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The crystal structure of tuscanite

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Abstract

The crystal structure of tuscanite

$[K_{0.88}Sr_{0.04}(H_2O)_{1.08}](Ca_{5.25}Na_{0.51}Fe^{+3}_{0.10}\ Mg_{0.11})(Si_{6.34}Al_{3.66})O_{22}(SO_4)_{1.38}(CO_3OH)_{0.55}(O_4H_4)_{0.11}]$

space group $P2_1/a$, a = 24.03(2), b = 5.11(1), c = 10.88(2) A, β = 106.94(9)°, was determined by direct methods using diffractometric data. Anisotropic thermal refinement led to a final R value of 0.034 (R_w = 0.038). The crystal structure is characterized by the presence of double layers of (Si,Al) tetrahedra; the double layer can be described, as in latiumite, as made up by five-membered rings, nearly parallel to (010), but whereas in latiumite each tetrahedron in the ring points upward, in tuscanite three tetrahedra in every ring point upward and two downward. These layers are connected by calcium and sulphate ions, as in latiumite; whereas in latiumite successive layers are related by unit translation, in tuscanite they are related by a glide plane. The substitutions of (CO_3OH) and (H_4O_4) for SO_4 are proposed and discussed.

Introduction

Tuscanite was discovered by Orlandi *et al.* (1977) in some ejected blocks found in a pumice deposit in Pitigliano, Tuscany, Italy. They pointed out the close relationships between tuscanite and latiumite, a sheet silicate described by Tilley and Henry (1953) and whose crystal structure was studied by Cannillo *et al.* (1973). Orlandi *et al.* (1977) noted the similarity of the X-ray powder diffraction patterns of tuscanite and latiumite; in fact, the unit cell of tuscanite is very similar to that of latiumite, from which it is obtainable by doubling the a parameter. We easily made the hypothesis that tuscanite and latiumite differ only in the stacking sequence, namely unit translation in latiumite and glide plane in tuscanite, of the same structural unit. To understand the precise structural relationships between the two minerals, namely whether the relation is polytypic or polymorphic, and to discover possible differences, we undertook the present structural analysis. A preliminary report on the main features of the crystal structure of tuscanite was presented at the 1975 meeting of Società Italiana di Mineralogia e Petrologia.

Experimental

A small (0.15 × 0.12 × 0.27mm) tabular crystal of tuscanite from Pitigliano was used for the X-ray structure analysis. Unit-cell data of our specimen, determined by the Philips PW-1100 single-crystal automatic diffractometer, are a = 24.03(2), b = 5.11(1), c = 10.88(2) A, β = 106.94(9)°, space group $P2_1/a$, in good agreement with the values found by Orlandi *et al.* (1977) by least-squares fitting of powder data. Intensity data were collected by the same diffractometer, using graphite-monochromatized Mo$K\alpha$ radiation (λ = 0.7107 A), ω scan, integration width 1.2°. 3724 independent reflections were collected from 2° to 30° ϑ; the corresponding values of F_{obs} and $\sigma(F_{obs})$ were obtained by the procedure of Davies and Gatehouse (1973); no absorption correction was made owing to the small dimensions of the crystal (μ = 19.7 cm^{-1}); also, no extinction correction was applied. 2420 reflections were classified as "observed,"

