# BLUESTREAKITE, K<sub>4</sub>Mg<sub>2</sub>(V<sup>4+</sup><sub>2</sub>V<sup>5+</sup><sub>8</sub>O<sub>28</sub>)·14H<sub>2</sub>O, A NEW MIXED-VALENCE DECAVANADATE MINERAL FROM THE BLUE STREAK MINE, MONTROSE COUNTY, COLORADO: CRYSTAL STRUCTURE AND DESCRIPTIVE MINERALOGY

ANTHONY R. KAMPF<sup>§</sup>

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

JOHN M. HUGHES

Department of Geology, The University of Vermont, 180 Colchester Ave., Burlington, VT 05405, U.S.A.

JOE MARTY

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

BARBARA P. NASH

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

YU-SHENG CHEN

Center for Advanced Radiation Sources, University of Chicago, 5640 S. Ellis Ave., Chicago, IL 60637-1434, U.S.A.

IAN M. STEELE

Department of Geophysical Sciences, University of Chicago, 5734 S. Ellis Ave., Chicago, IL 60637-1434, U.S.A.

#### Abstract

Bluestreakite,  $K_4Mg_2(V^{4+}_2V^{5+}_8O_{28})$ ·14H<sub>2</sub>O, is a new mineral species from the Blue Streak mine, Bull Canyon, Montrose County, Colorado, USA. Bluestreakite typically occurs as irregular polycrystalline coatings on rounded quartz grains or masses of montroseite, and rarely as tablets or blades. It grows on corvusite-montroseite-bearing sandstone blocks intimately associated with gypsum, huemulite, hummerite, metamunirite, and munirite. Bluestreakite is dark greenish blue, with a light blue streak. The mineral is transparent, with a subadamantine luster. Bluestreakite does not fluoresce in short- or long-wave ultraviolet radiation, and it has a hardness *ca*. 2. Bluestreakite has a brittle tenacity, irregular fracture, and no cleavage. Density (calc.) = 2.630 g·cm<sup>-3</sup> based on the empirical formula and single-crystal cell data. Bluestreakite is biaxial (-), with a 1.750(5),  $\beta$  1.800(5),  $\gamma$  1.829 (calc.) (white light);  $2V_{meas.} = 73(3)^{\circ}$ . Optic orientation is  $Z = \mathbf{b}$  (X and Y not determined). The pleochroism is slight, with  $X < Y \approx Z$  dark greenish blue. Electron probe microanalysis gave the empirical formula (based on O = 42) ( $K_{3.37}Na_{0.15})_{23.52}Mg_{1.94}(V^{4+}_{1.40}V^{5+}_{8.60})O_{28}$ ·14H<sub>2</sub>O. Bluestreakite is monoclinic,  $P_{21}/n$ , with *a* 12.2383(7), *b* 10.3834(4), *c* 14.1945(6) Å,  $\beta$  103.008(2)°, *V* 1757.48(14) Å<sup>3</sup>, and *Z* = 2. The strongest four lines in the diffraction pattern are [*d* in Å(*I*)(*hkl*)]: 10.34(57)(101), 8.27(100)(011,101), 7.90(21)(110), and 1.9814(22)( $\overline{117}$ , $\overline{217}$ ). The atomic arrangement of bluestreakite is a partially reduced decavanadate group, with an ideal composition of [( $V^{4+}_2V^{5+}_8$ )O<sub>28</sub>]<sup>8-</sup>; charge balance in the structure is maintained by the [K<sub>4</sub>Mg<sub>2</sub>·14H<sub>2</sub>O]<sup>8+</sup> interstitial complex. The interstitial complex with the oxygen atoms of the structural unit takes place through direct bonding of the K1 and K2 atoms of the interstitial complex with the oxygen atoms of the structural unit, as well as extensive hydrogen bond

*Keywords*: bluestreakite, new mineral species, decavanadate, crystal structure, Blue Streak mine, Montrose County, Colorado, USA.

<sup>&</sup>lt;sup>§</sup> Corresponding author e-mail address: akampf@nhm.org

#### INTRODUCTION

The Uravan mineral belt (Fischer & Hilpert 1952, Chenoweth 1981), which sits astride the Colorado-Utah border, consists of bedded or roll-front deposits in the sandstone of the Salt Wash member of the Jurassic Morrison Formation (Carter & Gualtieri 1965, Shawe 2011). Here, uranium and vanadium, transported as oxyanions of U<sup>6+</sup> and V<sup>4+</sup> in weakly alkaline, moderately reducing, CO2-rich ground water, were deposited largely as uraninite,  $U^{4+}O_2$ , and montroseite,  $V^{3+}O_2$ (OH), where the solutions encountered more reducing conditions caused by carbonaceous material and H<sub>2</sub>S (Evans & Garrels 1958, Hostetler & Garrels 1962). Subsequent exposure of these deposits to more oxidizing near-surface aqueous solutions (which in turn react with sulfides to make the solutions more acidic) has resulted in a variety of secondary phases. Of particular interest mineralogically are the many secondary vanadium phases containing V<sup>4+</sup> and/or V<sup>5+</sup>.

