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# AMMONIOLASALITE, [(NH<sub>4</sub>)<sub>2</sub>Mg<sub>2</sub>(H<sub>2</sub>O)<sub>20</sub>][V<sub>10</sub>O<sub>28</sub>], A NEW DECAVANADATE SPECIES FROM THE BURRO MINE, SLICK ROCK DISTRICT, COLORADO

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#### Abstract

Ammoniolasalite,  $[(NH_4)_2Mg_2(H_2O)_{20}][V_{10}O_{28}]$ , is a new decavanadate species (IMA2017-094) from the Burro mine, Slick Rock district, San Miguel County, Colorado, USA (38°2'42"N 108°53'23"W). The mineral occurs as crusts on montroseite- and corvusite-bearing sandstone in an NH<sub>4</sub>-rich secondary assemblage also containing ammoniozippeite and the NH<sub>4</sub>-bearing decavanadates schindlerite and wernerbaurite. The crusts, up to 2 mm or more across, exhibit consistently oriented faces. Ammoniolasalite crystals are bright orange to orange-yellow with a vitreous luster. The mineral has brittle tenacity, Mohs hardness of 1, no cleavage, and conchoidal fracture. The density (measured) is 2.28(2) g/cm<sup>3</sup>. Ammoniolasalite is biaxial (-),  $\alpha$ = 1.740(3),  $\beta$  = 1.769(3),  $\gamma$  = 1.771(3) as measured in white light. The measured 2V is 31(1)°. Dispersion is very strong, r > v, and the orientation is  $Y = \mathbf{b}, Z \land \mathbf{a} = 38^{\circ}$  in obtuse  $\beta$ . Pleochroism is X yellow, Y yellow orange, Z orange; X < Y < Z. Electron probe microanalysis gave the empirical formula (based on 48 O apfu) [(NH<sub>4</sub>)<sub>1.76</sub>K<sub>0.24</sub>]<sub>22.00</sub>Mg<sub>1.94</sub>[V<sup>5+</sup><sub>10</sub>O<sub>28</sub>]·20H<sub>2</sub>O. Ammoniolasalite is monoclinic, C2/c, with a 24.478(3), b 10.9413(4), c 17.5508(12) Å,  $\beta$  119.257(7)°, and V = 4100.9(6) Å<sup>3</sup>. The strongest four lines in the diffraction pattern are [d in Å(I)(hkl)]: 10.64(24)(200), 9.43(100)(110, 11), 7.62(26)(002,110), 7.62(26)(002,1 and  $6.80(32)(\overline{1}12,\overline{3}11)$ . The atomic arrangement of ammoniolasalite was solved and refined to  $R_1 = 0.0357$  for 3628 independent reflections with  $I > 2\sigma(I)$ . Ammoniolasalite is isostructural with lasalite,  $[Na_2Mg_2(H_2O)_{20}][V_{10}O_{28}]$ , from which it derives its name. The structural unit is the  $[V_{10}O_{28}]^{6-}$  decavanadate group; charge balance in the structure is maintained by the  $[(NH_4,K)_2Mg_2(H_2O)_{20}]^{6+}$  interstitial complex. Linkage between the structural unit and the components of the interstitial complex is attained by bonding between the  $(NH_4,K)$  site and three oxygen atoms of the decavanadate structural group.

*Keywords*: ammoniolasalite, new mineral, decavanadate, lasalite, crystal structure, Burro mine, San Miguel County, Colorado, USA.

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#### INTRODUCTION AND OCCURRENCE

The uranium and vanadium deposits of Colorado and Utah have been a rich source of these metals for more than a century. Beginning late in the 19<sup>th</sup> century, mining has occurred in a 120 km long arcuate belt (the "Uravan Mineral Belt") of bedded or rollfront deposits in sandstone of the Salt Wash member of the Jurassic Morrison Formation (Carter & Gualtieri 1965, Shawe 2011). The uranium and vanadium ore mineralization was deposited where solutions rich in U and V encountered pockets of strongly reducing solutions that had developed around accumulations of carbonaceous plant material.

These deposits have been a rich source of secondary vanadium minerals that form from the oxidation of primary vanadium ore minerals at low temperature. The numerous minerals that have been discovered within the Uravan deposits include a growing number of decavanadate minerals, or minerals that contain the  $[V_{10}O_{28}]^{6-}$  polyanion, as well as  $[H_xV_{10}O_{28}]^{(6-x)-}$  protonated decavanadate clusters and  $[(V^{4+}_xV^{5+}_{10-x})O_{28}]^{(6+x)-}$  mixed-valence decavanadate clusters. The majority of decavanadate minerals, including protonated and mixed-valence decavanadate species, have been discovered in the Uravan deposits.

Ammoniolasalite is the newest member of the decavanadate family of minerals, added to the growing

list of decavanadate, protonated decavanadate, and mixed-valence decavanadate minerals (Table 1). Ammoniolasalite was found underground at the Burro mine, Slick Rock district, San Miguel County, Colorado, USA (38°2′42″N 108°53′23″W). The Burro mine is the type locality for metamunirite (Evans 1991), burroite (Kampf *et al.* 2017a), and ammonio-zippeite (Kampf *et al.* 2018). The mine is located near the southern end of the Uravan Mineral Belt.

