# Carlfrancisite: Mn<sub>3</sub><sup>2+</sup>(Mn<sup>2+</sup>,Mg,Fe<sup>3+</sup>,Al)<sub>42</sub>(As<sup>3+</sup>O<sub>3</sub>)<sub>2</sub>(As<sup>5+</sup>O<sub>4</sub>)<sub>4</sub>[(Si,As<sup>5+</sup>)O<sub>4</sub>]<sub>6</sub>[(As<sup>5+</sup>,Si)O<sub>4</sub>]<sub>2</sub>(OH)<sub>42</sub>, a new arseno-silicate mineral from the Kombat mine, Otavi Valley, Namibia

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### ABSTRACT

Carlfrancisite,  $Mn_{3}^{2+}(Mn^{2+},Mg,Fe^{3+},Al)_{42}(As^{3+}O_3)_2(As^{5+}O_4)_4[(Si,As^{5+})O_4]_6[(As^{5+},Si)O_4]_2(OH)_{42}$ , is a new mineral from the Kombat mine, Otavi Valley, Namibia, and occurs as curved platy aggregates  $\sim$ 2 cm across on a matrix of Mn arsenates and oxides. It is yellowy orange to pale yellow with a very pale-yellow streak, translucent with a vitreous to opalescent luster, and does not fluoresce under ultraviolet light. Cleavage is micaceous on {001}, and no parting or twinning was observed. Mohs hardness is 3, and carlfrancisite is brittle with a hackly fracture. The calculated density is 3.620 g/ cm<sup>3</sup>. Optical properties were measured with a Bloss spindle stage for the wavelength 590 nm using a gel filter. The indices of refraction are  $\varepsilon = 1.756$ ,  $\omega = 1.758$ , and it is non-pleochroic. Carlfrancisite is trigonal, space group  $R\overline{3}c$ , a = 8.2238(2), c = 205.113(6) Å, V = 12013.5(4) Å<sup>3</sup>, Z = 6, c:a = 1:24.941. The seven strongest lines in the X-ray powder-diffraction pattern are as follows: d (Å), I, (hkl): 2.826, 100, (2 2 44); 2.371, 88, (2 3 40, 1 3 41); 1.552, 84, (1 5 0); 2.676, 63, (2 3 7); 3.243, 54, (0 1 56, 1 2 39); 4.107, 48,  $(\overline{1} 2 0)$ ; 2.918, 47, (0 2 40). Chemical analysis by electron microprobe and crystalstructure refinement gave As<sub>2</sub>O<sub>5</sub> 13.07, As<sub>2</sub>O<sub>3</sub> 3.18, P<sub>2</sub>O<sub>5</sub> 0.50, V<sub>2</sub>O<sub>5</sub> 0.74, SiO<sub>2</sub> 8.96, Al<sub>2</sub>O<sub>3</sub> 0.78, FeO 0.22, MnO 53.25, MgO 9.37, H<sub>2</sub>O(calc) 8.42, sum 98.49 wt%. The H<sub>2</sub>O content and the valence states of As were determined by crystal-structure analysis. The empirical formula is  $Mn_{33}^{2+}s_5Mg_{10,39}Fe_{3-14}^{2+}Al_{0.68}$  $As_{144}^{3+4}(Si_{667}P_{0.32}V_{0.37}^{5+}As_{5,18}^{5+})O_{54}$  (OH)<sub>42</sub> on the basis of 96 anions with (OH) = 42 apfu. The structure of carlfrancisite is closely related to that of mcgovernite and turtmannite.

