

Lindbergite, a new Mn oxalate dihydrate from Boca Rica mine, Galiléia, Minas Gerais, Brazil, and other occurrences

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ABSTRACT

The new mineral lindbergite, $\text{Mn}(\text{C}_2\text{O}_4)\cdot 2\text{H}_2\text{O}$, is a secondary mineral in the Lavra da Boca Rica granite pegmatite, Sapucaia do Norte, Galiléia Co., Minas Gerais, Brazil. It occurs as: (1) white short prismatic crystals 0.1 to 0.3 mm in length, interpreted as pseudomorphs after the orthorhombic trihydrate, with faces of $\{100\}$, $\{hk0\}$, $\{010\}$, and $\{0kl\}$ forms, rounded edges, and twinning on (010); and (2) grayish-white aggregates in 0.1 mm thick translucent crusts made up of interlocking irregularly contoured platelets up to 0.03 mm in length. Lindbergite is transparent, has a white streak, vitreous luster, a perfect cleavage parallel $\{010\}$, and is non-fluorescent. The Mohs' hardness is $2\frac{1}{2}$ and its tenacity crumbly. The measured density is $2.10(3) \text{ g/cm}^3$ (calculated 2.251 g/cm^3). Lindbergite is biaxial negative, with $n_\alpha = 1.424(3)$, $n_\beta = 1.550(3)$, $n_\gamma = 1.65(1)$ (white light), $2V$ (obs.) = $80(2)^\circ$, $2V$ (calc.) = 77° . Dispersion is not detectable or very weak with $r > v$. Orientation $Y \wedge c = 20^\circ$. The empirical composition is $(\text{Mn}_{1.11}\text{Na}_{0.01}\text{Al}_{0.01})_{\Sigma 1.13}(\text{C}_{1.94}\text{O}_4)\cdot 2.15\text{H}_2\text{O}$. The mineral is monoclinic, $C2/c$, $a = 11.995(5)$, $b = 5.632(2)$, $c = 9.967(7) \text{ \AA}$, $\beta = 128.34(4)^\circ$, $V = 528.1(5) \text{ \AA}^3$, $Z = 4$. Lindbergite is the Mn analogue of humboldtine and glushinskite. Associated minerals at the type locality are the phosphates triphylite, phosphosiderite, frondelite, strengite, cyrilovite, bermanite, rockbridgeite, huréaultite, tavorite, reddingite, heterosite, laueite, and unidentified minerals. Additional occurrences of lindbergite are from Parsettens, Grisons, Switzerland, the Lecht Mines, Banffshire, Scotland, and the Morefield Pegmatite, Amelia, Virginia. Lindbergite also has been recorded from three localities in the Black Forest, Germany: Clara Mine, Oberwolfach, Wolfach Valley, Baden-Württemberg; Ortenberg near Offenburg; and Gremmelsbach near Triberg, but the natural origin was not firmly established for the last one. An occurrence of orthorhombic $\text{Mn}(\text{C}_2\text{O}_4)\cdot 3\text{H}_2\text{O}$ has been reported at Falotta, Grisons, Switzerland.

INTRODUCTION

Monoclinic $\text{Mn}(\text{C}_2\text{O}_4)\cdot 2\text{H}_2\text{O}$ has been defined as the new mineral lindbergite. Both the description and name have been approved by the CNMMN–IMA (Nomenclature Proposal 2003-029). The first lindbergite samples were obtained from the Boca Rica mine, Sapucaia do Norte, Galiléia Co. ($19^\circ 0' \text{S}$ $41^\circ 32' \text{W}$, DMS), Minas Gerais, Brazil, in 2002 by one of us (LADMF). Additional samples were collected by DA and PAM, and obtained through the mineral dealer Ramiro Sardinha in Governador Valadares, Minas Gerais. The mineral is named lindbergite in honor of Marie Louise Lindberg (-Smith) (born 1918), from the USGS, who described several new mineral species from the nearby Sapucaia quarry (frondelite, faheyite, moraesite, barbosalite, and tavorite). The Boca Rica mine is taken as the type locality for lindbergite. Type material (number DR542) is deposited in the Museu de Geociências, Instituto de Geociências, Universidade de São Paulo, Rua do Lago, 562, 05508-080, São Paulo, SP, Brazil and in the Museu Jobas de Ciências Naturais “José Bonifácio de Andrada e Silva,” Rua Martim Afonso, 24, Biquinha, 11310-010, São Vicente, SP, Brazil.