The samples of the new mineral were first collected by one of the authors (PMA). The mineral is moderately rare. It occurs on montroseite- and corvusite-bearing sandstone in an NH<sub>4</sub>-rich secondary assemblage also containing ammoniozippeite and the NH<sub>4</sub>-bearing decavanadates schindlerite and wernerbaurite.

The new mineral forms from the oxidation of montroseite-corvusite assemblages in a moist environment. Mining operations have exposed unoxidized and oxidized phases. Under ambient temperatures and generally oxidizing near-surface conditions, water reacts with 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.*,  $NH_4^+$ ,  $Na^+$ ,  $Ca^{2+}$ ,  $Mn^{2+}$ ,  $Pb^{2+}$ ). The  $NH_4^+$  presumably derives from organic matter in the deposit.

Mineral species	Ideal formula*	First reference; best structure reference						
Decavanadates: [V <sub>10</sub> O <sub>28</sub> ] <sup>6-</sup>								
Ammoniolasalite	[(NH <sub>4</sub> ) <sub>2</sub> Mg <sub>2</sub> (H <sub>2</sub> O) <sub>20</sub> ][V <sub>10</sub> O <sub>28</sub> ]	This study						
Burroite	[(NH <sub>4</sub> ) <sub>2</sub> Ca <sub>2</sub> (H <sub>2</sub> O) <sub>15</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2017a)						
Huemulite	[Na <sub>4</sub> Mg(H <sub>2</sub> O) <sub>24</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Gordillo et al. (1966); Colombo et al. (2011)						
Hughesite	[Na <sub>3</sub> Al(H <sub>2</sub> O) <sub>22</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Rakovan <i>et al</i> . (2011)						
Hummerite	[K <sub>2</sub> Mg <sub>2</sub> (H <sub>2</sub> O) <sub>16</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Weeks et al. (1951); Hughes et al. (2002)						
Hydropascoite	[Ca <sub>3</sub> (H <sub>2</sub> O) <sub>24</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2017b)						
Kokinosite	[Na <sub>2</sub> Ca <sub>2</sub> (H <sub>2</sub> O) <sub>24</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2014a)						
Lasalite	[Na <sub>2</sub> Mg <sub>2</sub> (H <sub>2</sub> O) <sub>20</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Hughes <i>et al</i> . (2008)						
Magnesiopascoite	[Ca <sub>2</sub> Mg(H <sub>2</sub> O) <sub>16</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf & Steele (2008)						
Pascoite	[Ca <sub>3</sub> (H <sub>2</sub> O) <sub>17</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Hillebrand et al. (1914); Hughes et al. (2005)						
Postite	[MgAl <sub>2</sub> (OH) <sub>2</sub> (H <sub>2</sub> O) <sub>27</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2012)						
Schindlerite	[(NH <sub>4</sub> ) <sub>4</sub> Na <sub>2</sub> (H <sub>2</sub> O) <sub>10</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2013a); Kampf <i>et al.</i> (2016)						
Wernerbaurite	[(NH <sub>4</sub> ) <sub>2</sub> Ca <sub>2</sub> (H <sub>2</sub> O) <sub>16</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2013a); Kampf <i>et al.</i> (2016)						
Okieite	[Mg <sub>3</sub> (H <sub>2</sub> O) <sub>27</sub> ][V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al</i> . (in prep)						
	Protonated Decavanadates: [	$[H_x V_{10} O_{28}]^{(6-x)-}$						
Gunterite	[Na <sub>4</sub> (H <sub>2</sub> O) <sub>22</sub> ][H <sub>2</sub> V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al</i> . (2011b)						
Rakovanite	[Na <sub>3</sub> (H <sub>2</sub> O) <sub>15</sub> ][H <sub>3</sub> V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al</i> . (2011a)						
	Mixed-Valence Decavanadates: [(V	x <sup>4+</sup> x <sup>5+</sup> 10-x)O <sub>28</sub> ] <sup>(6+x)-</sup>						
Bluestreakite	[K <sub>4</sub> Mg <sub>2</sub> (H <sub>2</sub> O) <sub>14</sub> ][V <sup>4+</sup> <sub>2</sub> V <sup>5+</sup> <sub>8</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2014b)						
Nashite	$[Na_{3}Ca_{2}(H_{2}O)_{24}][(V^{4+}V^{5+}_{9})O_{28}]$	Kampf <i>et al.</i> (2013b)						

TABLE 1. DECAVANADATE FAMILY OF MINERALS

\* Expressed as the interstitial complex and the structural unit.



FIG. 1. Ammoniolasalite crystals, some exhibiting parallel growth; field of view 0.83 mm.

The name ammoniolasalite is based upon the mineral being the ammonium analogue of lasalite, with  $NH_4$  dominant at the site occupied by Na in lasalite. Five cotype specimens are deposited in the collections of the Natural History Museum of Los Angeles County, Los Angeles, California, USA, under catalogue numbers 67477, 67478, 67479, 67480, and 67481.