Table 1. Atomic coordinates and thermal anisotropic parameters of tuscanite

Atom	Mult.	x	y	z	Beq.	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
T(1)	1.0	0.4386(1)	0.2403(2)	0.0725(1)	0.50	25(1)	500(30)	112(6)	-6(5)	24(2)	-18(12)
T(2)	1.0	0.4420(1)	0.2296(2)	0.3529(1)	0.53	31(1)	510(30)	96(6)	8(6)	22(2)	18(13)
T(3)	1.0	0.4216(1)	0.7256(2)	0.4789(1)	0.54	31(1)	560(30)	96(6)	1(5)	22(2)	3(12)
T(4)	1.0	0.1619(1)	0.2724(2)	0.3540(1)	0.61	36(1)	590(28)	106(6)	17(6)	22(2)	18(12)
T(5)	1.0	0.0753(1)	0.2351(2)	0.0824(1)	0.49	26(1)	475(31)	102(6)	-7(6)	20(2)	-22(13)
O(1)	1.0	0.0969(1)	0.9179(5)	0.0505(2)	0.89	54(4)	760(78)	150(20)	11(15)	18(7)	-71(30)
O(2)	1.0	0.0070(1)	0.2297(6)	0.1003(2)	1.34	34(4)	2060(100)	290(19)	-2(20)	44(7)	-47(42)
O(3)	1.0	0.4194(1)	0.9354(5)	0.0466(2)	0.96	74(4)	650(80)	180(20)	-16(15)	64(8)	-119(32)
O(4)	1.0	0.4191(1)	0.3459(5)	0.1948(2)	0.87	46(4)	950(80)	160(18)	43(15)	32(7)	37(33)
O(5)	1.0	0.4197(1)	0.9016(5)	0.3514(2)	1.07	68(4)	862(85)	237(20)	-9(16)	59(7)	28(35)
O(6)	1.0	0.0161(1)	0.2409(6)	0.4152(2)	1.51	49(4)	2180(105)	255(20)	36(20)	11(7)	87(45)
O(7)	1.0	0.4027(1)	0.4228(5)	0.4342(2)	0.99	54(4)	835(84)	248(20)	-2(15)	52(8)	-115(33)
O(8)	1.0	0.3710(1)	0.8528(5)	0.5362(2)	1.06	63(4)	995(85)	232(19)	97(16)	67(7)	88(34)
O(9)	1.0	0.2200(1)	0.4482(5)	0.3948(2)	0.97	40(4)	1000(84)	234(20)	-7(15)	25(7)	-32(33)
O(10)	1.0	0.1702(1)	-0.0351(5)	0.3520(2)	1.55	139(6)	801(88)	201(21)	106(18)	60(9)	79(35)
O(11)	1.0	0.1231(1)	0.3853(5)	0.2155(2)	0.90	48(4)	870(83)	147(18)	-22(15)	2(7)	46(32)
Ca(1)	1.01	0.1906(1)	0.7236(1)	0.1885(1)	0.88	41(1)	879(23)	209(10)	10(4)	34(2)	29(10)
Ca(2)	0.90	0.3554(1)	0.7252(2)	0.1472(1)	1.04	46(1)	1113(27)	235(11)	-18(5)	39(2)	70(12)
Ca(3)	0.99	0.3082(1)	0.2396(1)	0.4565(1)	0.69	35(1)	657(22)	149(5)	29(4)	22(2)	-34(10)
K	0.51	0.0365(1)	0.7445(4)	0.2713(2)	2.51	146(4)	2050(72)	577(17)	-24(15)	116(7)	-11(3)
S	0.95	0.2692(1)	0.1979(2)	0.1403(1)	0.64	27(1)	614(27)	167(6)	-8(5)	25(2)	-64(11)
O(12)	1.0	0.2063(1)	0.1633(6)	0.0816(3)	1.70	53(4)	2120(115)	386(23)	-63(18)	30(8)	199(42)
O(13)	1.0	0.3024(1)	0.1469(1)	0.0489(3)	1.66	70(5)	1972(108)	387(23)	-87(19)	89(9)	-313(42)
O(14)	1.0	0.2819(1)	0.4639(5)	0.1968(3)	1.37	75(5)	930(87)	400(25)	-43(17)	82(9)	-142(38)
O(15)	1.0	0.2871(1)	0.0006(5)	0.2458(3)	1.54	100(5)	1110(95)	292(24)	33(18)	38(9)	57(38)

The values of x, y, z are given in fractional coordinates, the anisotropic thermal parameters ($\times 10^5$) are of the form: $\exp -(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})$. Estimated standard deviations are in parentheses.

having observed intensity greater than three times the corresponding standard deviations.

