CRYSTAL CHEMISTRY AND MINERALOGY OF THE LOMONOSOVITE GROUP IN THE LIGHT OF THE CRYSTAL STRUCTURE OF LOMONOSOVITE

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(ABSTRACT)

The composition of lomonosovite is $Na_2Ti_2Si_2O_9 \cdot NaPO_3$ or $Na_2Ti_2Si_2O_8 \cdot NaPO_4$ with much Na and Ti being replaced by Mn^{++}. There is no ramsayite nucleus in lomonosovite. New X-ray crystallographic data show a strong structural analogy between lomonosovite and spurrite, $Ca_2Ca_2Si_2O_8 \cdot CaCO_3$. In the lomonosovite structure there are layers of Na_3PO_4 between the individual parts of twinned crystals; these layers enter neither the structures of the individual parts of the twins nor the formula of a non-twinned crystal. When these layers are abundant they offer a reason for large discrepancies in "precise" chemical analyses.

Leaching removes large amounts of Na_3PO_4 from lomonosovite. This loss is accompanied by substitution of H_2O; the substituted substance is identical with murmanite, $Na_2Ti_2Si_2O_9 \cdot nH_2O$. The process is nonreversible and is not analogous to the processes in zeolites.

Lomonosovite was discovered in 1936 but a full mineralogical description of it did not appear until 1950 [1, 2]. Its accepted formula, $Na_2Ti_2Si_2O_9 \cdot Na_3PO_4$, proposed by Borneman [3], consists of the basic ramsayite "molecule" $Na_2Ti_2Si_2O_9$ and the "side chain" Na_3PO_4. Lomonosovite is very similar to a previously described mineral, murmanite, $(Na_2(Ti, Nb)_2Si_2O_9 \cdot nH_2O$ [4]) into which it grades through a series of intermediate compounds. According to Borneman the formula $n(Na, K, Ca)_2(Ti, Nb\ldots)_2Si_2O_9(n-x)Na_3PO_4 \cdot xH$ must be assigned to these compounds, emphasizing the possible loss of a part of the phosphate molecules by lomonosovite. In lomonosovite, the most highly phosphatic member of the group, x = 0, and in murmanite, the phosphate-free member, $Na_2Ti_2Si_2O_9 \cdot nH_2O$, x = n. Epistolite is the niobium analogue of murmanite (Semenov [5]).

According to Borneman, Na-phosphate is easily leached out of lomonosovite and the intermediate varieties even by cold water [3]. The water extract gives an alkaline reaction and has a $Na_2O:P_2O_5$ ratio of 3:1. Borneman points out that the leaching of Na-phosphate from lomonosovite by water indicates the lack of a strong "chemical bond" between the phosphate and silicate components, but the content of water increases in the intermediate leached varieties of the mineral.

According to our X-ray analysis, lomonosovite is triclinic and the parameters of its unit cell are: a = 5.4Å, b = 7.03Å, c = 14.3Å, $\alpha = 100°$, $\beta = 96°$, $\gamma = 90°$ and Z = 2. Thus the cell contains two formula units of $Na_2Ti_2Si_2O_9 \cdot Na_3PO_4$. Of the two possible space groups, $P\bar{1}$ and P_1, the second is the more probable, for the lomonosovite structure has no center of symmetry (it is not detected either on the Patterson diagrams or by statistical analysis of the distribution of structural factors).

Although two parameters, a = 5.4 and c = 14.3Å, of the lomonosovite unit cell are very near those of ramsayite (a = 14.54, c = 5.23Å), the other parameters are different and there is no indication of the presence of the metasilicate chains $(SiO_3)_\infty$, which in ramsayite [6] lie in the direction of the short axis c = 5.23Å. Neither the ordinary polarizing microscope nor the electron microscope shows any indications that lomonosovite is related to ramsayite (Semenov [5]).