Lindbergite also has been recorded at the Lecht Mines, Banff-

shire, Scotland (Wilson and Jones 1984), and at three localities in the Black Forest, Germany: Gremmelsbach near Triberg (Walenta 1989, 1992, 2003), Clara Mine, Oberwolfach, Wolfach Valley Baden-Württemberg (Blass and Graf 2000) and Ortenberg near Offenburg (Walenta 2003). The natural origin was not firmly established for the first German occurrence. The Gremmelsbach Mn oxalate dihydrate may have formed because someone used oxalic acid to clean a specimen. The oxalic acid is used by collectors to remove Fe oxides (rust stains) from specimens and may react with the other components present to form oxalates. Earlier, it was suspected that the use of concrete in underground workings played a role in the genesis of the Clara material, but there is no way of forming oxalic acid from concrete constituents. Essentially, this would necessitate carbonate reduction, which is chemically impossible. Oxalic acid originates from plants, where its derivatives oxaloacetate and oxalosuccinate are important intermediates in the citric acid cycle. Lindbergite from the Clara mine was found while cracking open a fresh boulder at the dumps, and the white prismatic crystals (pseudomorphs in fact) were immediately recognizable. The mineral also was identified at the Morefield Pegmatite, Amelia, Virginia (Lance E. Kearns, personal communication, 2003).

In 1977, one of us (S.G.) recorded the presence of excel-

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lent, completely colorless transparent crystals of a conspicuous “ship-like” morphology, at an abandoned Mn mine at Falotta, Oberhalbstein, Grisons, Switzerland. These crystals were identified by single-crystal analysis and Gandolfi films as orthorhombic $\text{Mn}(\text{C}_2\text{O}_4) \cdot 3\text{H}_2\text{O}$, identical with the synthetic compound quoted in the ICDD card 32-648. From EDS, it was learned that it contained Mn and C. When performing the single-crystal study, the crystal began to lose its transparency and turned milky white, while retaining its morphology. Using Debye-Scherrer films, this new material was identified as monoclinic $\text{Mn}(\text{C}_2\text{O}_4) \cdot 2\text{H}_2\text{O}$, as recorded in the ICDD card 25-544, equivalent to lindbergite. All the remaining trihydrate also changed to the dihydrate, and no additional material could be found. Lindbergite was found in nature as pseudomorphs after the trihydrate by SG in 1981 in an abandoned Mn mine at Parsettens, Oberhalbstein, Switzerland.

OCCURRENCE

Lindbergite occurs as a secondary mineral in the Lavra (= mine) da Boca Rica (a granite pegmatite), Sapucaia do Norte, Galiléia Co., Minas Gerais, Brazil (Fig. 1). It is associated with the phosphates triphylite, phosphosiderite, frondelite, strengite, cyrilovite, bermanite, rockbridgeite, huréaulite, tavorite, red-dingite, heterosite, laueite, and unidentified minerals.