Structure determination

The structure was solved by application of direct methods, using the MULTAN program (Main *et al.* 1971). The E-map showed distinct peaks for the four heavy cations, Ca(1), Ca(2), Ca(3), K, the five (Si,Al) tetrahedral cations, and the sulphur atom present in the asymmetric unit. Successive Fourier synthesis located all the oxygen atoms, either linked to (Si,Al) atoms or belonging to the sulphate group. Three cycles of full matrix least squares, with isotropic thermal parameters, lowered $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ to the value 0.05; the reflections were weighted according to the reciprocal of the variance, estimated from counting statistics. Further refinement, based on anisotropic thermal parameters, reduced R to the value 0.035 and $R_w = \Sigma(w||F_o| - |F_c||^2/\Sigma w|F_o|^2)^{1/2}$ to the value 0.038.

All the scattering factors used in the structure-factor calculations were taken from Hanson *et al.* (1964), with no contribution for anomalous scattering. Final atomic parameters are given in Table 1. Bond distances and angles are given in Tables 2 and 3. Observed and calculated structure factors are on deposit.[1]

Description and discussion of the crystal structure

As tuscanite is closely related to latiumite, we shall follow, as far as possible, in the description of its crystal structure, the corresponding paragraph in the paper by Cannillo *et al.* (1973). As can be seen from Figure 1, which shows a projection of the crystal structure of tuscanite in the [010] direction, the most characteristic feature of the mineral is the presence of corrugated double layers of (Si,Al) tetrahedra, bonded to each other by a layer made up by calcium cations and sulphate groups.

As in latiumite, the single layer, shown in Figure 2,

[1] To obtain a copy of the structure factors, order document Am-77-055 from the Business office, 1909 K Street, N.W., Washington, D.C. 20006. Please remit $1.00 in advance for the microfiche.

Table 2. Interatomic distances of tuscanite (A)

T(1)-O(1)vi	1.637(2)	Ca(1)-O(1)	2.519(2)
-O(2)vii	1.590(2)	-O(9)	2.569(3)
-O(3)ii	1.626(3)	-O(10)i	2.332(3)
-O(4)	1.626(2)	-O(11)	2.445(2)
mean	1.620	-O(12)i	2.609(3)
		-O(13)iv	2.668(3)
T(2)-O(4)	1.751(2)	-O(14)	2.544(3)
-O(5)ii	1.759(3)	-O(15)i	2.633(3)
-O(6)vii	1.718(3)	mean	2.540
-O(7)	1.770(2)		
mean	1.749	Ca(2)-O(3)	2.386(2)
		-O(4)	2.430(2)
T(3)-O(5)	1.643(3)	-O(5)	2.477(3)
-O(6)iii	1.604(3)	-O(12)iv	2.520(3)
-O(7)	1.646(3)	-O(13)i	2.573(3)
-O(8)	1.653(2)	-O(14)	2.397(3)
mean	1.636	-O(15)i	2.618(3)
		mean	2.486
T(4)-O(8)v	1.666(2)		
-O(9)	1.609(3)	Ca(3)-O(7)	2.534(2)
-O(10)	1.585(3)	-O(8)ii	2.485(3)
-O(11)	1.631(2)	-O(9)	2.290(2)
mean	1.623	-O(9)v	2.436(2)
		-O(10)iii	2.305(3)
T(5)-O(1)ii	1.767(3)	-O(14)	2.944(3)
-O(2)	1.708(2)	-O(15)	2.516(3)
-O(3)vi	1.773(2)	mean	2.501
-O(11)	1.743(2)		
mean	1.748	K-O(1)	3.271(3)
		-O(2)i	3.057(4)
S-O(12)	1.471(3)	-O(2)	3.182(3)
-O(13)	1.468(3)	-O(3)viii	3.277(3)
-O(14)	1.486(3)	-O(4)viii	3.414(3)
-O(15)	1.495(3)	-O(5)viii	3.259(3)
mean	1.480	-O(6)i	3.094(4)
		-O(6)	3.125(4)
		-O(7)iii	3.243(3)
		-O(8)v	3.260(3)
		-O(10)i	3.274(4)
		-O(11)	2.969(3)
		mean	3.202