**Keywords:** Carlfrancisite, new mineral species, arseno-silicate, Tsumeb mine, Otavi Valley, Namibia, electron microprobe analysis, optical properties, chemical analysis, mcgovernite, turtmannite

### INTRODUCTION

The mineralization at the Kombat mine, Namibia, consists of epigenetic, hydrothermal, and metasomatic replacement deposits, and fracture-fill Cu-Pb-(Ag) sulfides, and a strong metamorphic overprint has given rise to lenses of Mn-oxide, Fe-oxide, and Fesilicate minerals (Schneider 2004). Dunn et al. (1988) reported a mcgovernite-like mineral from this locality. As part of our ongoing interest in the basic Mn arsenates (Cooper and Hawthorne 1999, 2001, in preparation 2013), we have characterized the mineral described by Dunn et al. (1988) and report the results here. This mineral is named carlfrancisite after Carl A. Francis (born October 21, 1949, in Stafford Springs, Connecticut, U.S.A.), mineralogist and curator of the Harvard mineral collection, who was involved in the discovery and initial characterization of the chemical composition and unit cell of this mineral, and who has made many significant contributions to scientific mineralogy, mineral curation, and the mineral-collecting community. The new mineral and mineral name have been approved by the Commission on New Minerals, Nomenclature and Classification, International Mineralogical Association (IMA 2012-033). The holotype specimen of carlfrancisite has been deposited in the mineral collection of the Department of Natural History, Royal Ontario Museum, catalog number M56375.

OCCURRENCE

Carlfrancisite was found in 1982 in the E15-11 south stope, 11 level, at the 1241 m elevation, in the Asis West sector of the Kombat mine (Dunn et al. 1988), Otavi Valley, Namibia, latitude 19°45' S, longitude 17°45' E, associated with alleghanyite, chlorite, pyrochroite, spinels, and ribbeite. Carlfrancisite and associated Mn silicates and oxides occur as metamorphosed lenses associated with Cu-Pb-Ag sulfide ores in feldspathic sandstone that have subsequently been reworked by late hydrothermal solutions (Innes and Chaplin 1986).

### **PHYSICAL PROPERTIES**

Carlfrancisite occurs as curved platy aggregates ~2 cm across on a matrix of Mn arsenates and oxides (Fig. 1a). It is pale yellow to yellowy orange (Fig. 1b) with a very pale yellow streak, is translucent with a vitreous to opalescent luster, and does not fluoresce under ultraviolet light. Cleavage is micaceous on {001}, and no parting or twinning was observed. Mohs hardness is 3, and carlfrancisite is brittle with a hackly fracture. The calculated density is 3.620 g/cm<sup>3</sup>. Optical properties were measured with a Bloss spindle stage for the wavelength 590 nm using a gel filter. Carlfrancisite is non-pleochroic, uniaxial positive with indices of refraction  $\omega$ = 1.756,  $\varepsilon$  = 1.758, both ±0.002.

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FIGURE 2. The Raman spectrum of carlfrancisite.

### VIBRATIONAL SPECTROSCOPY

The Raman spectrum of carlfrancisite was collected in backscattered mode with a HORIBA Jobin Yvon-LabRAM ARAMIS integrated confocal micro-Raman system equipped with a 460 mm focal-length spectrograph and a multichannel air-cooled (-70 °C) CCD detector. A magnification of 100× was used with an estimated spot size of 1  $\mu$ m, a 1800 gr/mm grating, an excitation radiation of 532 nm, and a laser power between 5 and 12.5 mW. Calibration was done using the 520.7 cm<sup>-1</sup> line of Si metal, data were collected over the range 4000–100 cm<sup>-1</sup> for 20 s, and the final spectrum is the average of 2 scans. The FTIR spectrum was collected using a Bruker Hyperion IR microscopy equipped with a liquid-nitrogen-cooled detector on a thin film produced FIGURE 1. Images of carlfrancisite: (a) platy aggregates on a matrix of Mn arsenates and oxides; (b) yellow-orange platy aggregate of carlfrancisite.

with a diamond anvil. Spectra over the range 4000–400 cm<sup>-1</sup> were obtained by averaging 100 scans with a resolution of 4 cm<sup>-1</sup>. Baseline correction was done using the OPUS spectroscopic software (Bruker Optic GmbH).

