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Dessauite, (Sr,Pb)(Y,U)(Ti,Fe^{3+})$_{20}O_{38}$, a new mineral of the crichtonite group from Buca della Vena mine, Tuscany, Italy

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ABSTRACT

Dessauite, a new mineral in the crichtonite group, occurs within cavities in calcite veins as tabular {001} rhombohedral, black, millimeter-sized crystals at Buca della Vena Mine, Apuan Alps (Tuscany, Italy). It is associated with derbylite, hematite, rutile, karelianite, siderite, and calcite. Dessauite is trigonal, space group $R\bar{3}$, with a = 9.197(1) Å, α = 68.75(2)°. Optically, dessauite is opaque and shows low bireflectance and very weak pleochroism. Electron microprobe analyses led to the following simplified chemical formula: (Sr,Pb)(Y,U)(Ti,Fe^{3+})$_{20}O_{38}$. The crystal structure of dessauite was refined from single-crystal X-ray diffraction data to R = 0.065. Dessauite is isostructural with the others members of the crichtonite group; a peculiar structural feature is the presence of additional, partially occupied octahedral sites. A comparison of the crystal-chemical formulas of all the minerals within the crichtonite group is presented. In view of the structural information, the analytical data have been re-arranged on the basis of the crystal-chemical formula $ABC_{18}T_2O_{38}$, rather than $AM_{21}O_{38}$.

INTRODUCTION

The crichtonite group includes a series of minerals, all characterized by the general formula $AM_{21}O_{38}$: crichtonite, senaite, davidite-(La), davidite-(Ce), davidite-(Y), loveringite, landauite, lindsleyite, and mathiasite. The first two minerals and the davidite species have been known for more than 90 years, whereas the others have been described only very recently. However, little was known about the discriminating differences in their chemical composition until crystal-structure analyses, for most of them, allowed the unambiguous allocation of the different cations to the proper sites.

For completeness, three additions should be made to the above list. (1) A mineral from Italy referred to as "davidite from Pizzo Cervandone" by Stalder and Bühler (1987) probably deserves the status of a distinct mineral species because of its peculiar chemical composition. (2) A Re-rich unnamed mineral of uncertain origin preserved in the Natural History Museum, Geneva (Switzerland), was described by Sarp et al. (1981). That mineral, however, despite having the same cell constants and space group as the other crichtonite-group minerals, could be somewhat different since it has excess cations (more than 23 cations per 38 O atoms, instead of 22) and several differences exist in the X-ray diffraction pattern. (3) A poorly characterized mineral from Romania has been referred to as "romanite" (Dragila 1990), but it has not been submitted to the I.M.A. Commission on New Minerals and Mineral Names (Jambor and Puziewicz 1992) and therefore cannot be regarded as a distinct mineral species. Dragila (1990) assigned "romanite" to the davidite group, but such an assignment seems highly questionable.

The aims of this paper are the description of a new member of the crichtonite group, found in Tuscany, Italy, and a review and comparison of the crystal-chemical formulas of the other minerals in the group.

The new mineral has been named dessauite in memory of the late Gabor Dessau (1907–1983), professor of ore mineralogy at the University of Pisa and devoted expert of the ore deposits of Tuscany. Both the mineral and its name have been approved by the I.M.A. Commission on New Minerals and Mineral Names. The type material is deposited in the Museo di Storia Naturale e del Territorio, University of Pisa (catalog no. 16385).

OCCURRENCE

Dessauite was found in the Buca della Vena Mine, Apuan Alps, near the town of Stazzema (LU), northern Tuscany, Italy. It occurs in calcite veins hosted within dolomite ("grezzoni") and is associated with calcite, rutile, hematite, siderite, and derbylite. Dessauite is the latest in a long series of rare minerals identified within the Buca della Vena mine in the last decade, such as apuanite, versiliaite, stibivanite–2O, karelianite, robinsonite, tintinaite, and andorite. All these minerals originated from hydro-

