

# The crystal structure of kukisvumite, $\text{Na}_6\text{ZnTi}_4(\text{Si}_2\text{O}_6)_4\text{O}_4 \cdot 4\text{H}_2\text{O}$

S. Merlino, M. Pasero\* and O. Ferro

Università di Pisa, Dipartimento di Scienze della Terra, Via S. Maria 53, I-56126 Pisa, Italy

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**Abstract.** The crystal structure of kukisvumite, a rare mineral from Kola peninsula, Russia, has been refined in the *Pccn* space group with X-ray single-crystal data to  $R = 0.055$ . Kukisvumite, ideally  $\text{Na}_6\text{ZnTi}_4(\text{Si}_2\text{O}_6)_4\text{O}_4 \cdot 4\text{H}_2\text{O}$ , has  $a = 29.029(4)$  Å,  $b = 8.595(1)$  Å,  $c = 5.209(1)$  Å, and  $Z = 2$ . Its crystal structure is closely related to that of lintisite; the latter mineral is obtained from kukisvumite through the substitution  $\text{Zn}^{2+} + \square = 2\text{Li}^+$ . In the *Pccn* space group an average structural model is realized, resulting in columns of half-occupied  $[\text{ZnO}_4]$  tetrahedra and in zig-zag chains of half-occupied Na-centered octahedra. The actual structure of kukisvumite would imply a concerted ordering of zinc and sodium in neighbour cells. The polysomatic relationships of kukisvumite with the minerals of the lintisite family are shortly outlined.

## Introduction

Kukisvumite was described by Yakovenchuk, Pakhomovskii and Bogdanova (1991) from an arfvedsonite-microcline pegmatite vein in the Kukisvumtschorr deposit, Khibiny alkaline massif, Kola peninsula, Russia. Microprobe analyses of kukisvumite gave the following simplified chemical formula:  $\text{Na}_6\text{ZnTi}_4\text{Si}_8\text{O}_{28} \cdot 4\text{H}_2\text{O}$ . The chemical composition of kukisvumite is very similar to that of lintisite, a mineral newly described from the nearby Lovozero massif (Khomyakov, Polezhaeva, Merlino, Pasero, 1990; Merlino, Pasero, Khomyakov, 1990). The chemical formula of lintisite can be written as  $\text{Na}_6\text{Li}_2\text{Ti}_4\text{Si}_8\text{O}_{28} \cdot 4\text{H}_2\text{O}$ , that is, the two minerals are related by the simple substitution  $\text{Zn}^{2+} + \square = 2\text{Li}^+$ .

The unit cell parameters of the two minerals are also closely related in spite of the different crystal systems, and the X-ray powder patterns are almost identical in all the strongest lines.

It seemed likely that close structural relationships exist between kukisvumite and lintisite. Therefore we undertook a structural study of kukisvumite from the type locality to clarify its relationships with lintisite. A preliminary account of the basic structural features of kukisvumite has been shortly outlined by Merlino and Pasero (1997). In this paper a full description of the kukisvumite structure is given.

## Experimental

Preliminary Weissenberg photographs taken on selected crystals of kukisvumite confirmed the orthorhombic symmetry and the space group *Pccn*. The following unit cell parameters for the crystal eventually chosen for the intensity data collection (dimensions  $0.44 \times 0.06 \times 0.02$  mm) were refined at the four-circle diffractometer:  $a = 29.029(4)$ ,  $b = 8.595(1)$ ,  $c = 5.209(1)$  Å.

The intensity data set was collected with a Siemens four-circle diffractometer. Operating conditions were the following: room temperature, 50 kV, 40 mA,  $\text{MoK}_\alpha$ ,  $\lambda = 0.71069$  Å, scan mode  $\theta - 2\theta$ , scan speed  $2^\circ/\text{min}$ , maximum  $2\theta = 50^\circ$ ,  $-1 \leq h \leq 34$ ,  $-1 \leq k \leq 10$ ,  $-1 \leq l \leq 6$ , refinement carried out against squared structure amplitudes (1138 independent reflections) with SHELXL-93 (Sheldrick, 1993) computer package, absorption effects accounted for through XABS2 (Parkin, Moezzi, Hope, 1995).

