CRYSTAL STRUCTURE OF A RUBIDIUM IRON FELDSPAR[1]

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ABSTRACT

$RbFeSi_3O_8$ is a synthetic hydrothermal intermediate microcline with Fe^{3+} substituting 38.9, 48.7, 10.2, and 11.0 atomic percent respectively for Si at tetrahedral positions. Lattice parameters for the triclinic cell ($C\bar{1}$) are $a = 8.952(4)$, $b = 13.127$ (5), $c = 7.359(2)$ Å, $\alpha = 90.05°$, $\beta = 116.47°$, and $\gamma = 89.35°$ at 24°C. The calculated density is 3.032 gm/cm³ and $Z = 4$. The Rb–O interatomic distances range 3.033(6)– 3.348(5) Å. The average T–O distances are 1.702, 1.734, 1.620, and 1.622 Å respectively for the four independent tetrahedral positions. An asymmetrical ($C1$) structure was also refined and the discrepancy factors $R(F_0{}^2)$ and $wR(F_0{}^2)$ for the two structures are not significantly different at the 0.01 level. The $C\bar{1}$ model is probably the correct structure because several of the T–O distances in the $C1$ model are abnormally short, *e.g.*, $T_2(Oi)$—$O_B(Oi)$, 1.50(3) Å.

INTRODUCTION

The results of careful refinement of the structures of sanidine (Cole, Sorum, and Kennard, 1949), orthoclase (Jones and Taylor, 1961), intermediate microcline (Bailey and Taylor, 1955, and Bailey, 1969) and maximum microcline (Brown and Bailey, 1961) are summarized by Brown and Bailey (1961) and discussed further by Jones (1968) and Jones and Taylor (1968). The distribution of the aluminum on tetrahedral sites in these feldspar structures is inferred from the tetrahedral bond distances. The aluminum distribution varies from completely random in high sanidine to a unique position in maximum microcline.

The substitution of iron for aluminum in alkali feldspars offers an opportunity to determine the distribution of cations in tetrahedral sites by criteria other than bond lengths. The multiplicity of the tetrahedral positions can be determined as parameters in the least–squares structure refinement and the total number of electrons can be determined for
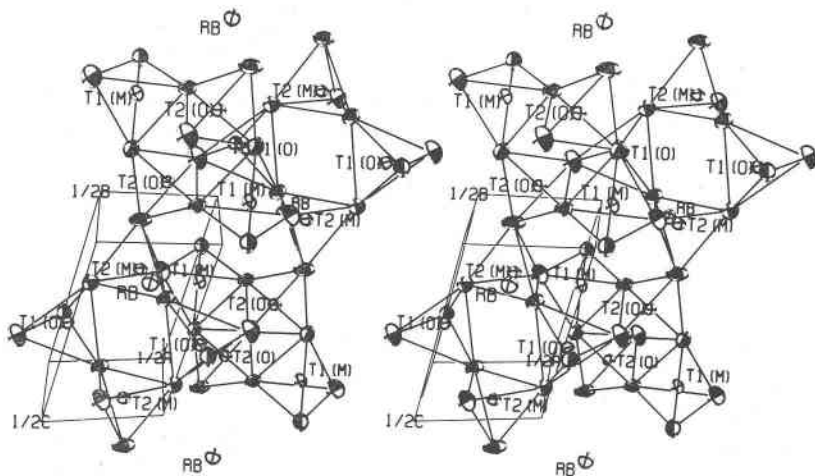
Fig. 1.   Stereoscopic view of the contents of one unit cell—$RbFeSi_3O_8$.

each position by numerically integrating a volume of the three–dimensional electron density map. These parameters have been applied to the determination of the structure of $RbFeSi_3O_8$, a synthetic intermediate microcline. The structures for the centrosymmetrical ($C\bar{1}$) and asymmetrical ($C1$) space groups were refined and the results of both refinements are presented.

