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CRYSTAL CHEMISTRY OF MILARITE-TYPE MINERALS

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ABSTRACT

Milarite-type minerals have the general formula

$$A_2{}^{[6]}B_2{}^{[9]}C^{[12]}D^{[18]}T(2)_3{}^{[4]}T(1)_{12}{}^{[4]}O_{30},$$

where the A-position can be occupied by Mg, Ca, Fe, Ti, Zr; the B-position by K, Na, Mg, H_2O (or H_3O); the C-position by K, Na, Ca, Ba; the D-position by H_2O; the $T(2)$-position by Al, Be, Mg, Fe, Li; and the $T(1)$-position by Si and Al; the coordination number is given in bracketed superscripts. The B-position which previously had not been recognized as being occupied in milarite-type minerals is 9-coordinated with three short and six longer bonds to the surrounding oxygen atoms. This position must be occupied in all milarite-type compounds in which the number of non-tetrahedral cations and/or H_2O, relative to the 15 tetrahedral cations, is greater than three. If this number is greater than five (as in armenite) the 18-coordinated sites (in 0, 0, 0) in the center of the $Si_{12}O_{30}$ double hexagonal ring has to be occupied. In yagiite, merrihueite, roedderite, and "Ca-cordierite" part of the Mg (or Fe^{2+}) must be in tetrahedral coordination in the $T(2)$-sites. The different groups of milarite-type minerals can be formally derived from a proto-milarite, $2MgO\cdot 2Al_2O_3\cdot 11SiO_2$ or $Mg_2Al_3Si_{11}AlO_{30}$, by simple cationic substitutions which alter the Al/Si ratio and which progressively fill the B- and C-positions.

INTRODUCTION

Milarite was found first in the Val Giuf, Graubünden, Switzerland and was described by Kenngott (1870). Since then several other minerals isostructural with milarite have been characterized (see Table 2). Milarite group minerals crystallize in space group $P6/m\ 2/c\ 2/c$ with approximate cell parameters of $a \approx 10$ Å and $c \approx 14$ Å. The crystal-chemical formula of the milarite-type minerals as deduced by Ito *et al.* (1952) and adopted by many subsequent workers is $A_2{}^{[6]}C^{[12]}T(2)_3{}^{[4]}T(1)_{12}{}^{[4]}O_{30}\cdot H_2O$, where the coordination numbers of the cations are indicated as superscripts in square brackets. The most prominent feature of the structure is a hexagonal double ring of silicate tetrahedra, of composition $Si_{12}O_{30}$. These rings are joined by tetrahedrally coordinated cations in sites $T(2)$ into a three-dimensional tetrahedral framework and further connected by octahedrally coordinated cations in the A-sites. The double rings are stacked in the c-direction thus forming the 12-coordinated C-site between them. The water molecules found in some milarite-type minerals have been assumed to be located inside the double hexagonal rings in large voids, with 18-coordination.

The synthetic compound $K_2Mg_5Si_{12}O_{30}$ (Roedder, 1951) contains twice as much potassium as can be accommodated in the *C*-sites. A structure determination of $K_2Mg_3Si_{12}O_{30}$ (Khan *et al.*, 1971) has shown that this excess potassium is located in 9-coordinated sites (with three short K-O bonds, and six longer ones) which previously had not been known to be occupied in milarite-type structures. The general formula for milarite group minerals is therefore:

$$A_2{}^{[6]}B_2{}^{[9]}C^{[12]}D^{[18]}T(2)_3{}^{[4]}T(1)_{12}{}^{[4]}O_{30}.$$

Thus, the six different sites (see Table 1) may contain up to 21 cations and/or water molecules per 30 oxygen atoms in the formula unit. Since the radii of the cations which can be accommodated in these different sites range from 0.4 Å (for Si^{4+}) up to 1.7 Å (for K^+ and Ba^{2+}), and since the Mg and Fe atoms can enter into both tetrahedral and octahedral coordinations (Khan *et al.*, 1971) the chemical variability of milarite-type minerals is large.

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In Table 2 we show all known species of the milarite group with the cations and water molecules distributed among the six positions described above. Included in this list are two "calcium-rich cordierites", mentioned by Miyashiro (1956) as being probably of the milarite-type. The assignment of the atomic species to the different

Table 1. Cation and/or water positions in milarite-type structures (C.N. = coordination number). The list of elements occupying the different positions includes those listed in Table 2, as well as trace elements identified by Černý (1968) in amounts larger than 0.01wt% in milarites from Věžná.