All attempts to solve the structure of lomonosovite were hindered by the large number of sodium atoms (10) which must be distributed in the unit cell if the formula given above is accepted. *

The situation changed radically in 1960 when Semenov gave a detailed description of a second form of lomonosovite whose existence had been suggested earlier (β-lomonosovite or metalomonosovite) and which, according to chemical analysis, contains the same basic ramsayite "molecule" but has a metaphosphate ($NaPO_3$) instead of the orthophosphate (Na_3PO_4) "side chain"; i.e. the formula of metalomonosovite is $Na_2Ti_2Si_2O_9 \cdot NaPO_3$. It was surprising to discover that the metalomonosovite unit cell has the same dimensions as the lomonosovite cell (a = 5.28Å, b = 7.05Å, c = 14.50Å, $\alpha = 102°24'$, $\beta = 96°50'$, $\gamma = 90°$, according to R. P. Shibayev) and only X-ray powder photographs show certain differences between the two minerals.

In the same year (1960) the structure of spurrite, $Ca_5CO_3[SiO_4]_2$, one of the calcium silicates of cement, was published [8, 9], and it developed that its unit cell is very similar to that of lomonosovite (a = 10.46Å, b = 6.70, c = 14.20Å, $\beta = 101°19'$). Spurrite is monoclinic with $\alpha = 90°$ and its parameter a = 10.46Å; i.e. it is twice as large as in lomonosovite. However, in spurrite there is a clear pseudo period a' = ½a along axis x, as can be clearly seen from the diagram of the spurrite structure in Fig. 1.

The formulas of the two minerals:

$$Ca_5\,CO_3\,[SiO_4]_2 = Ca_2Ca_2Si_2O_8 \cdot CaCO_3 \quad \text{(spurrite)}$$

and

$$Na_2Ti_2Si_2O_9 \cdot NaPO_3 = Na_2Ti_2Si_2O_8 \cdot NaPO_4 \quad \text{(metalomonosovite)}$$

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*This was noticed by Semenov, who, approaching the problem from the mineralogical point of view [5, 7], proposed a formula for lomonosivite with a smaller number of cations--$Na_8MnTi_3[Si_4P_2O_{24}]$.