EXPERIMENTAL

Synthetic intermediate microcline $RbFeSi_3O_8$ crystals were grown hydrothermally from a SiO_2–$RbOH$–H_2O–Fe_2O_3 mixture at $480 \pm 5°C$ and 1.6 ± 0.1 kbar pressure (Kopp and Harris, 1970). An untwinned crystal resembling a rectangular parallelepiped ($0.013 \times 0.046 \times 0.070$ mm) was mounted on a computer–operated Picker four–circle goniostat equipped with a scintillation–counter detector. Lattice parameters were determined by a least–squares adjustment of twelve $CuK\beta$ (1.39217Å) reflections between 59° and 102°2θ. At 24°C, $a = 8.952(4)$, $b = 13.127(5)$, $c = 7.359(2)$Å, $\cos \alpha = -0.0008(5)$ (90.05°), $\cos \beta = -0.4455(4)$ (116.47°), and $\cos \gamma = 0.0113(7)$ (89.35)°. The space group ($C\bar{1}$ or $C1$) and cell parameters are consistent with previous feldspar structures. The calculated density is 3.032 gm/cm³; $Z = 4$.

Independent reflections were measured in the 2θ range 30–80° by the θ–2θ scan technique and in the 2θ range 1–30° by the omega scan technique using Zr filtered $MoK\alpha$ radiation for both scans (Busing, *et al.*, 1968). The take–off angle was 1.5°. Each reflection was step–scanned at intervals of 0.01° with 1 sec count at each step for a total of 1.6° 2θ for the θ–2θ scan and 0.85° ω for the omega scans. The background for the 2θ scans was counted for 60 sec on each side of the peak. The background for the omega scans was scanned at 0.85° 2θ on each side of the peak (Busing, *et al.*, 1968). A standard reflection (060) was measured every 20 reflections to monitor X-ray source variations. The range of net count of (060) for the 2θ scans is 78000 to 82000. In addition to scaling the omega and 2θ data with the (060) standard re-

Table 2. Atomic and temperature parameters for RbFeSi$_3$O$_8$.