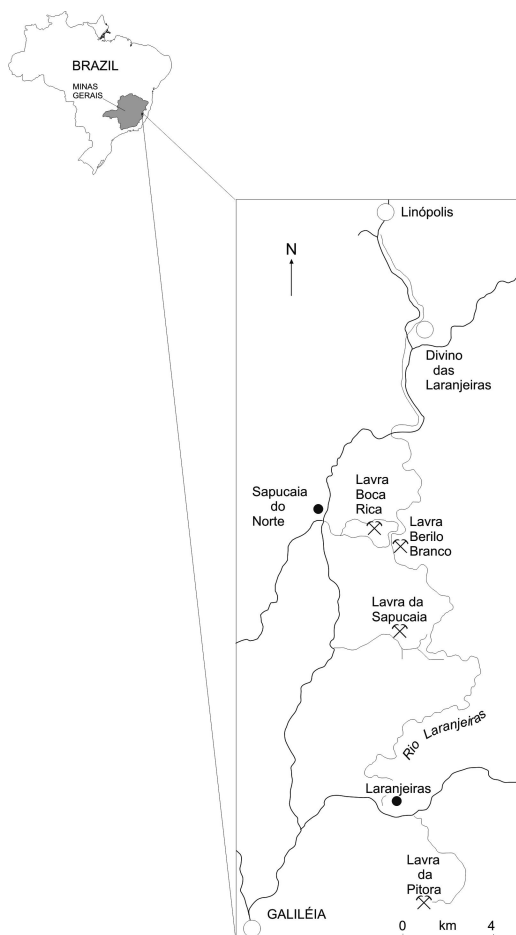


FIGURE 1. Location map showing the Boca Rica mine (Wilson 1999).

HABIT AND PHYSICAL PROPERTIES

Lindbergite from Boca Rica mine display two habits: (1) short prismatic crystals 0.1 to 0.3 mm in length with faces of $\{100\}$, $\{hk0\}$, $\{010\}$, and $\{0kl\}$ forms, rounded edges, and twinning on (010). This habit is interpreted as a pseudomorphous after the trihydrate. The “crystals” are actually groups of smaller crystals, some of which present lozenge-shaped section; and (2) aggregates in 0.1 mm thick translucent crusts consisting of interlocked irregularly contoured platelets up to 0.03 mm in length (Fig. 2). In this case, the mineral should have formed originally as dihydrate. The color is white (crystals) or greyish white (aggregates). Its streak is white and the luster is vitreous. Lindbergite is transparent and non-fluorescent; its Mohs’ hardness is $2\frac{1}{2}$ and cleavage along $\{010\}$ is perfect. Parting was not observed. Tenacity is crumbly. Density (obs.) is $2.10(3) \text{ g/cm}^3$; density (calc.) is 2.356 g/cm^3 (2.251 g/cm^3 for the ideal formula).

Optical data (Table 1) for lindbergite from Boca Rica mine were determined in white light. Indices of refraction were measured by immersion of grains in liquids calibrated with an Abbé refractometer. Lindbergite is biaxial negative.

MINERAL CHEMISTRY

Chemical analyses ($n = 10$) were carried out at the Laboratório de Microscopia Eletrônica do Departamento de Geologia Sedimentar e Ambiental do Instituto de Geociências da Universidade de São Paulo using an EDS system equipped with Si(Li) detector, 15 kV accelerating potential, 8.5 nA beam current, and a $5 \mu\text{m}$

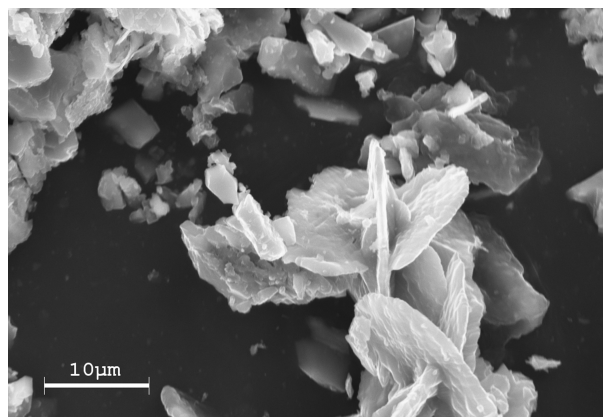


FIGURE 2. Secondary electron image of interlocked irregularly contoured platelets of lindbergite. Specimen from Boca Rica mine.

TABLE 1. Optical properties of lindbergite

	1	2
n_a	1.424(3)	1.434(3)
n_b	1.550(3)	
n_c	1.65(1)	1.662(3)
birefringence	0.226	0.228
$2V_{\text{obs}}$	$80(2)^\circ$	
$2V_{\text{calc}}$	77°	
dispersion	not detectable	
	or $r > v$ very weak	
Orientation	$Y \wedge c = 20^\circ$	
Pleochroism	none	

Notes: 1 = Boca Rica mine, Sapucaia do Norte, Galiléia, Minas Gerais, Brazil (this study). 2 = Ortenberg near Offenburg, Black Forest, Germany (Walenta 2003).

beam diameter, with Mn-metal, albite, and corundum as probe standards. C₂O₃ and H₂O were determined at the Laboratório de Química e ICP do Instituto de Geociências, Universidade de São Paulo by CHN analysis on 0.5 mg handpicked material. The mean analytical results are represented in Table 2. The low total is likely the result of impurities (mainly phosphosiderite) in the handpicked material for the CHN analysis, and EDS data uncorrected for C₂O₃ and H₂O matrix effects. The empirical formula (based on four oxalate O atoms) is (Mn_{1.11}Na_{0.01}Al_{0.01})_{Σ1.13}(C_{1.94}O₄)·2.15H₂O.

