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The chemical formula of natural staurolite

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Abstract

For 82 staurolite analyses taken from the literature, a strong correlation ($r = -0.96$) exists between Fe + Mg + Zn and Al′, where Al′ is the aluminum remaining after any Si deficiency below 8.0 atoms is made up by Al. The equation of the best fit line [$\text{Al}' = 20.5 - 0.80(\text{Fe+Mg+Zn})$] and charge balance requirements result in the generalized chemical formula

$$(\text{Fe,Mg,Zn})_{25.6-1.25x}\text{Al}_{1.5x-8.2}\text{Si}_{16.2-0.5x}\text{O}_{48}\text{H}_{\sim 4}$$

where $16.58 \leq x \leq 18.61$ for the 82 staurolites used. The average chemical formula for those specimens is $(\text{Fe,Mg,Zn})_{4.0}\text{Al}_{17.3}(\text{Si}_{7.6}\text{Al}_{0.4})\text{O}_{48}\text{H}_{\sim 4}$. Although it has not been possible to place rigid limits on the composition of natural staurolite, the availability of only 8 sites suitable for Si leads to a lower limit of 16.4 for x and the formula $(\text{Fe,Mg,Zn})_{\sim 5.1}\text{Al}_{\sim 16.4}\text{Si}_{8.0}\text{O}_{48}\text{H}_{\sim 4}$, which represents an upper limit for Si and (Fe+Mg+Zn) and a lower limit for Al.

Some staurolites of unusual compositions do not fit the curves of lattice parameters *vs.* unit cell Fe content proposed by Griffen and Ribbe (1973). By recasting the curves in terms of $\langle r \rangle$ (= mean radius of the cations in the $\text{Fe}_2\text{Al}_{0.7}\text{O}_2(\text{OH})_2$ monolayer) instead of Fe content, the crystal chemical rationale (Griffen and Ribbe, 1973) for lattice parameter variations in staurolite is found to be satisfactory.

Introduction

Despite its common occurrence in medium grade, regionally metamorphosed pelites, and limited occurrence in other geologic environments and rock types, much less is known with certainty about staurolite than about many other minerals of comparable abundance and petrogenetic importance. The crystal structure was solved (Náray-Szabó, 1929; Náray-Szabó and Sasvari, 1958) and refined (Hanisch, 1966; Smith, 1968), only to find that site occupancies and cation site assignments are too complex in natural specimens to be unequivocally determined by X-ray diffraction techniques. Mössbauer spectroscopy was employed to shed light on the assignment of iron to octahedral and tetrahedral sites (Bancroft *et al.*, 1967; Smith, 1968; Dowty, 1972; Dickson and Smith, 1976) but the data are inconclusive and have led to conflicting interpretations. The major cationic substitutions have been inferred from statistical analyses of staurolite compositions (Griffen and Ribbe, 1973).

The crystal structure of staurolite can be envisioned as consisting of layers of approximate kyanite structure and composition alternating parallel to {010} with layers one atom thick (monolayers) of approximate composition $\text{Fe}_2\text{Al}_{0.7}\text{O}_2(\text{OH})_2$. Figures 1a and 1b illustrate portions of the structure. Monolayers (Fig. 1b) coincide with mirror planes, and are sandwiched between "1/2 kyanite" layers (Fig. 1a). The sandwiches are repeated along *b*, after reflection and translation by an *a*-glide perpendicular to [010]. The stacking arrangement is shown schematically in Figure 2. Smith's (1968) site refinement shows that the **Si**, **Al(1)**, and **Al(2)** sites[2] of the "kyanite" layer are essentially fully occupied, but the **Fe**, **Al(3A)**, and **Al(3B)** sites of the monolayer are partially occupied (0.92, 0.42, and 0.28 atoms, respectively). Protons are bonded to **O(1A)** and

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[2] Smith's site names are printed in boldface to differentiate them from chemical symbols. Site names imply the predominant, but not necessarily the only, element in the site.