Atom	SG	Multiplicity	10^4x	10^4y	10^4z	$10^4\beta_{11}$[a]	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$	a[b]	b	c
Rb(i)	C1	1	2924.6[c]	-5.7	1477.2	78(1)	35.2(4)	115(2)	2.1(5)	41.8(9)	0.9(6)	159(1)	159(1)	176(1)
Rb(0)	C1	1	-2926(2)	5(1)	-1481(2)	d								
Rb	C$\bar{1}$	1	2924.8(7)	-5.4(5)	1478.4(9)	77(1)	35.2(4)	115(2)	1.6(4)	41.8(9)	158(1)	158(1)	160(1)	175(1)
T$_1$(m)	C1	0.74(3)	88(10)	-1928(6)	2275(11)	55(2)	21.9(7)	77(3)	-3.5(8)	35(2)	-8(1)	116(3)	128(3)	149(2)
T$_1$(mi)	C1	0.49(3)	-61(9)	1880(6)	-2186(10)	d								
T$_1$(m)	C$\bar{1}$	0.611(7)	73(2)	-1902.7(9)	2228(2)	57(2)	22.7(7)	79(3)	-2.9(8)	35(2)	-7.6(9)	119(3)	131(3)	150(2)
T$_1$(0)	C1	0.32(3)	70(8)	-8097(6)	2191(10)	53(2)	21.1(6)	73(3)	7.9(8)	32(2)	6.4(9)	117(2)	120(2)	148(2)
T$_1$(0i)	C1	0.69(3)	-72(10)	8072(7)	-2190(12)	d								
T$_1$(0)	C$\bar{1}$	0.507(7)	71(2)	-8085.4(8)	2190(2)	54(2)	21.3(6)	73(3)	8.2(7)	33(2)	6.7(8)	117(3)	120(2)	150(2)
T$_2$(m)	C1	0.96(3)	7226(12)	-1218(7)	3463(13)	64(3)	17.9(9)	68(3)	3(2)	28(2)	2(2)	120(3)	126(3)	148(3)
T$_2$(mi)	C1	0.84(3)	-7168(11)	1141(7)	-3425(12)	d								
T$_2$(m)	C$\bar{1}$	0.896(7)	7197(2)	-1178(1)	3443(2)	65(3)	19.1(8)	68(3)	4(1)	29(2)	3(2)	120(3)	128(3)	149(3)
T$_2$(0)	C1	0.88(3)	7192(11)	-8822(7)	3454(12)	65(3)	19.3(3)	69(3)	2(1)	30(3)	-1(2)	121(3)	130(3)	147(3)
T$_2$(0i)	C1	0.87(3)	-7213(11)	8794(7)	-3390(12)	d								
T$_2$(0)	C$\bar{1}$	0.873(7)	7202(2)	-8808(1)	3422(2)	68(3)	20.0(8)	71(3)	3(1)	31(2)	-1(2)	122(3)	132(3)	152(3)
O$_A$(2)	C1	1	83(29)	-1506(18)	-13(38)	59(10)	33(3)	217(18)	-2(4)	46(9)	-11(6)	137(13)	167(8)	229(10)
O$_A$(2i)	C1	1	83(29)	1457(18)	-111(38)	d								
O$_A$(2)	C$\bar{1}$	1	2(6)	-1482(4)	51(7)	68(7)	33(3)	228(13)	-4(4)	39(8)	-9(5)	146(8)	171(7)	239(6)
O$_A$(1)	C1	1	6575(30)	15(18)	2925(33)	141(16)	22(4)	117(15)	-3(6)	32(11)	4(5)	135(12)	160(8)	235(14)
O$_A$(1i)	C1	1	-6730(30)	-2(18)	-2724(33)	d								
O$_A$(1)	C$\bar{1}$	1	6657(6)	8(4)	2821(7)	165(10)	23(3)	128(11)	3(4)	11(8)	-3(4)	141(8)	160(7)	270(7)
O$_B$(m)	C1	1	8367(29)	-1561(20)	2293(38)	110(15)	54(5)	229(14)	20(6)	145(12)	23(6)	79(28)	200(9)	255(7)
O$_B$(mi)	C1	1	-8116(29)	1586(20)	-2258(38)	d								
O$_B$(m)	C$\bar{1}$	1	8242(6)	-1576(4)	2272(7)	133(9)	57(4)	213(13)	10(5)	135(10)	27(6)	121(8)	208(7)	252(7)
O$_B$(0)	C1	1	8285(29)	-8439(20)	2175(34)	114(10)	57(4)	162(15)	-1(5)	109(10)	-23(6)	104(21)	204(7)	235(7)
O$_B$(0i)	C1	1	-8149(29)	8407(20)	-2280(34)	d								
O$_B$(0)	C$\bar{1}$	1	8217(6)	-8422(4)	2228(7)	116(8)	57(4)	175(12)	-5(5)	100(9)	-21(5)	139(8)	200(7)	236(7)
O$_C$(m)	C1	1	519(28)	-3215(19)	2608(32)	72(8)	34(4)	100(16)	5(4)	36(9)	-5(6)	143(13)	150(8)	178(11)
O$_C$(mi)	C1	1	-390(28)	3132(19)	-2731(32)	d								
O$_C$(m)	C$\bar{1}$	1	458(5)	-3172(4)	2671(6)	78(7)	35(3)	115(10)	4(4)	31(7)	-8(4)	152(7)	160(7)	189(7)
O$_C$(0)	C1	1	484(32)	-6819(20)	2609(34)	90(7)	32(3)	114(12)	-1(4)	34(8)	2(5)	156(8)	166(8)	183(8)
O$_C$(0i)	C1	1	-452(32)	6775(20)	-2660(35)	d								
O$_C$(0)	C$\bar{1}$	1	467(6)	-6799(4)	2633(6)	90(7)	33(3)	116(10)	-3(4)	38(7)	3(4)	158(7)	165(8)	183(7)
O$_D$(m)	C1	1	1787(29)	-1298(17)	4280(29)	82(10)	26(3)	47(17)	-2(4)	-4(11)	-2(6)	94(20)	151(8)	196(6)
O$_D$(mi)	C1	1	-1614(29)	1231(17)	-3994(29)	d								
O$_D$(m)	C$\bar{1}$	1	1702(5)	-1264(3)	4138(6)	90(7)	26(3)	74(9)	0(4)	12(7)	2(4)	124(8)	152(7)	195(6)
O$_D$(0)	C1	1	1692(28)	-8750(17)	4057(29)	86(10)	27(3)	56(13)	3(5)	7(10)	-1(5)	105(15)	152(8)	191(7)
O$_D$(0i)	C1	1	-1788(28)	8748(17)	-4176(29)	d								
O$_D$(0)	C$\bar{1}$	1	1736(6)	-8749(3)	4114(6)	97(7)	28(3)	72(9)	5(4)	27(7)	3(4)	125(8)	156(7)	193(6)

[a]Coefficients in the temperature factor: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

[b]RMS component of thermal displacement along principal axis $\times 10^3$ A.