CRYSTALLOGRAPHIC DATA

The powder data for lindbergite from the Boca Rica mine were obtained at the Laboratório de Difração de Raios X do Departamento de Mineralogia e Geotectônica do Instituto de Geociências da Universidade de São Paulo on a Siemens D5000 diffractometer equipped with a Göbel mirror and a position-sensitive detector, using CuKα radiation at 40 kV and 40 μA. The X-ray diffraction (XRD) pattern for lindbergite from Parsettens, Oberhalbstein, Switzerland, was obtained in a Debye-Scherrer camera (90 mm diameter, FeKα radiation). Intensities were estimated visually. The powder XRD pattern

for lindbergite (Table 3) was indexed by analogy with that for synthetic Mn(C₂O₄)·2H₂O (ICDD card 25-544). Lindbergite is thus monoclinic, space group C2/c, Z = 4. Unit-cell parameters (Table 4) were refined from the powder data. The a:b:c ratio calculated from the unit-cell parameters is 2.1298:1:1.7697 for the Boca Rica lindbergite and 2.1322:1:1.7749 for the Parsettens lindbergite. Gladstone-Dale relationship calculations for the Boca Rica lindbergite yielded 1 - (K_p/K_c) = 0.089 (poor compatibility) using the empirical formula (see remarks under chemical data about the analysis quality), and 0.053 (good compatibility) using the ideal formula. Lindbergite is the Mn analogue of humboldtine (Fe) and glushinskite (Mg) (Table 5). The powder XRD data are practically identical with those of synthetic Mn(C₂O₄)·2H₂O. The crystal structure of the synthetic analogue was studied by Deyrieux et al. (1973).

THE TRIHYDRATE ORTHORHOMBIC, Mn(C₂O₄)·3H₂O

A natural occurrence of Mn oxalate trihydrate has been recorded from Falotta, Oberhalbstein, Grisons, Switzerland. The mineral occurs on small quartz crystals, associated with braunite and other Mn minerals. Presumably, the mineral formed by a reaction of humic acids with the primary Mn ore.

TABLE 2. Chemical composition of lindbergite (wt%)

	1	2	2 (range)
MnO	39.63	39.99	(37.46-42.38)
Al ₂ O ₃		0.24	(b.d-2.06)
Na ₂ O		0.21	(b.d.-0.46)
C ₂ O ₃	40.24	34.90	
H ₂ O	20.13	19.62	
Total	100.00	94.96	

Notes: 1 = Ideal formula, Mn(C₂O₄)·2H₂O. 2 = Boca Rica mine. b.d.: below detection (detection limit is probably 0.05 to 0.1 wt%).

TABLE 4. Cell parameters of lindbergite

	1	2	3
a (Å)	11.995(5)	12.032(8)	12.015(3)
b (Å)	5.632(2)	5.643(7)	5.6323(4)
c (Å)	9.967(7)	10.016(7)	9.961(2)
β (°)	128.34(4)	128.40(5)	128.37(7)
V(Å ³)	528.1(5)	532.9(3)	528.492(2)

Notes: 1 = Boca Rica mine, Sapucaia do Norte, Galiléia, Minas Gerais, Brazil (this study). 2 = Parsettens, Oberhalbstein, Switzerland (this study). 3 = Synthetic (Deyrieux et al. 1973; ICDD card 25-544).