## Structure refinement

On the basis of the reasonable hypothesis that kukisvumite was structurally very similar to lintisite, a starting set of fractional coordinates for kukisvumite was obtained on the basis of the monoclinic structure of lintisite, which has an approximately pseudo-orthorhombic metric ( $\beta \approx 91^\circ$ ), by taking into account the different origin and space group symmetry. The site occupancy factors of some atoms were lowered on the basis of crystal-chemical considerations. Therefore a zinc atoms with 50% occupancy was placed in the same site which is occupied by lithium in lintisite. Moreover, also the occupancy of Na2 atom was fixed at 50%, because the space group *Pccn* resulted in two incompatible equivalent positions for Na2, 2.60 Å apart. For the same reason, we fixed 50% occupancy for the water molecule, too: owing to the different symmetry in kukisvumite there are two independent, half occupied sites, O81 and O82, instead of a single O8 site in lintisite. The full occupancy at those sites, too, would have implied some additional impossible interatomic distances, e.g., 1.02 Å between O81 and O82.

Such a structural model was refined smoothly. The final reliability indices for the anisotropic model were  $R1$  (conventional  $R$  factor) = 0.055 for 588 reflections with

\* Correspondence author (e-mail: pasero@dst.unipi.it)

$F_o > 4\sigma(F_o)$  and 0.146 for all 1138 reflections,  $wR2$  (weighted  $R$  factor on  $F^2$ ) = 0.148; weighting scheme =  $1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.00P]$ , where  $P = [\text{Max}(F_o^2, 0) + 2F_c^2]/3$ .

The maximum and minimum electron density maxima in the final difference Fourier synthesis were  $+0.67$  and  $-0.64 \text{ e}/\text{\AA}^3$ , with all major positive electron density maxima in the surroundings of O81 and O82. Owing to the disordered nature of kukisvumite the hydrogen atoms were included in the refinement in calculated positions, and the relevant O—H and H...O distances were constrained to reasonable values during the refinement.

The final fractional coordinates and displacement parameters for kukisvumite are listed in Table 1. Selected bond distances are given in Table 2, and are compared with the corresponding distances in lintisite. The valence bond balance is reported in Table 3.

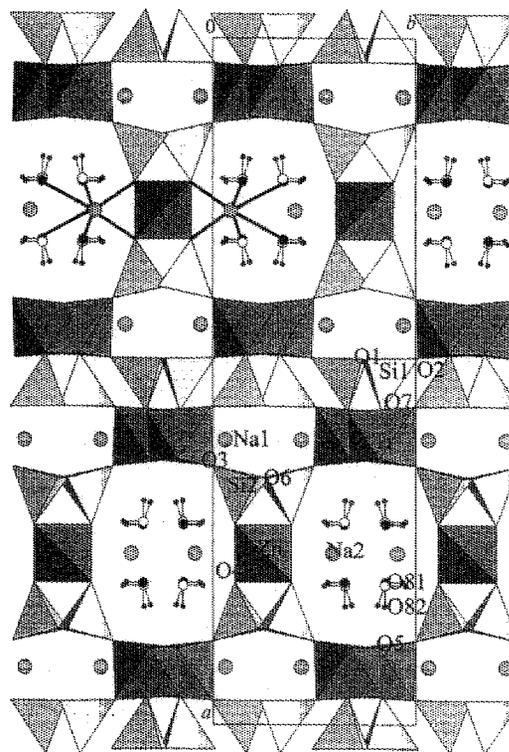
**Table 1a.** Final fractional coordinates and  $U_{eq}$  displacement parameters for kukisvumite; e.s.d.'s are given in parentheses and refer to the last digit.