## APPEARANCE AND PHYSICAL PROPERTIES

Crystals of ammoniolasalite occur as short prismatic (on  $[10\overline{1}]$ ) to equant crystals, often exhibiting stepped or skeletal faces (Fig. 1). The crystal forms measured by reflected-light goniometry are  $\{001\}$ ,  $\{110\}$ ,  $\{10\overline{1}\}$ ,  $\{111\}$ ,  $\{11\overline{1}\}$ ,  $\{201\}$ , and  $\{31\overline{1}\}$  (Fig. 2). Crystals typically form crusts in which areas up to 2 mm or more across exhibit consistently oriented faces. Ammoniolasalite is bright orange to orange-yellow, with a light orange streak and a vitreous luster; the mineral does not exhibit fluorescence in long- or short-wave ultraviolet light. The Mohs hardness is 1, based on scratch tests. No cleavage was observed. Ammoniolasalite exhibits brittle tenacity and has conchoidal fracture. The density is 2.28(2) g/cm<sup>3</sup> measured by floatation in methylene



FIG. 2. Crystal drawing of ammoniolasalite, clinographic projection.

iodide–toluene, and the calculated density is  $2.278 \text{ g/cm}^3$  for the empirical formula and  $2.271 \text{ g/cm}^3$  for the ideal formula. At room temperature ammoniolasalite is slowly soluble in H<sub>2</sub>O (minutes) and rapidly soluble in dilute HCl (seconds).

Optically, ammoniolasalite is biaxial (–), with  $\alpha = 1.740(3)$ ,  $\beta = 1.769(3)$ ,  $\gamma = 1.771(3)$  measured in white light. The 2*V* is 31(1)°, measured using extinction data analyzed with EXCALIBR (Gunter *et al.* 2004); the calculated 2*V* is 29.1°. Dispersion is very strong, r > v, and the optical orientation is  $Y = \mathbf{b}$ ,  $Z \wedge \mathbf{a} = 38^{\circ}$  in obtuse  $\beta$ . Ammoniolasalite is pleochroic with *X* yellow, *Y* yellow orange, *Z* orange; X < Y < Z. The Gladstone-Dale compatibility,  $1 - (K_p/K_c)$ , is 0.006 for the empirical formula, in the range of superior compatibility (Mandarino 1981).

### INFRARED SPECTROSCOPY

Fourier transform infrared (FTIR) spectra were recorded using a Thermo Nicolet model 6700 spectrometer equipped with a Continuµm microscope. The samples were analyzed in transmission mode with a micro diamond-compression cell using one diamond window as the background. The spectra of ammonio-

Constituent	Mean	Range	S.D.	Standard	Normalized
(NH <sub>4</sub> ) <sub>2</sub> O					3.26*
K <sub>2</sub> O	0.95	0.53-1.16	0.29	sanidine	0.81
MgO	6.53	6.36-6.79	0.19	diopside	5.56
$V_2O_5$ $H_2O$ Total	76.02	75.12–76.84	0.71	V metal	64.68 25.70* 100.01

TABLE 2. ANALYTICAL DATA (wt.%) FOR AMMONIOLASALITE

\* Based upon the crystal structure with V = 10 and O = 48 apfu.

<i>I</i> obs	$d_{\rm obs}$	$d_{calc}$	<i>I</i> calc	hkl	<i>I</i> obs	$d_{\rm obs}$	$d_{calc}$	<i>I</i> calc	h k l	<i>I</i> obs	$d_{\rm obs}$	$d_{calc}$	/ <sub>calc</sub>	h k l
24	10.64	10.6778	37	200			3.1276	1	115			2.1549	1	827
100	0.40	9.7377	57	110	0	0.000	3.0657	3	333	40	0 4050	(2.1373	5	604
100	9.43	9.1972	43	111	8	3.066	3.0601	3	133	13	2.1250	2.1219	7	152
21	8.57	8.4896	26	202			3.0211	1	802			2.0957	2	352
26	7.62	7.6560	29	002			2.9834	1	620	4	2.0793	2.0855	3	1116
		7.4952	5	111	12	2.970	2.9759	11	8 0 4			2.0670	1	152
32	6.80	6.7997	31	112			2.9445	1	4 2 5			2.0632	1	425
		6.5393	1	311			2.9387	2	710			2.0222	1	515
5	6.05	6.0619	5	312	13	2.891	2.8998	14	225			2.0146	1	335
		5.4566	1	112			2.8950	2	531			(1.9967	1	552
7	5.32	5.3389	6	400			2.8299	1	606	7	1.9976	{1.9901	2	1117
		5.1517	2	021			2.7970	1	316			1.9877	4	10 0 8
5	5.02	5.1442	1	202			2.7538	4	602			1.9839	1	12 0 6
5	5.02	4.9708	1	221	23	2 725	2.7353	7	040	7	1.9569	1.9598	7	406
_		4.9195	1	311	20	211 20	2.7262	10	621			1.9479	1	352
2	4.460	4.4570	2	512			\2.6927	4	041			1.9326	2	714
2	4.219	4.2448	2	4 0 4			2.6689	4	823			1.8778	1	1118
		4.0669	2	314			2.6633	1	711			1.8530	1	154
12	4 020	4.0554	3	4 2 1	5	2 621	2.6157	1	116			1.8473	1	227
12	1.020	3.9957	4	223	Ū	2.021	2.6136	1	513			1.8236	4	060
		3.9814	1	312			2.5786	1	806	10	1.8182	1.8204	4	12 0 8
		3.8682	2	114			2.5721	1	404			(1.8108	1	061
12	3 750	3.7526	1	423			2.5520	1	006			1.8066	1	516
12	0.100	3.7320	10	023			2.5152	1	915			1.7921	1	535
2	3.581	3.5658	2	131	2	2.479	2.4547	2	731			1.7511	1	6 1 10
3	3 4 18	3.4375	1	131	3	2.368	2.3696	4	916			1.7486	1	715
Ũ	0.110	3.3998	2	224			2.3140	1	244			(1.7273	1	445
	(	3.3356	2	3 1 5	5	2 282	2.2870	4	514	3	1.7175	{1.7147	2	606
5	3.326	3.3148	2	712	Ŭ	LILOL	2.2735	3	10 0 6			(1.7099	2	4 1 10
	(	3.3075	1	713			2.2421	1	641			1.6841	1	644
6	3 173	3.1833	5	621			2.1999	1	917			1.6595	1	14 0 2
0	51110	3.1760	1	711	8	2 1795	2.1829	1	10 2 5	7	1.6282	1.6253	4	956
		3.1364	1	024	0	2.1700	2.1769	4	150	4	1.5961	1.5978	4	554