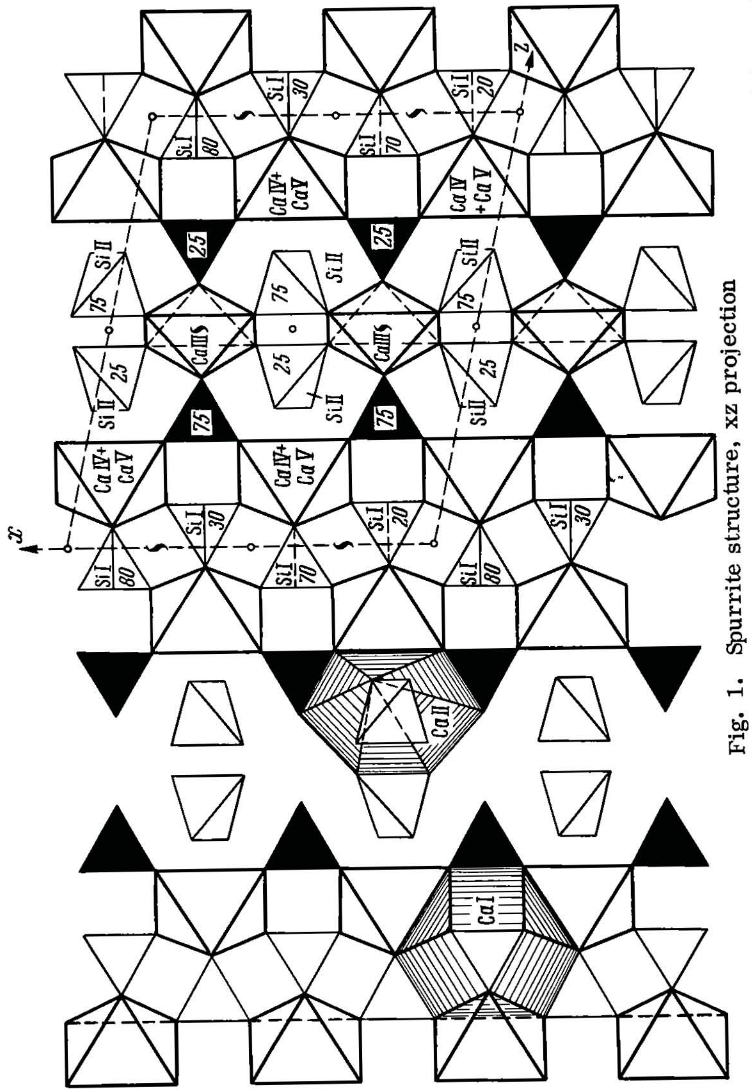

Fig. 1. Spurrite structure, xz projection

The true monoclinic cell is outlined against the background of the orthorhombic pattern. The height of the central ion (C or Si), i.e., coordinate y is given within each CO_3 triangle and each of the two kinds of SiO_4 tetrahedra. Two Ca III octahedra with coordinates $y = +0.05$ and $0.50 + 0.05$ are positioned vertically above each other in the unit cell. Octahedra Ca IV and Ca V with coordinates near $y = 0$ and $y = 0.5$ overlap each other. Polyhedra Ca I and Ca II are not shown in the monoclinic cell. They lie above and below the two kinds of SiO_4 tetrahedra (Si I and Si II) and are shown (one of each kind) in the left part of the diagram.

can be matched atom for atom with one important stipulation, that one oxygen ion be moved from the "basic" part of the metalomonosovite formula to the "side chain" so that the PO_4 tetrahedron of metalomonosovite corresponds to the triangular CO_3 anion of spurrite. Moreover, the four Ca ions in the basic part of the spurrite structure must be juxtaposed with 2Na + Ti in the basic ramsayite part of metalomonosovite. The juxtaposition of cations with different radii (Ca, Na ~1.0Å, Ti ~0.65Å) need cause no surprise, for in the past three years strong and sometimes complete analogies between the calcium silicates of cement and a number of alkali titanium and zirconium silicates has been demonstrated in the Institute of Crystallography [10]. The pattern of silicon-oxygen anions in the structural analogues, belonging to two classes of silicates always regarded as distinct, is identical, and there is geometric similarity in the cation pattern represented by indefinitely extended columns of octahedra standing on their edges. In the Ca-silicates all octahedra in the columns are occupied by Ca, but in the "colored" titanium and zirconium silicates, in each column octahedra with the "light" cations (Ca, Mg) alternate with the octahedra containing "colored" cations (Ti, Zr, Mn, Re). It is precisely in this geometric manner that the structure of cuspidine, $Ca_4[Si_2O_7]F_2$, is "identical" with the structure of lovenite ($CaZrNaFe[Si_2O_7]OF$), wöhlerite, rosenbuschite, seidozerite ($Na_4Zr_2TiMn[Si_2O_7]_2O_2F_2$) and a number of other minerals [10]. Similar "identity" is apparent between spurrite and (meta) lomonosovite although it is not possible as yet to give a completely solved structure of the second mineral.

In comparing the formulas of lomonosovite and spurrite we shortened the ramsayite part of the lomonosovite formula and extended the formula of its side chain, thus changing the metaphosphate radical PO_3 to the orthophosphate PO_4 without corresponding valence compensation in the side chain. This is probably correct from the geochemical point of view. In the spurrite structure all Si-tetrahedra are independent SiO_4 groups and so are the CO_3 triangles. But the existence of an independent PO_3 radical with trivalent P (not the flat triangular group but the group of the "umbrella" type) is still known only in the chemical laboratory and is apparently impossible in natural minerals (unlike SO_3) in which the metaphosphates may form only in the same way as metasilicates, i.e. by condensation of the PO_4 tetrahedra (with pentavalent P) into chains and rings. These structures are absent from spurrite and should not occur in lomonosovite. Moreover, the new analyses of metalomonosovite show that it contains more water than lomonosovite (ortholomonosovite). The problem of valence compensation in the basic molecule follows from an analogy with seidozerite. The strong and characteristic pleochroism of this mineral is explained by the fact that almost one half of its titanium is trivalent. (Meta) lomonosovite also has strong and characteristic pleochroism (unlike ramsayite. Semenov [7]) and must also contain trivalent titanium. Thus the "ramsayite" part of the lomonosovite molecule should be written $Na_2O \cdot Ti_2O_3 \cdot 2SiO_2 = Na_2Ti_2Si_2O_8$, and when the normal (neutral reaction with methyl orange) Na phosphate NaH_2PO_4 is added, we get the ideal formula of metalomonosovite $Na_2Ti_2^{(3)}Si_2O_8 \cdot NaH_2PO_4$.*

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*In reference [10] the stability of tervalent titanium on seidozerite (and in lovenite and other Zr and Ti silicates) was explained by the presence of large amounts of manganese in these minerals insuring equilibrium $Ti^{4+} + Mn^{2+} \rightleftarrows Ti^{3+} + Mn^{3+} + (Mn^{4+})$. Semenov's formula of lomonosovite cited above indicates that manganese is an essential component of the mineral.

