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AMERICAN CRYSTALLOGRAPHIC ASSOCIATION
Ottawa Meeting, August 20, 1970
SESSION ON MINERALOGICAL CRYSTALLOGRAPHY

As announced in the *Amer. Mineral.* **55**, 1080 (1970) the summer meeting of the A.C.A. included a Special Session on Mineralogical Crystallography. Fifteen papers were given in a full-day session; abstracts are printed below. In a brief discussion at the end of the day the participants, who included the presidents of the A.C.A., M.A.C., and M.S.A., voted unanimously that the Session on Mineralogical Crystallography should be made a permanent feature of the A.C.A. summer meeting. A mineralogical field trip in the region north of Ottawa on Saturday, August 22nd, 1970, was conducted by Dr. D. D. Hogarth.

TOPOTAXY

J. D. H. DONNAY, *The Johns Hopkins University, Baltimore, Md. 21218.*

Topotaxy has been variously defined. Essentially it designates the mutual orientation of crystals of two different crystalline species, either different chemical compounds or polymorphic forms of one chemical composition, when this orientation is the result of a chemical or physical reaction. Phenomena of mutual orientation have long been studied from the crystallographic viewpoint more than from the paragenetic one, and their nomenclature stresses the different types of reticular control: three-dimensional in syntaxy (H. Ungemach) and two-dimensional in epitaxy (L. Royer); one-dimensional control is unknown. Twinning deals with related phenomena (Mallard) where the mutually oriented crystals belong to the same crystalline species (*i.e.*, to the same form in case of polymorphism). Most twins obey three-dimensional control, whose degree of perfection forms the basis of their descriptive classification (G. Friedel); two-dimensional control has not been reported; one-dimensional control is known (in quartz, J. Drugman), but requires two pre-existing faces. Topotactic reactions may lead to syntactic or epitactic intergrowths; such a reaction may go to completion or not (partial topotaxy). Pseudomorphosis, which is independent of mutual orientation, may be involved. Deciphering the crystal morphology of a pseudomorph while heeding crystal growth, in case of total topotaxy, is the only way of determining directly what the original species was. Skeletal growth may simulate twinning and lead to erroneous conclusions regarding topotactic pseudomorphs, as the speaker has learned to his dismay.

SURFACE DECOMPOSITION OF Al_3Ni DURING ELECTRO-
POLISHING OF A DIRECTIONALLY SOLIDIFIED $Al-Al_3Ni$
EUTECTIC ALLOY.E. E. LAUFER AND J. T. JUBB, *Department of Energy, Mines and
Resources, Ottawa, Canada.*

Electron micrographs are presented which are interpreted as evidence of the surface decomposition of Al_3Ni to $AlNi$ during electropolishing of directionally solidified $Al-Al_3Ni$ eutectic alloys. This effect occurs for a wide range of polishing conditions.

TRANSFORMATION MECHANISM BETWEEN HIGH-QUARTZ AND
KEATITE PHASES OF $\text{LiAlSi}_2\text{O}_6$ COMPOSITION

CHI-TANG LI, *Owens-Illinois Technical Center, Toledo, Ohio.*

The high-quartz phase of $\text{LiAlSi}_2\text{O}_6$ composition reconstructively transforms into the keatite phase at elevated temperature. The transformation matrix was first derived from the powder data and then confirmed by the single crystal data. During the transformation, each independent high-quartz reflection transforms into one, two, or three corresponding independent keatite reflections, depending on the symmetry of each particular high-quartz reflection. (Si,Al)-tetrahedra form 6- and 8-membered rings in the high-quartz phase but form 5-, 7-, and 8-membered rings in the keatite phase. All the 6-membered rings and half of the 8-membered rings transform into 5- and 7-membered rings, or a combination of both types of rings of the keatite phase. The other half of the 8-membered rings remain unchanged linkagewise throughout the transformation. Transformation starts when either one common edge of two neighboring 6-membered rings is broken or the two adjacent edges, one at each ring, of the particular common edge are broken. During the transformation, most atoms undergo no change in linkage and little change in position. For example, only two out of twelve (Si,Al) atoms per enlarged high-quartz cell are involved in the bond-breakage and only four out of forty-eight (Si,Al)-O bonds are broken. The two (Si,Al) atoms which break away from the old framework both undergo a displacement of 2.6 Å, while the displacements of the remaining ten (Si,Al) atoms range only from 0.01 to 0.35 Å. This study of the transformation mechanism makes it possible to follow the movement of each atom during the transformation. To the writer's knowledge, this is the first time that a phase transformation is studied in such a great detail.