| Atom             | $x$       | $y$       | $z$       | $U_{eq}$  |
|------------------|-----------|-----------|-----------|-----------|
| Ti               | 0.5830(1) | 0.8450(4) | 0.1322(7) | 0.0055(8) |
| Si1              | 0.4863(2) | 0.8418(7) | 0.7887(9) | 0.005(1)  |
| Si2              | 0.6557(2) | 0.1484(6) | 0.0514(9) | 0.007(1)  |
| Zn <sup>a</sup>  | $3/4$     | $1/4$     | 0.791(2)  | 0.024(2)  |
| Na1              | 0.5840(3) | 0.0615(8) | 0.623(2)  | 0.015(2)  |
| Na2 <sup>a</sup> | 0.7500(7) | 0.591(2)  | 0.786(3)  | 0.031(4)  |
| O1               | 0.4652(4) | 0.763(1)  | 0.050(3)  | 0.009(3)  |
| O2               | 0.4641(4) | 0.011(1)  | 0.754(3)  | 0.007(3)  |
| O3               | 0.6223(4) | 0.000(1)  | 0.006(2)  | 0.009(3)  |
| O4               | 0.7917(4) | 1.108(1)  | 0.539(3)  | 0.022(4)  |
| O5               | 0.8879(4) | 0.820(1)  | 0.939(3)  | 0.006(3)  |
| O6               | 0.6398(4) | 0.274(1)  | 0.825(2)  | 0.011(3)  |
| O7               | 0.5420(4) | 0.833(1)  | 0.793(2)  | 0.007(3)  |
| O81 <sup>a</sup> | 0.7922(8) | 0.851(4)  | 0.934(5)  | 0.016(6)  |
| O82 <sup>a</sup> | 0.7965(9) | 0.849(4)  | 1.128(6)  | 0.025(7)  |
| H1 <sup>a</sup>  | 0.792     | 0.93      | 0.80      | 0.05      |
| H2 <sup>a</sup>  | 0.825     | 0.81      | 0.94      | 0.05      |
| H3 <sup>a</sup>  | 0.797     | 0.95      | 1.24      | 0.05      |
| H4 <sup>a</sup>  | 0.828     | 0.85      | 1.04      | 0.05      |

a: *s.o.f.* 0.5 (see text for explanation).

**Table 1b.** Anisotropic displacement parameters for kukisvumite.

| Atom | $U_{11}$  | $U_{22}$  | $U_{33}$  | $U_{23}$   | $U_{13}$   | $U_{12}$   |
|------|-----------|-----------|-----------|------------|------------|------------|
| Ti   | 0.008(2)  | 0.005(2)  | 0.004(2)  | 0.001(2)   | 0.000(1)   | 0.001(2)   |
| Si1  | 0.004(3)  | 0.005(2)  | 0.006(2)  | -0.001(3)  | 0.000(2)   | 0.002(3)   |
| Si2  | 0.006(2)  | 0.007(2)  | 0.008(2)  | 0.000(3)   | -0.001(2)  | 0.001(3)   |
| Zn   | 0.024(4)  | 0.025(4)  | 0.022(4)  | 0          | 0          | 0.001(4)   |
| Na1  | 0.020(4)  | 0.012(4)  | 0.013(4)  | -0.001(4)  | -0.001(4)  | -0.001(4)  |
| Na2  | 0.030(10) | 0.042(11) | 0.022(9)  | -0.001(9)  | 0.007(9)   | -0.009(11) |
| O1   | 0.004(6)  | 0.011(6)  | 0.010(6)  | 0.003(7)   | -0.003(6)  | -0.003(7)  |
| O2   | 0.008(7)  | 0.003(6)  | 0.012(7)  | -0.003(7)  | -0.005(7)  | 0.002(6)   |
| O3   | 0.006(6)  | 0.013(7)  | 0.009(8)  | 0.001(6)   | 0.005(6)   | 0.001(5)   |
| O4   | 0.007(7)  | 0.009(7)  | 0.050(10) | -0.001(7)  | 0.002(8)   | 0.006(6)   |
| O5   | 0.004(7)  | 0.007(7)  | 0.006(6)  | 0.001(6)   | 0.000(5)   | -0.002(5)  |
| O6   | 0.010(7)  | 0.009(7)  | 0.014(8)  | 0.009(6)   | 0.001(6)   | 0.002(6)   |
| O7   | 0.006(6)  | 0.006(7)  | 0.008(6)  | -0.001(7)  | 0.002(5)   | -0.005(6)  |
| O81  | 0.004(14) | 0.023(16) | 0.021(16) | 0.008(21)  | 0.003(12)  | -0.007(16) |
| O82  | 0.009(14) | 0.039(18) | 0.027(18) | -0.017(24) | -0.005(15) | 0.003(18)  |