Symbol	Equipoint	C.N.	Site Symmetry	Possible Occupants
T(1)	24(m)	4	1 - C_i	Si,Aℓ,Mg
T(2)	6(f)	4	222 - D_2	Aℓ,Be,Mg,Fe,Li,Ti,B
A	4(c)	6	32 - D_3	Mg,Ca,Fe,Ti,Zr,Mn,Sr,Co,Nb
B	4(d)	9(=3+6)	$\bar{6}$ - S_3	K,Na,H_2O,(H_3O?),Mg,Fe
C	2(a)	12	622 - D_6	K,Na,Ca,Ba,Y,Yb,Pb
D	2(b)	18(=12+6)	6/m C_6^h	(H_2O?)

Table 2. Distribution of chemical elements over the different sites in milarite-type minerals. All atom fractions are multiplied by a factor of 100. The sum of atoms exceeds in several cases the possible maximum occupancy of a given site. Such deviations are believed to be within experimental error of the chemical analyses.

Mineral	Locality	$A^{[6]}_{200}$	$B^{[9]}_{200}$	$C^{[12]}_{100}$	$D^{[18]}_{100}$	$T(2)_{300}$	$T(1)_{1200}$	Reference for Analysis
roedderite	Indarch	$Mg_{181}Fe^{2+}_{27}$	Na_{100}	$K_{69}Na_{30}$		Mg_{300}	$Si_{1188}A\ell_7Mg_5$	Fuchs et al, 1966
roedderite	Wichita County	$Mg_{180}Fe^{2+}_{10}$	Na_{150}	$K_{80}Na_{20}$		Mg_{300}	$Si_{1150}A\ell_{50}$	Olsen, 1967
$Na_2O\cdot 5MgO\cdot 12SiO_2$	synthetic	Mg_{200}	Na_{100}	Na_{100}		Mg_{300}	Si_{1200}	Seifert & Schreyer, 1969
merrihueite	Mezö-Madras	Fe_{200}	Na_{76}	$K_{94}Ca_{12}$		$Fe_{185}Mg_{127}$	Si_{1200}	Dodd et al, 1965
$K_2O\cdot 5MgO\cdot 12SiO_2$	synthetic	Mg_{200}	K_{100}	K_{100}		Mg_{300}	Si_{1200}	Khan et al, 1971
sodgianite	Central Asia	$Zr_{83}Fe^{3+}_{42}Ti_{38}Fe^{2+}_{18}$	$Na_{90}K_{10}$	K_{100}		$Li_{261}A\ell_{21}Fe^{3+}_{18}$	Si_{1200}	Dusmatov et al, 1968
milarite	Val Giuf	Ca_{209}	$(H_2O)_{57}Na_{15}$	K_{105}		$Be_{210}A\ell_{92}$	Si_{1198}	Palache, 1931
milarite	Piz Ault	Ca_{206}	$(H_2O)_{68}Na_{38}K_{15}$	K_{100}		$Be_{172}A\ell_{86}Fe_{19}$	Si_{1194}	Hügi, 1956
milarite	Kola Peninsula	Ca_{209}	$(H_2O)_{70}Na_{10}$	K_{103}		$Be_{145}A\ell_{153}$	Si_{1202}	Sosedko & Telesheva, 1962
milarite	Kazakhstan	$Ca_{192}Mn_6Fe_2$	$(H_2O)_{77}$	$K_{95}Na_5$		$Be_{226}A\ell_{73}$	Si_{1166}	Chistyakova et al, 1964
milarite	Central Asia	$Ca_{193}Sr_2$	$(H_2O)_{76}Na_{28}$	K_{98}		$Be_{186}A\ell_{114}$	$Si_{1059}A\ell_{141}$	Iovcheva et al, 1966
osumilite	Sakkabira	$Mg_{93}Fe^{2+}_{96}Mn_{11}$		$K_{65}Na_{33}Ca_2$		$A\ell_{263}Fe^{3+}_{24}$	$Si_{1038}A_{162}$	Olsen & Bunch, 1970
osumilite	Monte Arci	$Fe^{2+}_{118}Mg_{72}Mn_{10}$		$K_{66}Na_{12}Ca_3$		$A\ell_{272}Fe^{3+}_{22}$	$Si_{1034}A\ell_{166}$	Olsen & Bunch, 1970
osumilite	Oregon	$Fe^{2+}_{98}Mg_{88}Mn_{14}$		$K_{65}Na_8Ca_2$		$A\ell_{275}Fe^{3+}_{19}$	$Si_{1041}A\ell_{159}$	Olsen & Bunch, 1970
$K_2O\cdot 4MgO\cdot 5A\ell_2O_3\cdot 20SiO_2$	synthetic	Mg_{200}		K_{100}		$A\ell_{300}$	$Si_{1000}A\ell_{200}$	Schreyer & Seifert, 1967
yagiite	Colomera	Mg_{200}	Na_{50}	$Na_{70}K_{30}$		$A\ell_{196}Mg_{60}Fe_{34}Ti_{10}$	$Si_{1020}A\ell_{180}$	Bunch & Fuchs, 1969
"ca-cordierite"	Laacher See	$Mg_{126}Fe^{2+}_{64}Mn_{10}$	$(H_2O)_{92}Fe_{79}$	Ca_{95}		$A\ell_{132}Mg_{116}Fe_{52}$	$Si_{865}A\ell_{335}$	Miyashiro, 1956
"Ca-cordierite"	Celebes	$Fe^{2+}_{167}Fe^{3+}_{38}$	$Mg_{15}(H_2O)_3$	Ca_{80}		$A\ell_{306}$	$Si_{855}A\ell_{345}$	Miyashiro, 1956
armenite	Kongsberg	Ca_{203}	$(H_2O)_{191}Na_6K_3$	Ba_{95}	$(H_2O)_{25}$	$A\ell_{300}$	$Si_{880}A\ell_{316}$	Neumann, 1941
$4MgO\cdot 4A\ell_2O_3\cdot 7SiO_2$?	synthetic	Mg_{200}	Mg_{200}			$A\ell_{300}$	$Si_{700}A\ell_{500}$	Schreyer & Schairer, 1962