CRYSTAL STRUCTURES OF $\text{Ca}_3(\text{AsO}_4)_2$, $\text{Ca}_3(\text{VO}_4)_2$ AND WHITLOCKITE.

C. CALVO AND R. GOPAL, *Institute for Materials Research
McMaster University, Hamilton, Ontario.*

Four sets of data have been analyzed for these three nearly isostructural crystals.¹ The space group is $R3c$, $Z=7$, with lattice parameters of $a=14.09, 14.12, 13.67$ Å, and $\alpha=45.95^\circ, 44.95^\circ, 44.35^\circ$ for $\text{Ca}_3(\text{AsO}_4)_2$, $\text{Ca}_3(\text{VO}_4)_2$, and the mineral respectively. The unit-cell contents are formulated as $\text{Ca}_{18}\text{M}_2\text{B}(\text{XO}_4)_{14}$ with the M ions, Mg for the mineral, lying on the three-fold axis and B a disordered Ca in the former two structures and $2H$, according to Keppler, in the mineral. Small differences in the mineral structure are probably related to the H position. These structures are related to that of $\text{Ba}_3(\text{PO}_4)_2$ and the changes arise from the shorter cation oxygen bond lengths in the calcium salts.

THE REACTION OF STANNOUS FLUORIDE WITH HYDROXY-
APATITE—THE CRYSTAL STRUCTURE OF $\text{Sn}_3\text{PO}_4\text{F}_3$ ²

ALAN F. BERNDT, *University of Missouri, St. Louis, Missouri.*

The reaction of stannous fluoride with hydroxyapatite is of interest in determining the mechanism by which stannous-fluoride-containing toothpastes are useful in the prevention of cavities. This reaction, *in vitro*, yields a crystalline product, containing Sn, with a rhombohedral unit cell with $a=7.030$ Å, $\alpha=115^\circ 23'$. Equi-inclination Weissenberg data

¹ The diffractometer data for $\text{Ca}_3(\text{VO}_4)_2$ were provided by Mr. J. Rowland and Dr. E. Gabe.

² This research was supported by the Faculty Research Committee-UMSL.

were collected, with $\text{CuK}\alpha$ radiation, with the axis of rotation parallel to the 3-fold axis. A total of 160 reflections were observed. Identification of this compound was achieved by means of a complete structure determination.

A three-dimensional Patterson map showed the location of the tin atoms. With the heavy atoms located, difference electron-density maps were used to indicate the presence and location of a phosphate ion and three fluoride ions. The structure was refined by least squares to a value of R of 7.6 percent. The stoichiometry was identified to be $\text{Sn}_3\text{PO}_4\text{F}_3$. The space group is acentric, $R3$ and the calculated density is 4.49 g cm^{-3} .

The phosphate ions are aligned along the 3-fold axis. Each Sn atom has three nearest neighbors (one O and two F) at an average distance of 2.15 \AA , arranged in a trigonal pyramid. This geometry is similar to that observed in compounds containing the SnCl_3^- group.