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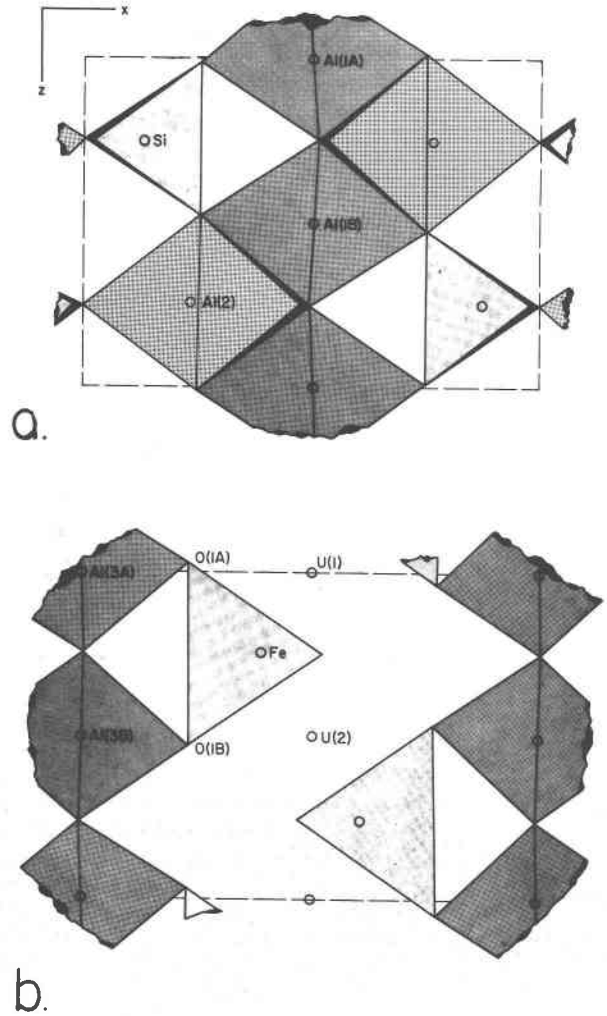

Fig. 1. Structure of staurolite. Dashed line is a unit cell boundary. (a) A half-thickness slab of the "kyanite" layer. The entire layer is obtained by stacking two such slabs across an *a*-glide perpendicular to [010]. (b) The "$Fe_2Al_{0.7}O_2(OH)_2$" monolayer. Only labelled atoms are actually in the monolayer (at y = 0), but polyhedra are completed for clarity.

O(1B), and lie nearly in faces of the **Al(3A)** and **Al(3B)** octahedra (Takéuchi *et al.*, 1972). The major cationic substitutions occur in the monolayer (Smith, 1968; Griffen and Ribbe, 1973).

Conspicuous in their uncertainty are an "ideal" chemical formula and compositional limits for staurolite. Lack of such basic information persists because (1) several cation sites are only partially occupied, making the total number of cations variable, (2) the fixed number of anions (48) in the topologically close packed unit cell thus does not fix the number of protons, and (3) several apparently nonessential elements are nearly ubiquitous in small amounts. The variability in the total number of cations almost certainly requires a variable number of protons for electrostatic neutrality. In a neutron diffraction study, Takéuchi *et al.* (1972) found three protons in the unit cell of a staurolite from an unknown locality. Griffen and Ribbe (1973) used densities of three analyzed specimens to calculate an average of 3.8 H^+, and suggested that $\sim 4H^+$ was therefore an appropriate approximation for the average number of protons per unit cell. In the absence of more definitive data, we have adopted that suggestion.

This study was undertaken in order to more concisely define the chemical formula of staurolite, and to investigate the feasibility of proposing chemical limits for that formula.

Methods

Data selection

The 82 staurolite analyses used in this study were selected from the literature solely because all five potentially major cations (Si, Al, Fe^{2+}, Mg, and Zn) were determined; in addition, nearly all included minor Mn and Ti. About 95% of the analyses were done by electron microprobe, the remainder by wet

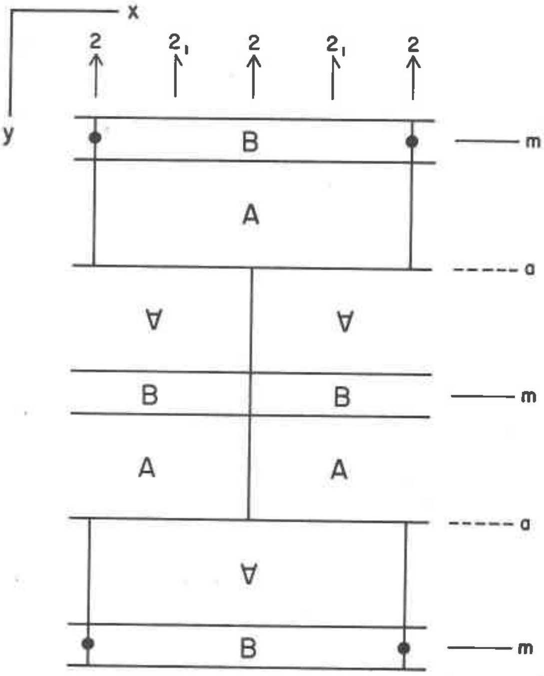

Fig. 2. The stacking scheme for layers shown in Figure 1. Standard notation is used for symmetry elements, dots mark unit cell corners.