sites is based on the published chemical analyses and on the sizes of the ions in question. The water molecules are assumed by us to be located in the 9-coordinated *B*-sites. Our reasons for this assignment are:

1. The H_2O molecule is of approximately the same size as the potassium ion which in $K_2Mg_5Si_{12}O_{30}$ prefers the *B*-sites over the 18-coordinated *D*-sites (which would be much too large for either K or H_2O).
2. Upon heating to 1000°C, and after losing H_2O, the *c* cell constant of milarite increases by approximately 0.06Å, while the *a* cell constant decreases by about 0.04Å (Černý, private communication, 1971; Iovcheva *et al.*, 1966). If the water molecules were located in the large *D*-sites as suggested by Iovcheva *et al.*, their loss should not affect the cell constants in any measurable degree. If they are, however, in the *B*-positions the changes in cell parameters could be interpreted as the result of an adjustment of the oxygen atoms around the *B*-site taking place after the removal of the H_2O (compare the discussion of the cell parameters of $K_2Mg_5Si_{12}O_{30}$ and osumilite in Khan *et al.*, 1971).

This assignment is at variance with the findings of Ito *et al.* (1952). However, their structure determination is of low accuracy ($R = 0.30$ for 105 observed structure factors) and cannot be taken as solid proof. Bakakin and Soloveva (1966) report in an abstract that they found in milarite "water molecules on threefold axes near the face of the Ca flattening octahedron". Their statement may possibly refer to what we call the *B*-sites, but since they do not give any further details, its meaning is uncertain. The presence of water in milarite-type minerals has been the subject of much discussion. Water is well authenticated in milarite and armenite only. Miyashiro (1956) assumed water to be present in osumilite. However, Olsen and Bunch (1970), who carefully analyzed osumilite from its three known occurrences, including the type locality, concluded that water is not a constituent of osumilite. Likewise there is no clear indication for the presence of water in merrihueite, roedderite, yagiite, or in the synthetic forms ($K_2Mg_5Si_{12}O_{30}$ and synthetic osumilite; Schreyer and Seifert, 1967).