Depending on prevailing Eh and pH, a variety of V-bearing oxy-anions may be present in these mineralizing solutions (Evans & Garrels 1958). The decavanadate anion,  $[V_{10}O_{28}]^{6-}$ , is stable at relatively high Eh in mildly acidic solutions, whereas its protonated derivatives,  $[H_x V_{10}O_{28}]^{(6-x)-}$ , occur at somewhat lower pH. Under more reducing conditions, conducive to the presence of both V<sup>4+</sup> and V<sup>5+</sup>, mixed-valence decavanadate anions,  $[(V^{4+}_x V^{5+}_{10-x})O_{28}]^{(6+x)-}$  may occur. Although the mineral-forming solutions are not available to sample or analyze, minerals of the three types have been characterized ( $[V_{10}O_{28}]^{6-}$ ,  $[H_xV_{10}O_{28}]^{(6-x)-}$ , and  $[(V^{4+}_xV^{5+}_{10-x})O_{28}]^{(6+x)-}$ ), suggesting that the various Eh and pH conditions noted above occurred during the evolution of the Uravan district secondary minerals (Table 1).

In this paper we describe the fourteenth natural decavanadate mineral and the second mixed-valence decavanadate, bluestreakite. All 14 natural decavanadate minerals (Table 1) occur in the mines of the Uravan Mineral Belt. All except pascoite and huemulite were first described from these mines and 11 of the 14 have been described since 2008. All 11 of the recent discoveries are the result of the collecting efforts of one of the authors (JM).

Bluestreakite is named for the place of its discovery, the Blue Streak mine, Montrose County, Colorado, USA. The new mineral and name were approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association (IMA2014–047). Two cotype specimens of bluestreakite are deposited in the Natural History Museum of Los Angeles County, 900 Exposition Boulevard, Los Angeles, CA 90007, USA under catalogue numbers 64174 and 64175.

#### **O**CCURRENCE

Bluestreakite was found on sandstone blocks in the Blue Streak mine, Bull Canyon, Montrose County, Colorado, USA (38°11′58″N 108°50′24″W).

Mineral species	Ideal formula*	First reference; best structure reference
Decavanadates: [V <sub>10</sub> O	28] <sup>6—</sup>	
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); Weeks <i>et al</i> . (1951)
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> ]	Hughes <i>et al</i> . (2002); Kampf <i>et al</i> . (2014)
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> ]	Hughes <i>et al</i> . (2008)
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> ]	Kampf & Steele (2008)
Magnesiopascoite	[MgCa <sub>2</sub> (H <sub>2</sub> O) <sub>16</sub> ] [V <sub>10</sub> O <sub>28</sub> ]	Hillebrand et al. (1914)
Pascoite	[Ca <sub>3</sub> (H <sub>2</sub> O) <sub>17</sub> ] [V <sub>10</sub> O <sub>28</sub> ]	Hughes <i>et al</i> . (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	[Na <sub>2</sub> (H <sub>3</sub> O) <sub>4</sub> (H <sub>2</sub> O) <sub>10</sub> ] [V <sub>10</sub> O <sub>28</sub> ]	Kampf <i>et al.</i> (2013a)
Wernerbaurite	[Ca <sub>2</sub> (H <sub>3</sub> O) <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)
Protonated Decavanad	dates: [H <sub>x</sub> V <sub>10</sub> O <sub>28</sub> ] <sup>(6-x)-</sup>	
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> (2011a)
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> (2011b)
Mixed-Valence Decava	nadates: [(V <sup>4+</sup> <sub>x</sub> V <sup>5+</sup> <sub>10-x</sub> )O <sub>28</sub> ] <sup>(6+x)-</sup>	
Nashite	[Na <sub>3</sub> Ca <sub>2</sub> (H <sub>2</sub> O) <sub>24</sub> ] [(V <sup>4+</sup> V <sup>5+</sup> <sub>9</sub> )O <sub>28</sub> ]	Kampf <i>et al.</i> (2013b)
Bluestreakite	$[K_4Mg_2(H_2O)_{14}][V^{4+}_2V^{5+}_8O_{28}]$	This study

TABLE 1. DECAVANADATE FAMILY OF MINERALS

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

The new mineral was collected by one of the authors (JM) on August 14, 2011. It was found growing on corvusite-montroseite-bearing sandstone blocks, closely associated with gypsum, huemulite, hummerite, metamunirite, and munirite. Other minerals found nearby include calciodelrioite, delrioite, hughesite, magnesiopascoite, metarossite, pascoite, powellite, and rossite.

The 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 environments, meteoric water reacts with pyrite in the deposit to form aqueous solutions with relatively low pH. The various secondary vanadate phases that formed depend upon the Eh-pH and presence of other cations (Na<sup>+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, Al<sup>3+</sup>) (*e.g.*, Evans & Garrels 1958).

## APPEARANCE AND PHYSICAL PROPERTIES

Crystals of bluestreakite most commonly occur in irregular polycrystalline coatings on rounded quartz grains or masses of montroseite (Figs. 1, 2). Rare distinct crystals (up to 0.3 mm in maximum dimension) are tablets or blades flattened on  $\{10\bar{1}\}$ , elongated on [010] and exhibiting the forms  $\{101\}$ ,  $\{10\bar{1}\}$ , and  $\{120\}$  (Figs. 3 and 4). Bluestreakite is very dark greenish blue, with a light blue streak. The mineral is transparent, with a subadamantine luster. Bluestreakite does not fluoresce in short- or long-wave ultraviolet radiation, and has a hardness of approximately 2.



