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Franciscanite and örebroite, two new minerals from California and Sweden, related to redefined welinite

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ABSTRACT

Franciscanite and örebroite are new mineral species isotypic with welinite.

Franciscanite, ideally $Mn_6[V,\square]_2Si_2(O,OH)_{\sim 14}$, is hexagonal, has space group *P*3, with $a = 8.148(1)$ and $c = 4.804(1)$ Å, $V = 276.2$ Å^3, Z = 1. It is dark brownish red, with vitreous luster, and no cleavage; $D_{meas} = 4.1$ and $D_{calc} = 3.93$ g/cm^3. It is found with sonolite, hausmannite, and gageite in the Franciscan Complex at the Pennsylvania mine in the San Antonio Valley, Santa Clara County, California.

Örebroite, ideally $Mn_6[Sb^{5+},Fe^{3+}]_2Si_2(O,OH)_{\sim 14}$, is hexagonal, has space group *P*3, with $a = 8.183(7)$ and $c = 4.756(9)$ Å, $V = 275.8$ Å^3, Z = 1. It is dark brown with vitreous luster and no cleavage; $D_{calc} = 4.77$ g/cm^3. It is found associated with calcite at the Sjö mine, Örebro, Sweden.

New data for welinite indicate the revised formula $Mn_6[W,Mg]_2Si_2(O,OH)_{\sim 14}$ and space group *P*3. Gageite, associated with franciscanite, has the composition $Mn_{38.84}Mg_{0.58}Fe_{0.16}Si_{15}(O,OH)_{90}$, supporting the occurrence of octahedral vacancies in gageite and balangeroite.

INTRODUCTION

During the course of describing the paragenesis of santaclaraite (Erd and Ohashi, 1984), a rare mineral related to welinite was found and briefly described. Our subsequent investigation of this mineral has shown it to be the V analogue of welinite. We have named it *franciscanite* for the *Franciscan Complex* (Fox, 1983) in which it was found.

Our investigation of this phase led us to the observations by Moore (1967, 1972) of "so-called" welinite from the Sjö mine in Sweden; it was first found by Igelström, but a description was not published. Unlike the type welinite from Långban, the Sjö mine material had not been studied in detail; it was identified by Moore on the basis of optical examination and X-ray powder diffractometry. Dr. Bengt Lindqvist of the Naturhistoriska Riksmuseet in Stockholm generously provided this material for our study, and a detailed examination shows it to be a new mineral, wherein (Fe^{3+} + Sb^{5+}) substitutes for the (Mg + W) in welinite and for the V in franciscanite. We have named this mineral *örebroite* for the town of Örebro, near which the Sjö mine is located.

The two new minerals, their names, and the redefinition of welinite were approved by the Commission on New Minerals and Mineral Names, IMA. Type materials for franciscanite and örebroite are preserved in the Smithsonian Institution (catalogue no. 163041 and no. 163008, respectively). Additional type örebroite is in the Naturhistoriska Riksmuseet in Stockholm (no. RM 93:0255).

EXPERIMENTAL PROCEDURES

Crystals of franciscanite and type welinite were studied using Weissenberg and precession methods; however, örebroite occurs in fine-grained, polycrystalline intergrowths, intimately associated with calcite, and therefore no suitable single crystals were found. Unit-cell parameters for both franciscanite and örebroite were refined using powder data obtained on a 114.6-mm-diameter Gandolfi camera, polycrystalline sample, Si as an internal standard, and Fe*Kα* X-radiation. Such powder-diffraction patterns in conjunction with the chemical data showed that franciscanite and örebroite are isotypic with welinite.

The two new minerals and welinite were analyzed using an ARL-SEMQ electron microprobe, utilizing an operating current of 15 kV and a sample current of 0.025 μA, measured on brass. The data were corrected using a modified version of MAGIC-IV (Colby, 1971). The standards used were manganite (Mn), Sb_2O_3 (Sb), synthetic olivenite (As), synthetic V_2O_5 (V), scheelite (W), and hornblende (Si, Fe, Mg, Al). Because of the extreme paucity of material, we were unable to obtain definitive evidence for the

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Table 1. X-ray powder-diffraction data