CYLINDRITE—CRYSTALLOGRAPHY AND CRYSTAL CHEMISTRY.¹

EMIL MAKOVICKY, *McGill University, Montreal, Quebec.*

The cylindrite structure is composed of alternating layers of two kinds, A and B , parallel to (100). Layers A and B diffract separately and thus have different lattices. Layer A approximates the following composition, calculated for 26 S: (Pb + Ag) 14.56, Sn 4.68, Sb 4.64, Fe 1.33; Me:S ratio = 1:1 having been obtained from structure determination; a subcell is pseudo-tetragonal, triclinic, $A1$, $a = 11.73 \text{ \AA}$, $b = 5.79 \text{ \AA}$, $c = 5.81 \text{ \AA}$, $\alpha = 90^\circ 10'$, $\beta = 92^\circ 23'$, $\gamma = 93^\circ 52'$; the true cell is triclinic, $F1$, $a = 140.67 \text{ \AA}$, $b = 5.79 \text{ \AA}$, $c = 75.52 \text{ \AA}$, $\alpha = \beta = 90^\circ$, $\gamma = 93^\circ 52'$; the layers are $\sim 3 \text{ \AA}$ thick and show deformed "PbS-like" structure, with two Sb-containing steps repeatedly offsetting part of the layer back and forth in the a direction by $\sim 1.1 \text{ \AA}$. Layer B approximates the following composition, calculated for 24 S: Sn 7.80, Sb 2.35, Fe 1.01; Me:S = 1:2; subcell pseudo-hexagonal, triclinic, $A1$, $a = 11.71 \text{ \AA}$, $b = 3.67 \text{ \AA}$, $c = 6.32 \text{ \AA}$, $\alpha = 90^\circ 2'$, $\beta = 92^\circ 35'$, $\gamma = 90^\circ 51'$; cell triclinic, $C1$, $a = 140.37 \text{ \AA}$, $b = 3.67 \text{ \AA}$, $c = 37.92 \text{ \AA}$, $\alpha = \beta = 90^\circ$, $\gamma = 90^\circ 51'$; layers SnS_2 are offset again by $\sim 1.1 \text{ \AA}$, offset parts have the same length as in A . Step regions accommodate Sb and Fe atoms. Many different pairs of lattices, such as the $F1$ and $C1$ triclinic ones given above, are observed for A and B . Step regions face each other and have opposite formal charges. Several cylindrite subtypes with different Sb contents in the step regions exist, with Sb-content increments equal in the two layers. The relative position of the two sets of layers in the cylinder-axis direction, perpendicular to the steps, is fixed. Cylindrical growth is caused by the flexibility of bond adjustments on interlayer boundaries along the step direction, the b axis, which is tangent to the cylinder. The a dimension is related to the thickness of one cylindrite growth layer. Cylindrical growth starts from a minute straight-layered nucleus. Central parts of cylinders have complicated structures showing a series of nuclei. Phases departing from the cylindrite A:B ratio are not cylindrical and vary in composition.

ORDERING OF CATIONS IN OLIVINE.²

L. W. FINGER, *Geophysical Laboratory, Carnegie Institution of Washington, Washington, D.C. 20008.*

The crystal structures of olivines from rock 10020 of the Apollo 11 lunar sample and a volcanic rock from Australia have been refined to determine if there is any cation ordering in the two octahedral positions. After intensity collection, the crystals were analyzed on the electron microprobe and the octahedral occupancies were constrained according to this chemistry. The refinement converged satisfactorily with the conventional R equal to 4.8

¹ Research partially sponsored by NRC through Prof. A. J. Frueh.

² This research was partially supported by N.A.S.A. contract NAS 9-9988.

and 2.6 percent for the lunar and terrestrial samples respectively. The refined values of the occupancies indicate partial ordering of iron into *M1* despite the smaller size of this octahedron. The effect is consistent and more pronounced in the terrestrial sample which probably has a slower cooling history and a lower temperature of equilibration than the lunar sample.

REFINEMENT OF THE CRYSTAL STRUCTURE OF APOPHYLLITE

ALAN A. COLVILLE, *California State College at Los Angeles, California*
AND CHARLES P. ANDERSON, *University of California*
at Riverside, Riverside, California.

The crystal structure of apophyllite, $(\text{Na}_{0.16}\text{K}_{0.84})\text{Ca}_4(\text{Si}_4\text{O}_{10})_2\text{F}\cdot 8\text{H}_2\text{O}$, with $a = 8.963(2)$, $c = 15.804(2)\text{Å}$, $P4/mnc$, $Z = 2$ has been refined by least-squares ($R = .054$) with 546 independent intensities collected with a diffractometer.

Apophyllite is a sheet structure with the bridging Si-O bond lengths 1.619(4), 1.626(4), and 1.630(4) Å, while the non-bridging bond length Si-O(3) is 1.589(4) Å. The two independent Si-O-Si angles are 139.9(2) and 140.6(2) degrees. The mean Si-O bond length is 1.616 Å. The interpretation of the role of the water in the structure is based on the charge balance approach of Donnay and Allmann (1970). Assuming that the fluorine ion is bonded to calcium, the sum of the cation valences for O(2) and O(3) would be 1.96 and 1.66 v.u. respectively. The water oxygen therefore would be hydrogen bonded to O(3) and O(2). The oxygen-oxygen distances are 2.75 and 3.17 Å respectively and the final sum of the cation valences reaching O(2) and O(3) would be 2.03 and 1.93 v.u.

Preliminary results from a neutron-diffraction study by E. Prince indicate that the fluorine may be bonded to an H^+ ion. This would eliminate fluorine from the calcium polyhedron and lead to a formula of $\text{KCa}_4(\text{Si}_4\text{O}_{10})_2\text{HF}(\text{OH})\cdot 7\text{H}_2\text{O}$ for apophyllite.