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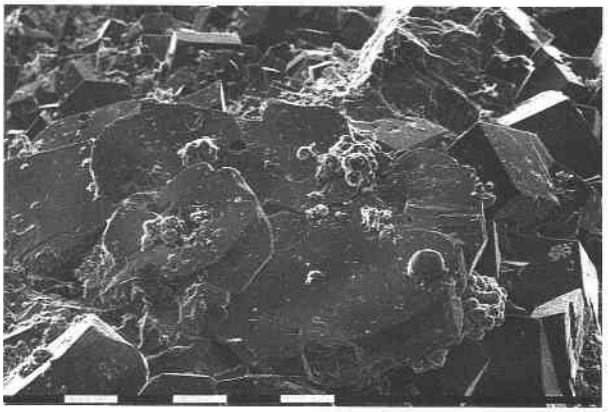

FIGURE 1. SEM photograph of tabular {001} dessauite crystals on calcite. Scale bar: 0.1 mm.

thermal fluids circulating through a small hematite-barite ore deposit within dolomite, during an alpine metamorphic event (Orlandi and Checchi 1986).

PHYSICAL AND OPTICAL PROPERTIES

Dessauite occurs as small, flattened rhombohedral crystals, tabular {001} with hexagonal outline (Fig. 1). Dimensions: diameter up to 1 mm, thickness up to 0.2 mm. The mineral is black and opaque with a metallic luster, and it is brittle. The Vickers hardness, VHN_{100}, is 1105 to 1782. Cleavage was not observed, and the fracture is conchoidal. The density could not be measured because of the small grain size; the calculated density is 4.68 g/cm^3. In reflected plane-polarized light the color is ash-grey with pale bluish tones. Internal reflections were not observed. The anisotropy is weak in shades of very dark blue and tan. The bireflectance is low, and the pleochroism is very weak. The reflectance values R1 and R2 were measured on a digital microphotometer EEL-165 in air and in oil with refractive index 1.5150(2) using SiC as a standard. The relevant values are listed in Table 1. The X-ray powder pattern of dessauite is reported in Table 2.

CHEMICAL COMPOSITION

A total of 21 chemical analyses were made using a JEOL JXA-8600 electron microprobe equipped with a Tracor Northern Series II system (at CNR-CSMGA, Firenze). No elements (with $Z > 9$) other than those reported here were detected by a preliminary 300 s energy-dispersive scan. The working conditions were: accelerating voltage 20 kV, beam current 30 nA (monitored on a Faraday cup), counting time 40 s for peak and 20 s for both left and right backgrounds. The following standards were used: metallic vanadium (V), rutile (Ti), monazite (Th, La, Ce, Pr, Nd), metallic uranium (U), almandine (Al, Si, Mg), chromite (Cr), ilmenite (Fe), YAG (Y), diopside (Ca), rhodonite (Mn), willemite (Zn), celestine (Sr), benitoite (Ba), and crocoite (Pb). The presence of Si, Pr, and Nd was always tested but these elements were never detected. The raw data were reduced using a Bence-Albee correction routine (Bence and Albee 1968).

Four of the 21 analyses were rejected because of major inhomogeneities. The analytical data and the resulting crystal-chemical formula are given in Table 3. On this

TABLE 1. Reflectance values (%) for dessauite

Wavelength (nm)	Air		Oil	
	R1	R2	R1	R2
470	17.3	17.7	5.6	6.0
546	16.6	17.1	5.0	5.5
589	16.1	16.6	4.8	5.3
650	16.0	16.5	4.6	5.1

TABLE 2. X-ray powder pattern of dessauite

d_{obs}	d_{calc}	*hkl*	*I*
3.412	3.414	220	m
3.064	3.068	$1\bar{3}1$	vw
2.998	2.997	$\bar{2}11$	w
2.902	2.901	231	m
2.846	2.845	232	mw
2.758	2.755	030	vw
2.624	2.641	230	vw
2.499	2.495	$\bar{3}31$	mw
2.431	2.433	$\bar{1}31$	vw
2.250	2.249	241	w
2.140	2.144	140	w
1.916	1.917	340	mw
1.805	1.808	251	mw
1.712	1.712	150	vw
1.603	1.606	$4\bar{5}1$	m
1.548	1.551	$\bar{5}21$	w
1.511	1.511	$\bar{3}63$	w
1.441	1.441	$\bar{4}31$	m

Notes: Gandolfi camera, diameter 114.6 mm, Fe$K\alpha$ radiation, λ = 1.93728 Å. Intensities were estimated by eye and are given as follows: m = medium; mw = medium weak, w = weak, vw = very weak.