TABLE 3. Powder X-ray diffraction data for lindbergite

hkl	1			2		3		4	
	d _{calc.} (Å)	d _{obs.} (Å)	I _{obs.}	d _{obs.} (Å)	I _{obs.}	d _{obs.} (Å)	I _{obs.}	d _{obs.} (Å)	I _{obs.}
1 1 1	4.861	4.855	26					4.861	14
2 0 2	4.813	4.803	100	4.839	100	4.87	100	4.814	100
2 0 0	4.704	4.699	84	4.715	70	4.73	100	4.714	95
0 0 2	3.909	3.907	23	3.925	40	3.91	50	3.903	30
1 1 2	3.663							3.662	11
1 1 1	3.626	3.622	22	3.639	50	3.63	40	3.627	12
3 1 2	3.203					3.20	5	3.207	2
3 1 1	3.179							3.184	7
4 0 2	2.998	2.996	58	3.007	80	2.99	90	3.003	70
0 2 0	2.816	2.816	4			2.79	5	2.818	13
1 1 3	2.678	2.679	14	2.682	60	2.66	80	2.676	35
1 1 2	2.654	2.652	12					2.653	60
2 2 1	2.549							2.551	2
2 2 2	2.431					2.44	10	2.431	6
2 0 2	2.370	2.369	4			2.36	5	2.370	3
4 0 0	2.352							2.354	1
0 2 2	2.285	2.286	10	2.291	30	2.28	30	2.284	25
3 1 1	2.235	2.235	5					2.236	6
5 1 3	2.192	2.191	7					2.194	4
5 1 2	2.178							2.182	1
2 2 3	2.145	2.146	5			2.13	10	2.145	10
2 2 1	2.125							2.126	10
4 2 2	2.053							2.054	7
1 1 3	2.038			2.046	10	2.02	10	2.037	18
5 1 1	2.016							2.020	9
4 2 3	1.993							1.993	4
4 2 1	1.977							1.979	3
6 0 4	1.947			1.949	10	1.946	5	1.952	17
6 0 2	1.925	1.927	5					1.929	12
0 2 3	1.913	1.914	5	1.919	30	1.914	40	1.919	16
3 1 5	1.859							1.858	4
1 3 1	1.843	1.842	6	1.846	20	1.836	40	1.843	30
4 2 4	1.829							1.829	18

Notes: 1 = Boca Rica mine, Sapucaia do Norte, Galiléia, Minas Gerais, Brazil (this study). 2 = Parsettens, Oberhalbstein, Switzerland (this study). 3 = Ortenberg near Offenburger, Black Forest, Germany (Walenta 2003; includes the additional lines 1.505 and 1.464Å, both with I = 5). 4 = Synthetic (Deyrieux et al. 1973; ICDD card 25-544; includes 11 additional lines to 1.515 Å).

TABLE 5. Comparative data for lindbergite, humboldtine and glushinskite

	Lindbergite Boca Rica mine (this study)	Synthetic Deyrieux et al. (1973)	Humboldtine Caric (1959)	Glushinkite Wilson et al. (1980)
chemical formula	Mn(C ₂ O ₄)·2H ₂ O	Mn(C ₂ O ₄)·2H ₂ O	Fe(C ₂ O ₄)·2H ₂ O	Mg(C ₂ O ₄)·2H ₂ O
crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
space group	C2/c	C2/c	C2/c	C2/c
unit-cell parameters	<i>a</i> = 11.995(5) Å <i>b</i> = 5.632(2) Å <i>c</i> = 9.967(7) Å β = 128.34(4)°	<i>a</i> = 12.015(3) Å <i>b</i> = 5.632(4) Å <i>c</i> = 9.961(2) Å β = 128.37(7)°	<i>a</i> = 12.060 Å <i>b</i> = 5.550 Å <i>c</i> = 9.804 Å β = 127.97°	<i>a</i> = 12.675 Å <i>b</i> = 5.406 Å <i>c</i> = 9.984 Å β = 129.45°
strongest powder pattern lines [<i>d</i> (Å) – <i>l</i>]	4.855–26 4.803–100 4.699–84 3.907–23 2.996–58	4.814–100 4.714–95 3.000–70 2.676–35 2.653–60	4.80–100 4.70–65 3.88–25 3.004–50 2.654–30	4.890–100 3.168–80 2.379–60 2.036–80 1.863–60
optical data	<i>n_a</i> = 1.424(3) <i>n_b</i> = 1.550(3) <i>n_c</i> = 1.65(1) 2 <i>V</i> = 80°	Not reported	<i>n_a</i> = 1.494 <i>n_b</i> = 1.561 <i>n_c</i> = 1.692 2 <i>V</i> = 70°	<i>n_a</i> = 1.365 <i>n_b</i> = 1.530 <i>n_c</i> = 1.595 2 <i>V</i> = 58°