Örebroite				Franciscanite			
I/I_0*	d_{obs}	d_{calc}	*hkl*	I/I_0*	d_{obs}	d_{calc}	*hkl*
1	7.01	7.09	100**	20	4.07	4.07	110**
30	4.08	4.09	110**	30	3.97	3.97	101**
20	3.94	3.95	101**	90	3.105	3.107	111
1	3.54	3.54	200**	90	2.844	2.844	201**
100	3.096	3.102	111**	40	2.668	2.667	210**
50	2.838	2.841	201**	10	2.404	2.402	002**
40	2.680	2.679	210**	100	2.331	2.332	211**
20	2.370	2.378	002	5	2.111	2.113	301**
		2.362	300	5	2.071	2.069	112**
70	2.334	2.334	211**	1	1.959	1.957	310**
10	2.116	2.116	301**	20	1.812	1.813	311**
2	2.056	2.056	112	70	1.785	1.785	212**
		2.046	220	15	1.680	1.681	302**
5	1.971	1.975	202	15	1.656	1.656	401**
		1.966	310	5	1.619	1.619	320**
10	1.818	1.817	311**	50	1.538	1.540	140
90	1.778	1.778	212			1.534	321
		1.772	400	2	1.490	1.490	113**
10	1.677	1.676	302**	2	1.465	1.466	411
2	1.663	1.660	401	30	1.373	1.373	123**
50	1.550			2	1.306	1.307	331
2	1.475			5	1.297	1.296	412
1	1.446			1	1.285	1.285	421
5	1.364			2	1.240	1.239	133
2	1.346			5	1.226	1.225	511
1	1.317			10	1.166		
5	1.300			2	1.139		
2	1.234			2	1.128		
5	1.1705			2	1.0501		
1	1.1505			5	1.0452		
5	1.1364			10	1.0225		
1	1.1251			10	0.9864		
5	1.0866						

* Intensities estimated visually.

** Reflections used in least-squares refinement.

oxidation state of Mn, Fe, V, W, or Sb; the valences were inferred as discussed in the text. The paucity of material also precluded a direct water determination for franciscanite. Thermal analyses were performed on type welinite and impure örebroite to determine water contents. A Mettler Thermoanalyzer–Inficon IQ200 mass spectrometer system was used. The welinite provided a reliable water value by TGA. However, the water content of örebroite could only be estimated by comparison of peak areas on evolved gas curves for örebroite and a brucite standard. Water contents of franciscanite and örebroite were therefore calculated, based on the determined Si:H ratio for type welinite.

FRANCISCANITE

Single-crystal X-ray diffraction photographs displayed apparent Laue symmetry $6/m$ and extinctions for $00l$ reflections as consistent with space groups $P6_3$ and $P6_3/m$. The former is compatible with the structure of welinite (Moore, 1968a), with which franciscanite is isotypic, as discussed below. However, very weak reflections were observed that violate the 6_3-axis extinction rule. The space group must therefore be a subgroup of $P6_3$. The only space groups consistent with the diffraction data and crystal structure (Moore, 1968a) are $P3$ and $P1$. Because the optical and other properties are consistent with trigonal symmetry, the space group is inferred to be $P3$, and the lattice parameters were refined accordingly; they are $a = 8.148(1)$ and $c = 4.804(1)$ Å, $V = 276.2$ Å^3; Z = 1. The powder-diffraction data are given in Table 1.

Table 2. Microprobe analyses

	Franciscanite	Örebroite	Welinite*
SiO_2	18.4	15.2	15.7
MnO	64.9	54.6	55.5
MgO	0.0	0.8	2.5
Fe_2O_3	0.0	9.2	0.8
Al_2O_3	0.0	0.3	0.0
WO_3	0.0	0.0	21.7
Sb_2O_5	0.0	15.8	1.6
As_2O_5	0.0	1.6	0.0
V_2O_5	13.5	0.0	0.0
H_2O	3.4**	2.8**	2.9†
Total	100.2	100.3	100.7
	Cations per 2 Si		
Si	2.00	2.00	2.00
Mn	5.96	6.09	5.99
Mg	0.00	0.16	0.47
Fe	0.00	0.91	0.07
Al	0.00	0.05	0.00
W	0.00	0.00	0.72
Sb	0.00	0.77	0.08
As	0.00	0.11	0.00
V	0.98	0.00	0.00
H	—	—	2.46

* Moore (1968a) gave Mn 40.1, Fe 0.6, Mg 1.7, Sb 0.7, Si 5.1, W 17.6, H_2O 3.2, with 7.6% Mn as Mn^{4+}. The equivalent metal oxides are MnO 42.0, MnO_2 9.3, Fe_2O_3 0.9, MgO 2.8, Sb_2O_5 0.9, WO_3 22.2, SiO_2 10.9 wt%.

** Water calculated from the Si:H ratio of type welinite.

† Water determined by TGA-EGA.