The milarite group minerals may be divided into four subgroups on the basis of their Al contents. The roedderite subgroup is virtually Al-free, milarite contains between one and three Al atoms per formula unit, osumilite has five, and armenite as well as the "Ca-cordierites" of Miyashiro (1956) have six Al atoms. In all four groups, the octahedrally coordinated *A*-position is occupied by a divalent cation. An exception is sodgianite, where this site appears to be occupied by 3- and 4-valent cations (sodgianite has been recognized by Strunz (1970) to belong to the milarite-type minerals). The *B*-sites are usually populated by alkalis and water and occupancy of this position is very variable between the groups. It is most fully occupied in armenite. The roedderite group contains up to 1.5 atoms in this position. In the osumilites the *B*-site appears to be empty. Brown and Gibbs (1969), in their crystal structure determination, do not report to have found any atoms to be located there. The composition of the Ca-cordierites requires that divalent cations, either Mg or Fe, are in this position, thus making the coordination of these cations rather unusual. However, one should keep in mind that an eight-coordination for Mg is commonly found in garnets.

Because water molecules and alkali ions seem to replace each other in the *B*-sites of milarite we actually may not be dealing with water molecules but instead with hydronium ions (H_3O^+). If this is the case we would expect the oxygen atom of the hydronium ion not to be exactly in the *B*-position (1/3 2/3 0), but slightly displaced from it along the threefold axis (1/3 2/3 *z*), so that H_3O could form three hydrogen bonds in a trigonally pyramidal configuration to the three neighboring O(1) oxygen atoms (Khan *et al.*, 1971).

The *C*-position is occupied by alkali ions in all minerals but armenite, where it is filled by barium. The *T*(2)-sites are marked by considerable variation. They may be occupied by Mg, Fe^{2+}, Fe^{3+}, Be, Al, or Li. Magnesium in tetrahedral coordination is very rare in silicates. In the system K_2O-MgO-SiO however, there are two phases in which Mg is 4-coordinated: Mg-merrihueite, $K_2Mg_5Si_{12}O_{30}$ (Khan *et al.*, 1971), where the four-coordination has been substantiated by the structure determination (with a bond length Mg-O of 1.955Å), and in $K_2O \cdot MgO \cdot 3SiO_2$, which according to Roedder (1951) has an X-ray powder pattern strongly resembling the pattern of kalsilite ($KAlSiO_4$). Consequently, $K_2O \cdot MgO \cdot 3SiO_2$ may be formulated as $K(Mg_{0.5}Si_{0.5})SiO_4$, with Mg and Si substituting for Al.

Schreyer and Schairer (1962) have shown that alkali-free milarite-type phases can be synthesized within the system MgO-Al_2O_3-SiO_2. As these authors recognized, the milarite structure theoretically could form from the composition $2MgO \cdot 2Al_2O_3 \cdot 11SiO_2$, *i.e.*, only the *A*, *T*(2), and *T*(1) positions would be filled. If we consider 2:2:11 to represent a "proto"-milarite composition, the main groups of milarite minerals may be derived by simple cationic substitutions. This is shown schematically in Table 3. As can be seen, numerous variations are possible, simply by altering the Al/Si ratio and consequently the

Table 3. Formal derivation of milarite-type minerals from the proto-milarite-type.

Replacement	$A_2^{[6]}$	$B_2^{[9]}$	$C^{[12]}$	$D^{[18]}$	T(2)	T(1)	O_{30}	Type
(starting composition)	M_2^{2+}				M_3^{3+}	$Si_{11}A\ell$	O_{30}	proto-milarite
$(Si+2M^{1+}+3M^{2+})$ for $4M^{3+}$	M_2^{2+}	M^{1+}	M^{1+}		M_3^{2+}	Si_{12}	O_{30}	roedderite
$(Si+5M^{1+}+M^{4+}+M^{3+})$ for $(2M^{2+}+3M^{3+}+A\ell^{3+})$	$M^{4+}+M^{3+}$	M^{1+}	M^{1+}		M_3^{1+}	Si_{12}	O_{30}	sodgianite
$(Si+M^{1+}+2M^{2+})$ for $3M^{3+}$	M_2^{2+}	(H_2O)	M^{1+}		$M_2^{2+}M^{3+}$	Si_{12}	O_{30}	milarite
$(A\ell+M^{1+})$ for Si	M_2^{2+}		M^{1+}		M_3^{3+}	$Si_{10}A\ell_2$	O_{30}	osumilite
$(2A\ell+M^{2+})$ for 2Si	M_2^{2+}	(H_2O)	M^{2+}	$(H_2O)_x$	M_3^{3+}	$Si_9A\ell_3$	O_{30}	armenite

alkali content. It should be mentioned, however, that this starting composition does not produce a milarite-type phase under the experimental conditions investigated by Schreyer and Schairer (1962).

