one instance and KrF_2 and XeF_2 in the other instance). However, astatine and radon follow the long rare earth series of elements and are probably more "metallic" than anticipated. Appelman *et al.* (*7*) observed the volatile halides AtI, AtBr, and AtCl in mass spectrometric experiments but were unable to observe the corresponding fluoride after treating astatine with chlorine trifluoride. Probably an involatile ionic fluoride was formed, with properties similar to radon fluoride. (AtF would be isoelectronic with RnF^+, the complex ion postulated to be present in the radon solutions.)

Practical applications may be found for the new radon solutions in the preparation of radiation sources, collection of radon for analysis, and removal of radon from the air of uranium mines. For medical use, radon "seeds" or "needles" are prepared at the present time, as in the past, by sealing radon gas into gold capillary tubes. The solutions offer new possibilities for metering and handling radon conveniently in nonvolatile form. By evaporating the solutions to dryness from metal foils, porous materials, or plastics, and either coating or encapsulating the deposits to absorb alpha particles, gamma radiation sources can probably be made in a variety of new forms. The oxidizing solutions offer possibilities for collecting radon from large volumes of air in bubble trains or other gas-liquid contact devices. In uranium mines, high concentrations of radon constitute a major health hazard to miners who are exposed to the atmospheres for long periods of time. Forced ventilation is used to reduce the radon concentrations, but alternatively, chemical scrubbers can probably now be devised which will allow the air to be purified and recirculated.

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Pyroxferroite: Stability and X-ray Crystallography of Synthetic $Ca_{0.15}Fe_{0.85}SiO_3$ Pyroxenoid

Abstract. *Synthetic $Ca_{0.15}Fe_{0.85}SiO_3$ pyroxenoid has the same (pyroxmangite) structure and very nearly the same composition as pyroxferroite, a new mineral found in Apollo 11 lunar samples. The synthetic material is not stable below pressures of approximately 10 kilobars. It appears likely that the lunar pyroxferroite has persisted in a metastable state for some billions of years.*

An unidentified yellow mineral reported by the preliminary examination team (*1*) in lunar samples from Mare Tranquillitatis is a pyroxenoid with the pyroxmangite structure, but it contains very little Mn or Mg (*2–5*). This mineral, which has been named pyroxferroite (*6*), has not been found in terrestrial rocks. We report here data on the stability and crystallography of a synthetic pyroxenoid with the pyroxmangite structure and the composition $Ca_{0.15}Fe_{0.85}SiO_3$, which is very close to that reported for some of the lunar samples.

Ferrosilite III, one of the several polymorphs of pure $FeSiO_3$ synthesized at high pressures (*7*), was shown by Burnham (*8*) to be structurally related to the pyroxenoid minerals. Indications of triclinic diffraction symmetry, and one unit cell dimension of 22.61 Å, led Burnham to hypothesize that ferrosilite III represents an extension of previously known pyroxenoid structures to one with single silicate chains having a repeat length consisting of nine silica tetrahedra—a *Neunerkette* (*9*).

The synthesis and identification of ferrosilite III fitted well with Liebau's (*10*) suggestion that repeat lengths of silicate chains in pyroxenoids are at least partially controlled by average octahedral cation size. Pure wollastonite ($CaSiO_3$) is a pyroxenoid with a repeat length of three silica tetrahedra (a *Dreierkette*) (*11*). We anticipated that pyroxenoids containing intermediate amounts of Ca and Fe might have the *Fünferketten* structure, as found in rhodonite, and that, as the iron content increases and the average octahedral cation size decreases, the pyroxenoids might have the *Siebenerketten* pyroxmangite structure.

Bowen *et al.* (*12*) synthesized pyroxenoids containing Ca and Fe ranging in composition from pure $CaSiO_3$ to approximately $Ca_{0.28}Fe_{0.72}SiO_3$ (mole ratio), but the fine grain size and limited x-ray equipment available to them precluded any identification more definite than "wollastonite solid solution." To our knowledge, no modern study has confirmed that their iron-rich pyroxenoids do in fact have the *Dreierketten* (wollastonite) structure. It was subsequently shown (*13*) that pyroxenoids more rich in iron than approximately $Ca_{0.37}Fe_{0.63}SiO_3$ are not stable at low pressures (below 2 kb), yet with increasing pressure pyroxenoids progressively more rich in iron become stable (*14*). By careful choice of pressure and temperature for each composition, we have succeeded in synthesizing a complete range of pyroxenoids from $Ca_{0.5}Fe_{0.5}SiO_3$ to $FeSiO_3$. We have found by single-crystal precession photography that a synthetic pyroxenoid of composition $Ca_{0.15}Fe_{0.85}SiO_3$ has the predicted *Siebenerketten* silicate chain configuration. Thus this phase is a manganese- and magnesium-free analog of pyroxmangite and has the same structure as well as very nearly the same composition as the lunar pyroxferroite (*2–5*).