FIG. 2. SEM image of bluestreakite crystals coating the surface of a quartz crystal.

Bluestreakite has a brittle tenacity. No cleavage or parting was observed, and the mineral displays irregular fracture. Bluestreakite is very slowly soluble in  $H_2O$  (days) and easily soluble in dilute HCl with immediate loss of color. The density of bluestreakite could not be measured because the mineral dissolves in aqueous density liquids and there is insufficient material for direct measurement. The calculated density is 2.350 g·cm<sup>-3</sup> based on the empirical formula and single-crystal cell data.



FIG. 1. Crystalline coating of bluestreakite covering a quartz grain associated with huemulite and gypsum. FOV: 0.3 mm.



FIG. 3. Crystals of bluestreakite with huemulite and gypsum on quartz. FOV: 3 mm.



FIG. 4. Crystal drawing of bluestreakite (clinographic projection in nonstandard orientation, *b* vertical).

#### OPTICAL PROPERTIES

The optical properties of bluestreakite were determined in white light. The mineral reacts in index liquids with n > 1.8, so  $\gamma$ , which is > 1.8, was calculated based on  $\alpha$ ,  $\beta$ , and 2V. The dark color and typically poor quality of crystals made extinction positions and indices of refraction difficult to discern. Bluestreakite is biaxial (–), with  $\alpha$  1.750(5),  $\beta$  1.800(5), and  $\gamma$  1.829 (calc). The 2V was measured as 73(3)° from extinction data using EXCALIBR (Gunter *et al.* 2004). No dispersion was observed. The optic orientation was only partially determined:  $Z = \mathbf{b}$ . The mineral exhibits slight pleochroism:  $X < Y \approx Z$ , all dark greenish blue. The Gladstone-Dale compatibility index, 1 – (K<sub>p</sub>/K<sub>c</sub>) is 0.021, in the range of excellent compatibility (Mandarino 2007).

### THE COMPOSITION OF BLUESTREAKITE

Analyses of bluestreakite (42 analyses from 17 crystals) were performed at the University of Utah on a Cameca SX-50 electron microprobe with four wavelength-dispersive spectrometers. Analytical conditions were 15 keV accelerating voltage, 10 nA beam current, and a beam diameter of 10 µm. Counting times were 10 seconds for each element and in the analytical routine Na, K, Mg, and V were counted simultaneously. Potassium experiences a time-dependent decrease in intensity under the electron beam that was accounted for by an exponential fit to the intensity versus time measurements and extrapolation to zero-time intensity. Raw X-ray intensities were corrected for matrix effects with a phi-rho-z algorithm (Pouchou & Pichoir 1991). There was no visible damage from the electron beam. Because insufficient material is available for a direct determination of H<sub>2</sub>O, the H<sub>2</sub>O

TABLE 2.	ANALYTICAL RESULTS FOR
	BLUESTREAKITE

Const.	wt.%	Range	SD	EMP Standard
Na <sub>2</sub> O K <sub>2</sub> O MgO V <sub>2</sub> O <sub>5</sub> VO <sub>2</sub> * V <sub>2</sub> O <sub>5</sub> * H <sub>2</sub> O <sup>†</sup> Total	0.34 11.45 5.64 65.61 8.38 56.42 18.20 100.43	0.08–0.77 10.50–12.49 5.23–6.06 62.13–68.00	0.14 0.51 0.18 1.14	albite sanidine diopside Y vanadate (syn)

\* Apportioned in accord with the structure.

<sup>†</sup> Based upon crystal structure.

content was calculated based upon the structure determination. Analytical data are given in Table 2.

The empirical formula (based on 42 O *apfu*) is  $(K_{3,37}Na_{0,15})_{\Sigma_3,52}Mg_{1.94}(V^{4+}_{1.40}V^{5+}_{8.60})O_{28}\cdot14H_2O$ . As noted in the crystal structure section, bluestreakite exhibits compositional variability similar to that observed in vanadium oxide bronze minerals, *i.e.*, charge balance is maintained by oxidation of one V<sup>4+</sup> atom to V<sup>5+</sup> for each cation charge deficiency resulting from partial occupancy of a cation site. EMP analyses showed the Mg content to generally be close to 2 *apfu*, corresponding to full occupancy of that structural site. Conversely, the analyses showed total K+Na to vary between 3.16 and 3.80 *apfu*, with 4 *apfu* corresponding to full occupancy of the K site. For bluestreakite, the compositional variability can be represented by the general formula  $K_{4-x}Mg_2(V^{4+}_{2-x}V^{5+}_{8+x})O_{28}\cdot14H_2O$ , where  $x \approx 0$  to 1.

We suggest that the ideal formula corresponds to the the composition when the K site is fully occupied (x = 0), K<sub>4</sub>Mg<sub>2</sub>(V<sup>4+</sup><sub>2</sub>V<sup>5+</sup><sub>8</sub>)O<sub>28</sub>·14H<sub>2</sub>O, which requires K<sub>2</sub>O 13.32, MgO 5.70, VO<sub>2</sub> 11.73, V<sub>2</sub>O<sub>5</sub> 51.43, H<sub>2</sub>O 17.83%, total 100 wt.%.