**Fig. 1.** The crystal structure of kukisvumite, as seen along [001]. The unit cell is outlined. The coordination of Na2 is sketched only twice, to avoid confusion with the atom labels, which are in keeping with the fractional coordinates of Table 1.

## Description of the structure

The structure of kukisvumite (depicted in Fig. 1) is closely related to that of lintisite (Merlino, et al., 1990). All coordination polyhedra are similar in the two structures (cf. Table 2). The titanium is octahedrally coordinated by six oxygen atoms, the octahedra giving rise to zig-zag chains through sharing of edges. The titanium atoms occupy eccentric positions within the coordination polyhedra. The two independent silicon atoms occur within tetrahedra arranged in pyroxene-like chains, having O—O—O angles of  $171^\circ$  (Si1 chains) and  $162^\circ$  (Si2 chains). Zinc is in tetrahedral coordination as well. Unlike lithium in lintisite, in

**Table 2a.** Bond lengths (Å) in kukisvumite. The corresponding distances in lintisite (Merlino, et al., 1990) are reported for the sake of comparison.

|                | kukisvumite | lintisite       |
|----------------|-------------|-----------------|
| Ti polyhedron  |             |                 |
| O5             | 1.82(1)     | 1.807(3)        |
| O3a            | 1.87(1)     | 1.874(3)        |
| O5b            | 1.93(1)     | 1.923(3)        |
| O2c            | 1.94(1)     | 1.956(3)        |
| O7b            | 2.11(1)     | 2.116(3)        |
| O7d            | 2.13(1)     | 2.141(3)        |
| (Ti—O)         | 1.968       | 1.969           |
| Si1 polyhedron |             |                 |
| O2e            | 1.60(1)     | 1.597(3)        |
| O7d            | 1.62(1)     | 1.628(3)        |
| O1             | 1.64(1)     | 1.638(3)        |
| O1b            | 1.65(1)     | 1.648(3)        |
| (Si1—O)        | 1.628       | 1.628           |
| Si2 polyhedron |             |                 |
| O4a            | 1.56(1)     | 1.586(3)        |
| O3a            | 1.62(1)     | 1.629(3)        |
| O6b            | 1.64(1)     | 1.653(3)        |
| O6e            | 1.66(1)     | 1.663(3)        |
| (Si2—O)        | 1.622       | 1.633           |
| Zn polyhedron  |             |                 |
|                |             | (Li-polyhedron) |
| O4f            | 2.15(1)     | 2.013(7)        |
| O4a            | 2.15(1)     | 2.013(7)        |
| O4g            | 2.16(1)     | 2.014(6)        |
| O4b            | 2.16(1)     | 2.014(6)        |
| (Zn—O)         | 2.154       | 2.014           |
| Na1 polyhedron |             |                 |
| O3a            | 2.34(1)     | 2.355(4)        |
| O1h            | 2.42(1)     | 2.419(3)        |
| O5d            | 2.43(1)     | 2.448(3)        |
| O7d            | 2.47(1)     | 2.464(3)        |
| O2i            | 2.49(1)     | 2.500(4)        |
| O6j            | 2.65(1)     | 2.589(3)        |
| O6e            | 2.66(1)     | 2.649(3)        |
| O1k            | 2.69(1)     | 2.687(3)        |
| (Na1—O)        | 2.519       | 2.514           |
| Na2 polyhedron |             |                 |
| O81l           | 2.26(3)     | 2.328(4)        |
| O82m           | 2.29(4)     | 2.328(4)        |
| O4g            | 2.46(2)     | 2.513(3)        |
| O4a            | 2.48(2)     | 2.513(3)        |
| O81n           | 2.66(4)     | 2.638(4)        |
| O82n           | 2.72(4)     | 2.638(4)        |
| (Na2—O)        | 2.481       | 2.493           |