Franciscanite occurs as glassy, irregular grains, up to 1.0 mm in diameter. It has brownish-red color (cherry-red when fresh, darkening with time), brownish-red streak, vitreous luster, moderate transparency, no cleavage, an uneven fracture, and a Mohs' hardness of approximately 4. Franciscanite seems to be weakly magnetic, but some grains have extremely small opaque inclusions that may be responsible for this. The density, determined using heavy-liquid techniques, is 4.1 (meas.) as compared with 3.93 (calc.) g/cm^3. Franciscanite is uniaxial (+) with indices of refraction $\omega = 1.856(3)$ and $\epsilon = 1.882(3)$. Freshly collected material has marginally lower indices of refraction [$\omega = 1.859(3)$ and $\epsilon = 1.876(3)$]. Hence, the higher indices of the type material may reflect oxidation of Mn^{2+} to Mn^{3+} or Mn^{4+}. Franciscanite is strongly pleochroic, with O = wine red, and E = dark red to nearly black; absorption is intense, $E \gg O$.

Chemical analytical data are listed in Table 2. The empirical formula, calculated on the basis of Si = 2, is $Mn_{5.96}[V_{0.98}\square_{1.02}]_{\Sigma 2.00}Si_{2.00}(O,OH)_{\sim 14}$. Together with the crystallographic data, this composition indicates that franciscanite is the V analogue of welinite.

Franciscanite occurs as irregular, very sparse, segregations in a sheared sonolite-hausmannite-gageite assemblage in chert of the Franciscan Complex, on the southwest side of the San Antonio Valley, Santa Clara County, California. The type specimen was found on the dump of the Pennsylvania mine (Trask, 1950), an area now prohibited to mineral collecting. Franciscanite occurs in the chert together with the manganese ore mineral, braunite. This is also the type locality for santaclaraite (Erd and Ohashi, 1984), but franciscanite is not typically found with the santaclaraite-rich veins and masses. The mineral is rare at this, the type and only, locality.

The type specimen consists of a 1.0-mm grain of franciscanite within sonolite in a sheared assemblage of sonolite, hausmannite, and a caryopilite-like mineral. This assemblage is crosscut by a 0.5-mm veinlet of fibrous gageite, somewhat pale violet in color, which is younger than the host assemblage.

Inasmuch as this is only the second published description of gageite, we provide additional data. Microprobe analysis, using the same standards as for the welinite-group minerals, yielded SiO_2 22.8, FeO 3.3, MgO 0.6, MnO 69.7, with H_2O (by difference) 6.6 wt%. This water value may be low owing to the fibrous nature of the aggregate or may reflect a variable water content for gageite, as may exist in balangeroite, the Mg analogue (Compagnoni et al., 1983). Calculation of a formula, on the basis of Si = 15, yields $[Mn_{38.84}Mg_{0.58}Fe_{0.16}\square_{2.42}]_{\Sigma 42}Si_{15.00}(O,OH)_{90}$. The low value for the sum of octahedral cations (39.58 of the theoretical 42.00) (Compagnoni et al., 1983) is expected. Dunn (1979) found 39.87 M^{2+} cations per 15 Si in the average of 7 analyses of Franklin gageite, and Compagnoni et al. (1983) found 38.93 octahedral cations in balangeroite, the Mg-dominant analogue. Additional study of these phases, particularly with respect to vacancies, is clearly needed, but this California material (in warped fibers) is less well-suited to crystallographic study than material from the other localities. Additionally, we mention for the record that at least five occurrences of gageite have been found in Japan (S. Matsubara and A. Kato, pers. comm.). In our discussion of this California material, we have used the name "gageite" in a general sense.

ÖREBROITE

Örebroite is hexagonal, space group *P*3, with unit-cell parameters $a = 8.183(7)$ and $c = 4.756(9)$ Å, $V = 275.8(0.4)$ Å^3, and Z = 1. The space group is assumed to be *P*3 or *P*1 by analogy with franciscanite and welinite. Although the powder-diffraction data (Table 2) are consistent with this assignment, it cannot be taken as definitive because of the lack of single-crystal diffraction data.

Örebroite is massive and polycrystalline. It has dark-brown color, reddish-brown streak, vitreous luster, no cleavage, an irregular fracture, and Mohs' hardness estimated to be approximately 4. Örebroite is nearly opaque. Like franciscanite, örebroite seems to be weakly magnetic, but the magnetism is probably due to inclusions of magnetite, because there is no reasonable source of this property in the chemistry of örebroite. The density could not be measured owing to the intimately associated calcite; $D_{calc} = 4.77$ g/cm^3. Optically, örebroite is uniaxial (+) with indices of refraction $\omega = 1.857(3)$ and $\epsilon = 1.875(3)$. These values compare well with those of Moore (1972) ($\omega = 1.85$, $\epsilon = 1.87$). Pleochroism is imperceptible.