Both infrared and Raman spectra have an intense envelope of absorption centered on ~3550 cm<sup>-1</sup> (Figs. 2 and 3) and showing extensive fine structure with numerous sharp bands characteristic of O-H stretching modes, in accord with the presence of numerous (OH) groups in the structure. The IR spectrum (Fig. 4) shows a strong absorption centered at ~890 cm<sup>-1</sup> with two shoulders at 952 and 932 cm<sup>-1</sup>, an intense peak at 802 cm<sup>-1</sup> and weaker peaks at 1024, 741, and 670 cm<sup>-1</sup>, whereas the Raman spectrum shows a strong band at ~790 cm<sup>-1</sup> with shoulders at 822 and 843 cm<sup>-1</sup> and separate weak peaks at 897 and 732 cm<sup>-1</sup> (Fig. 2). In carlfrancisite, there are three anionic groups,  $(SiO_4)^{4-}$ ,  $(AsO_4)^{3-}$ , and  $(AsO_3)^{3-}$  that will each have a symmetric stretch and an asymmetric stretch. However, the frequency separations between the bands due to the asymmetric and the symmetric stretches vary strongly from one structure to another, and cannot be assigned with conviction. The Raman spectrum of the much simpler mineral dixenite, Cu<sup>+</sup>Mn<sub>14</sub><sup>2+</sup>  $(As^{3+}O_3)_5(SiO_4)_2(As^{5+}O_4)$  (OH)<sub>6</sub>, has been assigned by Bahfenne and Frost (2009), but some features of the assignment are not compatible with the known structure of dixenite. Any assignment of the vibrational spectra of carlfrancisite at the present time would be speculative at best.

### **CHEMICAL COMPOSITION**

Carlfrancisite (not the crystal used for its structure determination) was analyzed with a Cameca SX-100 electron microprobe operating in wavelength-dispersion mode with an accelerating voltage of 15 kV, a specimen current of 10 nA, and a beam diameter of 10 µm. The following standards were used: cobaltite, As; apatite, P; synthetic VP<sub>2</sub>O<sub>7</sub>, V; diopside, Si; andalusite, Al; fayalite, Fe; spessartine, Mn; forsterite, Mg. The data were reduced and corrected by the PAP method of Pouchou and Pichoir (1985) and are given in Table 1. The As<sub>2</sub>O<sub>5</sub> and As<sub>2</sub>O<sub>3</sub> contents were derived from the corresponding site occupancies from the crystal-structure refinement and the As content determined by electron-microprobe analysis. The presence and quantity of (OH) groups were established by crystal-structure solution and refinement. In addition, both Raman and infrared spectroscopy (Fig. 3) show the presence of numerous sharp peaks assigned to O-H stretching modes. Table 1 gives the chemical composition (mean of 10 determinations). The empirical formula was calculated on the basis of 96 anions with (OH) = 42 anions per formula unit (apfu) as indicated by



FIGURE 3. The Raman (top) and infrared (bottom) spectra of carlfrancisite in the principal OH-stretching region.

<b>IABLE 1.</b> Chemical composition of carifrancisi
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Constituent	wt%	Range
As <sub>2</sub> O <sub>5</sub> *	13.07	-
As <sub>2</sub> O <sub>3</sub> *	3.18	-
$P_2O_5$	0.50(5)	0.41-0.57
SiO <sub>2</sub>	8.96(8)	8.86-9.10
Al <sub>2</sub> O <sub>3</sub>	0.78(3)	0.73-0.83
V <sub>2</sub> O3	0.61(4)	0.54-0.67
FeO	0.22(3)	0.18-0.26
MnO	53.25(26)	52.88-53.65
MgO	9.37(11)	9.20-9.52
H <sub>2</sub> O	8.58	-
Total	98.52	

crystal-structure solution and refinement, giving  $Mn_{33,55}^{2+}Mg_{10,39}$  $Fe_{0.14}^{2+}Al_{0.68}As_{1.44}^{3+}(Si_{6.67}P_{0.32}V_{0.37}^{5+}As_{5.08}^{5+})O_{54}(OH)_{42}$ . The general structural formula is ideally  $Mn_3^{2+}(Mn^{2+},Mg,Fe^{3+},Al)_{42}(As^{3+}O_3)_2(As^{5+}O_4)_4$  $[(Si,As^{5+})O_4]_6[(As^{5+},Si)O_4]_2(OH)_{42}$ .