In Tables 2 and 3 the atoms of the different asymmetric units are related to the symmetry equivalent atoms of the fundamental unit as follows:

i	atom at	x	1+y	z
ii	" "	x	1-y	z
iii	" "	1/2-x	1/2+y	1-z
iv	" "	1/2-x	1/2+y	-z
v	" "	1/2-x	-1/2+y	1-z
vi	" "	1/2-x	-1/2+y	-z
vii	" "	1/2+x	1/2-y	z
viii	" "	-1/2+x	3/2-y	z
ix	" "	-1/2+x	1/2-y	z

is formed by six-and eight-membered rings of tetrahedra. Whereas in latiumite the two sheets which build up the double layer are related each other by the screw rotations, in tuscanite they are related by inversion centers. The double layer, which in latiumite is repeated by the [100] translation, is repeated by the glide plane *a* in tuscanite.

The double layer can be described, as in latiumite, as made up by five-membered rings, nearly parallel to (010), but while in latiumite each tetrahedron in the ring points upward, in tuscanite (Fig. 1) three tetrahedra in every ring point upward and two downward. On the basis of the bond distances (Table 2), an ordered distribution of tetrahedral cations in the layer can be inferred, with silicon predominantly located in *T*(1), *T*(3), and *T*(4) sites and aluminum in *T*(2) and *T*(5).

Three independent cations, Ca(1), Ca(2), and Ca(3), occur between the tetrahedral layers. In both minerals, whereas Ca(3) connects directly two aluminosilicate layers, Ca(1) and Ca(2) connect them indirectly with sulphate groups as bridges between Ca(1) and Ca(2). Atomic scattering factors for calcium were used during the least-squares refinement for the three cationic sites, although the chemical data, given in Table 4, suggested a substitution of sodium, and minor iron and magnesium, for calcium. The multipliers of the Ca atoms were allowed to vary, and their final values as well as the thermal parameters obtained (Table 1) indicate that sodium atoms concentrate in the Ca(2) site.

A feature that distinguishes tuscanite from latiumite is the more open coordination around the potassium cation; in both minerals it is located in the cavity between two succeeding five-membered rings, but whereas in latiumite it is ten-coordinated with bond distances ranging from 2.91 to 3.24 A and an average K–O distance of 3.12 A, in tuscanite it is eleven (or twelve) coordinated, with bond distances ringing from 2.969 to 3.277 A (or 3.414 A) and an average K–O distance of 3.183 A (or 3.202 A). The multiplier of potassium atom, as given by the least-squares refinement, is 0.51. The chemical data indicated 0.44 potassium atoms in this site. The low occupancy of potassium atoms, together with high water content of tuscanite and the suitable dimensions of the site, indicates that water molecules can substitute for potassium cations in the cavity between five-membered rings.

The mean S–O bond length in the SO_4^{2-} tetrahedron (1.480 A) matches that found in latiumite (1.47 A) and is in perfect agreement with the value given by Shannon and Prewitt (1969) as sum of the effective ionic radii of $^{IV}S^{6+}$ (sulphur cation in tetrahedral coordination) and $^{III}O^{2-}$ (oxygen anion with coordination number three). The multipliers of all the atoms in the sulphate group were refined, but whereas the multipliers of the oxygen atoms indicated full occupancy, that of the sulphur atom was 0.95. It should be observed (Table 2) that the thermal parameter for the sulphur atom (B eq. = 0.65 A^2) is higher than those corresponding to aluminum and silicon in more open tetrahedral sites; thus a lower occupancy factor should be obtained by adjusting the thermal