#### **CRYSTAL STRUCTURE: EXPERIMENTAL**

Powder X-ray diffraction data for bluestreakite were obtained with a Rigaku R-Axis Rapid II curved imaging plate microdiffractometer using monochromatized MoK $\alpha$  radiation. The powder data presented in Table 3 are in good agreement with those calculated from the structure data. Observed *d* values and intensities were derived by profile fitting using JADE 2010 software (Materials Data Inc.). Unit cell parameters refined from the powder data using JADE 2010 with whole pattern fitting are *a* 12.241(2), *b* 10.377(2), *c* 14.181(2) Å,  $\beta$  103.076(4)°, and *V* 1754.6(5) Å<sup>3</sup>.

I <sub>obs</sub>	$d_{\rm obs}$	$d_{\rm calc}$	I <sub>calc</sub>	h k l	I <sub>obs</sub>	d <sub>obs</sub>	$d_{\rm calc}$	I <sub>calc</sub>	hkl	I <sub>obs</sub>	$d_{\rm obs}$	$d_{\rm calc}$	I <sub>calc</sub>	hkl	
57	10.34	10.2429	58	Ī 0 1	4	2.889	2.8763	3	<b>4</b> 12			( 1.9056	1	525	
100	0.07	∫ 8.3036	48	011			2.8677	1	<u>2</u> 32	0	1 0000	1.8998	2	441	
100	8.27	8.1675	52	101			2.8653	1	410	8	1.8982	1.8960	5	531	
21	7.90	7.8307	28	110	6	2.844	2.8388	7	<u>1</u> 05			1.8927	1	ē 2 1	
		7.2920	3	<u>1</u> 11	15	2.781	2.7802	15	<u>1</u> 33			1.8146	1	442	
7	6.93	6.9151	6	002			2.7679	1	033			( 1.7795	2	5 4 1	
6	5.773	5.7556	5	012			( 2.7383	2	<u>1</u> 15		1 7070	1.7704	4	<b>208</b>	
		5.6112	1	<u>1</u> 12			2.7357	4	204	8	1.7678	1.7583	1	253	CRY
		5.1917	1	020	14	2.719	2.7225	4	303			1.7562	6	540	ſST
6	5.170 <	5.1704	1	210			2.7107	7	<b>4</b> 13			( 1.7432	1	Ī 1 8	Æ
		5.1215	3	<b>202</b>			L 2.6950	2	322			1.7388	1	701	STI
11	4.871	4.8605	9	021			2.6924	1	411	15	1.7345 <	1.7340	9	236	RUC
		4.8405	1	112			2.6728	1	015			1.7306	4	060	UL
		( 4.6678	1	Ī 0 3	_		∫ 2.6404	3	232			1.7289	1	245	RE
7	4.616	4.6308	2	<u>1</u> 21	7	2.641	2.6370	4	<u>3</u> 31			1.7126	1	160	AN
-		4.5931	2	<b>2</b> 12			2.6335	1	313			1.6969	1	533	DI
		4.5599	1	211	5	2.582	2.5852	3	420			1.6930	1	161	DES
3	4.369	4.3814	1	121			2.5710	1	<u>3</u> 24			1.6810	1	710	RI
5	4.188	4.1518	5	022			2.5607	2	<b>ā</b> 04			1.6756	1	<b>2</b> 28	PTI
		4.0966	1	<u>1</u> 22			( 2.5163	1	<u>1</u> 41			1.6534	1	137	VE
13	4.102	4.0837	8	202	14	2.501	2.4985	11	331			1.6260	1	711	M
		4.0711	2	<u>3</u> 01		2.001	2.4949	1	115			( 1.6145	1	<b>4</b> 46	NEF
		( 3.8325	2	<b>2</b> 13			2.4739	1	141			1.6081	2	545	A
5	3 807	3.8004	1	212			2.3791	1	511	5	1.6094 〈	1.6035	1	724	G
•	0.001	3.7902	1	<u>3</u> 11	3	2.369	2.3655	2	512			1.6006	2	632	×
		3.6293	1	221			2.3445	1	503			1.5934	1	262	
3	3.634	3.6105	2	301			2.3339	8	<b>2</b> 06			1.5780	1	640	
		( 3.4711	2	123	9	2.329	2.3055	3	116			1.5510	1	218	
		3.4576	3	004			2.3004	1	314	5	1 5455	1.5471	1	733	
12	3.444	3.4472	4	023			( 2.2771	5	216	0	1.0400	1.5432	5	537	
		3.4143	5	303	16	2 266	2.2641	1	<b>4</b> 32			1.5310	1	ē 3 6	
		3.3513	1	114	10	2.200	2.2587	11	430	3	1.5233 🕚	1.5240	3	546	
		( 3.2790	3	<u>1</u> 31			2.2503	1	016			1.5083	1	2 2 9	
7	3 277	3.2435	2	<u>3</u> 13			2.1734	10	340			1.5061	1	722	
,	5.211	3,2291	3	$\bar{2}23$	12	2.174	2.1671	2	520			( 1.4905	2	800	
		3.2036	1	<u>3</u> 21			2.1609	1	514	4	1.4896	1.4824	2	109	10

TABLE 3. POWDER X-RAY DIFFRACTION DATA FOR BLUESTREAKITE

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TABLE 3. (CONTINUED)