A microprobe analysis is presented in Table 2. TGA-EGA of a 10-mg sample in vacuum showed a complex loss involving H_2O, CO_2 (from the calcite impurity), and volatile(s) undetected by the mass spectrometer. The total loss to 900°C was 6.4 wt%. A single water peak at 570°C strongly suggests that H is present as hydroxyl in örebroite. An estimated water content of 3 wt%, derived from EGA data, compares well with the calculated value of 2.8 wt% given in Table 2. A thermal event characterized by a minor burst of H_2O occurred at 320°C. Calculation of a formula, based on Si = 2, yields $Mn_{6.09}[Fe^{3+}_{0.91}Sb_{0.77}As_{0.11}Mg_{0.16}Al_{0.05}]_{\Sigma 2.00}Si_{2.00}(O, OH)_{\sim 14}$. There is, however, a possibility that Mg occupies the site of rank 6, with Mn^{2+}, and that some Mn^{3+} or Mn^{4+} is present. These data, together with the crystallographic data, indicate that örebroite is a Fe^{3+}-Sb analogue of welinite and franciscanite. There may be a complex charge-balance effect with Fe^{3+} and (Sb + As) occurring in near-equal proportions and giving a net 4^+ charge to the site. An analogous substitution occurs in melanostibite, which also occurs at the Sjö mine. Moore (1968b) showed that Sb^{5+} and Fe^{3+} substitute for Ti^{4+} in the ilmenite structure to give the formula $Mn^{2+}(Fe^{3+},Sb^{5+})O_3$. Because As^{5+} does not ordinarily have octahedral coordination, its inclusion in the substitution scheme in örebroite is problematical. Assuming that örebroite has the welinite structure (Moore, 1968a), the small amount of As^{5+} either has that coordination, or it occurs in domains with variation in coordination.

Örebroite was found among Igelström's Sjö mine antimonates and was known to him as "retinostibian." Moore (1972) noted that it is associated with hausmannite and dolomite. The material described here as örebroite is intimately associated with fine-grained calcite, the two minerals forming a compact, fine-grained aggregate. It is known only from the Sjö mine, near Örebro, Sweden.

WELINITE

As part of our study of the two new minerals, it was necessary to re-examine welinite type material, for which the space group had been reported as $P6_3$ by Moore (1967, 1968a). We studied type welinite using Weissenberg and precession methods and found it to have space group *P*3 due to the presence of very weak 00*l* reflections having $l = 2N + 1$ and as compatible with the structure of Moore (1968a). These results are identical with those for franciscanite. Because of very unusual apparent chemistry of welinite (Mn, W, and vacancies existing on two different sites) and because the chemistry resulted from a microprobe analysis done when ZAF corrections were only approximate (Moore, 1967), we re-analyzed the type material. TGA analysis of a 2-mg-sample in N showed a loss of 2.9 ± 0.5 wt% between 550–750°C, with a maximum rate of loss near 670°C. Evolved gas analysis of a 180-μg sample in vacuum confirmed an H_2O peak at approximately 625°C. The loss is therefore ascribed to H_2O, and the high release temperature suggests the presence of hydroxyl, as in örebroite. The resultant analysis is given in Table 2, together with Moore's original analysis. There is good agreement between the two results. Our analysis, when calculated on the basis of Si = 2, yields $Mn_{5.99}[W_{0.72}Sb_{0.08}Fe_{0.07}Mg_{0.47}\square_{0.66}]_{\Sigma 2.00}Si_{2.00}(O,OH)_{\sim 14}$.

DISCUSSION

Moore (1968a) determined the crystal structure of welinite, gave the unit-cell formula as $[Mn_{1.6}W_{0.3}\square_{0.1}]$ $[Mn_{4.0}Mg_{0.5}W_{0.5}\square_{1.0}]_{\Sigma 6}Si_{2.0}(O_{14-x}OH_{x})_{\Sigma 14}$, and proposed that the endmember formula is $Mn^{4+}Mn^{2+}Si_2O_7$. The first two positions of the formula correspond to octahedrally coordinated cations and vacancies on sites of rank 2 and 6 with Mn^{4+} ordered at M(1) and Mn^{2+} at M(2). Moore further pointed out that of the three anions, O(1) and O(2) are oxygen atoms, each coordinated to Si and 3 M(2); if M(2) has charge +2, there is therefore exact charge balance for O(1) and O(2).