TABLE 3. Electron microprobe analysis (average of 17 points) of dessauite from Buca della Vena Mine

Oxide	wt%	Range
V_2O_5	0.89	0.83–1.06
TiO_2	55.71	52.25–59.62
ThO_2	0.11	0.02–0.19
UO_2	4.52	2.63–8.74
Al_2O_3	0.08	0.04–0.20
Cr_2O_3	0.21	0.05–0.36
Fe_2O_3	27.24	26.26–28.18
Y_2O_3	2.02	1.37–2.64
La_2O_3	1.14	0.89–1.40
Ce_2O_3	0.68	0.48–0.84
MgO	0.03	0.00–0.16
CaO	0.12	0.07–0.22
MnO	0.56	0.31–0.97
ZnO	0.42	0.36–0.47
SrO	2.34	2.19–2.45
BaO	0.83	0.61–1.04
PbO	3.18	2.90–3.39
Total	100.08	

Notes: Empirical formula (calculated on the basis O = 38): $(Sr_{0.41}Pb_{0.26}Ba_{0.10}Ca_{0.04}Th_{0.01})_{\Sigma=0.82}(Y_{0.33}U_{0.31}Mn_{0.14}La_{0.13}Ce_{0.08})_{\Sigma=0.99}(Ti_{12.78}Fe_{6.25}V_{0.169}Zn_{0.0}Cr_{0.05}Al_{0.03}Mg_{0.01})_{\Sigma=19.39}O_{38}$. Simplified formula: $(Sr,Pb)(Y,U)(Ti,Fe^{3+})_{20}O_{38}$.

TABLE 5. Positional and displacement parameters for dessauite

Site	Occupancy	*x*	*y*	*z*	U^*
M0	$Sr_{0.42}Pb_{0.24}Ba_{0.12}\square_{0.22}$	0	0	0	0.0186(6)
M1	$Y_{0.30}U_{0.30}Mn_{0.12}La_{0.12}Ce_{0.06}\square_{0.10}$	½	½	½	0.0063(4)
M2	$Fe_{0.93}\square_{0.07}$	0.3095(2)	0.3095(2)	0.3095(2)	0.0089(7)
M3	$Fe_{0.77}Ti_{0.19}\square_{0.04}$	0.3479(2)	0.1263(2)	0.0207(2)	0.0063(4)
M4	$Ti_{0.96}\square_{0.04}$	0.3088(3)	0.7208(3)	0.1464(3)	0.0068(5)
M5	$Ti_{0.96}\square_{0.04}$	0.4740(3)	0.0791(3)	0.6426(3)	0.0063(5)
M6	$Fe_{0.07}\square_{0.93}$	0.396(2)	0.396(2)	0.396(2)	0.010(7)
M7	$Fe_{0.12}\square_{0.88}$	0.264(2)	0.423(2)	0.806(2)	0.007(2)
M8	$Fe_{0.06}\square_{0.94}$	0.226(4)	0.138(4)	0.456(4)	0.009(6)
M9	$Fe_{0.06}\square_{0.94}$	0.926(4)	0.237(3)	0.158(3)	0.005(5)
O1		0.304(1)	0.628(1)	0.380(1)	0.006(2)
O2		0.153(1)	0.238(1)	0.938(1)	0.004(2)
O3		0.918(1)	0.457(1)	0.303(1)	0.005(1)
O4		0.144(1)	0.519(1)	0.990(1)	0.006(1)
O5		0.391(1)	0.480(1)	0.138(1)	0.009(2)
O6		0.7062(9)	0.2379(9)	0.0714(9)	0.001(1)
O7		0.2150(8)	0.2150(8)	0.2150(8)	0.011(3)

* U_{eq} for cations M0–M5; U_{iso} for cations M6–M9 and O atoms.

basis, the following simplified chemical formula may be assumed for dessauite: $(Sr,Pb)(Y,U)(Ti,Fe^{3+})_{20}O_{38}$.

CRYSTAL STRUCTURE REFINEMENT

Due to the conspicuous presence of radioactive elements, it proved difficult to find well-crystallized dessauite. A crystal (dimensions 0.5 × 0.5 × 0.1 mm) was chosen for the X-ray intensity data collection on the basis of its Weissenberg photographs. Before starting the collection of intensity data the crystal was heated at 1000 °C for 24 h.