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$d_{calc}$	( 1.4777	1.4749	1.4740	1.4720	1.4680	( 1.4625	1.4605		1.4572	1.4572 1.4461	1.4572 1.4461 1.4393	1.4572       1.4461       1.4393       1.4348       1.4348
$d_{ m obs}$			1.4719	_					C R7C4.	1 67CH.	( R704.1	אסרא. אסרא.
l <sub>obs</sub>			o				Ċ	0		I	I	· •
h к I	<u>3</u> 42	116	341	135	432	<u>1</u> 07	126	7 7 7		217	2	2
$l_{calc}$	-	~	с	~	с	с	-	<del>,</del>	-	22	- 22 -	- 22
$d_{ m calc}$	2.1505	2.1277	2.1076	2.0635	2.0466	2.0240	2.0051	1.9866		1.9789	1.9789 1.9577	1.9789 1.9577 1.9220
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$l_{ m obs}$		•	4	•	4					77	77	77
н к І	131	2 14	320	213	032	$\bar{3} 2 2$	312	114		$\overline{4} 0 2$	4 0 2 4 0 0	402 320 102
$I_{calc}$	-	~	8	9	12	~	4	-		4	4 -	4 – w
$d_{ m calc}$	3.1868	3.1747	3.1560	3.1491	3.0951	3.0871	3.0170	3.0032		2.9934	2.9934 2.9811	2.9934 2.9811 2.9642
$d_{ m obs}$		)	3.162 {			3.100 {	)	3.003		_	_	
l <sub>obs</sub>			14		0	12		<u>б</u>				

The initial structure dataset was recorded on the diffractometer noted above and was solved by direct methods using SIR2004 (Burla et al. 2005). Because the limited nature of this dataset did not allow the determination of H atom positions, data were subsequently recorded using the same crystal at GSECARS and ChemMatCARS (CARS = Consortium for Advanced Radiation Sources) sectors 13 and 15 at the Advanced Photon Source, Argonne, IL. Data were recorded using a Bruker 6000 SMART CCD mounted on a Huber 4-circle diffractometer. We regard the cell parameters determined in the initial study to be more accurate and have used them in the final refinement of the structure.

Data were integrated and corrected for Lorentzpolarization and background effects using Bruker

## TABLE 4. CRYSTAL DATA, DATA COLLECTION, AND STRUCTURE REFINEMENT DETAILS FOR **BLUESTREAKITE**

X-ray radiation	0.49594 Å
Structural formula K <sub>3.8</sub> Mg <sub>2.0</sub> (\	/ <sup>4+</sup> <sub>1.8</sub> V <sup>5+</sup> <sub>8.2</sub> )O <sub>28</sub> ·14H <sub>2</sub> O
Space group	$P2_1/n$
Unit-cell dimensions	a = 12.2383(7) Å
	b = 10.3834(4) Å
	c = 14.1945(6) Å
	β = 103.008(2)°
V	1757.48(14) Å <sup>3</sup>
Ζ	2
Density (for above formula)	2.658 g·cm <sup>−3</sup>
Absorption coefficient	1.049 mm <sup>-1</sup>
Crystal size	30 × 25 × 15 mm
θ range	1.95 to 24.45°
Index ranges	<i>−</i> 19 ≤ <i>h</i> ≤ 15,
	<i>−</i> 15 ≤ <i>k</i> ≤ 16,
	$-22 \le \ell \le 23$
Reflections collected/unique	41421/7386;
_	$R_{\rm int} = 0.088$
GoF on $F^2$	0.968
Reflections with $F > 4\sigma(F)$	5403
Completeness to q = 24.45°	98.5%
Refinement method	Full-matrix least-
	squares on F <sup>2</sup>
Data / restraints / parameters	7386 / 21 / 307
Final R indices $[F_o > 2\sigma(I)]$	$R_1 = 0.0339,$
	$wR_2 = 0.0739$
R indices (all data)	$R_1 = 0.0563,$
	$wR_2 = 0.799$
Extinction coefficient	0.0090(10)
Largest diff. peak/hole	+1.08/-1.08e A <sup>-3</sup>

 $\begin{array}{l} \hline R_{\rm int} = \Sigma |F_{\rm o}^2 - F_{\rm o}^2 ({\rm mean})| \Sigma [F_{\rm o}^2]. \ {\rm GoF} = S = \{\Sigma [w(F_{\rm o}^2 - F_{\rm c}^2)^2]/(n-p)\}^{1/2}. \ R_1 = \Sigma ||F_{\rm o}| - |F_{\rm c}||/\Sigma |F_{\rm o}|. \ wR_2 = \{\Sigma [w(F_{\rm o}^2 - F_{\rm c}^2)^2]/\Sigma [w(F_{\rm o}^2)^2]\}^{1/2}; \ w = 1/[\sigma^2(F_{\rm o}^2) + (aP)^2 + bP] \ {\rm where} \ a \ {\rm is} \ 0.0389, \ b \ {\rm is} \ 0 \ {\rm and} \ P \ {\rm is} \ [2F_{\rm c}^2 + {\rm Max}(F_{\rm o}^2, 0)]/3 \end{array}$ 