which columns of edge-sharing [LiO<sub>4</sub>] tetrahedra occur, in the case of kukisvumite every second tetrahedral site is empty. This is related to the electrostatic valence balance, Zn<sup>2+</sup> having a doubled charge with respect to Li<sup>+</sup>, and is accomplished, as stated above, by setting 50% occupancy at the zinc site, in close agreement with the chemical composition of kukisvumite. Of the two sodium atoms, Na1 is eight-fold coordinated by eight oxygens, Na2 is octahedrally coordinated by two oxygen atoms and four water molecules [NaO<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>], giving rise to zigzag chains. In this latter case, too, the occupancy of sodium (Na2) and water molecules (O81 and O82) is halved.

Therefore the only major difference is the presence, in kukisvumite, of a zinc and sodium slab, instead of a lithium and sodium slab as in lintisite. These two structural units are compared in Fig. 2.

**Table 2b.** Distances (Å) and angles (°) involving water molecules in kukisvumite.

|                           | O—H distance | O...O distance | O—H—O angle |
|---------------------------|--------------|----------------|-------------|
| <i>O81 water molecule</i> |              |                |             |
| O81—H1...O4               | 1.00         | 3.02(3)        | 178         |
| O81—H2...O5               | 1.00         | 2.79(3)        | 142         |
| H1—O81—H2 angle           |              |                | 104         |
| <i>O82 water molecule</i> |              |                |             |
| O82—H3...O4 <sup>n</sup>  | 1.04         | 3.10(3)        | 178         |
| O82—H4...O5               | 1.01         | 2.84(3)        | 158         |
| H3—O82—H4 angle           |              |                | 105         |

Symmetry codes for equivalent positions: a:  $x, y + 1, z$ ; b:  $x, -y + 3/2, z - 1/2$ ; c:  $-x + 1, -y + 1, -z + 1$ ; d:  $x, y, z - 1$ ; e:  $x, y + 1, z - 1$ ; f:  $-x + 3/2, -y + 3/2, z$ ; g:  $-x + 3/2, y + 1, z - 1/2$ ; h:  $-x + 1, y + 1/2, -z - 1/2$ ; i:  $-x + 1, -y + 1, -z$ ; j:  $x, -y + 3/2, z - 3/2$ ; k:  $-x + 1, -y + 2, -z$ ; l:  $-x + 3/2, y, z - 3/2$ ; m:  $-x + 3/2, y, z - 1/2$ ; n:  $-x + 3/2, -y + 3/2, z - 1$ ; o:  $x, y, z + 1$ .

The half-occupancy of some atoms (Zn, Na2, O81, O82) in the structure of kukisvumite means that the real symmetry of the mineral is lower than *Pccn*, and that the average structure only can be assessed in this space group, in which two different, although geometrically equivalent, configurations are in superposition. In the actual structure either of these two configurations occurs at every single unit cell. As an hypothesis, the possible ordering of the two configurations should give rise to the doubling of the *b* cell parameter. However, careful inspection of long-exposed Weissenberg photographs, and of a number of selected area electron diffraction patterns, did not provide evidences of long-range ordering in kukisvumite. Therefore the disordered model better describes the real structure of the mineral.

It is worth noting that, unlike the other cations, the average (Zn—O) distance is significantly longer than ex-

**Table 3.** Bond valence balance given in conventional valence units (v.u.) and computed according to Brese & O'Keeffe (1992). The contribution of hydrogen bonds has been computed following Ferraris & Ivaldi (1988). Left and right superscripts indicate the number of equivalent bonds for each cation and each anion, respectively.