## X-ray powder diffraction

X-ray powder-diffraction data were obtained as indicated in Table 2. We indexed the powder pattern from intensities calculated with the routine XPOW embedded in the Bruker system of programs, and used this indexing to refine the unit-cell dimensions using the program CELREF (Appleman and Evans 1973).



FIGURE 4. The infrared spectrum of carlfrancisite from 1200 to  $650 \text{ cm}^{-1}$ .

 TABLE 2.
 X-ray powder-diffraction pattern for carlfrancisite

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I <sub>obs</sub>	$d_{\rm obs}$ (Å)	d <sub>calc</sub> (Å)	hkl	I <sub>obs</sub>	$d_{ m obs}$ (Å)	$d_{\rm calc}$ (Å)	h	k	1
25	17.086	17.055	0 0 12	88 B	2.371*	2.371	3	0	0
27	11.386	11.370	0 0 18	"	2.371*	2.369	3	0	3
9	6.837	6.853	018	18	2.341	2.341	2	3	43
14	6.221	6.216	1 1 16	11	2.287	2.287	1	3	47
36	5.689	5.685	0 0 36	16	2.276	2.274	0	0	90
"	5.689	5.650	1 1 22	15 B	2.245	2.247	1	3	50
15	5.275	5.277	0 1 26	19	2.209	2.211	1	2	78
11	5.091	5.097	1 1 28	"	2.209	2.206	1	3	53
11	4.880	4.873	0 0 42	9	2.180	2.179	2	3	55
8	4.754	4.756	0 1 32	12 B	2.128	2.132	0	3	42
8	4.593	4.595	1 1 34	"	2.128	2.125	1	3	59
21 B	4.286	4.294	0 1 38	13	2.044	2.044	1	3	65
48	4.107	4.106	120	14	2.018	2.018	2	3	67
15	3.925	3.932	1 2 15	14	2.009	2.005	1	3	68
11	3.889	3.893	0 1 44	13	1.968	1.967	1	1	100
17	3.783	3.784	1 2 21	8 B	1.939	1.940	2	3	73
15	3.616	3.611	1 2 27	24	1.890	1.890	1	3	77
8 B	3.522	3.522	228	9	1.857	1.858	1	4	37
"	3.522	3.518	1 2 30	18	1.817	1.817	1	3	83
43	3.430	3.444	1 1 52	12	1.795	1.794	2	3	85
"	3.430	3.424	1 2 33	7 B	1.776*	1.774	0	2	100
11	3.329	3.329	1 2 36	"	1.776*	1.774	0	4	8
54 B	3.243	3.251	0 1 56	11 B	1.761*	1.761	1	1	105
"	3.243	3.234	1 2 39	"	1.761*	1.761	2	0	101
10	3.194	3.198	0 2 28	31	1.725	1.725	2	3	91
37	3.139	3.140	1 2 42	30 B	1.698	1.699	3	3	84
43 B	3.051	3.062	0 2 34		1.698	1.699	0	3	84
	3.051	3.048	1 2 45	11 B	1.682	1.681	1	3	95
10	2.996	2.994	0 1 62	5	1.660	1.661	0	4	44
10	2.967	2.968	2 2 38		1.660	1.660	2	3	97
4/	2.918	2.920	0 2 40	16	1.649	1.649	1	3	98
86	2.869	2.870	1 2 51	27	1.629	1.628	2	3	100
100	2.826	2.825	2 2 44	10	1.629	1.629	3	5	/
32	2.778	2.778	0246	19	1.619	1.618	1	3	101
14	2.706	2.704	1 1 70	9 B "	1.000	1.008	-	1	74
<b>6</b> 2	2.706	2.703	1 2 5/	10.0	1.000	1.606	3	4	74
63 12	2.676	2.677	23/	10 B	1.598	1.599	2	4	50
13	2.045	2.044	1 3 14 $\overline{1} 2 17$	C D	1.598	1.598	2	3	103
23	2.022	2.024	131/	<u>ю</u> В "	1.580	1.588	-	5	21
15	2.579	2.578	$\frac{0}{1}$ $\frac{1}{2}$ $\frac{74}{2}$	12.0	1.580	1.584	2	2	31
20 0	2.545	2.544	1 2 20 7 2 20	12 D #	1.505	1.500	2	2	27
7 10	2.524	2.323	2 3 20	04	1.505	1.505	5	5	5/
20	2.433	2.450	2 2 2 2 7	04	1.552	1.552	-	5	0
20	2.410	2.410	2 3 3/ 1 3 38	15	1.540	1.540	2	J	-++
	2 mm 1 / 1	2 mm 1 / 1							