We obtained single crystals from an experiment in which synthetic clinopyroxene of $Ca_{0.15}Fe_{0.85}SiO_3$ composition was inverted to pyroxenoid at 12.5 kb and 1175°C. Precession photographs showed patterns similar to those seen on photographs of ferrosilite III,

Table 1. Unit-cell dimensions of *Siebenerketten* pyroxenoids. Numbers in parentheses (columns 2 through 8) are least-squares standard deviations in the last significant figure. Subscript numbers are estimates only.

Pyroxenoid	*a* (Å)	*b* (Å)	*c* (Å)	α (deg)	β (deg)	γ (deg)	Volume (Å³)	Reference
Synthetic $Ca_{0.15}Fe_{0.85}SiO_3$	6.6317(3)	7.5628(5)	17.380(1)	114.309(3)	82.755(5)	94.585(9)	787.6(1)	
Lunar pyroxenoid	6.61	7.54	17.3	114.6	82.7	94.6	777.3	(*3*)
Lunar pyroxenoid	6.623(1)	7.543(2)	17.354(6)	114.34(2)	82.72(2)	94.51(2)	783.2	(*4*)
Lunar pyroxenoid	6.62	7.55	17.38	114.34	82.69	94.53	784.7	(*5*)
Pyroxmangite from iron slag	6.81	7.49	17.2	113.28	82.8	94.33	799.2	(*17*)
Pyroxmangite from Ida, S.C.	6.67_1	7.55_7	17.4_5	113.7	84.0	94.3	800.6	(*16*)

the only major difference being the reciprocal translation parallel to c^*. Photographs of both materials exhibit a pattern of several strong reflections arising from the pyroxene-like substructure common to long-chain pyroxenoids. Unit cell dimensions were refined by least-squares analysis of 153 measurements from precision back-reflection Weissenberg films taken about each of the three axial directions (*15*). Results are compared in Table 1 with previous cell determinations for natural pyroxmangite with composition corresponding to $(Ca_{0.03}Mn_{0.27}Fe_{0.35}Mg_{0.35})SiO_3$ from Ida, South Carolina (*16*), pyroxmangite from iron slag (*17*), and lunar pyroxferroite (*3–5*). The slag pyroxmangite was termed "iron rhodonite" by Whiteley and Hallimond (*18*) but was equated with pyroxmangite on the basis of optical properties by Tilley (*19*) and on the basis of unit cell size by Perutz (*17*). Unfortunately it was only partially analyzed for Mn and Fe content, although it is similar to another of Whiteley's "iron rhodonites" that has the composition $(Ca_{0.13}Mn_{0.30}Fe_{0.51}Mg_{0.06})SiO_3$. These slag materials are the most iron-rich pyroxmangites previously reported. Because of the paucity of determinations, it is not possible at this time to correlate unit cell parameters with composition.

Indexed powder diffraction data are listed in Table 2. Indexing was carried out by comparison of observed 2θ values for CuKα radiation with a list of 2θ values calculated on the basis of the refined cell parameters and checked against qualitative intensity observations on precession photographs.

As is not uncommon with triclinic phases, three different crystallographic orientations have been employed in pyroxmangite studies. Tilley (*19*) and others have used a morphological orientation in which the *c* axis lies in the zone of the two major cleavages that were assigned indices (110) and (110) by analogy to pyroxene morphology. Perutz (*17*), however, adapted an orientation similar to that used by Gossner and Brückl (*20*) for rhodonite in which the major cleavages are parallel to (100) and (001). Liebau (*16*) used the Perutz cell with an interchange of axes in his structure analysis of pyroxmangite. The matrix for transforming from the Perutz to the Liebau orientations is (001/100/010); the transformation places the *c* axis parallel to the direction of the silicate chains and the cleavages parallel to (010) and (100). We list the cell parameters (Table 1) and the powder diffraction data (Table 2) in the Liebau orientation, although comparison of the structure with those of pyroxenes will be facilitated by use of a *C*-centered cell, which is the morphological cell of Tilley (*19*).