TABLE 5. ATOM COORDINATES AND DISPLACEMENT PARAMETERS  $(\mbox{\AA}^2)$  FOR BLUESTREAKITE

	x/a	y/b	z/c	$U_{ m eq}$	U <sup>11</sup>	U <sup>22</sup>	U <sup>33</sup>	U <sup>23</sup>	<i>U</i> <sup>13</sup>	U <sup>12</sup>
V1	0.93866(2)	0.07887(2)	0.90155(2)	0.00778(6)	0.01069(13)	0.00601(12)	0.00621(13)	-0.00008(9)	0.00098(10)	-0.00041(9)
V2	0.79785(2)	0.94064(3)	0.03155(2)	0.00870(7)	0.01041(13)	0.00807(13)	0.00752(13)	-0.00042(10)	0.00183(10)	-0.00116(9)
V3	0.93302(3)	0.09340(3)	0.21442(2)	0.01201(7)	0.01500(14)	0.01296(14)	0.00906(14)	-0.00396(11)	0.00479(11)	-0.00466(10)
V4	0.07623(2)	0.22990(3)	0.08804(2)	0.00892(7)	0.01199(13)	0.00672(12)	0.00808(13)	-0.00145(10)	0.00235(10)	-0.00152(9)
V5	0.81559(2)	0.24235(3)	0.02496(2)	0.01229(7)	0.01298(14)	0.00797(13)	0.01675(16)	-0.00157(11)	0.00508(12)	0.00058(10)
K1*	0.73710(4)	0.59525(4)	0.07015(3)	0.01541(13)	0.0191(2)	0.0123(2)	0.0136(2)	-0.00028(15)	0.00125(16)	0.00184(14)
K2*	0.76138(3)	0.43194(4)	0.79692(3)	0.01227(12)	0.01371(19)	0.01117(19)	0.0110(2)	0.00033(13)	0.00084(14)	0.00052(13)
Mg	0.52903(5)	0.07479(5)	0.21913(5)	0.01018(12)	0.0116(3)	0.0083(3)	0.0107(3)	-0.0001(2)	0.0027(2)	-0.0001(2)
01	0.94361(10)	0.07374(11)	0.05348(9)	0.0096(2)	0.0121(5)	0.0084(5)	0.0084(6)	-0.0009(4)	0.0025(5)	-0.0011(4)
O2	0.07012(10)	0.18196(11)	0.94888(8)	0.0100(2)	0.0130(6)	0.0089(5)	0.0077(6)	0.0001(4)	0.0018(4)	-0.0010(4)
O3	0.83803(10)	0.93738(11)	0.90262(9)	0.0097(2)	0.0127(5)	0.0077(5)	0.0086(6)	-0.0006(4)	0.0021(4)	-0.0015(4)
O4	0.82916(10)	0.21988(11)	0.16144(9)	0.0113(2)	0.0139(5)	0.0099(5)	0.0101(6)	-0.0014(4)	0.0027(5)	0.0007(4)
O5	0.72184(11)	0.08998(11)	0.00016(9)	0.0121(2)	0.0132(5)	0.0114(6)	0.0110(6)	-0.0008(5)	0.0012(5)	0.0009(4)
O6	0.05667(10)	0.20121(12)	0.21122(9)	0.0109(2)	0.0140(6)	0.0102(5)	0.0084(6)	-0.0019(4)	0.0023(5)	-0.0001(4)
07	0.84511(10)	0.20150(12)	0.89153(9)	0.0114(2)	0.0139(6)	0.0098(5)	0.0094(6)	0.0008(4)	0.0007(5)	0.0004(4)
O8	0.95386(10)	0.05556(11)	0.78509(9)	0.0106(2)	0.0132(6)	0.0089(5)	0.0093(6)	0.0000(4)	0.0020(5)	-0.0013(4)
O9	0.95563(10)	0.33272(11)	0.04803(9)	0.0116(2)	0.0149(6)	0.0080(5)	0.0113(6)	0.0006(4)	0.0015(5)	0.0001(4)
O10	0.82602(10)	0.95864(12)	0.16405(9)	0.0105(2)	0.0134(5)	0.0101(5)	0.0081(6)	-0.0004(4)	0.0026(4)	-0.0007(4)
O11	0.92899(11)	0.09111(12)	0.32864(10)	0.0160(3)	0.0201(6)	0.0177(6)	0.0113(6)	-0.0023(5)	0.0059(5)	-0.0016(5)
012	0.17802(10)	0.33356(11)	0.10458(9)	0.0126(2)	0.0151(6)	0.0102(5)	0.0123(6)	-0.0019(5)	0.0025(5)	-0.0017(4)
O13	0.69867(11)	0.83422(12)	0.00993(10)	0.0142(2)	0.0148(6)	0.0137(6)	0.0141(6)	-0.0015(5)	0.0033(5)	-0.0030(5)
O14	0.72723(11)	0.35941(13)	0.99388(10)	0.0172(3)	0.0197(6)	0.0140(6)	0.0184(7)	-0.0001(5)	0.0051(5)	0.0029(5)
OW1	0.83893(11)	0.45233(12)	0.24539(10)	0.0142(3)	0.0169(6)	0.0101(6)	0.0179(7)	-0.0042(5)	0.0085(5)	-0.0021(5)
H1A	0.8482(19)	0.3739(19)	0.2248(16)	0.017						
H1B	0.7905(18)	0.447(2)	0.2844(16)	0.017						
OW2	0.58854(11)	0.07122(11)	0.81330(10)	0.0125(2)	0.0156(6)	0.0086(6)	0.0112(6)	0.0015(5)	-0.0016(5)	-0.0019(4)
H2A	0.6350(18)	0.082(2)	0.8664(15)	0.015	. ,	. ,				
H2B	0.5738(17)	0.1500(19)	0.7881(15)	0.015						
OW3	0.89900(10)	0.29400(12)	0.69409(10)	0.0123(2)	0.0140(6)	0.0094(5)	0.0123(6)	0.0012(5)	0.0006(5)	-0.0007(4)
H3A	0.9127(17)	0.2124(18)	0.7130(15)	0.015						
H3B	0.8557(17)	0.291(2)	0.6354(14)	0.015						
OW4	0.54149(12)	0.09639(14)	0.07456(10)	0.0169(3)	0.0140(6)	0.0228(7)	0.0138(6)	0.0020(5)	0.0031(5)	0.0023(5)
H4A	0.6043(16)	0.094(2)	0.0545(18)	0.020						
H4B	0.5034(18)	0.160(2)	0.0546(17)	0.020						