chemical methods. References are cited in the caption of Figure 3. To facilitate comparisons, analyses were normalized to staurolite formulas based on 44(O) + 4(OH), where the original authors had not already done so (see Griffen and Ribbe, 1973). Cation ranges for analyses used in this study, expressed in terms of the staurolite formula, are

$$Fe_{1.86\text{–}3.66}Mg_{0\text{–}1.44}Zn_{0\text{–}1.54}Ti_{0\text{–}0.17}Mn_{0\text{–}0.15}Al_{16.56\text{–}19.24}Si_{7.18\text{–}8.09}O_{44}(OH)_4.$$

Computational procedures

The fitting of straight lines to geologic data is most often done by linear regression (unweighted least squares), even though that method is unsuited to many of the kinds of data involved. An assumption implicit in unweighted least squares is that all of the error resides in the dependent variable, and that is often not the case. If both variables are subject to error, either maximum likelihood estimation or a weighted least squares procedure is appropriate (Madansky, 1959; Jones, 1979).

In Figure 3, which displays "chemistry *vs.* chemistry" with neither variable clearly the independent one, the errors in both variables are of the same order of magnitude, and we chose to estimate the slopes and intercepts of best fit lines by the maximum likelihood method. The respective solutions are:

$$\hat{\beta} = \{S_{yy} - \lambda S_{xx} + [(S_{yy} - \lambda S_{xx})^2 + 4\lambda S_{xy}^2]^{1/2}\}/2S_{xy} \qquad (1)$$

and

$$\hat{\alpha} = \bar{y} - \hat{\beta}\bar{x} \qquad (2)$$

where $S_{yy} = \Sigma(y_i - \bar{y})^2/(n-1)$,
$S_{xx} = \Sigma(x_i - \bar{x})^2/(n-1)$,
$S_{xy} = \Sigma(y_i - \bar{y})(x_i - \bar{x})/(n-1)$,
$\lambda = \phi^2/\tau^2$,
$\bar{y} = \Sigma y_i/n$,
and $\bar{x} = \Sigma x_i/n$.
Here $x_i = X_i + \delta_i$ (true value + error),
$y_i = Y_i + \varepsilon_i$ (true value + error),
$\tau^2 = \mathrm{Var}(\delta_i)$,
and $\phi^2 = \mathrm{Var}(\varepsilon_i)$.

Although neither ϕ^2 nor τ^2 were known, their ratio, λ, could be estimated from the relative scatter of the data in the Y and X directions (Jones, 1979). (The solution for $\lambda = \infty$ is the unweighted least squares solution.) The values calculated for $\hat{\alpha}$ and $\hat{\beta}$ are fairly insensitive to variations in λ if the correlation coefficient (r) is high. This is so in Figure 3.

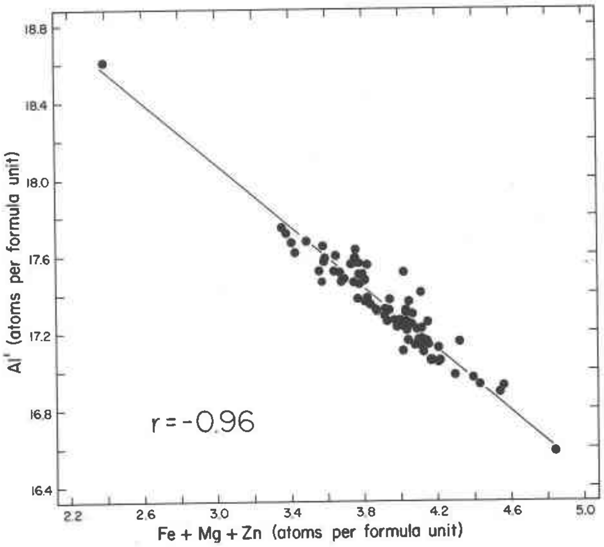

Fig. 3. Fe + Mg + Zn *versus* Al', where Al' is the number of Al atoms in the formula unit exclusive of those occupying the Si site. Line shown was estimated by maximum likelihood procedure. Data are from Ashworth (1975), Dickson and Smith (1976), Foster (1977), Fox (1971), Gibson (1978), Griffen and Ribbe (1973), Guidotti (1970), Hietenan (1969), Hollister (1970), Hollister and Bence (1967), Juurinen (1956), Kwak (1974), Leake (1958), Stoddard (1979), and von Knorring *et al.* (1979).