[c]Coordinates not varied to establish origin. Standard errors in parentheses, corresponding to the last significant digit, are given by the variance-covariance matrix.

[d]Same as preceding line.

flection, separate scale factors and extinction coefficients were adjusted for each data set in the least–squares refinement. Table 1[1] is a listing of scaled F_0 and scaled $\sigma^2(F_0^2)$ for the 4819 reflections that were scanned. Reflections with $F_0^2 < \sigma^2(F_0^2)$ are considered to be unobserved and are omitted from the least–squares refinement. The reflections were corrected for Lorentz and polarization effects and absorption ($\mu = 289.7$ cm^{-1}, correction range 0.58–0.81).

Starting parameters and the same sequence of labeling of the atoms in the asymmetric unit of the centrosymmetric model were taken from Bailey (1969).

The structures were refined by iterative least–squares adjustment using a modification of the Busing, Martin, and Levy (1962) computer program. The quantity minimized by the least–squares program was $\sum w||F_0^2| - |sF_0^2|$ with weights (w) equal to the reciprocals of $\sigma^2(F_0^2)$ where the estimate of $\sigma^2(F_0^2)$ is $\{T + B + [0.03 (T-B)]^2\}/\{A(Lp)^2\}$, where T = total counts, B = background counts, A = ab-

[1] Table 1 may be obtained from: Laboratory Records, Oak Ridge National Laboratory, P.O. Box X, Oak Ridge, Tennessee 37830 by asking for ORNL–DWG–72–2342.

sorption correction, and Lp = Lorentz and polarization corrections (Brown and Levy, 1964). Scattering factors for the ions are from Cromer and Waber (1965) and the anomalous dispersions for Rb and Fe are from Cromer (1965).

The multiplicities of the tetrahedral positions are variable parameters in both the centrosymmetrical ($C\bar{1}$) and asymmetrical ($C1$) models. The positional parameters of the two ions (Fe^{3+} and Si^{4+}) in the same tetrahedron were constrained to be the same and those of Fe^{3+} were varied. The multiplicity, m, of the iron was a variable parameter and the multiplicity of Si^{4+} was constrained to be $1-m$. Anisotropic temperature factors were also adjustable parameters for both models but because of the

Table 3.   Interatomic Distances and Angles—$RbFeSi_3O_8$-$C\bar{1}$.