Olsen and Bunch (1970) studied the compositions of natural osumilites and found that the K/(K + Na + Ca) ratio varies with the occupancy of the *C*-position. Actually their diagram can be extended to include the contents of the *B*-position as well, thus allowing the addition of yagiite (Fig. 1). In Figure 2, we have plotted total Al + Fe^{3+} against Σ(K + Na + Ca). It appears that as occupancy of the *B*-

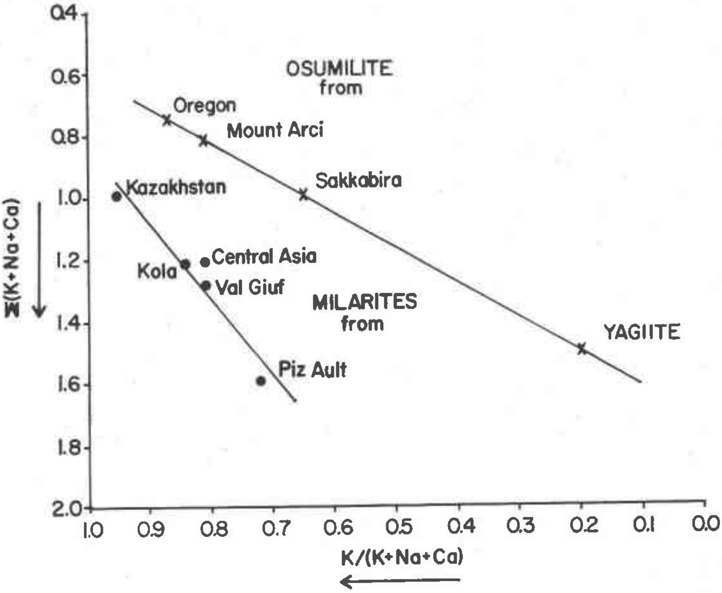

FIG. 1. Ratio K/(K + Na + Ca) versus total K + Na + Ca located in the *B*- and *C*-positions in osumilite and yagiite (crosses) and milarites (circles).

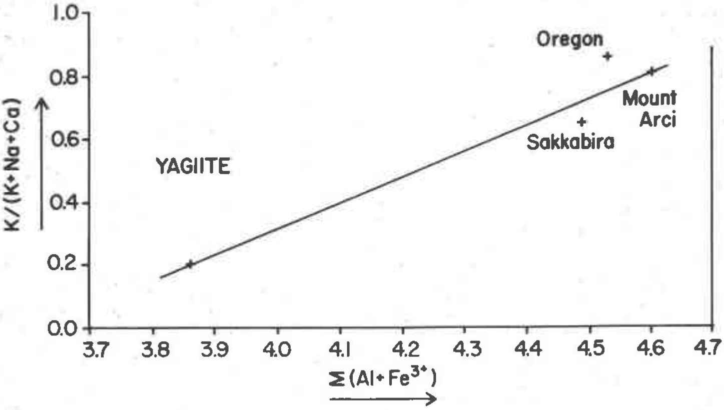

FIG. 2. Total Al + Fe^{3+} versus ratio K/(K + Na + Ca) in osumilite and yagiite.

and C-sites increases, the Al + Fe^{3+} content decreases and the sodium content increases (see Fig. 1). Thus, natural osumilites and yagiite are members of a solid solution series, related by the substitution $K + Al \rightleftharpoons 2Na + Mg$. Idealized end members of this series are $KMg_2Al_3(Si_{10}Al_2)O_{30}$ and $Na_2Mg_2(Al_2Mg)(Si_{10}Al_2)O_{30}$. The sodic end member may be related to roedderite through the substitution $4Al = 2Si + 2Mg$. However, intermediate members of the series are lacking. A similar relationship between the K/K + Na + Ca ratio and total cation occupancy of the $B + C$ sites is found for milarites as well (Fig. 1). The Ca represents the excess over 2 in the A site. However, there are no corresponding changes in the abundances of other cations as was found for osumilites. Possibly this means that variation in K/K + Na + Ca ratio is correlated with the postulated hydronium content of the B-site. Unfortunately, there is no direct evidence bearing on this possibility.