Structure analysis of rhodonite (*21*) shows that calcium is restricted to one of five metal sites in the *Fünferketten* arrangement. The Ca-containing site has irregular seven coordination, whereas the other four sites, containing Mn with minor amounts of Fe and Mg, have distorted octahedral coordination. If Ca can only be accommodated in the seven-coordinated site, then, in theory, rhodonite could contain up to 20 mole percent $CaSiO_3$. The metal coordination in the *Siebenerketten* struc-

Table 2. X-ray powder diffraction data for $Ca_{0.15}Fe_{0.85}SiO_3$ pyroxenoid. Measured 2θ values are the means of two scans each of two samples of synthetic $Ca_{0.15}Fe_{0.85}SiO_3$ pyroxenoid; scans were made with copper radiation, a scan speed of 1° 2θ per minute, a time constant of 4 seconds, and a chart speed of 2.54 cm per minute. Silicon powder was used as a standard; however, there appears to be a systematic error of $-0.03°$ 2θ relative to the calculated values, which are based on the unit-cell parameters given in Table 1. We left the powder data in terms of 2θ because it is meaningless to compute *d* values for multiple peaks. Measured intensities are based on the means of peak height above background of the four scans described above. There is a strong preferred orientation effect in these values, which are presented only as a convenience to other workers who may wish to index powder patterns.

hkl	$2\theta_{meas}$ (deg)	I_{meas}	$2\theta_{calc}$ (deg)	d_{calc} (Å)
010	12.83	11	12.85	6.889
100	13.46	20	13.47	6.576
110	18.94	50	18.94	4.685
$\bar{1}11$	21.06	6	21.08	4.214
020	25.85	19	25.87	3.444
$1\bar{2}3$	26.69	4	26.76	3.332
200	27.11	17	27.12	3.288
$\bar{2}01$	28.29	18	28.29	3.154
021 $2\bar{1}2$	28.72	42	28.69 28.80	3.112 3.100
120 $0\bar{2}5$	29.58	43	29.65 29.65	3.013 3.013
$2\bar{1}3$	29.98	33	30.01	2.978
014 210 203	30.36	100	30.37 30.49 30.56	2.943 2.932 2.925
211	31.50	7	31.49	2.841
121	31.86	5	31.91	2.804
$0\bar{2}6$	33.45	24	33.47	2.677
$\bar{1}14$	34.13	47	34.16	2.625
$2\bar{2}2$	34.59	24	34.69	2.586
$0\bar{3}3$	35.71	22	35.75	2.511
$0\bar{3}2$	35.99	8	36.02	2.493
$0\bar{3}1$	37.20	1	37.20	2.417
024	41.17	28	41.23	2.189
206 $3\bar{1}2$	41.76	44	41.76 41.82	2.163 2.160
124 303	42.99	14	43.05 43.06	2.101 2.101
$2\bar{3}2$	44.08	7	44.11	2.053
$\bar{1}36$	44.27	2	44.29	2.045
207	46.50	1	46.50	1.953
230	49.02	9	49.09	1.856
$\bar{3}04$	49.63	4	49.63	1.837
$\bar{3}22$ $\bar{2}32$	53.60	17	53.61 53.67	1.710 1.708
$0\bar{1}10$	54.14	18	54.19	1.693
$2\bar{3}9$ 134	55.29	19	55.31 55.37	1.661 1.659
402	55.90	21	55.95	1.643
316	57.32	2	57.36	1.606
$2\bar{4}8$	58.75	9	58.79	1.571
330	59.13	2	59.17	1.562

ture is presumably analogous to that in rhodonite, except that only one in seven metal sites is suitable for Ca (*22*). The composition of our synthetic pyroxenoid differs by less than 1 mole percent from the 1 : 6 ratio of Ca to Fe in which one out of seven metal sites could be totally occupied by Ca.

Analyses of natural pyroxmangites (*23*) show ratios of Ca to (Fe + Mg + Mn) of less than 1 : 6; none shows a higher ratio. Likewise no rhodonites have Ca contents greater than that required to completely occupy one out of five metal sites, and most contain less Ca. Because of its size and coordination requirements, calcium appears to exert a limiting influence on the compositional range of pyroxenoid structure types similar to the limiting influence that it exerts in pyroxenes, where it can occupy *M2* sites but not *M1* sites. If the structural analogy between pyroxmangite and rhodonite is correct, then the $Ca_{0.15}Fe_{0.85}SiO_3$ composition should represent a CaFe end member for the *Siebenerketten* structure.

The fact that the *Neunerketten* structure forms at a Ca-free composition suggests that Ca is not essential to *Siebenerketten* structure formation and that Ca-free pyroxmangites may be stable on the $FeSiO_3$-$MnSiO_3$ join, particularly at Mn-rich compositions. However, the precise role that crystal chemical factors such as mean octahedral cation size play in controlling pyroxenoid structure type is at present unknown.