The presence of tervalent titanium in lomonosovite answers the question how Ti may alternate with Na in the polyhedra represented in Fig. 1, which are larger than the octahedra. This is difficult to imagine if Ti^{4+} (0.64Å) alternates with Na (0.96Å) but simple if Ti^{3+} (0.83Å) alternates with Na.

What, then, is lomonosovite (ortholomonosovite) in which chemical analysis reveals additional Na_2O and much smaller H_2O content than in metalomonosovite although the unit cell is the same in both? This question will be answered if we ascribe to lomonosovite those structural characteristics of spurrite which determine its cleavage and particularly the mechanism of twinning and polysynthetic intergrowth. It will be seen from Fig. 1 that in spurrite cations Ca IV and Ca V are surrounded by coordination polyhedra having the shape of incomplete octahedra lacking one vertex. If these octahedra are completed in some layers (on the "cleavage" plane), then as Fig. 2 shows, the corresponding layer in the spurrite structure becomes a twinning seam, a concrete symmetry plane or twinning plane which is also a cleavage plane. From the stoichiometric point of view these will be introduced, extra layers, not considered in general in the calculation of the formula. It is easy to see from Fig. 2 that if the details of the spurrite structure detailed on the left side of Fig. 1 are taken into account the formula of such a twinning layer will be balanced: Ca_2SiO_4 (one octahedron each of Ca IV and Ca V plus two Ca I polyhedra per two SiO_4 tetrahedra).

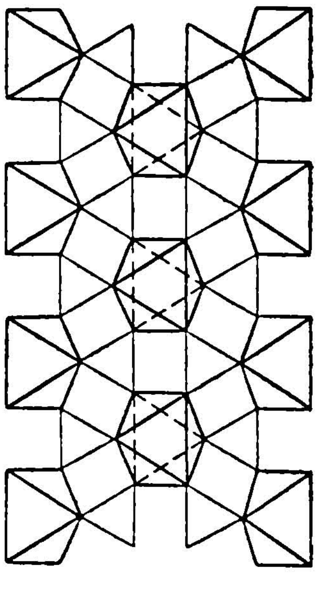

Fig. 2. Mechanism of polysynthetic twinning in spurrite.

In the twinning plane the incomplete Ca IV octahedra become complete. On each side of the complete octahedra lie the Ca IV octahedra lacking one vertex. They are typical of spurrite and belong to the individuals of the twin.

It is very significant, as convincingly shown by grossularite garnets (plazolite, hibschite) and by olivines [11], that in the orthosilicates a considerable part of the independent tetrahedra $[SiO_4]^{4-}$ may be replaced by "empty" tetrahedra composed of four hydroxyls with the same volume and charge, i.e. by $[(OH)_4]^{4-}$. It is much easier to prepare synthetic garnets composed entirely of the $[(OH)_4]^{4-}$ groups (isometric Ca-hydroaluminates) than grossularites, $Ca_3Al_2[SiO_4]_{3-x}(OH)_{4x}$, in which only a part of the SiO_4 groups is replaced by $(OH)_4$ [11].

In other words, the twinning seams in spurrite may be produced by "foreign" layers with the formula Ca_2SiO_4 or with the composition of portlandite, $Ca_2(OH)_4$ but completely analogous in structure to the layered hydroxide. It is known that the nonstoichiometric layers of portlandite are very characteristic of the simplest cement silicate, hillebrandite [11].