|                | Ti   | Si1  | Si2  | Zn   | Na1  | Na2  | H-bonds $\Sigma_v$                         |
|----------------|------|------|------|--|------|--|--|
| O1             |      | 0.95 |      |  | 0.19 |  | 2.16                                       |
|                |      | 0.93 |      |  | 0.09 |  |  |
| O2             | 0.72 | 1.07 |      |  | 0.15 |  | 1.94                                       |
| O3             | 0.85 |      | 1.01 |  | 0.23 |  | 2.09                                       |
| O4             |      |      | 1.18 | <sup>2</sup> 0.30 <sup>0.5</sup><br><sup>2</sup> 0.29 <sup>0.5</sup> |      | 0.17 <sup>0.5</sup><br>0.16 <sup>0.5</sup> | 0.13 <sup>0.5</sup><br>0.11 <sup>0.5</sup> |
| O5             | 0.99 |      |      |  | 0.18 |  | 0.19 <sup>0.5</sup><br>0.17 <sup>0.5</sup> |
|                | 0.73 |      |      |  |      |  | 2.08                                       |
| O6             |      |      | 0.95 |  | 0.10 |  | 2.05                                       |
|                |      |      | 0.90 |  | 0.10 |  |  |
| O7             | 0.45 | 1.02 |      |  | 0.16 |  | 2.05                                       |
|                | 0.42 |      |      |  |      |  |  |
| O81            |      |      |      |  |      | 0.28                                       | -0.13                                      |
|                |      |      |      |  |      | 0.10                                       | -0.19                                      |
| O82            |      |      |      |  |      | 0.26                                       | -0.11                                      |
|                |      |      |      |  |      | 0.08                                       | -0.17                                      |
| $\Sigma_{c,v}$ | 4.16 | 3.97 | 4.04 | 1.18   | 1.20 | 1.05                                       |  |

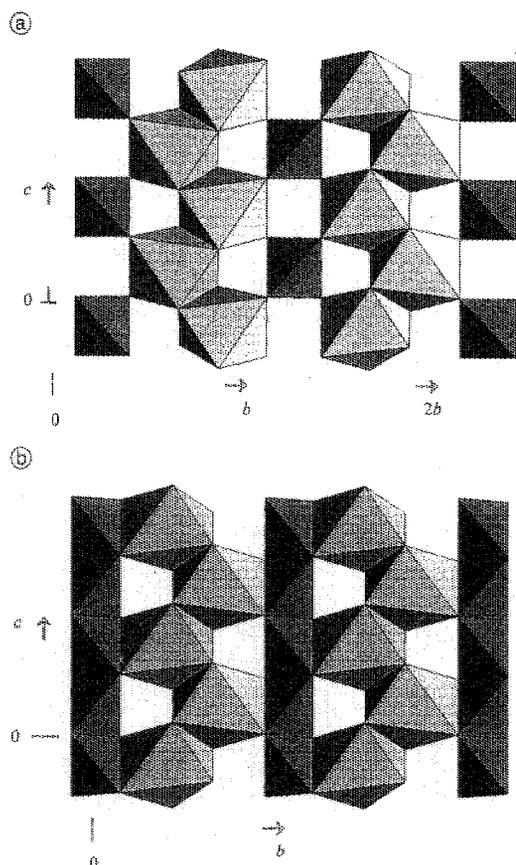


Fig. 2. Possible ordering mechanism within the zinc-sodium slab in kukisvumite, which would result in the doubling of the  $b$  axis (a), and the lithium-sodium slab in lintsite (b). [100] projections. See text for explanation.

pected for tetrahedrally coordinated  $Zn^{2+}$ . The distance computed according to the ionic radii by Shannon (1976) should be 1.96 Å, whereas it is actually 2.15 Å in kukisvumite, resulting in a low bond valence sum for Zn (1.18 instead of 2 v.u.; cf. Table 3). The explanation of this behaviour lies in the partial occupancy at the Zn site: the average structure of kukisvumite consists of columns of 50% occupied  $[ZnO_4]$  tetrahedra, whereas in the actual structure there is alternation along [001] of completely filled and empty tetrahedra. The former should therefore be smaller, and the latter larger. This behaviour implies that the oxygen (O4) moves from the empty site towards the Zn-filled site, and accounts for the high displacement parameters of O4, as well as of O81 and O82, which correspond to water molecules.