The crystal was completely transparent and colorless. The synthetic analogue obtained by Huizing et al. (1977) was pink. Figure 3 shows a photograph of the altered crystal. This milky material is dihydrate, but formed by water loss while retaining the trihydrate orthorhombic shape. Figure 4 shows a crystal drawing (SHAPE) of the trihydrate material from Falotta, based on the measurement on an optical goniometer. Based on the orientation of the unit-cell in the ICDD card 32-648, the pointed ends mark the *c*-axis.

Under the polarizing microscope, the mineral appears colorless and shows strong birefringence. The refractive indices of two directions were determined for Na_D light: a higher value along the *c*-axis [1.560(3)] and a lower one along the *a*-axis [1.460(3)]. Qualitative EDS analysis data showed the presence of Mn and C. XRD data for the natural trihydrate were obtained with a Gandolfi camera (114.6 mm, FeK α). Intensities were estimated visually. The results are compared with those for the synthetic analogue in Table 6. The unit cell is orthorhombic, space group *Pcca*. Cell parameters are represented in Table 7.

SYNTHESIS AND STABILITY

Based on synthesis experiments, Huizing et al. (1977) studied the stability of the Mn oxalate trihydrate and the two dihydrates, α (equivalent to lindbergite, monoclinic) and γ (orthorhombic) (Fig. 5). According to these experimental data, the less-stable Mn oxalate is the trihydrate. Especially when it is not completely dry, conversion into the α -dihydrate occurs within a few days. The α -dihydrate is stable in air for unlimited periods, provided the material is thoroughly dried. The γ -dihydrate is completely stable and drying is not necessary.

Oxalate occurrences have been described in other igneous rocks. Humboldtine, the Fe analogue of lindbergite, was quoted in pegmatite fractures at Santa Maria de Itabira, Minas Gerais, Brazil (Matioli et al. 1997) and in veins in a rhyolite from Hartkoppe Quarry, Bavaria, Germany (Lorenz 1995). Natroxalate was described from the Shomiokitovoye agpaitic pegmatite, at the northern flank of the Umbozero mine, Lovozero, Kola Peninsula, crystallized from hydrothermal hyperalkaline oxalate-carbonate solution (Khomyakov 1996).



FIGURE 3. Monoclinic dihydrate—Mn(C₂O₄)·2H₂O—pseudomorph after orthorhombic trihydrate Mn(C₂O₄)·3H₂O. Crystal size is about 1 × 0.5 mm and the crystallographic orientation is identical with that in the drawing of Figure 4. Specimen from Falotta, Switzerland.

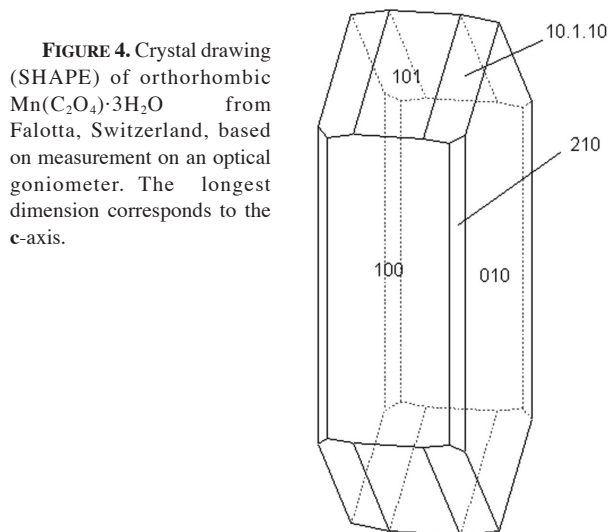


FIGURE 4. Crystal drawing (SHAPE) of orthorhombic Mn(C₂O₄)·3H₂O from Falotta, Switzerland, based on measurement on an optical goniometer. The longest dimension corresponds to the *c*-axis.