Like all other members of the crichtonite family, dessauite is rhombohedral, space group $R\bar{3}$. The following unit-cell parameters were obtained by least-square fitting of 2ϑ values of 30 reflections ($19° < 2\vartheta < 30°$) accurately centered on the diffractometer: a = 9.197(1) Å, α = 68.75(2)°. The corresponding parameters in the hexagonal setting are: a = 10.385 (1), c = 20.921 (2) Å.

The intensity data were collected with an Ital Structures four-circle automatic diffractometer, operating at 48 kV and 28 mA, using graphite-monochromated Mo$K\alpha$ radiation (λ = 0.71069 Å), $\omega-2\vartheta$ scan mode, ϑ_{max} = 30°, scan range ± (0.7 + 0.15 tan ϑ)°, and minimum scan speed of 1.5°/min, proportionally raised on the basis of the intensity of a pre-scan of the peaks. A total of 2983 reflections were measured and corrected for Lorentz and polarization factors. By considering as unobserved those reflections with $I < 3\sigma_I$, and by merging equivalents, a set of 1169 unique reflections was obtained. Many doubtful weak observed reflections, probably caused by diffuse scattering related to the semi-metamictic nature of the crystal, led to a further cutoff for reflections having $F_o < 15\sigma_{Fo}$, thus obtaining a definitive set of 891 observed reflections. The absorption effects were corrected by means of DIFABS (Walker and Stuart 1983). The starting atomic fractional coordinates were those of davidite (Gatehouse et al. 1979). The same site labeling was adopted. The cation site occupancies were set in such a way as to give the best agreement between the chemical composition and the structural information (namely, the electron density values, the bond distances, and the electrostatic charge balance for all sites). As in the case of mathiasite (Gatehouse et al. 1983), the difference-Fourier synthesis revealed a pair of additional electron density maxima, ~12 and 10 e/Å^3 in height, which were recognized as suitable sites for octahedrally coordinated cations. Such sites were called M6 and M7 and introduced with low occupancies in the refinement. For the sake of simplicity only Fe was allocated in the M6 and M7 sites; however, such sites are likely to be occupied by other minor cations (V, Zn, Cr) as well. The introduction of 8% Fe in M6 and 12% Fe in M7 caused the lowering of the R factor of approximately 2%. At this stage a new difference-Fourier synthesis featured two further electron density maxima, ~5 e/Å^3 in height, corresponding to sixfold- and fivefold-coordinated sites, respectively; 6% Fe was assigned to in each of the two sites, which were called M8 and M9. The R factor dropped further by approximately 1%. Using anisotropic displacement parameters for cation sites M0–M5, and the neutral atomic scattering factors incorporated in the least-square program SHELXL-93 (Sheldrick 1993), the structure was refined to the following reliability indices for 891 structure amplitudes: R_1 (conventional R factor) = 0.065, $R_{w(2)}$ (weighted R factor computed on F^2) = 0.168, and S (goodness of fit) = 1.38. The weighting scheme was $w = 1/[\sigma^2_{F_o^2} + (0.0544P)^2 + 27.94P]$, where $P = [\text{Max}(F_o^2, 0) + 2F_c^2]/3$. Observed and calculated structure factors are listed in Table 4[1].

DISCUSSION

Description of the structure of dessauite

The final atomic positional and displacement parameters are given in Table 5. Bond distances are given in

[1] For a copy of Table 4, order Document AM-97-640 from the Business Office, Mineralogical Society of America, 1015 Eighteenth Street, Suite 601, Washington, DC 20036, U.S.A. Please remit $5.00 in advance for the microfiche. Deposit items may also be available on the American Minerologist web site, refer to the inside back cover of a current issue for web address.