For Figure 4 the mean ionic radius is clearly the independent variable, and it was assumed to be known without error (that is, errors in the staurolite analyses and in ionic radii were ignored). Unweighted least squares was therefore used, although maximum likelihood estimation would have given very similar lines, and the correlation coefficients are independent of the best fit procedure employed.

Results and discussion

The chemical formula of staurolite

All but two of the 82 staurolites have fewer than 8 silicon atoms per 44(0) + 4(OH); the two exceptions, with 8.02 Si and 8.09 Si, were wet chemical analyses and may therefore reflect small amounts of the quartz inclusions common in staurolite. Because Smith (1968) found the **Si** site to be essentially fully occupied, we assumed that site to be filled by all of the silicon and enough aluminum to total 8.0 atoms (see Griffen and Ribbe, 1973, footnote 4). In Figure 3 we have plotted the remaining aluminum (designated Al') against the sum of Fe, Mg, and Zn. The high correlation (r = −0.96) suggests that the choice of aluminum to make up the silicon deficiency was justified. The equation of the maximum likelihood line, with $\lambda = 0.7$, is $Al' = 20.5 - 0.80(Fe+Mg+Zn)$. If we designate the number of Al′ atoms as x and the number of Si atoms y, then a general formula can be written as

$$(Fe,Mg,Zn)_{(20.5-x)/0.8}Al_x(Si_yAl_{8-y})O_{44}(OH)_4.$$

By requiring the sum of cation charges to equal 92 e.s.u., y may be found in terms of x, but it is necessary to account for the charge contributions of small amounts of Ti and Mn. For the 75 analyses that included Ti and the 79 that included Mn, average values are 0.11 Ti and 0.04 Mn, yielding an average cation charge contribution of 0.52 e.s.u. from these two elements. When this is taken into account, $y = 16.23 - 0.5x$. Omitting the small amounts of Ti and Mn (and other possible nonessential substituents) from the "ideal" formula, and recognizing the probability that the number of protons is somewhat variable, we propose the following general chemical formula for natural staurolite:

$$(Fe,Mg,Zn)_{25.6-1.25x}Al_{1.5x-8.2}Si_{16.2-0.5x}O_{48}H_{\sim4}.$$

For the 82 analyses used, $16.58 \leq x \leq 18.61$, and the average chemical formula ($\bar{x} = 17.3$) is

$$(Fe,Mg,Zn)_{4.0}Al_{17.3}(Si_{7.6}Al_{0.4})O_{48}H_{\sim4}.$$

The standard deviations of the absolute errors in the unit cell cation contents predicted for the 82 specimens by the general formula are 0.12 atoms for Si and Al, and 0.06 atoms for (Fe+Mg+Zn).

Neither Figure 3 nor the placement of the divalent metals within the same parentheses in the general formula is intended to imply that those atoms occupy the same site. Smith's (1968) X-ray site refinement and the statistical treatment of Griffen and Ribbe (1973) indicate a complex cation distribution with the **Fe** site principally occupied by Fe, Zn, and Al, and the **Al(3)** sites by Al and Mg. The simultaneous substitutions $(Fe,Zn) \leftrightarrows Al$ and $Al \leftrightarrows Mg$ at the two kinds of sites result in the overall variation seen in Figure 3.

Compositional limits of natural staurolite

Although the compositional range of these 82 staurolites is considerable, there is obviously no reason to suppose that the extreme compositions observed define compositional limits. Richardson (1967), Schreyer and Seifert (1969), and Griffen (1981) have synthesized Fe-, Mg-, and Zn-staurolites, respectively, from starting mixtures corresponding to 4 metal atoms per staurolite formula unit. Although these contain more Fe, Mg, or Zn (respectively) than any of the 82 natural specimens, Figure 3 suggests that even higher metal concentrations could be achieved by reducing the amount of aluminum. Limiting compositions have apparently not yet been observed, but some relevant deductions can be made from the general staurolite formula (this paper) and crystal structure (Hanisch, 1966; Smith, 1968).