					TABLE 5. (CO	NTINUED)				
	x/a	d/y	z/c	U <sub>eq</sub>	U <sup>11</sup>	$U^{22}$	U <sup>33</sup>	U <sup>23</sup>	$U^{13}$	U <sup>12</sup>
OW5	0.65005(11)	0.22041(12)	0.25467(10)	0.0151(3)	0.0168(6)	0.0099(6)	0.0208(7)	-0.0044(5)	0.0087(5)	-0.0033(5)
H5B	0.6965(18)	0.225(2)	0.2174(16)	0.018						
0W6	0.01627(11)	0.43470(12)	0.86453(10)	0.0138(2)	0.0223(7)	0.0084(6)	0.0108(6)	0.0010(5)	0.0039(5)	-0.0008(5)
H6A	0.0222(18)	0.5027(18)	0.8993(16)	0.017						
H6B	0.0147(19)	0.3686(18)	0.8999(16)	0.017						
0W7	0.5269(2)	0.6242(2)	-0.04645(16)	0.0622(8)	0.0869(17)	0.0556(13)	0.0286(11)	0.0183(10)	-0.0195(11)	-0.0506(13)
H7A	0.502(3)	0.570(3)	-0.092(2)	0.075						
H7B	0.513(3)	0.615(4)	0.0098(18)	0.075						
*Occu	pancies: K1 = 0	).920(2), K2 = 0.	.977(2).							

software (SAINTPLUS). SHELXL-2013 (Sheldrick 2008) was used for the refinement of the structure. Difference Fourier synthesis located all H atom positions and the H positions were refined with a soft restraint on the O-H distances of 0.9(3) Å and with their  $U_{eq}$  set to 1.2× that of the O atoms to which they are associated. All non-hydrogen atoms were refined with anisotropic displacement parameters. Data collection and refinement details are given in Table 4, atom coordinates and equivalent isotropic displacement parameters in Table 5, and selected bond distances and cation bond valence sums in Table 6. A table of structure factors, a CIF file, and a table of anisotropic displacement parameters are available from the Depository of Unpublished Data on the MAC website [document Bluestreakite CM52\_ 10.3749/canmin.14000721.

### ATOMIC ARRANGEMENT OF BLUESTREAKITE

The structure of bluestreakite (Fig. 5) consists of two distinct parts, a structural unit and an interstitial complex, as suggested by Schindler & Hawthorne (2001) for minerals with structural units of higher bond valence. The structural unit consists of the  $[(V^{4+}_{x}V^{5+}_{10-x})O_{28}]^{(6+x)-}$  modification of the wellknown decavanadate group  $(V_{10}^{5+}O_{28})^{6-}$  polyanion (Table 1), wherein a portion of the pentavalent vanadium atoms is reduced to V4+. The bond valence sums for V1 to V5 indicate that the V<sup>4+</sup> occurs predominantly at the sites V3 and V5 (Table 6). The blue color of bluestreakite is an indicator of the mixed-valence of vanadium, as opposed to the yellow-orange color of the pure pentavalent phases (Ramasarma 2007). Mixed-valence ( $V_{10}O_{28}$ ) groups are not unknown; Ramasarma (2007) described a mixed-valence blue decavanadate compound, and the mineral nashite (Kampf et al. 2013b) is a naturally occurring mixed-valence decavanadate compound, also with the typical blue color that derives from charge transfer between V ions.

The interstitial complex is formed of irregular  $K1[O_4(H_2O)_4]$  and  $K2[O_6(H_2O)_3]$  polyhedra, and a  $Mg(H_2O)_6$  octahedron. Bonding between the interstitial complex and the structural unit takes place through direct bonding of the K1 and K2 atoms of the interstitial complex with the oxygen atoms of the structural unit (Table 6) as well as extensive hydrogen bonding between oxygen atoms of the structural unit and hydrogen atoms of the interstitial complex H<sub>2</sub>O molecules. Structure refinement suggested the K sites are nearly full (Table 5), but microprobe analyses indicated that the K content varies over a rather large range (Table 2); the structure crystal was not analyzed by EMPA. We subsequently concluded that bluestreakite exhibits compositional variability similar to that observed in