TABLE 6. Selected bond distances (Å) in dessauite

M0-O2	2.802(8) × 6
M0-O6	2.827(8) × 6
M1-O1	2.194(8) × 6
M2-O5	1.925(9) × 3
M2-O7	1.976(18)
M3-O4	1.954(9)
M3-O3	1.981(8)
M3-O2	1.983(9)
M3-O7	2.026(9)
M3-O4	2.036(9)
M3-O2	2.088(8)
M4-O2	1.878(9)
M4-O6	1.947(8)
M4-O3	1.963(8)
M4-O1	1.994(9)
M4-O6	2.071(8)
M4-O5	2.089(9)
M5-O4	1.853(9)
M5-O1	1.905(9)
M5-O3	1.926(9)
M5-O5	2.002(10)
M5-O6	2.055(8)
M5-O5	2.158(9)
M6-O1	1.96(2) × 3
M6-O5	2.23(3) × 3
M7-O4	1.97(2)
M7-O3	2.02(2)
M7-O1	2.05(2)
M7-O2	2.07(2)
M7-O1	2.13(2)
M7-O3	2.13(2)
M8-O3	1.88(3)
M8-O4	2.01(3)
M8-O7	2.09(3)
M8-O6	2.19(3)
M8-O5	2.19(3)
M8-O5	2.24(3)
M9-O6	1.89(3)
M9-O2	1.93(3)
M9-O2	1.95(3)
M9-O4	2.09(3)
M9-O6	2.19(3)

Table 6. Moreover, average cation-oxygen distances in crichtonite-group minerals, for which structural data are available, are compared in Table 7. The basic features of the structure of dessauite are identical to those of the other members of the crichtonite group; therefore they will not be considered in detail here. The main difference with respect to the other minerals is the occurrence, in dessauite, of three additional octahedral sites (M6, M7, and M8) and of a site in square pyramidal coordination (M9), all with low occupancies. The M6 site is also present in mathiasite (Gatehouse et al. 1983). The occupancies of the M2–M5 sites were slightly reduced, because a 100% occupancy in these sites was not compatible with non-empty M6–M9 sites, because of unreliable cation-cation contacts. As explained in the previous section, the additional sites were introduced in two different steps. The mutual exclusion between M0–M5 sites (those of the basic structure) and M6–M9 sites (the additional sites) was strictly accounted for during the first step, namely the introduction of M6 and M7 cations. The introduction of M8 and M9 would actually have required an even slightly lower occupancy in M3–M5 octahedral sites. However, the strong correlation coefficients between the overall scale factors and the occupancy factors within

TABLE 7. Comparison of average bond distances (Å) in minerals of the crichtonite group

	M0	M1	M2	M3	M4	M5
Crichtonite	2.792	2.205	1.972	2.006	1.969	1.967
Senaite	2.817	2.227	1.996	1.998	1.976	1.964
Loveringite	2.789	2.166	1.994	1.983	1.971	1.970
Landauite	2.822	2.211	1.963	1.997	1.973	1.969
Davidite	2.769	2.243	1.981	2.010	1.975	1.972
Mathiasite	2.832	2.136	1.981	1.980	1.978	1.971
Dessauite	2.814	2.194	1.938	2.011	1.990	1.983

sites occupied by two or more different cations, makes the occupancies of M6–M9 sites highly sensitive to certain structural parameters (e.g., the U/Y ratio in M1, or the Pb/Sr ratio in M0). Therefore, although the model presented here for dessauite has excess cations (22.4 per 38 O atoms), it represents the best compromise between the structural data and crystal-chemical constraints. An excess of total cations with respect to the theoretical 22 is also observed in lindsleyite and mathiasite (Haggerty et al. 1983).

It is suggested that the cation disorder over additional octahedral sites may be related to the heating processes used to improve the crystalline state of dessauite. To confirm this, structural studies on other minerals of the crichtonite group, treated in the same way, are planned.

Crystal chemistry of the crichtonite-group minerals

As a general rule for these minerals, it seems better to indicate the chemical compositions of crichtonite-group minerals by means of the crystal-chemical formula $ABC_{18}T_2O_{38}$ rather than by the formula previously adopted, $AM_{21}O_{38}$. In fact, in the latter formula, the general symbol M includes very different cations, namely T cations, which are in tetrahedral coordination in the M2 crystallographic site, and B and C cations, which have octahedral coordination. The present distinction between B and C is based on the markedly different dimensions of the respective crystallographic sites: B cations are located in the larger, high-symmetry M1 site ($M-O_{av}$ = 2.20 Å), whereas C cations are located in the smaller M3, M4, and M5 sites ($M-O_{av}$ = 1.98 Å). The chemical differences among the various end-members are better appreciated by considering the nature of all cations, not only the large A cations (twelvefold-coordinated M0 crystallographic site). Previously, only the A cations were used to discriminate between the crichtonite-group minerals. On these grounds, the distinction between crichtonite and dessauite would have been impossible, because both have Sr as the dominant A cation.