All that has been said of spurrite can be applied mutatis mutandis to lomonosovite. It can be concluded from the identity between the lomonosovite and metalomonosovite unit cells, on the one hand, and their close similarity to the spurrite cell, on the other, that the true lomonosivite is metalomonosovite, $Na_2Ti_2Si_2O_8 \cdot NaH_2PO_4 = Na_2Ti_2Si_2O_8 \cdot Na_2PO_2(OH)_2$, with its strong tendency [1, 2] to form twins and polysynthetic intergrowths by adding layers which must be given a (neutral) formula, Na_2HPO_4 or $Na_2H_2SiO_4$. The second formula is more probable, because the availability of the necessary amounts of $[PO_4]$ ions is not likely. However, as chemical analyses show, the most characteristic introduced layers in lomonosovite are those containing quadrivalent Mn, which stabilizes tervalent Ti.* In its neutral form the introduced layer should be $Mn_2O_2SiO_4$, but each of the two extra oxygen ions (in excess of four) takes two H ions from the basic structure, freeing lomonosovite from the water molecules.

The paradox that metalomonosovite and lomonosovite have the same unit cell but different formulas can be resolved only after considering the basic difficulty of X-ray analysis familiar only to specialists in structural analysis. Usually the beginning of analysis is much delayed because of the difficulty of finding untwinned crystals (even though they measure only a fraction of a millimeter). Often the desired crystal is found after examining several hundred crystals under a binocular microscope and checking by the X-ray pattern. A special section of modern X-ray analysis is devoted to the methods of overcoming the difficulties presented by twinning. The necessity for selecting untwinned crystals is completely ignored in preparing "pure" material for chemical analysis. Considering the characteristic tendency of lomonosovite and metalomonosovite toward twinning and polysynthetic intergrowths [1, 2] which both introduce additional layers, it is not surprising that, despite the chemical (but not structural) purity of the selected material, chemical analysis shows an excess of Na_2O (excess of SiO_2 is usually ignored),** which after rounding off the analytical figures changes metalomonosovite into ortholomonosovite to puzzle the structural analyst.

The twinning seams are the weakest zones in (ortho) lomonosovite and are easily leached out, the leaching affecting not only the introduced layers but also the adjacent phosphatic layers (Fig. 2), and according to the formula given above the dissolved material must be $NaH_2SiO_4 + Na_2HPO_4$. After substracting SiO_2, which is insoluble and does not attract attention, the analyst finds that the $Na_2O{:}P_2O_5$ ratio in the water extract is 3:1, but this does not prove that Na_2PO_4 has been leached from the whole mass of lomonosovite and that it has been converted to murmanite.

Unfortunately we have no complete (X-ray) geometry of murmanite, but two of its parameters are identical with those of lomonosovite, a = 5.38Å and b = 7.03Å (Kh. S. Mamedov). In the Nb variety, epistolite, a = 5.41Å and b = 7.12Å (A. V. Nikitin). Parameter c is given by Mamedov with some reservations as 12.34Å.*** If this is confirmed, then, since in lomonosovite

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*The appearance of pink color when murmanite is dissolved indicates, according to Borneman, that a part of its Mn has a high oxidation state and appears as Mn_2O_3 [4].

**Note, however, in reference [3] a certain surprise expressed by Borneman concerning the increase in SiO_2 content in lomonosovite with increasing P_2O_5 content.

***The same value for c (smaller than in lomonosovite) is indicated by comparison of powder photographs of lomonosovite and murmanite-epistolite (N.I. Organova).

c = 14.50Å, murmanite could be regarded as "shortened" lomonosovite and explained very simply by the argument used in explaining the nature of ortholomonosovite. If the latter is a result of increased symmetry caused by introduction into metalomonosovite of a third layer of octahedra symmetrical with the already existing complete octahedra, then the symmetry of murmanite could be achieved by coalescence into one layer of the two octahedral layers characteristic of the metalomonosovite structure.

But as a result of recent analyses [7] both chemists and mineralogists are unanimous in asserting that in passing from lomonosovite to murmanite, in spite of the removal of phosphorus or of considerable reduction of its content, the total number of oxygen ions increases. The removed tetrahedron $PO_4[PO_2(OH)_2]$ is replaced by about eight hydroxyl ions, and further difference between metalomonosovite and murmanite lies in the removal not of NaH_2PO_4 from the side chain but of Na_3PO_4; i.e., two Na ions are removed from the basic ramsayite molecule.