TABLE 6. X-ray diffraction data for MnC₂O₄·3H₂O

hkl	1			2	
	d _{calc} (Å)	d _{obs} (Å)	I _{obs}	d _{obs} (Å)	I _{obs}
010	6.626	6.630	100	6.64	100
111	4.865	4.860	10	4.87	<1
201	4.635	4.635	60	4.63	3
210	4.121	4.124	50	4.12	9
012	3.935	3.935	60	3.93	2
211	3.798	3.801	90	3.80	10
202	3.583	3.585	30	3.58	1
020				3.31	<1
212	3.152	3.153	80	3.15	3
121				3.00	1
311	2.956	2.959	60	2.953	4
113	2.819	2.819	50	2.815	1
220				2.798	2
022				2.738	<1
221	2.695	2.697	60	2.693	8
312	2.619	2.622	70	2.616	3
213	2.557	2.558	20	2.552	1
222	2.433	2.439	30	2.444	2
321	2.339	2.333	30	2.337	1
402				2.315	1
123	2.269	2.268	20		
030	2.209	2.208	30	2.206	3
412				2.186	<1
322	2.161	2.159	40	2.158	1
223	2.126	2.122	60	2.124	2
420	2.061	2.059	10	2.059	1
230				2.033	<1
032	2.013	2.012	40	2.011	3
231	1.994	1.992	20	1.991	3
511	1.965	1.960	30	1.965	1
422	1.899	1.898	50	1.898	3
205				1.831	<1
215				1.765	<1
233	1.727	1.726	70	1.726	3
610				1.695	1
430	1.692	1.689	30	1.690	2
611				1.670	1
431	1.667	1.667	40	1.666	1
315				1.653	1
034				1.636	<1
504	1.596	1.596	20	1.597	1
240	1.580	1.579	10	1.578	1
234	1.565	1.564	20	1.567	1
241				1.558	1
620	1.550	1.548	30		
306	1.479	1.479	20		
342	1.432	1.432	20		
425	1.419	1.419	10		
440	1.402	1.401	20		
720	1.369	1.369	20		
525	1.315	1.315	10		
		1.278	50		
		1.213	10		
		1.195	30		
		1.128	20		
		1.122	30		
		1.073	40		
		1.064	30		
		0.990	50		
		0.978	30		

Notes: 1 = Falotta, Oberhalbstein, Grisons, Switzerland. (this study). 2 = Synthetic (Huizing et al. 1977, ICDD card 32-648).

TABLE 7. Cell parameters for MnC₂O₄·3H₂O.

	1	2
a(Å)	10.527(5)	10.524(1)
b(Å)	6.626(2)	6.614(1)
c(Å)	9.783(6)	9.769(1)
V(Å ³)	682.38	679.98

Notes: 1 = Falotta, Oberhalbstein, Grisons, Switzerland. 2 = Synthetic (Huizing et al. 1977, ICDD card 32-648).

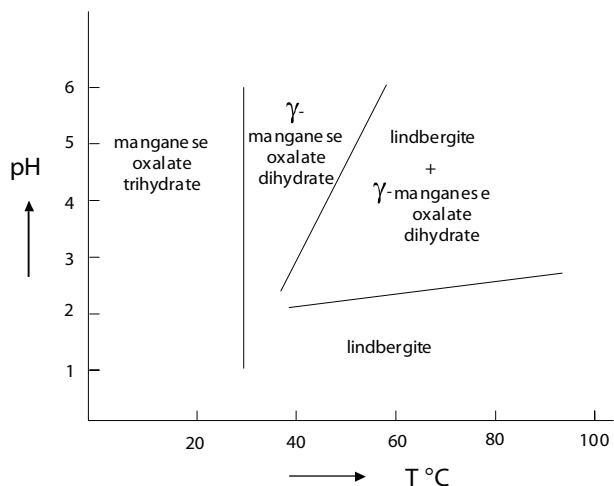


FIGURE 5. Stability field of lindbergite (α), the orthorhombic polymorphous (γ), and the trihydrate (Huizing et al. 1977).

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