TABLE 3. CALCULATED\* AND OBSERVED POWDER X-RAY DIFFRACTION DATA (*d* IN Å) FOR AMMONIOLASALITE

\* The calculated intensities have been scaled (normalized) such that the sum of the intensities of the 110 and  $\overline{1}11$  reflections (57 + 43) = 100% because these two reflections together account for the observed line of 100% intensity. Note that profile fitting yields observed intensities that are based on the integration of the areas under the peaks.

lasalite from the Burro mine and lasalite from the Packrat mine (Mesa County, Colorado) are shown in Figure 3, with the general band assignments labeled. The spectra are almost identical, except for the band attributed to  $NH_4$  deformation observed in the spectrum of ammoniolasalite.

#### CHEMICAL COMPOSITION

Analyses of ammoniolasalite (four points on three crystals) were performed at the University of Utah with a Cameca SX-50 electron microprobe equipped with four wavelength-dispersive spectrometers and

### TABLE 4. DATA COLLECTION AND STRUCTURE REFINEMENT DETAILS FOR AMMONIOLASALITE

Diffractometer	Rigaku R-Axis Rapid II
X-ray radiation/power	$MoK\alpha$ (I = 0.71075 Å)/50 kV, 40 mA
Temperature	293(2) K
Structural Formula	$\{[(NH_4)_{1.30}K_{0.70}]_{\Sigma 2.00}Mg_2(H_2O)_{20}\}\{V^{5+}_{10}O_{28}\}$
Space group	C2/c
Unit cell dimensions	<i>a</i> = 24.478(3) Å
	b = 10.9413(4) Å
	<i>c</i> = 17.5508(12) Å
	$eta=$ 119.257(7) $^\circ$
V	4100.9(6) Å <sup>3</sup>
Ζ	4
Density (for above formula)	2.295 g/cm <sup>3</sup>
Absorption coefficient	2.406 mm <sup>-1</sup>
<i>F</i> (000)	2821
Crystal size	200 $ imes$ 170 $ imes$ 160 $\mu$ m
θ range	3.12 to 27.48°
Index ranges	$-28 \le h \le 31, -14 \le k \le 14, -22 \le l \le 22$
Reflections collected/unique	17595/4684; <i>R</i> <sub>int</sub> = 0.030
Reflections with $l > 2\sigma l$	3628
Completeness to $\theta = 27.48^{\circ}$	99.50%
Refinement method	Full-matrix least-squares on $F^2$
Parameter/restraints	341/30
GoF	1.070
Final <i>R</i> indices $[l > 2\sigma l]$	$R_1 = 0.0357, wR_2 = 0.0831$
R indices (all data)	$R_1 = 0.0458, \ wR_2 = 0.0884$
Largest diff. peak/hole	$+1.39/-0.63 e/A^3$

 $\overline{R_{int} = \Sigma |F_o^2 - F_o^2(\text{mean})|/\Sigma[F_o^2]}. \text{ GoF} = S = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}. R_1 = \Sigma ||F_o| - |F_c||/\Sigma|F_o|.$  $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}; w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP] \text{ where } a \text{ is } 0.0506, b \text{ is } 0, \text{ and } P \text{ is } [2F_c^2 + Max(F_o^2, 0)]/3.$ 

Probe for EPMA software. Analytical conditions were 15 kV accelerating voltage, 10 nA beam current, and beam diameters of 5 and 10  $\mu$ m. There was no visible damage to the sample under the electron beam. Counting times were 30 s on peak and 30 s on background for each element. Special care was taken to obtain the best possible accuracy for the N analyses using the methodology of Bastin & Heijligers (1991) and the same primary standard for N that they used (Cr<sub>2</sub>N). Time-dependent intensity corrections were applied to Na, Ca, and V; the N intensity was time independent. Raw X-ray intensities were corrected for matrix effects with a  $\varphi\rho(z)$  algorithm (Pouchou & Pichoir 1991).