Inasmuch as the volume of a mineral is determined largely by the volume of its anions, particularly of the oxygen and hydroxyl ions, it would seem that there could be no diminution of the murmanite cell as compared with the (meta) lomonosovite cell, but on the contrary the murmanite cell would be called upon to accommodate additional oxygen ions (of the hydroxyls).

Considering the large size of the majority of the cation polyhedra in the spurrite structure shown in Fig. 1, we see that the extra oxygen ions are easily disposed of in the form of hydronium cations $(OH_3)^+$, which substitute in the large polyhedra for two Na^+ ions, one from the "basic" ramsayite structure and the other from the "side chain."* This does not increase the dimensions of the cell, and the preservation of its normal lomonosovite size is achieved by the same scheme as in hydrogrossularite; i.e., when phosphoric acid becomes deficient (by leaching) the PO_4 tetrahedra are replaced by empty $(OH)_4$ tetrahedra. To refine the valence relations, consider that the $[PO_2(OH)_2]$ tetrahedron in metalomonosovite has only one negative charge while the tetrahedron $(OH)_4{}^{4-}$ has four negative charges. The necessary compensation, three extra positive charges, may be introduced by adding H^+ protons to the silicon-oxygen tetrahedra, i.e., by replacement of the oxygen vertices in a part of the SiO_4 tetrahedra by OH, which, of course, does not contribute to the stability of murmanite. More stable forms are obtained by the return of Ti to the quadrivalent state or even by replacement of one of the two Ti^{4+} by Nb^{5+}, which gives us a more stable variety of murmanite, Nb-epistolite, for which there is no lomonosovite analogue. Because of the normal oxidation state of the high valence ions in the murmanite-epistolite groups, these minerals have considerably weaker pleochroism, etc. Thus the ideal structural formulas must be:

(мета) lomonosovite $Na_2\,Ti_2^{(3)}\,Si_2O_8 \cdot NaH_2PO_4$

(мета!) murmanite $(OH_3)_2\,Ti_2^{(4)}\,Si_2O_7OH \cdot Na\,(OH)_4$

(мета!) epistolite $(OH_3)_2\,TiNbSi_2O_8 \cdot Na\,(OH)_4$.

All three minerals are related to the simplest cement silicate, spurrite—$Ca_2Ca_2Si_2O_8 \cdot CaCO_3$.

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*Borneman also suggests this [3].

Good though these formulas look, it is obvious that the last two cannot represent stable forms, for they contain both OH_3 and OH (even if they are placed at the opposite ends of the formula). The thermal curve of metamurmanite is characterized by peaks caused by liberation of low temperature water. The powder photographs showing decrease in parameter c in the murmanite-epistolite group require removal of the components of water and evidently as an entire layer. But four OH groups cannot be compensated even by two OH_3 ions. Moreover, even the "shortened" murmanite and epistolite retain water. Evidently the simplest solution lies in removal of the whole $Ti(OH)_4$ group, or if Ti is tervalent, of the $NaTi(OH)_4$ group.*

Although we began this article by enclosing in quotation marks the terms "basic ramsayite part of the molecule" and "side chain" or phosphatic part of lomonosovite, thus underlining the fact that these concepts are obsolete in modern structural chemistry based on X-ray analysis, we kept these terms to the end, freed them from quotation marks and very likely failed to disabuse any one concerning them. We cited a statement by Borneman [3], one of our most eminent analytical chemists, that there is no strong "chemical bond between the two parts of lomonosovite." In the same work [3] Borneman speaks of the impossibility of solid solutions between the two component parts of lomonosovite because, as we understand it now, solid solution means isomorphous entry into a single crystal of molecules necessarily possessing identical "structures," i.e., identical pattern of spatial distribution of equal numbers of similar diadochic atoms of equal size. It is evident that there is no such relation between the ramsayite and the phosphatic parts of lomonosovite.