The M(1) cations play an unusual role in the structure in that each octahedron shares faces with adjacent M(1) octahedra to form an infinite column parallel to **c**. Such a column of Al octahedra is also found in the structure of dumortierite (Moore and Araki, 1978), although the octahedra are incompletely occupied. In welinite the O(3) anion is coordinated to two such octahedrally coordinated M(1) cations plus two M(2) cations. The charge balance at O(3) thus depends on the occupancy of the M(1) sites; local cation vacancies and/or the presence of cations of low charge require that O(3) be OH; the presence of cations of high charge requires O at O(3).

The rather simple chemistry of franciscanite provides a key to understanding cation-ordering schemes in all three minerals (franciscanite, örebroite, and welinite). Table 2 shows that, within error, there are 6 Mn^{2+} and 1 V per cell. Placing all Mn^{2+} on M(2) results in exact charge balance for O(1) and O(2). Assuming that all V occupies M(1), exactly half of the face-sharing octahedra are occupied, and assuming that occupied and vacant octahedra are ordered, face-sharing of octahedra with cations of high valence is avoided. Lastly, such a scheme is consistent with space group $P3$ rather than $P6_3$; in the latter, all M(1) sites are equivalent. Assuming that such ordering causes M(1) to be divided into two sites, each of rank one, causing the space group to be $P3$, the major elements of the structure, which are well defined, are retained with only the minor modification implied by the very weak $00l$ reflections having $l = 2N + 1$. Dr. F. Pertlik (pers. comm.) has refined the structure of franciscanite and has verified that this model is correct.

Both örebroite and welinite have 6 Mn^{2+}, and we infer that M(2) is principally occupied by Mn^{2+} in both structures. There is minor Mg in both, and this may occupy M(1) in part, as consistent with local charge-balance considerations for M(1) and M(2).

In örebroite there are nearly equal numbers of Fe (0.91) and Sb + As (0.88) per cell. We infer that these cations occupy alternate M(1) sites analogous to the possible (V,$\square$) ordering in franciscanite. Such a scheme results in charge balance for O(3) if that anion is O^{-2}; (OH) substitution would occur in proportion to the occurrence of +2 cations or vacancies on M(1). Such substitutions are minimal in type örebroite.

Lastly, we infer that in welinite the M(2) site is occupied by Mn^{2+}, probably with some Mg in solid solution. Such a scheme implies that all W occupies M(1), and we suggest that the 0.72 W may be distributed over the site so that the 0.66 vacancies occur in face-sharing octahedra adjacent to W. Moore (1968a) proposed that some Mn^{4+} occupies this site; if so, this implies that some Mg occupies M(1), in that there are 5.99 Mn and 0.47 Mg per cell.

The results of the refinement of the welinite structure (Moore, 1968a) are consistent with these site occupancies. For example, the average M(2)–O distance (2.21 Å) is in excellent agreement with other Mn^{2+}–O distances and is compatible with occupancy of that site only by Mn^{2+}. In addition, the site occupancies are calculated to correspond to 143 and 150 electrons for Moore's and the above occupancies, respectively, on the basis of neutral atoms. Given the errors or ambiguities inherent in such calculations and in the refined occupancies, the agreement is reasonable. Furthermore, as Moore (1968a) pointed out, the M(1)–O average distance (1.96 Å) is compatible with octahedrally coordinated W^{6+}. Calculations of the numbers of electrons for the M(1) site for Moore's occupancies $(Mn_{1.6}W_{0.3}\square_{0.1})$ and those of this paper $(W_{0.72}Sb_{0.08}Fe_{0.07}Mg_{0.47}\square_{0.66})$ give 62 and 52 electrons respectively, in good agreement. Especially insofar as the *R* factor of the crystal-structure determination was high (10.6%) and because the space group used ($P6_3$) is not valid for the full detailed structure, these data imply that W preferentially occupies M(1) and that M(2) is at least dominantly occupied by Mn^{2+}.

There is ambiguity in the valences of V in franciscanite, Mn in welinite, and Fe and Sb in örebroite. The formal cation charges determine, in each case, the charge balance for O(3), which may be O or OH. Because accurate water determinations cannot be made for franciscanite and örebroite, valences cannot be directly inferred. Lastly, we note that short-range ordering in the M(1) site as proposed for all three phases need not result in violations of extinction rules for space group $P6_3$. Reversals in the ordering sequence may occur within a given column or between columns, giving rise to apparent long-range disorder. Such relations may be reflected in TEM studies by antiphase domains. It is also possible that domains associated with different cations on M(1) and anions on M(3) have locally different structures and symmetries. Clearly, the data presented in this paper suggest that much work, especially with respect to structure refinements and TEM observations, remains to be carried out on these phases in order to define the detailed crystal-chemical relations.

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