V1-	07	1.6967(12)	1.33	V2-	O13	1.6188(13)	1.65
	O8	1.7217(13)	1.25		O5	1.8116(12)	0.98
	O3	1.9193(12)	0.73		O10	1.8438(13)	0.90
	O2	1.9233(12)	0.72		O3	1.9992(13)	0.59
	01	2.1402(12)	0.40		O2	2.0265(12)	0.55
	01	2.1433(12)	0.40		01	2.2224(12)	0.32
Mean, sum:		1.924	4.83	Mean, sum:		1.920	4.97
V3-	O11	1.6333(14)	1.52	V4-	012	1.6229(12)	1.63
	O4	1.8643(12)	0.76		O9	1.8064(12)	0.99
	O6	1.8911(12)	0.71		O6	1.8417(12)	0.90
	O10	1.9397(12)	0.62		O3	2.0181(12)	0.56
	O8	2.0747(12)	0.44		O2	2.0222(12)	0.55
	01	2.3261(12)	0.24		01	2.2684(12)	0.28
Mean, sum:		1.955	4.29	Mean, sum:		1.930	4.91
V5-	O14	1.6206(13)	1.58	Mg-	OW1	2.0288(14)	0.40
	O9	1.9164(12)	0.66		OW3	2.0639(14)	0.37
	O4	1.9211(13)	0.65		OW2	2.0695(13)	0.36
	O5	1.9395(12)	0.62		OW5	2.0971(14)	0.34
	07	2.0507(13)	0.47		OW4	2.1037(15)	0.33
	01	2.3234(12)	0.24		OW6	2.1069(15)	0.33
Mean, sum:		1.962	4.23	Mean, sum:		2.078	2.12
K1–	O13	2.6321(13)	0.26	K2–	O3	2.8205(13)	0.15
	O14	2.6693(14)	0.23		07	2.8212(13)	0.15
	011	2.7409(14)	0.19		012	2.8226(13)	0.15
	OW7	2.7444(25)	0.19		OW3	2.8505(13)	0.14
	OW5	2.8693(15)	0.14		O6	2.8802(13)	0.13
	OW1	2.9235(15)	0.12		08	2.9261(13)	0.12
	OW6	2.9676(14)	0.10		O14	3.0139(14)	0.09
	012	2.9895(13)	0.10		OW2	3.0341(14)	0.09
Mean, sum:		2.817	1.33		OW6	3.0503(15)	0.08
•				Mean, sum:		2.913	1.12
				,			

TABLE 6. SELECTED BOND DISTANCES (Å) AND BOND-VALENCE VALUES (VU) IN BLUESTREAKITE

Bond valences are based upon constants for  $V^{5+}$ , Mg, and K from Brese & O'Keeffe (1991), except V3 and V5, which use the mixed-valence,  $V^{4+}/V^{5+}$  constants of Brown & Altermatt (1985).

vanadium oxide bronze minerals (Hughes & Finger 1983, Evans & Hughes 1990). The seeming disparity between the K content of the structure crystal as determined by site refinement ( $K_{3.80}$ ) and the average of 42 chemical analyses of 17 crystals determined by electron microprobe ( $K_{3.37}$ ) illustrates that compositional variability.

In compounds such as the oxide bronzes, cations can partially occupy sites, in non-stoichiometric amounts. Charge balance for the non-stoichiometric cation charges is maintained by reduction of one V<sup>5+</sup> atom to V<sup>4+</sup> for each cation charge in a partially occupied site. In bluestreakite, charge balance is maintained by the coupled substitution  $\Box + V^{5+} \leftrightarrow$  $K^+ + V^{4+}$  and the general formula can be written  $K_{4-x}Mg_2(V^{4+}_{2-x}V^{5+}_{8+x})O_{28}\cdot14H_2O$ , where  $x \approx 0$  to 1 (the range 0.16 to 0.80 is provided by the EMP analyses). In the structure refinement, the Mg site refined to full occupancy and the K1 and K2 site occupancies refined to 0.920(2) and 0.977(2), respectively, neglecting the contribution of minor Na. This provides the formula  $K_{3,8}Mg_{2,0}(V^{4+}{}_{1.8}V^{5+}{}_{8.2})$   $O_{28}\cdot14H_2O$  for the structure crystal. Considering that *x* values less than 1.0 correspond to the K sites being, on average, more than  ${}_{34}$  occupied, we propose the ideal formula  $K_4Mg_2$   $(V^{4+}{}_2V^{5+}{}_8O_{28})\cdot14H_2O$ .

It is worth noting that an x value of 2 would theoretically yield a formula in which all V is 5+:  $K_2Mg_2(V_{10}^{5+}O_{28})\cdot 14H_2O$ . Except for two fewer  $H_2O$  groups *pfu*, this is the same as the formula of hummerite,  $K_2Mg_2(V_{10}^{5+}O_{28})\cdot 16H_2O$ , a phase that is associated with bluestreakite. However, the structure of hummerite (Hughes *et al.* 2002), although



FIG. 5. The atomic arrangement of bluestreakite. The unit is cell shown by dashed lines. O atoms of isolated H<sub>2</sub>O groups are shown as large white spheres. H atoms are shown as small white spheres.

also containing the decavanadate polyanion, is distinct from that of bluestreakite, with different coordination of the K atom(s) in the interstitial complex.

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