The pressure-temperature stability field determined for $Ca_{0.15}Fe_{0.85}SiO_3$ pyroxenoid is shown in Fig. 1. A similar diagram for this pyroxenoid (*14*) was based mainly on synthesis; the present diagram is based entirely on new data,

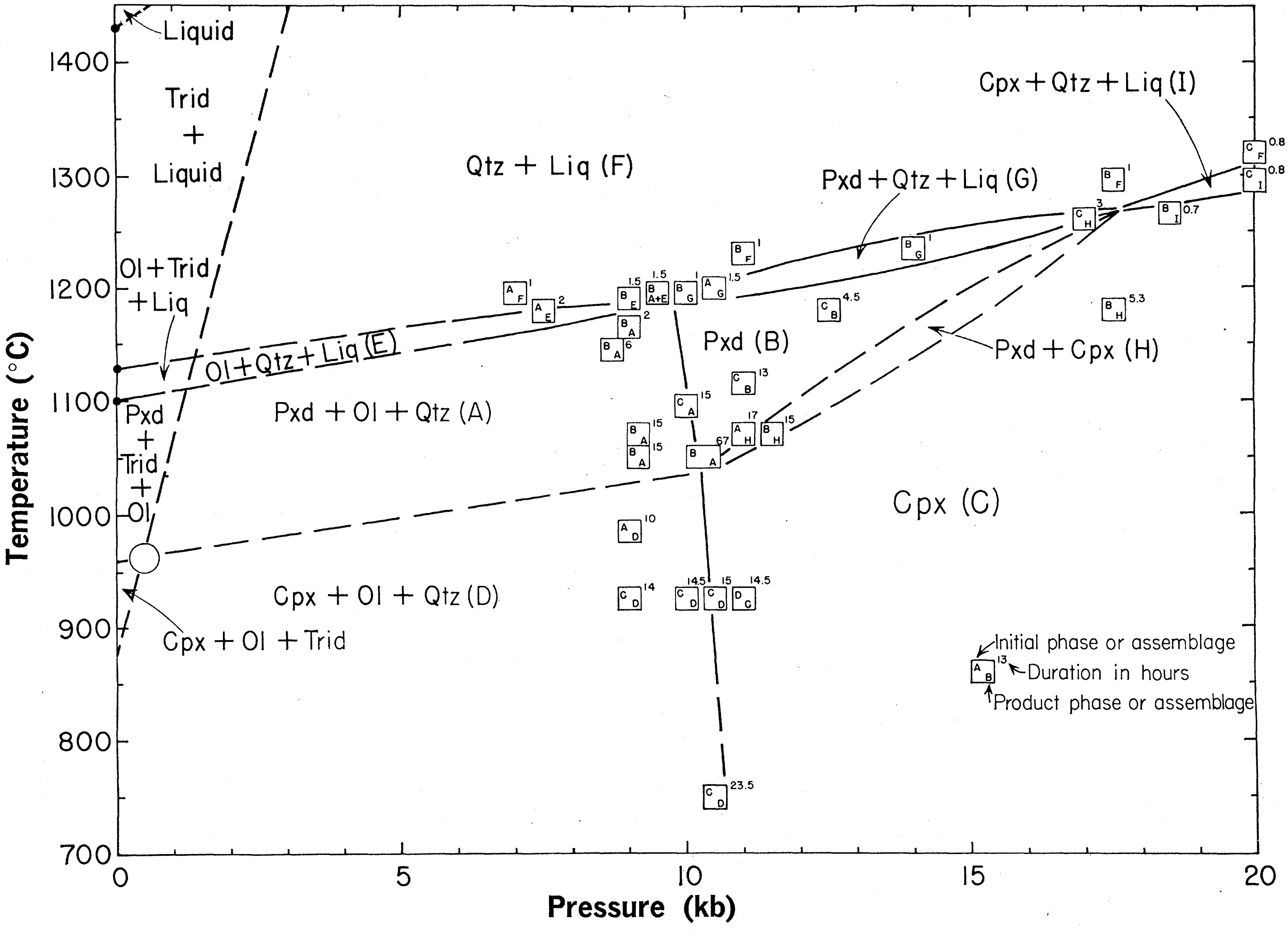

Fig. 1. Pressure-temperature equilibrium diagram for $Ca_{0.15}Fe_{0.85}SiO_3$ bulk composition, based on experiments with a solid-media, piston-and-cylinder pressure apparatus. Letters within each data symbol indicate for each experiment the initial and product phases or assemblages, and adjacent numerals give the duration in hours. The height of each symbol represents the estimated uncertainty in temperature for each experiment; no correction has been made for the effect of pressure on the platinum versus platinum-90–rhodium-10 thermocouples. The width of each symbol represents the estimated uncertainty in pressure for each experiment (*28*). Experiments above 925°C were carried out in iron capsules; the loaded capsules were dried overnight under vacuum at 170°C. For experiments at and below 925°C silver capsules were used; to each charge (10 to 15 mg) 1 mg of distilled water and approximately 0.5 mg of pure silica glass were added to saturate the vapor phase during the run (*13*). These hydrothermal experiments were necessary to obtain suitable rates for the reaction: clinopyroxene→Ca-enriched clinopyroxene plus olivine plus SiO_2. (Closed circles) Data at a pressure of 1 atm of Bowen *et al.* (*12*); (open circle) data from Lindsley and Munoz (*13*). *Liq*, liquid containing small amounts of Fe_2O_3; *Qtz*, quartz; *Trid*, tridymite; *Ol*, olivine rich in Fe_2SiO_4; *Cpx*, clinopyroxene; *Pxd*, pyroxenoid. The iron content of pyroxenoid and clinopyroxene that coexist with olivine and a silica phase in this diagram must always be less than $Ca_{0.15}Fe_{0.85}SiO_3$ and will decrease with decreasing pressure. The structures of these iron-depleted pyroxenoids are unknown; they may be of the *Fünferketten* or *Dreierketten* type, or both.

and the solid-solid phase boundaries are bracketed by reversed reactions. Only the pyroxenoid-clinopyroxene transformation is binary; the remaining solid-solid reactions fall in the ternary system $CaFeSiO_4$-Fe_2SiO_4-SiO_2. The liquids must contain small amounts of Fe_2O_3 (*12*), but inasmuch as all melting experiments were carried out in equilibrium with metallic iron, the variance remains unchanged, and, to a good approximation, the liquids may also be considered ternary systems (*24*).