Table 3. Tetrahedral bond angles of tuscanite

O(1)vi -T(1)-O(2)vii	111.4(1)	O(4) -T(2)-O(5)ii	107.9(1)
O(1)vi -T(1)-O(3)ii	109.6(1)	O(4) -T(2)-O(7)	104.0(1)
O(1)vi -T(1)-O(4)	106.3(1)	O(4) -T(2)-O(6)vii	110.5(1)
O(2)vii-T(1)-O(3)ii	110.6(1)	O(5)ii-T(2)-O(7)	109.3(1)
O(2)vii-T(1)-O(4)	109.9(1)	O(5)ii-T(2)-O(6)vii	111.4(1)
O(3)ii -T(1)-O(4)	108.9(1)	O(7) -T(2)-O(6)vii	113.3(1)
O(5) -T(3)-O(7)	109.1(1)	O(8)v -T(4)-O(10)	110.0(1)
O(5) -T(3)-O(8)	105.1(1)	O(8)v -T(4)-O(11)	108.3(1)
O(5) -T(3)-O(6)iii	111.8(1)	O(8)v -T(4)-O(9)	102.7(1)
O(7) -T(3)-O(8)	108.2(1)	O(10) -T(4)-O(11)	112.2(1)
O(7) -T(3)-O(6)iii	112.2(1)	O(10) -T(4)-O(9)	117.1(1)
O(8) -T(3)-O(6)iii	110.2(1)	O(11) -T(4)-O(9)	105.9(1)
O(1)ii -T(5)-O(2)	110.7(1)	T(1)iv-O(1)-T(5)i	123.8(1)
O(1)ii -T(5)-O(3)vi	106.3(1)	T(1)ix-O(2)-T(5)	162.0(2)
O(1)ii -T(5)-O(11)	114.0(1)	T(1)i -O(3)-T(5)iv	127.4(2)
O(2) -T(5)-O(3)vi	113.2(1)	T(1) -O(4)-T(2)	127.6(2)
O(2) -T(5)-O(11)	109.6(1)	T(2)i -O(5)-T(3)	125.5(2)
O(3)vi -T(5)-O(11)	102.8(1)	T(2)ix-O(6)-T(3)v	158.7(2)
		T(2) -O(7)-T(3)	122.1(2)
O(12)-S-O(14)	110.6(2)	T(3) -O(8)-T(4)iii	136.9(2)
O(12)-S-O(15)	106.4(2)	T(4) -O(11)-T(5)	132.4(2)
O(12)-S-O(13)	112.1(2)		
O(14)-S-O(15)	108.7(2)		
O(14)-S-O(13)	110.9(2)		
O(15)-S-O(13)	108.1(2)		

factor of sulphur to a more realistic lower value, say 0.40 A². Table 4 indicates an occupancy of nearly 0.7 for the sulphate group, together with an occupancy of nearly 0.3 for the carbonate group; Table 4 indicates also the presence of a substantial quantity of hydroxyl anions in the structure. To reconcile all these chemical data with the result of the structure analysis a substitution of sulphate anions by (CO_3OH) groups may be suggested. In this substitution a planar carbonate group takes its place near the position corresponding to a face of the tetrahedral sulphate group, whereas a hydroxyl anion, or a water molecule in some cases, locates itself near the position corresponding to the fourth vertex of the sulphate anion,

