04788(19)	0.24205(14)	0.0599(5)	0.0590(9)	0.0706(11)	0.0599(10)	0.0224(8)	0.0251(7)	0.0366(8)
V3	0.0610(2)	0.25859(19)	0.94375(15)	0.0595(5)	0.0573(9)	0.0652(11)	0.0658(10)	0.0228(8)	0.0269(7)	0.0343(8)
V4	0.3879(2)	0.32623(19)	0.17727(15)	0.0621(5)	0.0572(9)	0.0670(11)	0.0682(11)	0.0207(8)	0.0254(8)	0.0333(8)
V5	0.0295(2)	0.2883(2)	0.19460(15)	0.0632(5)	0.0633(10)	0.0703(12)	0.0689(11)	0.0211(9)	0.0310(8)	0.0388(9)
9	0.0892(7)	0.1318(7)	0.0793(5)	0.0570(16)	0.054(3)	0.066(4)	0.059(4)	0.019(3)	0.024(3)	0.034(3)
02	0.1022(7)	0.1021(7)	0.8636(6)	0.0578(15)	0.053(3)	0.067(4)	0.065(4)	0.022(3)	0.029(3)	0.034(3)
ő	0.1830(8)	0.9261(7)	0.1179(5)	0.0578(15)	0.055(3)	0.069(4)	0.062(4)	0.027(3)	0.021(3)	0.039(3)
64	0.3972(8)	0.1664(8)	0.0650(6)	0.0619(16)	0.058(3)	0.078(4)	0.065(4)	0.028(3)	0.029(3)	0.040(3)
05	0.2135(8)	0.8944(8)	0.9002(6)	0.0601(16)	0.058(3)	0.077(4)	0.065(4)	0.024(3)	0.031(3)	0.042(3)
06	0.3788(7)	0.1988(7)	0.2768(6)	0.0599(16)	0.050(3)	0.066(4)	0.066(4)	0.018(3)	0.021(3)	0.030(3)
07	0.0690(8)	0.1681(8)	0.2935(6)	0.0647(17)	0.062(3)	0.075(5)	0.062(4)	0.015(3)	0.023(3)	0.038(3)
08 0	0.3026(8)	0.3725(8)	0.0323(6)	0.0632(16)	0.053(3)	0.076(4)	0.068(4)	0.027(3)	0.027(3)	0.034(3)
60	0.2787(8)	0.4075(7)	0.2452(6)	0.0630(16)	0.060(3)	0.055(4)	0.072(4)	0.011(3)	0.024(3)	0.028(3)
010	0.9906(8)	0.3397(7)	0.0497(6)	0.0648(17)	0.069(4)	0.067(4)	0.074(4)	0.026(3)	0.029(3)	0.042(3)
011	0.1945(8)	0.9773(8)	0.3544(6)	0.0611(16)	0.061(3)	0.070(4)	0.066(4)	0.025(3)	0.030(3)	0.037(3)
012	0.5966(8)	0.4494(8)	0.2327(6)	0.0703(18)	0.058(3)	0.068(4)	0.087(5)	0.035(4)	0.029(3)	0.028(3)
013	0.0268(9)	0.3355(8)	0.8343(6)	0.0688(18)	0.071(4)	0.078(5)	0.071(4)	0.030(4)	0.025(3)	0.047(4)
014	0.9644(9)	0.3807(8)	0.2664(6)	0.0742(19)	0.081(4)	0.080(5)	0.081(5)	0.018(4)	0.036(4)	0.053(4)
OW1	0.5885(8)	0.1365(7)	0.4671(6)	0.0673(17)	0.064(4)	0.069(4)	0.078(4)	0.023(3)	0.029(3)	0.038(3)
OW2	0.6986(9)	0.7712(8)	0.2727(7)	0.0754(19)	0.083(4)	0.079(5)	0.093(5)	0.030(4)	0.054(4)	0.048(4)
OW3	0.5381(8)	0.9707(8)	0.2187(6)	0.0701(18)	0.064(4)	0.079(5)	0.074(4)	0.024(4)	0.026(3)	0.040(4)
OW4	0.9107(8)	0.0812(9)	0.4488(6)	0.0745(19)	0.058(4)	0.099(5)	0.079(4)	0.033(4)	0.032(3)	0.042(4)
OW5	0.3179(9)	0.6916(8)	0.2738(6)	0.0726(18)	0.065(4)	0.074(5)	0.081(5)	0.028(4)	0.030(3)	0.033(3)
OW6	0.6084(10)	0.6867(8)	0.4765(6)	0.078(2)	0.094(5)	0.077(5)	0.074(5)	0.031(4)	0.032(4)	0.049(4)
OW7	0.3442(10)	0.6571(10)	0.0247(8)	0.089(2)	0.080(5)	0.090(6)	0.109(6)	0.031(5)	0.045(4)	0.045(4)
OW8	0.9705(13)	0.3505(12)	0.5740(10)	0.137(4)	0.112(7)	0.124(9)	0.132(8)	-0.022(7)	0.056(6)	0.025(6)
*6WO	0.736(3)	0.473(2)	0.4850(18)	0.125(7)	0.134(15)	0.104(15)	0.119(15)	0.038(12)	0.045(12)	0.043(12)