Notes: Powder data collected on Siemens D5000 powder diffractometer from 4–62° 20; step width = 0.020°; step time = 20 s; divergence and anti-scatter slits = 1.0 mm; CuK\alpha radiation (h = 1.54178 Å); 40 V/40 mA; no internal standard used, mean standard deviation on *d*-values is estimated as 0.002 Å. Indexed on *a* = 8.2128(14), *c* = 204.66(5) Å, *V* = 11955.2(3.5) Å<sup>3</sup>. B = broad reflection. \* Not used in refinement. However, this resulted in lines that could not be indexed on the resulting unit-cell of carlfrancisite. However, detailed examination of the single-crystal diffraction frames did not show the presence of any reflections that could not be indexed on the carlfrancisite cell. Eventually, we realized that the routines we were using to calculate reflection intensities did not assign *hkl* indices greater than 50. Normally this is not an issue, but for carlfrancisite with its *c* dimension of ~205 Å, indices for *l* can considerably exceed this value. When the pattern was directly indexed from single-crystal data (rather than calculating the resultant powder-pattern intensities), all lines could be indexed. Data are listed in Table 2. Unit-cell parameters refined from the powder data are as follows: a = 8.2148(2), c = 204.54(4) Å, V = 11953(1) Å<sup>3</sup>, close to the values obtained by single-crystal diffraction (see below).

### Single-crystal diffraction

A crystal was attached to a tapered glass fiber and mounted on a Bruker D8 three-circle diffractometer equipped with a rotatinganode generator (MoK $\alpha$ ), multilayer optics and an APEX-II detector. A total of 111 042 intensities was collected to 60° 20 using 10 s per 0.2° frames with a crystal-to-detector distance of 5 cm. The following unit-cell dimensions were obtained by leastsquares refinement of the positions of 9631 reflections with I >10 $\sigma I$ : a = 8.2238(2), c = 205.113(6) Å, V = 12013.5(4) Å<sup>3</sup>, Z = 6, c:a = 1:24.941. Systematic absences are in accord with the space group  $R\overline{3}c$ . The crystal structure is extremely complicated and will be described in detail elsewhere, together with the structure of mcgovernite.