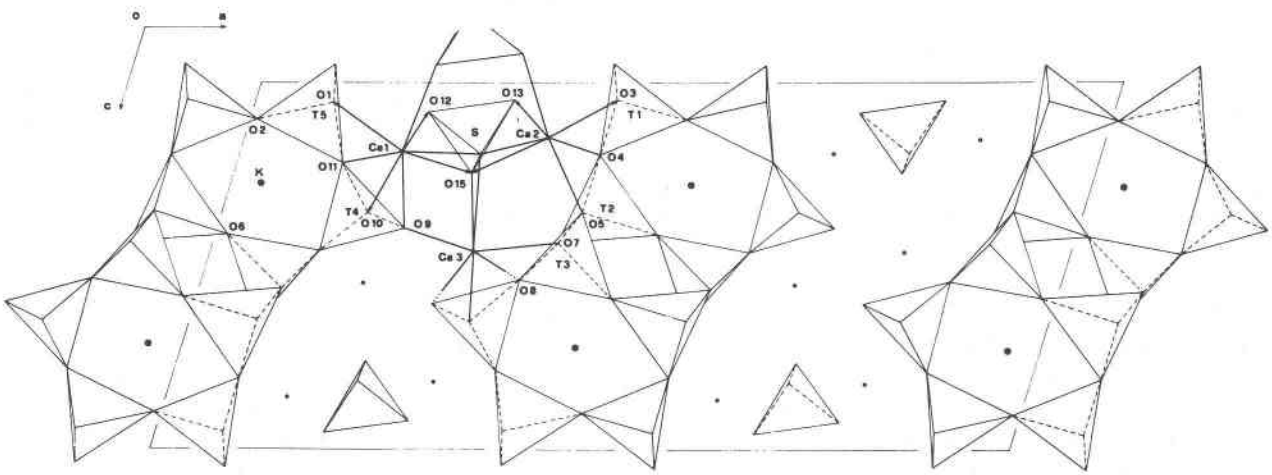

Fig. 1. Crystal structure of tuscanite, as seen along (010). Arrows indicate that the bond is associated with atoms translated one unit above, dashed lines with atoms translated one unit below.

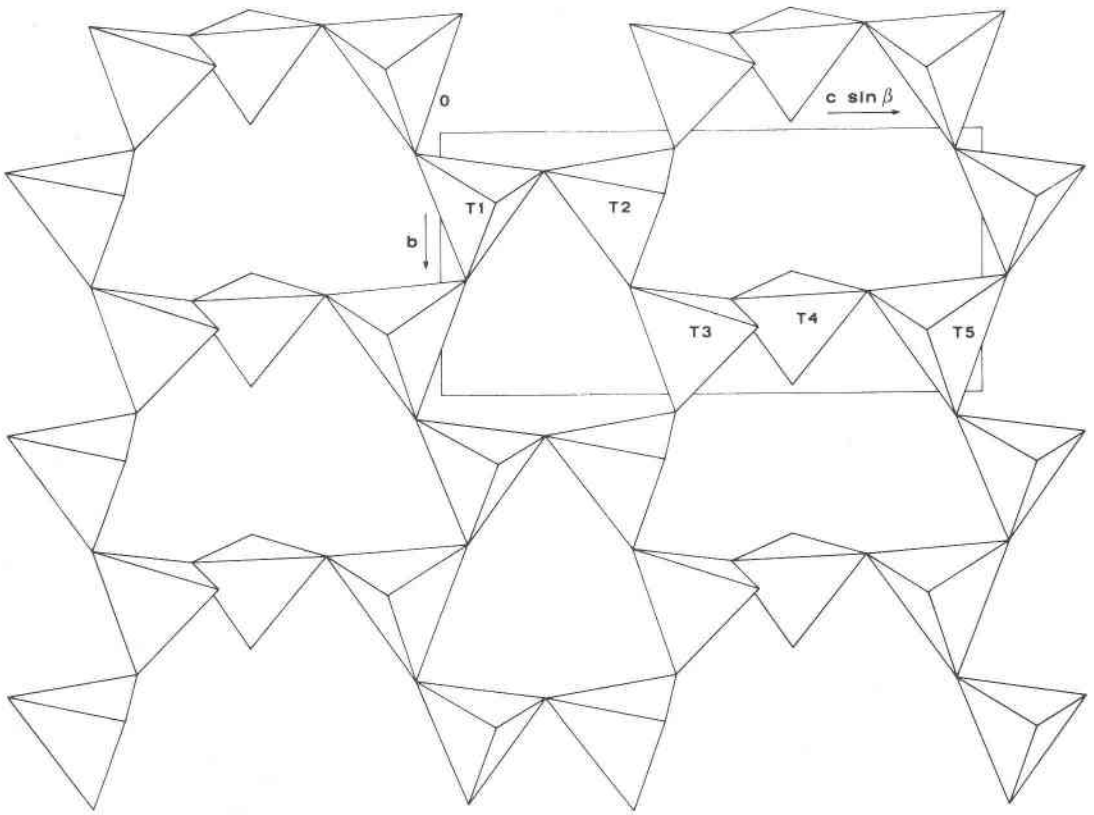

Fig. 2. (Si,Al) single layer of tuscanite, as seen along (100).