TABLE 4. ATOM COORDINATES AND DISPLACEMENT PARAMETERS (\mathring{A}^2) FOR BURROITE

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* OW9 occupancy = 0.5

Bond	Distance	BV	Bond	Distance	BV	Bond	Distance	BV
V1–O5	1.692(6)	1.35	V2-011	1.629(6)	1.60	V3–O13	1.633(7)	1.58
V1–O4	1.693(7)	1.35	V2–07	1.822(6)	0.95	V3–O8	1.819(6)	0.96
V1–O3	1.925(6)	0.72	V2–O6	1.822(6)	0.95	V3–O10	1.833(7)	0.92
V1–O2	1.937(6)	0.70	V2–O3	1.988(7)	0.61	V3–O2	1.999(6)	0.59
V1–O1	2.103(6)	0.44	V2–O2	2.001(6)	0.59	V3–O3	2.027(6)	0.55
V1–O1	2.110(6)	0.44	V2–O1	2.250(6)	0.30	V3–O1	2.229(6)	0.32
Mean, sum	1.910	5.00	Mean, sum	1.919	5.00	Mean, sum	1.923	4.92
V4–O12	1.600(7)	1.73	V5–O14	1.592(7)	1.77	Ca–OW4	2.393(7)	0.32
V4–O9	1.806(6)	0.99	V5–O10	1.861(7)	0.85	Ca–OW5	2.419(7)	0.29
V4–O6	1.874(7)	0.83	V5–O9	1.862(6)	0.85	Ca–OW3	2.437(7)	0.28
V4–08	1.885(6)	0.80	V5–07	1.880(7)	0.81	Ca–OW6	2.456(7)	0.27
V4–O4	2.053(7)	0.51	V5–O5	2.030(7)	0.54	Ca–OW2	2.490(7)	0.24
V4–O1	2.352(6)	0.23	V5–O1	2.328(6)	0.24	Ca–OW1	2.541(7)	0.21
Mean, sum	1.928	5.09	Mean, sum	1.926	5.06	Ca–OW1	2.559(7)	0.20
						Ca–O11	2.700(7)	0.14
						Mean, sum	2.499	1.95
Possible NH ₄	sites							
OW7–08	2.820(11)	0.20	OW8–OW4	2.758(13)	0.24	OW9–OW8	2.82(2)	0.20
OW7-010	2.821(9)	0.20	OW8–OW9	2.82(2)×½	0.10	OW9–OW8	2.85(2)	0.19
OW7-04	2.853(10)	0.18	OW8–OW9	2.85(2)×½	0.10	OW9–OW6	2.86(2)	0.18
OW7–OW5	3.073(11)	0.10	OW8–OW1	2.899(11)	0.16	OW9-012	2.86(2)	0.18
OW7–OW3	3.181(10)	0.08	OW8-014	2.915(13)	0.16	OW9–OW6	3.00(2)	0.12
OW7–OW2	3.255(10)	0.06	OW8–O13	3.062(14)	0.10	OW9-014	3.02(2)	0.12
OW7–013	3.283(11)	0.06	OW8-012	3.350(12)	0.05	OW9–OW1	3.11(2)	0.09
OW7-05	3.423(10)	0.04	OW8-011	3.414(14)	0.04	OW9-06	3.23(2)	0.07
Mean, sum	3.089	0.92	Mean, sum	3.033	0.95	Mean, sum	2.969	1.15

TABLE 5. SELECTED BOND DISTANCES (Å) AND BOND VALENCES (vu) IN BURROITE

 V^{5+} –O and Ca²⁺–O bond valence parameters from Brese & O'Keeffe (1991). NH₄⁺–O bond-valence parameters from García-Rodríguez *et al.* (2000).

linked by a shared OW1–OW1 edge; each of the Ca atoms in the dimer also bonds to the decavanadate group through the sharing of an oxygen atom at the O11 position.

Because of the marginal quality of the X-ray diffraction data, it was not possible to differentiate the isoelectronic N and O atoms based upon site scattering, and H atom sites could not be unambiguously located in the difference-Fourier maps. Three sites, designated OW7, OW8, and OW9, were found to be consistent with occupancy by either the O of an H₂O group or the N of an NH₄ group based upon coordination (bond-valence sums for the sites when assigned as NH4⁺ are 0.92, 0.95, and 1.15 vu, respectively; Table 5). The OW7 and OW8 sites refined to approximately full occupancy (and were assigned as such) and the OW9 site refined to approximately half occupancy and was assigned as such. The OW7, OW8, and OW9 sites account for five O or N apfu; one fully occupied site is sufficient to accommodate two NH_4 groups pfu, as indicated by the EPMA and ideal formula. Therefore, we have allocated two NH₄ groups and three H₂O groups *pfu* to the OW7, OW8, and OW9 sites, but have refined them all as O atoms. The three sites were refined with anisotropic displacement parameters.

SUMMARY

The discovery of burroite adds an additional decavanadate mineral to the growing list of pascoite-family minerals discovered in the Uravan Mineral Belt of the Colorado Plateau. Whereas the decavanadate structural group is a constant among the minerals, the local chemistry at the site of mineral genesis is reflected in the chemistry of the interstitial group, and yields the large variety seen among the newly discovered minerals. In the case of burroite, the interstitial composition of $[Ca_2(NH_4)_2 \cdot 15H_2O]^{6+}$ yields the second known calcium-ammonium hydrate decavanadate, joining wernerbaurite, $[Ca_2(NH_4)_2(H_2O)_{16}][V_{10}O_{28}]$ (Kampf *et al.* 2016) in that category.



FIG. 3. Crystal structure of burroite viewed along [100]. The unit cell is shown by dashed lines. OW sites that could be occupied by NH_4^+ are shown as yellow spheres, Ca atoms are blue, and oxygen atoms are white. The orange structural unit is the $[V_{10}O_{28}]^{6-}$ decavanadate cluster.

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