A lower limit may be placed on x by considering the occupancy of the **Si** site. Smith (1968) found the site to be essentially fully occupied and assumed, as have we, that any silicon deficiency was made up by aluminum. As x of the general formula decreases, however, the number of Si atoms increases and reaches 8.0 when $x = 16.4$. If $x < 16.4$, then the number of Si atoms exceeds 8.0 and those that cannot be accommodated in the **Si** site must be accommodated elsewhere. The octahedral **Al** sites are clearly inappropriate for silicon, and the **Fe** site, although tetrahedrally coordinated, is much too large ($\overline{\mathbf{Fe}\text{–O}} = 2.02$Å). From a crystal chemical viewpoint, then, there is no site available for excess Si, and 8.0 silicon atoms ($x = 16.4$) constitutes one compositional limit. In terms of the chemical formula, this limit is

$$(Fe,Mg,Zn)_{\sim5.1}Al_{\sim16.4}Si_{8.0}O_{48}H_{\sim4},$$

which represents an upper limit for Si and (Fe+Mg+Zn), and a lower limit for Al.

If Smith's (1968) site population factors are assumed to be valid for all staurolites, then an increase in x leads to a decrease in occupancy of the **Al(3)** sites. An upper limit for x is elusive, because there appears to be no *a priori* reason to define a minimum occupancy for sites in the monolayer. Moreover, the site population factors may well vary with composition. One can make arguments based on local charge imbalance as x increases, but these do not permit the specification of a definite upper bound for x, either.

Lattice parameters

Griffen and Ribbe (1973) plotted lattice parameters for 15 natural staurolites *versus* unit cell Fe content, and found that b and c increased with iron concentration whereas a was insensitive to it. Correlation coefficients for a, b, and c *vs.* Fe were 0.10, 0.90, and 0.65 respectively. They rationalized as follows. Increased Fe (the largest of the substituent cations) increases the thickness of the $Fe_2Al_{0.7}$

$O_2(OH)_2$ monolayer independent of the kyanite layer, expanding the "kyanite-monolayer-kyanite" sandwich in the *b* direction (Fig. 2). Along *c* the edge-sharing chain of partially filled **Al(3)** sites expands with increased substitution of Fe, but is somewhat restricted because it must still "fit" the kyanite layers. Along *a*, in the monlayer, **Al(3)** chains alternate with nearly empty octahedral chains (the **U** sites, Fig. 1b) which are easily collapsed, resulting in little variation in *a*.

Of the 82 staurolite analyses used in this study, only Juurinen's (1956) #6 and the specimen of von Knorring *et al.* (1979), besides the 15 specimens of Griffen and Ribbe (1973), were accompanied by refined lattice parameters. The first two are of unusual compositions (high Zn and high Al, respectively), however, and fall markedly off the regression lines calculated by Griffen and Ribbe. We have therefore reexamined the lattice parameter variations. In doing so, we have included the specimen of Smith (1968) and Juurinen's (1956) #2, even though they were not analyzed for zinc, because lattice parameters were available for them, and the oxide totals were high enough to preclude the presence of all but very minor zinc.

Figure 4 shows lattice dimensions as a function of the mean ionic radius of cations in the monolayer. Mean ionic radius was taken as the weighted average of the radii of monolayer cations,

$$\langle r\rangle = \frac{{}^{IV}r_{Fe}{}^{IV}Fe + {}^{IV}r_{Zn}{}^{IV}Zn + {}^{IV}r_{Al}{}^{IV}Al + {}^{VI}r_{Al}{}^{VI}Al + {}^{VI}r_{Mg}{}^{VI}Mg}{Fe + Mg + Zn + Al},$$

where Roman numerals are coordination numbers, chemical symbols represent numbers of atoms, and r represents the radius for the appropriate coordination of the subscript element, taken from Shannon (1976) for Fe, Mg, and Al, and Griffen (1981) for Zn. In assigning cations to sites in the monolayer, the following assumptions were made:

(1) The population of the **Fe** site is 4.0 atoms, and includes all of the available Fe and Zn, and enough Al to make up the remainder.

(2) All available Mg is in the **Al(3)** sites.

(3) The **Al(1)** and **Al(2)** sites are fully occupied by Al, so that the **Al(3)** sites contain (in addition to the Mg of assumption 2) Al′-16-(4-Fe-Zn) aluminum atoms.