and in this manner each of the calcium polyhedra maintains its complete coordination. A substitution of this kind was introduced by Borneman-Starinkevic and Belov (1953), who suggested that when a planar carbonate ion substitutes for a tetrahedral phosphate ion in carbonate apatites, the vacant site is occupied by a fluorine ion. A minor substitution of H_4O_4 for SO_4 is also possible: this is a well-known kind of substitution in silicates and phosphates. The absence of any relevant peak in the final difference Fourier synthesis indicates that the carbonate group substitutes with equal probability on each face of the sulphate tetrahedron. Thus the crystal structure analysis supports the crystal chemical formula proposed for tuscanite by Orlandi *et al.* (1977):

$$[K_{0.88}Sr_{0.04}(H_2O)_{1.08}](Ca_{5.25}Na_{0.51}Fe^{3+}_{0.10}\ Mg_{0.11})$$
$$(Si_{6.34}Al_{3.66})O_{22}[(SO_4)_{1.38}(CO_3OH)_{0.55}(O_4H_4)_{0.11}]$$

The balance of electrostatic valence, computed using the method of Donnay and Allmann (1970), is reported in Table 5. As can be seen, the balance is fairly satisfactory, with deviations smaller than 10 percent from the theoretical values. The balance was calculated assuming for the potassium site an occupancy of 0.5 and for Ca(2) site an occupancy 2/3 Ca + 1/3 Na.

Table 4. Chemical composition of tuscanite (from Orlandi *et al.*, 1977)

	wt %	Atoms per unit cell
SiO_2	34.64	12.68
Al_2O_3	16.95	7.32
Fe_2O_3	0.76	0.20
FeO	-	-
MgO	0.40	0.22
MnO	-	-
CaO	26.76	10.50
SrO	0.38	0.08
Na_2O	1.45	1.02
K_2O	3.79	1.76
H_2O	2.61	3.20
SO_3	10.04	2.76
CO_2	2.20	1.10
Cl	0.02	-

Table 5. Estimated bond valences (V.U.)

	T(1)	T(2)	T(3)	T(4)	T(5)	Ca(1)	Ca(2)	Ca(3)	K	S	Σc_v
O(1)	0.966				0.722	0.257			0.025		1.971
O(2)	1.061				0.811				0.072+0.040		1.984
O(3)	0.988				0.713		0.274		0.024		1.999
O(4)	0.988	0.748					0.257		0.001		1.994
O(5)		0.736	0.987				0.241		0.027		1.991
O(6)		0.798	1.069						0.061+0.052		1.980
O(7)		0.720	0.981					0.273	0.030		2.004
O(8)			0.967	0.915				0.292	0.027		2.200
O(9)				1.028		0.240		0.384+0.312			1.963
O(10)				1.078		0.339		0.376	0.025		1.818
O(11)				0.984	0.757	0.286			0.111		2.139
O(12)						0.226	0.227			1.533	1.985
O(13)						0.205	0.209			1.544	1.958
O(14)						0.249	0.270	0.117		1.479	2.114
O(15)						0.217	0.195	0.280		1.446	2.138

Σc_v = sum of bond valences reaching the anions (V.U.)