THE HYDROGEN POSITIONS IN APOPHYLLITE

E. PRINCE, *Center for Radiation Research, National Bureau*
of Standards, Washington, D.C. 20234.

The crystal structure of apophyllite, $\text{KCa}_4(\text{Si}_4\text{O}_{10})_2\text{F}\cdot 8\text{H}_2\text{O}$, was refined by X-ray diffraction by Colville and Anderson (above). On the basis of the positions of all of the atoms heavier than hydrogen, and using the valence-summation technique of Donnay and Allmann (1970), they have proposed positions for the hydrogen atoms. A further refinement of the structure by neutron diffraction has been carried out. A least-squares refinement, using 671 independent reflections and anisotropic temperature factors, led to an R index of .046. The structure contained a water molecule with a reasonable configuration hydrogen-bonded to the silicate framework. However, a difference Fourier revealed a region of significant negative scattering density in the vicinity of the fluorine atom. A model in which 1/8 of the water molecules were replaced by OH^- ions, with the remaining proton attached to fluorine to form an HF molecule, was therefore tried, and the structure quickly refined down to an R index of .037. This result suggests that the double peaks in DTA and TGA curves may be explained by the evolution of water and HF at different temperatures. The structural formula should therefore be written $\text{KCa}_4(\text{Si}_4\text{O}_{10})_2\cdot\text{HF}(7\text{H}_2\text{O}\cdot\text{OH})$.

THE CRYSTAL STRUCTURE OF CARLETONITE, $\text{KNa}_4\text{Ca}_4\text{Si}_8\text{O}_{18}(\text{CO}_3)_4(\text{OH},\text{F})\cdot\text{H}_2\text{O}$

G. Y. Chao, *Carleton University, Ottawa, Ontario, Canada.*

Carletonite, a new mineral from Mt. St. Hilaire, is tetragonal with $a = 13.178\text{ Å}$ (3), $c = 16.695(4)$, space group $P4/mbm$, $Z = 8$, $D_x = 2.426$, $D_m = 2.45(1)$. 1220 symmetry non-

equivalent reflections were measured on a Supper manual single crystal diffractometer, using $\text{MoK}\alpha$ radiation. A cleavage fragment in the shape of a cube (0.08 mm) was used. The structure was solved by three-dimensional Patterson synthesis and refined by full matrix least squares method. Occupancy factors for the cations are also included as least squares variables. The final agreement index R is 0.050.

In carletonite the SiO_4 tetrahedra share three of their four oxygen atoms to form sheets consisting of 4 and 8-membered rings, similar to the sheets in apophyllite. However, the structure of carletonite is unique in that pairs of such sheets are cross-linked by half of the oxygen atoms not shared in the single sheets, forming "double sheets" of the composition Si_8O_{18} . Thus half of the SiO_4 tetrahedra share all four oxygen atoms and the other half share only three. K, Na, Ca atoms and the CO_3 groups are sandwiched between the "double sheets". The ideal composition of carletonite is established as $\text{KNa}_4\text{Ca}_4\text{Si}_8\text{O}_{18}(\text{CO}_3)_4(\text{OH},\text{F})\cdot\text{H}_2\text{O}$ in contrast to that calculated from the analytical data, $(\text{K},\text{Na},\text{Ca})_8\text{Si}_8\text{O}_{18}(\text{CO}_3,\text{OH},\text{F})_4\cdot\text{H}_2\text{O}$. The mineral is non-stoichiometric, being deficient in Na (~ 13 atomic %), Ca ($\sim 6.5\%$), K ($\sim 13\%$), CO_3 ($\sim 9\%$) and (OH,F) ($\sim 30\%$). The least squares occupancy factors agree with the analytical values within 5%.

CRYSTAL STRUCTURE OF A NEW MINERAL, SÖHNGEITE

J. D. SCOTT, *Department of Geological Sciences,
Queen's University, Kingston, Ontario.*

Söhngeite, $8[\text{Ga}(\text{OH})_3]$, a recently discovered mineral from the second oxidized zone at the Tsumeb Mine, South West Africa, has a rather unusual crystal structure. The unit cell, though dimensionally orthorhombic $a=7.4865$, $b=7.4379$, $c=7.4963$ Å, displays strong pseudo-cubic symmetry ($Pn3n$). There is also a pronounced pseudo-cubic subcell with $a=3.74$ Å ($Pm3m$).