Ammoniolasalite is very sensitive to dehydration and deammoniation in air and particularly under vacuum. During vacuum deposition of the conductive carbon coat required for the EPMA study, the ammoniolasalite was subjected to an ultimate vacuum of  $2 \times 10^{-5}$  torr for approximately 5 min. This evidently resulted in the loss of much of the weakly held H<sub>2</sub>O and NH<sub>4</sub>, and no further loss was detected during the EPMA study. The EPMA value obtained for (NH<sub>4</sub>)<sub>2</sub>O was 1.64 wt.% (range 1.34–1.75); when combined with the analyzed  $K_2O$ , 3.26 wt.% (NH<sub>4</sub>)<sub>2</sub>O is required for the full occupancy of the NH<sub>4</sub>/K site. This suggests that 50% of the NH<sub>4</sub> was lost prior to the EPMA study. For comparison, the difference of the EPMA analytical total from 100% is 14.86 wt.%; assuming this to be the total H<sub>2</sub>O remaining in the sample, and considering that the calculated H<sub>2</sub>O based on the structure is 25.70 wt.%, there appears to have been a 42% loss of H<sub>2</sub>O under vacuum.

To better quantify  $(NH_4)_2O$  and  $H_2O$ , ammoniolasalite was subjected to CHN analysis, which provided 2.65 wt.%  $(NH_4)_2O$  and 19.76 wt.% H<sub>2</sub>O. These values are lower than the calculated values by 19% and 23%, respectively. This is indicative of significant dehydration and deammoniation in air prior to the analysis, which is not surprising considering that separating the crystals from matrix and obtaining a pure sample for CHN analysis required breaking the crystals into very small fragments. The well-documented loss of NH<sub>4</sub> and H<sub>2</sub>O prior to the analyses makes it necessary to use calculated values for  $(NH_4)_2O$  and H<sub>2</sub>O. The NH<sub>4</sub> and H<sub>2</sub>O losses result in higher concentrations for the remaining constituents than are to be expected for the fully hydrated and

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TABLE 5. ATOM COORDINATES AND DISPLACEMENT PARAMETERS (Å $^2)$  FOR AMMONIOLASALITE

	x∕a	y/b	z/c	$U_{ m eq}$
NH <sub>4</sub> *	0.19594(8)	0.18515(16)	0.54271(9)	0.0775(8)
Mg	0.11231(4)	0.59803(6)	0.59049(4)	0.02225(17)
V1	0.01373(2)	0.09591(3)	0.85111(2)	0.01581(10)
V2	-0.07104(2)	-0.10769(3)	0.71750(2)	0.01835(11)
V3	0.07055(2)	0.30260(3)	0.78148(2)	0.01857(10)
V4	0.12815(2)	0.09627(3)	0.71820(2)	0.02047(11)
V5	0.15482(2)	0.09919(3)	0.90732(2)	0.02149(11)
01	-0.03813(7)	0.09791(12)	0.88788(9)	0.0214(3)
02	0.08604(7)	0.09625(12)	0.94082(9)	0.0213(3)
O3	-0.01123(7)	-0.07523(12)	0.67332(9)	0.0174(3)
04	0.01115(6)	0.26853(12)	0.82651(8)	0.0175(3)
O5	-0.06153(7)	0.09706(12)	0.72373(8)	0.0163(3)
06	-0.07131(7)	-0.25453(14)	0.71741(9)	0.0289(4)
07	-0.13640(7)	-0.06908(13)	0.61253(9)	0.0227(3)
08	-0.11202(7)	-0.06959(13)	0.77895(9)	0.0217(3)
09	0.13631(7)	0.26565(13)	0.88920(9)	0.0223(3)
010	0.11222(7)	0.26450(13)	0.72161(9)	0.0211(3)
011	0.16648(8)	0.09591(14)	0.66609(10)	0.0315(4)
012	0 18838(7)	0.09829(13)	0.83404(9)	0.0233(3)
013	0.07059(7)	0.44921(15)	0.78067(9)	0.0293(4)
014	0.21472(8)	0.09774(14)	0.00352(10)	0.0200(4)
OW1	0 11240(9)	0.40853(15)	0.59515(11)	0.0316(4)
H1A	0 1170(12)	0.369(2)	0.6379(13)	0.038
H1B	0.0967(13)	0.376(2)	0 5545(13)	0.038
OW2	0.04240(9)	0.40908(16)	0.96422(10)	0.0316(4)
H2A	0.0327(12)	0.3592(19)	0.9265(14)	0.038
H2B	0.0230(12)	0.4696(18)	0.9389(15)	0.038
OW3	0 17764(9)	0.60190(16)	0.54574(12)	0.0329(4)
НЗА	0.2147(10)	0.614(2)	0.5774(15)	0.039
H3B	0.1684(11)	0.647(2)	0.5028(14)	0.039
OW4	0.17833(10)	0.59674(16)	0.71752(11)	0.0404(5)
H4A	0 1940(13)	0.6553(19)	0.7409(17)	0.048
H4B	0 1960(13)	0.5390(19)	0.7491(16)	0.048
OW5	0.03886(9)	0.60281(15)	0.62316(11)	0.0311(4)
H5A	0.0517(13)	0.604(2)	0.6740(13)	0.037
H5B	0.0166(11)	0.655(2)	0 5998(14)	0.037
OW6	0 11169(9)	0.21342(15)	0.09362(10)	0.0367(5)
H6A	0 1131(13)	0.171(2)	0.1301(14)	0.044
H6B	0.0992(12)	0.173(2)	0.0506(13)	0.044
OW7	0.26938(10)	-0.31096(18)	0 19557(14)	0.0495(5)
H7A	0.2586(14)	-0.345(3)	0.2262(18)	0.059
H7B	0.2948(13)	-0.360(3)	0.1939(19)	0.059
OW8	-0.04867(9)	-0.21599(16)	0.52786(11)	0.0376(4)
Н8А	-0.0470(12)	_0 176(2)	0.4873(14)	0.0070(4)
H8B	-0.0400(13)	-0.166(2)	0.5650(14)	0.045
	0.17776(15)	-0.3853(3)	0.0000(14)	0.045
Над	0.178(2)	_0 449(3)	0.0200(2)	0.0000(11)
HOR	0.170(2)	-0.364(4)	0.846(2)	0.115
OW10	0.22757(0)	0.61262(17)	0.000(2)	0.110
	0.22707(9)	0.616(2)	0.31000(12)	0.0412(0)
H10R	0.2479(13)	0.664(2)	0.3014(14)	0.049
HIUD	0.2007(11)	0.004(2)	0.3020(10)	0.045