On these premises, the simplified structural formula of dessauite is $(Sr,Pb)(Y,U)(Ti,Fe^{3+})_{18}Fe^{3+}{}_2O_{38}$, and the crystal-chemical formulas of all members of the crichtonite group can be rewritten as in Table 8. Accordingly, the new mineral species dessauite is chemically intermediate between crichtonite and davidite and could be described both as a (Y,U)-dominant variant of crichtonite and as a (Sr,Pb)-dominant variant of davidite.

TABLE 8. Cation distributions in minerals of the crichtonite group

M0	M1	M2	M3-M4-M5	Ref.
$Sr_{0.70}(Pb,REE)_{0.20}\square_{0.10}$	$Mn_{0.70}Fe_{0.30}$	$Fe_{2.00}$	$Ti_{13.60}Fe_{4.40}$	1
$Pb_{0.83}\square_{0.17}$	$Mn_{1.00}$	$Fe_{2.00}$	$Ti_{13.66}Fe_{4.34}$	2
$Ca_{0.72}REE_{0.23}(Y,Th,U,Pb)_{0.05}$	$Zr_{0.58}Mg_{0.32}REE_{0.10}$	$Fe_{1.23}Mg_{0.60}\square_{0.17}$	$Ti_{12.48}Cr_{2.24}Fe_{2.19}Al_{0.38}V_{0.21}\square_{0.50}$	3
$Na_{0.70}K_{0.15}Pb_{0.15}$	$Mn_{1.00}$	$Zn_{2.00}$	$Ti_{15.84}Fe_{2.16}$	4
$REE_{0.54}Ca_{0.20}(Sr,Th,Pb)_{0.09}\square_{0.17}$	$REE_{0.37}U_{0.33}Y_{0.30}$	$Fe_{1.58}Mg_{0.24}\square_{0.18}$	$Ti_{12.67}Fe_{4.57}Cr_{0.21}\square_{0.55}$	5
$K_{0.82}Na_{0.14}Ba_{0.14}Sr_{0.10}$	$Zr_{0.70}Ca_{0.30}$	$Fe_{0.90}Mg_{0.90}\square_{0.20}$	$Ti_{12.90}Cr_{3.10}Fe_{1.30}(V,Nb,Mg)_{0.70}$	6
$Sr_{0.42}Pb_{0.24}Ba_{0.12}\square_{0.22}$	$Y_{0.30}U_{0.30}REE_{0.18}Mn_{0.12}\square_{0.10}$	$Fe_{1.64}\square_{0.16}$	$Ti_{12.66}Fe_{4.62}\square_{0.72}$	7
$Pb_{0.90}Sr_{0.80}$	$Y_{0.50}Mn_{0.30}\square_{0.20}$	$Fe_{2.00}$	$Ti_{14.00}Fe_{3.75}Re_{0.25}$	8*
$Ba_{0.60}Sr_{0.40}$	$Zr_{0.90}Ca_{0.10}$	$Fe_{1.00}Mg_{1.00}$	$Ti_{11.60}Cr_{4.00}Fe_{2.00}Mg_{0.40}$	9*
$U_{0.50}Pb_{0.40}(Sr,Ca)_{0.10}$	$(Mn,Y,Nb)_{0.90}Sn_{0.10}$	$Fe_{1.40}(Zn,As,V)_{0.60}$	$Ti_{12.4}Fe_{5.6}$	10*

Notes: Distributions made on the basis of single-crystal X-ray structure analyses. (1) Crichtonite (Grey et al. 1976). (2) Senaite (Grey and Lloyd 1976). (3) Loveringite (Gatehouse et al. 1978). (4) Landauite (Grey and Gatehouse 1978). (5) Davidite-(La) (Gatehouse et al. 1979). (6) Mathiasite [including $Mg_{0.20}$ in M6] (Gatehouse et al. 1983). (7) Dessauite [including $Fe_{0.42}$ in M6, $Fe_{0.72}$ in M7, $Fe_{0.36}$ in M8, $Fe_{0.36}$ in M9] (this study). (8) Unnamed (Sarp et al. 1981). (9) Lindsleyite (Haggerty et al. 1983). (10) "Davidite from Pizzo Cervandone" (Stalder and Bühler 1987).

* Structural study not carried out; a tentative cation distribution has been set up one the basis of the chemical data.

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