### Polysomatic description of kukisvumite

Merlino and Pasero (1997) discussed the lintsite polysomatic family starting from three basic structural modules, denoted as  $L$  [ $Na_4Ti_4(Si_2O_6)_2O_6$ ],  $S$  [ $Na_2Li_2Si_4O_{10} \cdot 4H_2O$ ], and  $Z$  [ $(Na,K)Si_3AlO_8 \cdot 2H_2O$ ]: by stacking such modules singularly, or in regularly alternating pairs, six different polysomes were described, corresponding to the structures of the minerals lorenzenite [ $L$ :  $Na_4Ti_4(Si_2O_6)_2O_6$ ; Sund-

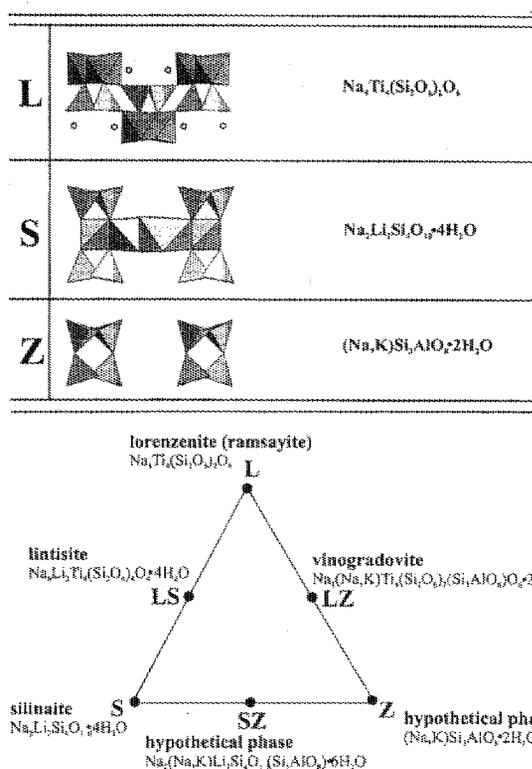


Fig. 3. The three polysomatic models introduced by Merlino and Pasero (1997), and the ideal chemical formulae of the resulting polysomes.

berg, Lehtinen, Kivekäs, 1987], silinaite [ $S$ :  $Na_2Li_2 \cdot Si_4O_{10} \cdot 4H_2O$ ; Grice, 1991], lintsite [ $LS$ :  $Na_6Li_2 \cdot Ti_4(Si_2O_6)_4O_4 \cdot 4H_2O$ ; Merlino, et al., 1990], vinogradovite [ $LZ$ :  $Na_4(Na,K)Ti_4(Si_2O_6)_2(Si_3AlO_8)_2O_6 \cdot 2H_2O$ ; Rastsvetaeva, Simonov, Belov, 1968; Rastsvetaeva and Andrianov, 1984; Kalsbeek and Rønso, 1992], plus the two hypothetical structures  $Z$  [ $(Na,K)Si_3AlO_8 \cdot 2H_2O$ ] (a zeolite-like structure) and  $SZ$  [ $Na_2(Na,K)Li_2Si_4O_{10} \cdot (Si_3AlO_8) \cdot 6H_2O$ ] (Fig. 3).

In this picture kukisvumite can be described as formed by alternation of the same  $L$  modules which occur in lintsite and in lorenzenite and of a modified  $S$  module (hereafter  $K$  module) with zinc instead of lithium (with formula  $Na_2ZnSi_4O_{10} \cdot 4H_2O$ ).

Although in kukisvumite the stacking sequence is the same as in the related structure lintsite, a different layer group symmetry for the  $K$  module,  $P(n)c2$ , is observed with respect to the layer group symmetry of the  $S$  module,  $P(1)2_1/c1$ . The polysomatic formula of kukisvumite can be written as  $LK$ . Moreover, it is possible to hypothesize two other structures, which should represent variants of the above-mentioned polysomes, namely the Zn analogue of silinaite and the hypothetical structure  $KZ$ .

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