Particular care has been taken to establish that the pyroxenoid does break down at pressures below approximately 10 kb to the assemblage: Ca-enriched pyroxenoid (or clinopyroxene) plus fayalitic olivine plus a SiO_2 phase. Previous work (*13, 14*) indicates that, as pressure decreases, the $CaSiO_3$ content of the pyroxenoid (or clinopyroxene) in equilibrium with olivine plus SiO_2 increases. Addition of $MnSiO_3$ to the system would expand the stability field of pyroxenoid to lower pressures, inasmuch as terrestrial and slag pyroxmangites are stable at low pressure. Addition of $MgSiO_3$ would expand the stability field of clinopyroxene to lower pressures but, by analogy with more Ca-rich pyroxenoids (*12, 13*), would probably restrict the stability field for pyroxenoid. Finally, addition of any components (for example, alumina or alkalies) that preferentially enter the liquid rather than the solid phases (Fig. 1) will tend to depress the solidus at the expense of the pyroxenoid and clinopyroxene stability fields. The conclusion is inescapable, then, that pyroxenoids of compositions approaching $Ca_{0.15}Fe_{0.85}SiO_3$ have crystallized either at high pressures or under metastable conditions.

The available petrographic evidence —for example, the presence of cristobalite and of vesicles (*1*)—strongly indicates that lunar rocks containing pyroxferroite crystallized under low-pressure, high-temperature (volcanic) conditions. It thus appears that the lunar pyroxferroite precipitated from an iron-enriched residual liquid, probably during fairly rapid cooling. In this regard it is instructive to note that Bowen, Schairer, and Posnjak synthesized (metastable) iron-rich pyroxenoids at a pressure of 1 atm by crystallizing liquids or glasses for periods of a few hours (*12, 25*), whereas experiments of 13 to 40 days duration were necessary to demonstrate that dry iron-rich pyroxenoids and clinopyroxenes are metastable at low pressures (*13*). Thus, even at temperatures close to the solidus, the inversion to the stable assemblage is sluggish; we suggest that at lower temperatures there is insufficient activation energy to permit transformation from pyroxenoid to the stable assemblage, even over long periods of time. Ware and Lovering (*26*) report the probable discovery of the stable breakdown assemblage fayalite plus iron-depleted pyroxene plus a silica mineral in a lunar sample; in this case, evidently, cooling was sufficiently slow to permit decomposition of the primary metastable phase. But it is apparent that the undecomposed lunar pyroxferroite has persisted in the metastable state since it crystallized—a period in excess of 3 billion years (*1, 27*). We suggest that the stability of lunar pyroxferroite be tested by holding a specimen in vacuum at 950° to 1000°C for a period of 1 to 2 months.

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