	$U^{11}$	$U^{22}$	$U^{33}$	U <sup>23</sup>	$U^{13}$	U <sup>12</sup>
NH <sub>4</sub>	0.0942(14)	0.0969(15)	0.0443(9)	-0.0178(8)	0.0360(8)	-0.0341(10)
Mg	0.0232(4)	0.0226(4)	0.0196(4)	0.0025(3)	0.0094(3)	-0.0008(3)
V1	0.0182(2)	0.01678(19)	0.01317(18)	-0.00030(12)	0.00826(15)	0.00041(14)
V2	0.0206(2)	0.0161(2)	0.0196(2)	-0.00106(13)	0.01081(16)	-0.00259(14)
V3	0.0190(2)	0.0163(2)	0.0210(2)	-0.00075(13)	0.01022(16)	-0.00226(14)
V4	0.0201(2)	0.0230(2)	0.0216(2)	0.00094(14)	0.01280(16)	0.00063(15)
V5	0.0170(2)	0.0273(2)	0.01721(19)	-0.00050(14)	0.00602(15)	0.00094(15)
01	0.0237(8)	0.0242(8)	0.0190(7)	-0.0002(6)	0.0126(6)	0.0003(6)
02	0.0209(8)	0.0250(8)	0.0171(7)	-0.0004(5)	0.0086(6)	-0.0004(6)
O3	0.0196(8)	0.0176(8)	0.0165(7)	-0.0011(5)	0.0100(6)	0.0000(6)
04	0.0192(8)	0.0156(7)	0.0190(7)	-0.0023(5)	0.0104(6)	-0.0010(6)
O5	0.0160(7)	0.0177(8)	0.0149(7)	0.0000(5)	0.0074(6)	0.0005(6)
O6	0.0349(10)	0.0192(8)	0.0326(9)	0.0004(6)	0.0166(8)	-0.0030(7)
07	0.0224(8)	0.0245(8)	0.0194(7)	-0.0039(6)	0.0088(6)	-0.0040(6)
08	0.0247(8)	0.0214(8)	0.0233(8)	0.0001(6)	0.0152(7)	-0.0036(6)
09	0.0195(8)	0.0242(8)	0.0209(7)	-0.0037(6)	0.0081(6)	-0.0038(6)
O10	0.0227(8)	0.0199(8)	0.0235(7)	0.0004(6)	0.0135(6)	-0.0035(6)
011	0.0316(10)	0.0392(10)	0.0323(9)	0.0031(7)	0.0223(8)	0.0022(8)
012	0.0176(8)	0.0278(9)	0.0246(8)	0.0013(6)	0.0104(6)	0.0027(6)
O13	0.0324(10)	0.0200(8)	0.0376(10)	-0.0006(6)	0.0187(8)	-0.0032(7)
014	0.0230(9)	0.0427(11)	0.0239(9)	0.0006(7)	0.0043(7)	0.0010(7)
OW1	0.0448(12)	0.0247(9)	0.0245(9)	0.0024(7)	0.0162(9)	-0.0030(8)
OW2	0.0359(10)	0.0302(10)	0.0200(9)	-0.0048(6)	0.0070(7)	0.0096(8)
OW3	0.0246(9)	0.0410(11)	0.0334(10)	0.0089(7)	0.0144(8)	0.0012(8)
OW4	0.0429(12)	0.0304(10)	0.0278(10)	0.0038(7)	0.0016(8)	-0.0030(8)
OW5	0.0340(10)	0.0344(10)	0.0265(9)	0.0011(7)	0.0158(8)	0.0014(7)
OW6	0.0664(13)	0.0205(9)	0.0264(9)	-0.0018(7)	0.0253(9)	-0.0030(8)
OW7	0.0376(12)	0.0409(13)	0.0679(14)	0.0068(10)	0.0243(10)	0.0075(9)
OW8	0.0564(12)	0.0319(10)	0.0293(9)	-0.0082(7)	0.0247(9)	-0.0088(9)
OW9	0.084(2)	0.0641(19)	0.086(2)	0.0252(15)	0.0002(16)	0.0011(16)
OW10	0.0341(11)	0.0421(12)	0.0327(10)	-0.0046(8)	0.0049(8)	0.0113(8)

TABLE 5. CONTINUED.