### **RELATED MINERALS**

Carlfrancisite, ideally  $Mn_3^{2+}(Mn^{2+},Mg,Fe^{3+},Al)_{42}(As^{3+}O_3)_2(As^{5+}O_4)_4[(Si,As^{5+})O_4]_6[(As^{5+},Si)O_4]_2(OH)_{42}$ , is related to mcgovernite (Palache and Bauer 1927; Wuensch 1960, 1968; Cooper and Hawthorne 2001), ideally  $(Zn,Mn^{2+})_3(Mn^{2+},Mg,Fe^{3+},Al)_{42}[(As^{3+},Zn)O_3]_2(As^{5+}O_4)_4[(Si,As^{5+})O_4]_8(OH)_{42}$ , and turtmannite (Brugger

 TABLE 3.
 Comparison of the properties of carlfrancisite, mcgovernite, and turtmannite

	Carlfrancisite <sup>*,a</sup>	Mcgovernite <sup>b</sup>	Turtmannite <sup>c</sup>			
Formula	see below*	see below	see below			
Symmetry	Rhombohedral	Rhombohedral	Rhombohedral			
Space group	R <del>3</del> c	R3c	R <del>3</del> c			
a (Å)	8.2238	8.2061	8.259			
с	205.113	204.118	204.3			
Ζ	6	6	6			
V (ų)	12013.5	11851.3	12068.5			
Color	shades of yellow	shades of brown-orange	bright yellow			
Cleavage	{001} perfect	{001} perfect	{001} perfect			
Hardness	3	3	-			
Occurrence	Mn deposit	Mn-Zn deposit	Mn deposit			

Notes: The characteristic differences between carlfrancisite and mcgovernite are as follows. Essential [5]-coordinated Zn in mcgovernite is replaced by Mn in carlfrancisite. In addition, in mcgovernite, there are two tetrahedrally coordinated sites fully occupied by As<sup>5+</sup> and four tetrahedrally coordinated sites occupied by Si and As<sup>5+</sup> with Si dominant at all four sites; in carlfrancisite, there are two tetrahedrally coordinated sites occupied by Si and As<sup>5+</sup> with Si dominant at all four sites; in carlfrancisite, there are two tetrahedrally coordinated sites occupied by Si and As<sup>5+</sup> with Si dominant at only three of these four sites. Turtmannite is distinct from both carlfrancisite and mcgovernite by having essential tetrahedrally coordinated V<sup>5+</sup>.

\* Carlfrancisite:  $Mn_3^{++}(Mn^{2+},Mg)_{34}(Mg,Fe,Al)_8[(As^{3+}O_3),(Mn^{2+}O_6)]_2(As^{5+}O_4)_8[(Si,As^{5+})O_4]_6[(As^{5+},Si)O_4]_2(OH)_{42}; mcgovernite: (Zn,Mn^{2+})_3(Mn^{2+},Mg,Fe^{3+},Al)_{42}[(^{31}As^{3+},^{[51}Zn)O_3]_2(As^{5+}O_4)_4[(Si,As^{5+})O_4]_8(OH)_{42}; turtmannite: (Mn,Mg)_{45}Mg_{6(1-3)}(VO_4)_6(SiO_4)_6(AsO_3)_x O_{10(1-3)}(OH)_{40+x}.$ 

<sup>a</sup> This study.

<sup>b</sup> Cooper and Hawthorne (2001).

<sup>c</sup> Brugger et al. (2001).

et al. 2001), ideally (Mn,Mg)<sub>22.5</sub>Mg<sub>3-3x</sub>(VO<sub>4</sub>)<sub>3</sub>(SiO<sub>4</sub>)<sub>3</sub>(AsO<sub>3</sub>)<sub>x</sub>O<sub>5-5x</sub> (OH)<sub>20+x</sub>. All three structures have the same space group  $R\overline{3}c$  and similar unit-cell dimensions, and consist of close-packed and not-so-close-packed layers of octahedra and tetrahedra stacked along the **c** direction. Comparative data are given in Table 3. These structures are the most complicated of a family of minerals based on dominantly close-packed layers (Wuensch 1968), including arakiite (Cooper and Hawthorne 1999), dixenite (Araki and Moore 1981), franciscanite (Pertlik 1986), hematolite (Moore and Araki 1978), kraissilite (Moore and Ito 1978; Cooper and Hawthorne 2013), örebroite (Dunn et al. 1986), and welinite (Moore 1967).

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