Although these assumptions lead to an oversimplified picture of the cation site assignments, the mean radii are a reasonable first approximation.

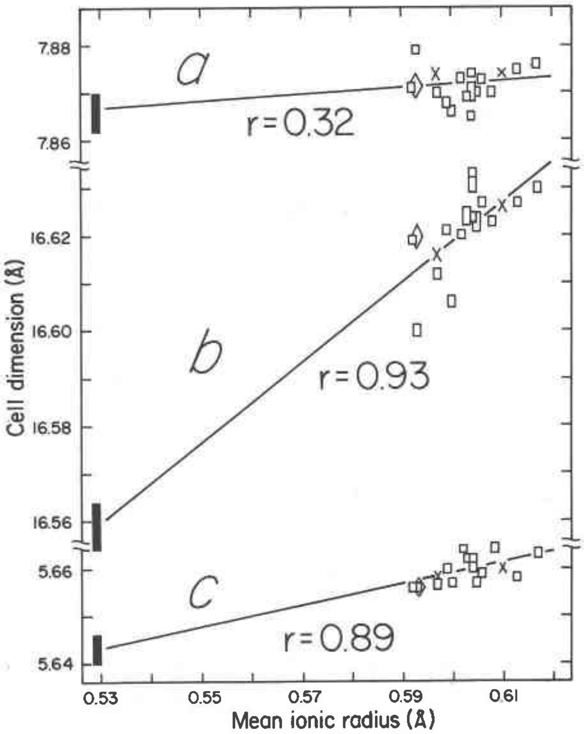

Fig. 4. Mean ionic radius of cations in the monolayer *versus* unit cell dimensions for 19 natural staurolites. Lines plotted were estimated by least-squares procedures. Symbols: Open rectangles, Griffen and Ribbe (1973); diamonds, Smith (1968); crosses, Juurinen (1956); solid rectangles, von Knorring *et al.* (1979).

The correlations seen in Figure 4 are consistent with the essential features of the model of Griffen and Ribbe (1973) for staurolite lattice parameter variations, and tend to confirm their interpretation. However, for specimens with unusual compositions (for example, the two that did not fall near their regression lines), the use of ⟨r⟩ provides more insight into the variations than does Fe content. (Although the specimen of von Knorring *et al.* (1979) appears at first glance to control the position of the regression lines, lines were also calculated without that specimen, and differed by <0.002Å from the plotted lines for all three cell edges over the entire range of ⟨r⟩ shown.)

Conclusion

A strong correlation between Fe + Mg + Zn and Al′ (where Al′ is the aluminum remaining after any Si deficiency is made up by allocating Al) has permitted a generalized chemical formula for staurolite to be written as follows:

$$(Fe,Mg,Zn)_{25.6-1.25x}Al_{1.5x-8.2}Si_{16.2-0.5x}O_{48}H_{\sim 4}$$

where x is the number of Al′ atoms, and ranges from ~16.6 to ~18.6 for 82 staurolite analyses from the literature. For these 82 analyses, $\bar{x} = 17.3$, yielding an average staurolite formula of

$$(Fe,Mg,Zn)_{4.0}Al_{17.3}(Si_{7.6}Al_{0.4})O_{48}H_{\sim 4}$$

(Small amounts of Ti and Mn (±Co, ±V, *etc.*) are nearly always present in natural specimens, but are ignored here as nonessential). A value of $x = 16.4$ [*i.e.*, $(Fe,Mg,Zn)_{\sim 5.1}Al_{\sim 16.4}Si_{8.0}O_{48}H_{\sim 4}$] represents a compositional limit for staurolite because lower values would require more than 8 Si atoms, and appropriate crystallographic sites are not available for more than eight. An upper limit for x must also exist, but is not evident.

Some staurolites of unusual compositions (*e.g.*, low Fe and high Zn or low (Fe+Mg+Zn) and high Al) do not fit the curves of lattice parameters *vs.* unit cell Fe content constructed by Griffen and Ribbe (1973). If the curves are recast as lattice parameters *vs.* ⟨r⟩, where ⟨r⟩ is the mean ionic radius of cations in the $Fe_2Al_{0.7}O_2(OH)_2$ monolayer, the correlations are greatly improved, leaving the rationale for lattice parameter variations in staurolite (Griffen and Ribbe, 1973) intact.

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