\* The refined joint occupancy of the NH<sub>4</sub> site is N<sub>0.650(4)</sub>K<sub>0.350(4)</sub>.

ammoniated phase; therefore, the other analyzed constituents were normalized to provide a total of 100%. Analytical data are given in Table 2.

The empirical formula (based on 48 O *apfu*) is  $[(NH_4)_{1.76}K_{0.24}]_{\Sigma 2.00}Mg_{1.94}[V^{5+}_{10}O_{28}]\cdot 20H_2O$  (+0.12 H for charge balance). The simplified structural formula is  $[(NH_4,K)_2Mg_2(H_2O)_{20}][V_{10}O_{28}]$  and the endmember formula is  $[(NH_4)_2Mg_2(H_2O)_{20}][V_{10}O_{28}]$ , which requires  $(NH_4)_2O$  3.71, MgO 5.75, V<sub>2</sub>O<sub>5</sub> 64.85, H<sub>2</sub>O 25.69, total 100.00 wt.%.

## X-ray Crystallography and Structure Refinement

Powder and single-crystal X-ray diffraction data were obtained with a Rigaku R-Axis Rapid II curved imaging plate microdiffractometer using monochromatized MoK $\alpha$  radiation. X-ray powder diffraction data were recorded using a Gandolfi-like motion on the  $\varphi$  and  $\omega$  axes, which were used to randomize the sample position. Observed *d* values and intensities were derived by profile fitting using JADE 2010 software (Materials Data, Inc.). Data (in Å for MoK $\alpha$ ) are given in Table 3. Unit-cell parameters refined from the powder data using JADE 2010 with whole pattern fitting are *a* 24.471(9), *b* 10.935(9), *c* 17.456(9) Å,  $\beta$  119.051(14)°, and *V* 4083(4) Å<sup>3</sup>, in good agreement with those obtained from the single-crystal data.

The Rigaku Crystal Clear software package was used for processing the 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 methods using SIR2011 (Burla *et al.* 2012). SHELXL-2013 (Sheldrick 2015) was used for the refinement of the structure. Following the solution of the structure, the atom coordinates were transformed to correspond to those in the structure of lasalite. The occupancy of the



FIG. 3. The FTIR spectra of lasalite and ammoniolasalite.

large cation site was refined with joint occupancy by K and N, providing  $(NH_4)_{0.65}K_{0.35}$ . The difference between this and the occupancy indicated by the empirical formula,  $(NH_4)_{0.88}K_{0.12}$ , is probably due to a real variation in the relative amounts of  $NH_4$  and K; however, the structure refinement is clearly consistent with the strong dominance of  $NH_4$  at this site.

All non-hydrogen atoms were refined with anisotropic displacement parameters. Difference Fourier syntheses located all H atom positions, except those associated with the (NH<sub>4</sub>,K) site; however, residual electron density in the vicinity of the (NH<sub>4</sub>,K) site is consistent with several disordered H sites. 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 4, atom coordinates and displacement parameters in Table 5, and selected bond distances and bond-valence sums in Table 6.

### Atomic Arrangement of Ammoniolasalite

The atomic arrangement of ammoniolasalite (Fig. 4) consists of two distinct parts, a structural unit and an

interstitial complex, 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, which is also found in numerous other new Uravan Mineral Belt minerals, as summarized in Table 1. Ammoniolasalite is, in fact, isostructural with lasalite,  $[Na_2Mg_2(H_2O)_{20}][V_{10}O_{28}]$  (Hughes *et al.* 2008), first found at the Vanadium Queen mine in the La Sal district, about 36 km NNW of the Burro mine.

The interstitial complex in ammoniolasalite has the formula  $[(NH_4)_2Mg_2(H_2O)_{20}]^{6+}$ , the Na site in the lasalite structure being replaced by a slightly larger (NH<sub>4</sub>,K) site in ammoniolasalite. The Mg atoms bond to interstitial-unit water molecules and do not share any bonds with the decavanadate group or other polyhedra of the interstitial unit. The (NH<sub>4</sub>,K) site links to four H<sub>2</sub>O molecules and three oxygen atoms of the decavanadate structural group, yielding (NH<sub>4</sub>,K)(H<sub>2</sub>O)<sub>4</sub>O<sub>3</sub> coordination.