OCCURRENCE

Milarite-type minerals are typically found in low pressure environments. Milarite itself as well as armenite occurs as a fissure-filling or pegmatitic mineral. Osumilite is found only in volcanic rocks, where it occurs within vesicles as well as in the groundmass. Milarite-type phases found in synthetic systems usually occur at low pressures. Roedderite, $Na_2Mg_5Si_{12}O_{30}$, was found as a decomposition product of richterite only at pressures below 50 bars $P(H_2O)$ (Forbes, 1971). Iron-bearing roedderite, again the decomposition product of a sodic amphibole, occurs only at pressures below 500 bars $P(H_2O)$ (Ernst, 1960). Seifert and Schreyer (1969) have synthesized Mg-merriheuite at pressures up to 32.5 Kbar; however, from consideration of the phase equilibria of this phase, they conclude that this compound is restricted to low pressure environments.

While field relations and experimental data indicate that milarite-minerals form under low-pressure conditions, it is surprising to note that the molar volume change for the breakdown of osumilite under volcanic conditions (Schreyer and Seifert, 1967)

$$\underset{\substack{\text{osumilite}\\ 378.8\ \text{cm}^3/\text{mole}}}{\mathrm{KMg_2Al_3Si_{10}Al_2O_{30}}} = \underset{\substack{\text{sanidine}\\ 108.98}}{\mathrm{KAlSi_3O_8}} + \underset{\substack{\text{cordierite}\\ 232.81}}{\mathrm{Mg_2Al_4Si_5O_{18}}} + \underset{\substack{\alpha\text{-cristobalite}\\ 25.74}}{\mathrm{2SiO_2}}$$

is $+14.5$ cm^3/mole, which suggests that osumilite in this instance would be stable at higher pressures. If orthoclase and α-quartz are used, instead of sanidine and cristobalite, ΔV is reduced to 8.4 cm^3/mole but it is still positive. It is possible that changes in the phase compositions and/or nature of the phases might alter the sign of the volume change. The molar volume data for osumilite were obtained from Schreyer and Seifert (1967), for the other phases the compilation of Robie *et al.* (1966) was used.

As Miyashiro (1956) has recognized, milarite-type minerals, particularly osumilite, may be quite common in nature but are mistaken for cordierite. Several instances of optically anomalous cordierites were discussed by him. Two additional reports of optically positive "cordierite" (Rutherford, 1933; Conant, 1935) support the view that osumilite may be a fairly common mineral. It might be profitable to study such occurrences further.

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Note added in proof. Professor Schreyer called to our attention a paper (Borchert and Petzenhauser, 1966) in which the synthesis of four additional members of the milarite group is reported. These could be formulated in accord with the general formula proposed by us, as

$$Mg_2{}^{[6]}Na^{[9]}Rb^{[12]}Mg_3{}^{[4]}Si_{12}O_{30},\ Mg_2{}^{[6]}Ba^{[12]}Al_3{}^{[4]}Al_3Si_9O_{30},$$

$$Mn_2{}^{[6]}Ba^{[12]}Al_3{}^{[4]}Al_3Si_9O_{30},\quad \text{and}\quad Mg_2{}^{[6]}Ba^{[12]}Fe_3{}^{[4]}Al_3Si_9O_{30}.$$

The last compound could also be $Fe_2{}^{[6]}Ba^{[12]}(Mg_2Fe)^{[4]}Al_3Si_9O_{30}$. Of particular interest is the Na-Rb compound because the authors report that they were not able to synthesize the corresponding pure Rb compound. This may mean that Rb is too large to enter the nine coordinated *B* sites, which however can be occupied by potassium ions.

BORCHERT, W., AND I. PETZENHAUSER (1966) Osumilithbildung in verschiedenen silikatischen Systemen. *Ber. Deutsch. Keram. Ges.* **43,** 572–576.