The $Pn3n$ cell has been refined to $R=0.16$ with Ga in 8(c) and OH in 24(h) ($x=0.0877$). The isotropic B of the oxygen, however, diverges to very large values and the shortest O-O bond approaches 1.55 Å. A similar situation pertains in the centric orthorhombic derivative of this cell ($Pmnm$).

The correct space group is $Pmn2_1$ with an almost centric arrangement of Ga in 4(b) general positions, very slightly displaced about $\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{4}$, and $\frac{1}{4}$, $\frac{1}{4}$, $\frac{3}{4}$. The oxygen atoms form distorted octahedra about the gallium, which octahedra share corners to form an infinite framework. Two of the oxygen atoms lie near $z=0$, $\frac{1}{2}$, the other four atoms forming a rough plane centered on $z=0.30$. The present R index is 0.09 for 213 observed planes.

CRYSTAL GROWTH OF CaWO_4 AND $\text{Nd}_2(\text{CO}_3)_3\cdot 8\text{H}_2\text{O}$ BY SILICA GEL TECHNIQUE

J. W. McCauley, and H. M. Gehrhardt, *Army Materials and
Mechanics Research Center, Watertown, Massachusetts 02172.*

Spherulites (up to 2mm in diameter) and single crystals of CaWO_4 have been synthesized at ambient conditions by reacting $\text{CaCl}_2\cdot 2\text{H}_2\text{O}$ and $\text{NaWO}_4\cdot 2\text{H}_2\text{O}$ within acetic acid- $\text{Na}_2\text{SiO}_3\cdot 9\text{H}_2\text{O}$ gels. High calcium concentrations favored the growth of clear spherulites, whereas high tungstate concentrations resulted in almost opaque spherulites. The clear, highly translucent spherulites consisted of radiating single crystals. On the other hand, the nearly opaque CaWO_4 spherulites seemed to have formed by both radiating single crystals (in the inner part), and concentric banding or an agglomeration process in the outer half. After withdrawal from the gel, most of the spherulites developed crack patterns which, in general, divided the spherulites into halves or quarters. The addition of NH_4Cl to

the gel system led to the formation of single crystal needles (up to 2mm in length) of CaWO_4 .

Lavender-colored rhombic plates ($\{001\}$, $\{210\}$, $\{101\}$ forms), dendrites, and spherulites of $\text{Nd}_2(\text{CO}_3)_3 \cdot 8\text{H}_2\text{O}$ were also synthesized in a silica gel by reacting $\text{NdCl}_3 \cdot 6\text{H}_2\text{O}$ with Na_2CO_3 . Utilizing low pH $\text{HCl-Na}_2\text{SiO}_2 \cdot 9\text{H}_2\text{O}$ gels the crystals formed by recrystallization from an initial precipitate (perhaps $\text{Nd}_2(\text{CO}_3)_3 \cdot \text{Na}_2\text{CO}_3 \cdot n\text{H}_2\text{O}$) or at gel-gel and gel-bubble interfaces. Single crystal X-ray diffraction studies demonstrated that the crystals are orthorhombic with $a=8.88 \text{ \AA}$, $b=9.43 \text{ \AA}$, and $c=16.88 \text{ \AA}$. The diffraction symbol *mmmPccn* uniquely determines the space group as *Pccn*. These data are consistent with those determined for $\text{La}_2(\text{CO}_3)_3 \cdot 8\text{H}_2\text{O}$ by Shinn and Eick (*Inorg. Chem.*, **7**, 1340). Observation of the crystals with a petrographic microscope revealed that they consist of a domain structure and a resulting strain birefringence. Differential thermal analysis on the material showed that six waters of hydration are lost beginning at 77°C and the last two beginning at 121°C ; infrared and visible absorption spectra were also obtained from selected single crystals and compare favorably with the spectra for other neodymium and carbonate compounds.

BUERGERITE: REFINEMENT OF THE STRUCTURE BY NEUTRON DIFFRACTION¹

WALTER C. HAMILTON AND ARMIN TIPPE, *Chemistry Department,
Brookhaven National Laboratory, Upton, New York 11973.*