Polymorphic relations between tuscanite and latiumite

As previously noted, the crystal structure of tuscanite, and that of latiumite, can be conveniently described as made up by double layers of silicon and aluminum tetrahedra and layers of calcium cations and sulphate groups. However, to emphasize their polymorphic relations, the structures of these minerals can be formally described as built up by a different kind of structural units, made up by a sheet of

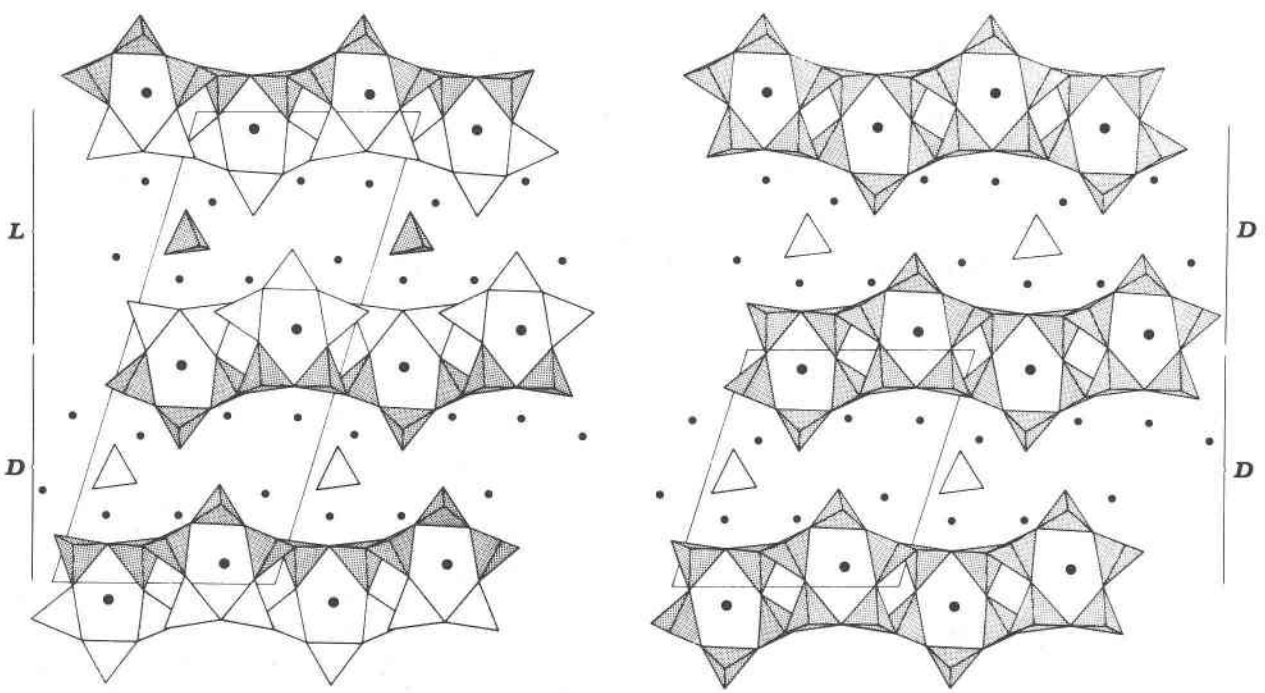

Fig. 3. Crystal structures of tuscanite and latiumite, as seen along (010).

calcium cations and sulphate–carbonate anions sandwiched between two tetrahedral single sheets such as those represented in Figure 2.

Whereas in latiumite successive three-sheet structural layers are related by [100] translation, in tuscanite they are related by inversion center (Fig. 3). Thus if we call *D* a three-sheet structural unit and *L* its enantiomorphous counterpart, the crystal structures of latiumite and tuscanite can be schematically described as *DDD*. . . . and *DLDL*. . . . sequences respectively. This indicates clearly that the two minerals cannot be considered as polytypic. In fact, according to the general usage, only such polymorphs can be regarded as polytypes which while differing in the relative positions of distant parts do not differ as regards relative positions of nearest neighbors. In these minerals the pairs of tetrahedra which assure the connection between adjacent layers are in eclipsed conformation in latiumite and in staggered conformation in tuscanite.

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