Table 7 compares the properties of lasalite and ammoniolasalite. As shown therein, the largest differences in unit-cell parameters are in *a* and *c*. The *b* lattice parameter, similar in lasalite and ammoniolasalite, is controlled by the thickness of the  $[V_{10}O_{28}]$  group,

V1–	Distance	BV	V2-	Distance	BV	V3-	Distance	BV
01	1.6821(16)	1.34	O6	1.6066(16)	1.62	O13	1.6042(17)	1.62
02	1.6994(15)	1.28	07	1.8031(15)	0.99	O9	1.8314(14)	0.92
O3	1.9153(14)	0.75	O8	1.8441(15)	0.89	O10	1.8348(15)	0.91
O4	1.9315(14)	0.72	O3	1.9951(15)	0.61	O4	2.0001(14)	0.61
O5	2.0896(14)	0.48	O3′	2.0202(14)	0.58	O4′	2.0064(14)	0.59
O5′	2.1439(14)	0.42	O5	2.2495(13)	0.32	O5	2.2571(13)	0.32
Mean, $\Sigma$ :	1.910	4.99	Mean, $\Sigma$ :	1.920	5.01	Mean, $\Sigma$ :	1.923	4.97
V4-	Distance	BV	V5-	Distance	BV	Mg–	Distance	BV
011	1.5984(16)	1.66	O14	1.6066(16)	1.62	OW4	2.0151(19)	0.40
O12	1.8378(15)	0.91	O12	1.8337(16)	0.92	OW2	2.0345(17)	0.38
O8	1.8630(15)	0.85	O9	1.8662(15)	0.84	OW6	2.0640(18)	0.36
O10	1.8885(15)	0.80	07	1.8869(15)	0.8	OW1	2.0750(17)	0.35
01	2.0766(16)	0.50	O2	2.0368(16)	0.55	OW3	2.099(2)	0.33
O5	2.3072(14)	0.28	O5	2.3176(14)	0.27	OW5	2.138(2)	0.30
Mean, $\Sigma$ :	1.929	5.00	Mean, $\Sigma$ :	1.925	5.00	Mean, $\Sigma$ :	2.071	2.12
NH <sub>4</sub> ,K–	Distance	BV(NH <sub>4</sub> )	BV(K)					
07	2.6980(19)	0.28	0.20					
OW7	2.759(2)	0.24	0.17					
011	2.773(2)	0.23	0.16					
OW9	2.841(4)	0.19	0.14					
OW9′	2.987(4)	0.13	0.09					
OW8	3.206(3)	0.07	0.06					
O14	3.252(2)	0.06	0.05					
Mean, $\Sigma$ :	2.93	1.20	0.87					

TABLE 6. SELECTED BOND DISTANCES (Å) AND BOND VALENCES\* (vu) IN AMMONIOLASALITE

\* NH<sub>4</sub><sup>+</sup>–O bond valence parameters are from García-Rodríguez *et al.* (2000). All other bond valence parameters are from Gagné & Hawthorne (2015).



FIG. 4. Crystal structure of ammoniolasalite viewed down [010].  $VO_6$  octahedra are orange,  $Mg(H_2O)_6$  octahedra are blue,  $NH_4^+$  groups are turquoise balls, O atoms of  $H_2O$  groups are large white balls, and H atoms are small white balls.  $NH_4^+$ –O bonds are shown as sticks. The unit cell outline is shown by dashed lines.

Mineral	Lasalite	Ammoniolasalite
Formula Symmetry	Na₂Mg₂[V <sub>10</sub> O₂8]·20H₂O Monoclinic, <i>C</i> 2/ <i>c</i>	(NH <sub>4</sub> ,K) <sub>2</sub> Mg <sub>2</sub> [V <sub>10</sub> O <sub>28</sub> ]·20H <sub>2</sub> O Monoclinic, <i>C</i> 2/ <i>c</i>
Cell parameters	a = 23.9019(7) Å b = 10.9993(3) Å	a = 24.478(3) Å b = 10.9413(4) Å
	c = 17.0504(5)  A $\beta = 118.284(1)^{\circ}$ $\lambda = 2947 4(2) \text{ Å}^3$	c = 17.5508(12)  A $\beta = 119.257(7)^{\circ}$ $M = 4100.9(6) \text{ Å}^3$
Ζ	4 = 3947.4(2) A	4 4 100.9(0) A
Density (meas.)	2.38(2) g/cm <sup>3</sup>	2.28(2) g/cm <sup>3</sup>
Optics	Biaxial (-); 589 nm $\alpha = 1.743(5)$ $\beta = 1.773(5)$ $\gamma = 1.780(5)$ 2 <i>V</i> (meas.) = 43(1)°	Biaxial (–); white light $\alpha = 1.740(3)$ $\beta = 1.769(3)$ $\gamma = 1.771(3)$ $2V$ (meas.) = $31(1)^{\circ}$
Reference	Hughes <i>et al.</i> (2008)	This study

TABLE 7. COMPARISON OF LASALITE AND AMMONIOLASALITE

essentially a constant between the structures. The *a* and *c* parameters, however, reflect the large cation in the interstitial complex, Na in lasalite and  $(NH_{4},K)$  in ammoniolasalite; the expansion of the unit cell in the  $\{010\}$  plane of the ammoniolasalite structure reflects the accommodation of the larger cations.

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