The structural parameters of buergerite (ideally $\text{NaFe}_3\text{B}_3\text{Al}_6\text{Si}_6\text{O}_{30}\text{F}$) have been refined, using three-dimensional single-crystal neutron diffraction data. The occupancy factors in the cation sites have been refined and provide confirmation of the substitution of Fe for Al in the $18c$ sites of space group *R3m*. Because of the differences in scattering amplitudes, some of the bond distances have been determined to slightly higher precision than in the X-ray study (Barton, in press)² In general the position and thermal parameters agree extraordinarily well with those of the X-ray study. The most significant result was the location of a partial hydrogen atom from a difference map; the occupancy factor of 0.083(11) implies a substitution of OH for O eight percent of the time in one of the oxygen sites (O(3)) which surround the iron atom octahedrally.³ This amounts to 0.75 H atoms per unit cell. A weak hydrogen bond with O-H 0.94(5), H . . . O 2.46(5) and $\angle\text{O-H} \dots \text{O}$ $154^\circ(2)$ is formed. The final $R(F^2)$ is 0.034 for 391 independent reflections; the maximum error in the final difference synthesis is 0.4 f \AA^{-3} , about one percent of the maximum peak height in the observed scattering density map; the bond lengths are in general precise to 0.002 \AA . A comparison of powder diffraction patterns at 300° K and 4.2° K provides no evidence for magnetic ordering or other structural changes between these temperatures.

REFINEMENT OF THE CRYSTAL STRUCTURE OF ELBAITE

R. BARTON, JR., *Carothers Research Laboratory, Experimental Station, E. I. du Pont de Nemours and Co., Wilmington, Delaware,* and G. DONNAY, *Geophysical Laboratory, 2801 Upton Street, N. W., Washington, D. C.*

The crystal structure of elbaite has been refined by least-squares by use of single crystal X-ray intensity data collected by counter diffractometry. Elbaite crystallizes in

¹ Research performed under the auspices of the U. S. Atomic Energy Commission.

² Barton, R. (1969) Refinement of the crystal structure of buergerite and the absolute orientation of tourmalines. *Acta Cryst.* B25, 1524-1533.

³ OH substitution in this site was predicted by G. Donnay (private communication) on the basis of charge-balance considerations.

$R3m$; the hexagonal cell dimensions for the specimen used are: $a=15.838(1)$, $c=7.1032(2)$ Å at 21°C. $D_m=3.05(1)$; with $Z=3$, $D_x=3.07$ g.cm⁻³. Of 1600 independent reflections measured, 1142 were significantly above background; the residual R for the observed reflections is 0.058. Elbaite is a species of tourmaline of ideal composition $\text{Na}(\text{Li,Al})_3\text{B}_3\text{Al}_6\text{Si}_6\text{O}_{27}(\text{OH,F})_4$. This investigation was conducted because an earlier study of elbaite (Ito and Sadanaga, 1951) is now thought to have been performed on a tourmaline of dravite-schorl composition, and a detailed knowledge of the structure of elbaite might explain why no solid solution occurs between elbaite and dravite. The results of the refinement show that the atomic coordinates of elbaite are very similar to those of dravite (Buerger, Burnham, and Peacor, 1962) and also to those of buergerite (Barton, 1969), so much so that no explanation of the lack of solid solution presents itself.

MAGNETIC PROPERTIES OF THE TOURMALINE GROUP

T. TSANG AND ARTHUR N. THORPE, *Howard University, Washington, D. C.*; FRANK E. SENFTLE, *U. S. Geological Survey, Washington, D. C.*; GABRIELLE DONNAY, *Geophysical Laboratory, Carnegie Institution of Washington, Washington, D. C.*

Tourmalines free from iron and manganese can only be paramagnetic, whereas those tourmalines containing magnetic ions can be expected to show evidence of magnetic exchange interactions. Magnetic susceptibilities of three iron-rich tourmaline crystals of different, known, chemical compositions have been studied from 8° to 300°K. These are specimens on which careful checking showed the absence of ferromagnetic inclusions. One sample studied is a ferric-oxytourmaline or buergerite (Barton 1969), which may be described from a magnetic viewpoint as having isolated triangles with Fe^{3+} ions at the corners. Its exchange constant $J/k=7.5^\circ\text{K}$ may be derived from the measured susceptibility data. The susceptibility measurements are also consistent with the X-ray and spectral evidence that, in spite of the presence of just enough aluminum to fill the Al position, a small fraction (~8%) of Fe^{3+} ions are substituting for aluminum. From the other two tourmaline samples, an iron-rich, aluminum-deficient specimen from Madagascar, and a magnesium-iron specimen from Pierpont, N.Y., it is possible to estimate the exchange interactions between the iron atoms substituting for aluminum atoms. Our results are again consistent with the optical spectral evidence that there is considerable deviation from octahedral symmetry at both the 9(b) and 18(c) positions of space group $R3m$. The distortion is most pronounced for the general position.