However, in the same work, Borneman speaks of a phenomenon which a modern crystal chemist might call interstitial solid solution, although Borneman avoids using this term. He speaks of zeolites and zeolite-like substances in which the basic part is a tectosilicate structure with a strong three-dimensional (aluminosilicate) framework and large voids accommodating foreign, by no means isomorphic molecules such as water, ammonia and the simplest organic molecules of suitable volume. These "tenants" can be removed or exchanged for others without changing the basic chemical nature or the structure of the host zeolite. It is well known [10] that natural and synthetic zeolites are much used now in chemical technology under the name of "molecular sieves" [11]. These compounds include not only the zeolitic substances with closed voids familiar to mineralogists, such as sodalite-lazurite, chabazite and other minerals discussed by Borneman, but also layered and platy zeolitic materials of the heulandite and especially montmorillonite type in which the foreign molecules are captured and released as whole layers theoretically infinite in two dimensions. The stoichiometric proportions which may exist to some extent in zeolitic materials with voids of finite volume are completely absent from the solid solutions of the second type.

The concept of lomonosovite-murmanite as a structure composed of basic layers of metalomonosovite, an analogue of spurrite (Fig. 1) alternating with nonperiodic $Na_2HPO_4—Ca_2SiO_4$ (Fig. 2) layers of variable thickness is very similar although not identical with the concept of layered

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*The fresh perfect cleavage surface of murmanite is violet and has metallic luster, but on weathering it becomes pink or silver with a yellowish or pinkish tinge [4].

zeolites, it destroys stoichiometry but still demands structural identity in two dimensions between the introduced layers and the expandable lattice of the host mineral.

The crystal structure of Na_2HPO_4 is still unknown, because sodium phosphates are hygroscopic and are usually represented by hydrates, but very probably it is quite similar to the structure of Ca_2SiO_4 or larnite (and of its almost undistinguishable modification—bredigite). From the diagram of the larnite structure (Fig. 3) it can easily be seen that, except for a slight flattening, the structure reproduces almost exactly the "theoretical" structure of a twin seam connecting (in polysynthetic intergrowths) the layers of metalomonosovite and ortholomonosovite. The two "essential" parameters of larnite are very near the two parameters of spurrite and lomonosovite: a = 5.48Å and b = 6.76Å. The larnite structure was deciphered seven years before that of spurrite [12]. Larnite shows such characteristic features of spurrite as the presence, in addition to the Ca columns, of the larger polyhedra (Ca I in Fig. 1) located exactly beneath each SiO_4 tetrahedron (along the y axis).

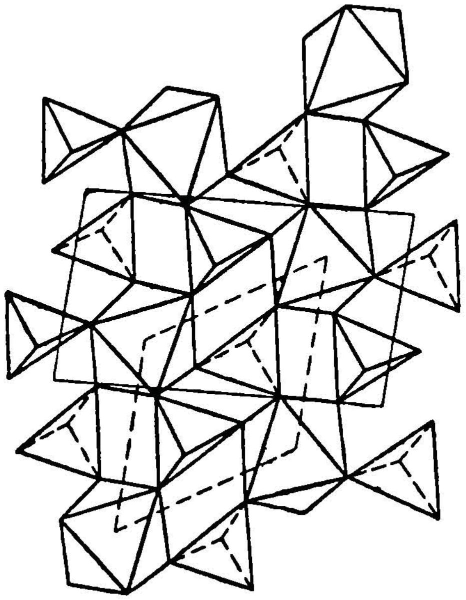

Fig. 3. Crystal structure of larnite.

Thus the nonstoichiometric entry of the Ca_2SiO_4 (Na_2HPO_4) layers discussed above is completely justified by analysis of appropriate structures.* At one time [13] the concept of nonstoichiometric layers (but similar in two dimensions) was used by one of the authors to explain the cleavage in corundum. At first these layers were supposed to have the composition of diaspore, but direct observation (Frank-Kamenetskii [11]) showed that they are muscovite sheets in which the layer of Al-octahedra is structurally identical with the "corundum" pattern [13].

In corundum these layers are very few and do not in any way change the formula Al_2O_3, but in lomonosovite they are very numerous and give the impression that there is a real difference even between the formulas of metalomonosovite and ortholomonosovite.

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*Note the high specific gravity of larnite (3.4) and the considerable difference in the specific gravity of murmanite (2.8) and lomonosovite (3.2).

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