$Rb-O_A(1)$	3.033(6)	$Rb-O_A(2)$	3.034(5)	$Rb-O_A(2)$	3.062(5)
$Rb-O_D(0)$	3.067(4)	$Rb-O_C(0)$	3.099(5)	$Rb-O_D(m)$	3.117(5)
$Rb-O_C(m)$	3.171(5)	$Rb-O_B(0)$	3.210(5)	$Rb-O_B(m)$	3.226(6)
$Rb-O_A(1)$	3.348(5)	$T_1(m)-O_A(2)$	1.669(5)	$T_1(m)-O_C(m)$	1.700(5)
$T_1(m)-O_B(m)$	1.704(5)	$T_1(m)-O_D(m)$	1.733(4)	$T_1(m)-T_2(0)$	3.011(3)
$T_1(m)-T_2(0)$	3.187(3)	$T_1(m)-T_1(0)$	3.196(2)	$T_1(m)-T_2(m)$	3.214(3)
$T_1(0)-O_A(2)$	1.717(5)	$T_1(0)-O_C(0)$	1.730(5)	$T_1(0)-O_B(0)$	1.735(5)
$T_1(0)-O_D(0)$	1.755(5)	$T_1(0)-T_2(m)$	3.047(3)	$T_1(0)-T_2(m)$	3.193(3)
$T_1(0)-T_2(0)$	3.232(2)	$T_2(m)-O_B(m)$	1.612(5)	$T_2(m)-O_D(0)$	1.619(4)
$T_2(m)-O_C(0)$	1.620(5)	$T_2(m)-O_A(1)$	1.629(5)	$T_2(m)-T_2(0)$	3.111(3)
$T_2(0)-O_B(0)$	1.605(5)	$T_2(0)-O_D(m)$	1.621(4)	$T_2(0)-O_C(m)$	1.625(5)
$T_2(0)-O_A(1)$	1.635(5)	$O_A(2)-O_D(m)$	2.716(6)	$O_A(2)-O_B(m)$	2.732(7)
$O_A(2)-O_D(0)$	2.765(6)	$O_A(2)-O_B(0)$	2.784(7)	$O_A(2)-O_C(m)$	2.844(7)
$O_A(2)-O_C(0)$	2.906(7)	$O_A(1)-O_C(0)$	2.590(6)	$O_A(1)-O_C(m)$	2.590(7)
$O_A(1)-O_B(m)$	2.635(7)	$O_A(1)-O_D(0)$	2.636(6)	$O_A(1)-O_B(0)$	2.645(7)
$O_A(1)-O_D(m)$	2.647(6)	$O_B(m)-O_C(0)$	2.634(7)	$O_B(m)-O_D(0)$	2.685(6)
$O_B(m)-O_C(m)$	2.793(7)	$O_B(m)-O_D(m)$	2.807(7)	$O_B(0)-O_C(m)$	2.647(7)
$O_B(0)-O_D(m)$	2.675(6)	$O_B(0)-O_D(0)$	2.848(7)	$O_B(0)-O_C(0)$	2.872(7)
$O_C(m)-O_D(m)$	2.677(6)	$O_C(m)-O_D(m)$	2.768(6)	$O_C(0)-O_D(0)$	2.687(6)
$O_C(0)-O_D(0)$	2.805(7)				

	$T_1(m)$	$T_1(0)$	$T_2(m)$	$T_2(0)$
$O(A)-T-O(B)$	115.2(3)	114.9(3)	112.4(3)	112.0(3)
$O(A)-T-O(C)$	108.2(3)	107.5(3)	109.1(3)	110.1(3)
$O(A)-T-O(D)$	105.9(3)	105.6(3)	108.8(3)	109.5(3)
$O(B)-T-O(C)$	110.3(3)	112.0(3)	112.1(3)	111.1(3)
$O(B)-T-O(D)$	107.5(2)	107.2(2)	108.5(3)	108.8(3)
$O(C)-T-O(D)$	109.5(3)	109.4(3)	105.7(3)	105.2(3)

$T_1(m)-O_A(2)-T_1(0)$	141.4(3)	$T_2(m)-O_A(1)-T_2(0)$	144.8(4)
$T_2(m)-O_B(m)-T_1(m)$	151.5(4)	$T_2(0)-O_B(0)-T_1(0)$	150.9(4)
$T_2(0)-O_C(m)-T_1(m)$	129.8(3)	$T_2(m)-O_C(0)-T_1(0)$	130.8(3)
$T_2(0)-O_D(m)-T_1(m)$	143.7(3)	$T_2(m)-O_D(0)-T_1(0)$	142.3(3)

A–D refer to the tetrahedral oxygen associated with the T in order of increasing bond length.

Table 4. Interatomic Distances and Angles—$RbFeSi_3O_8$-Cl.

$Rb(i)-O_A(1)$	2.95(3)	$Rb(i)-O_A(2i)$	2.96(3)	$Rb(i)-O_A(2)$	3.03(3)
$Rb(i)-O_D(0)$	3.06(3)	$Rb(i)-O_C(0)$	3.13(3)	$Rb(i)-O_C(m)$	3.16(3)
$Rb(i)-O_D(m)$	3.18(3)	$Rb(i)-O_B(mi)$	3.24(3)	$Rb(i)-O_A(1i)$	3.24(3)
$Rb(i)-O_B(mi)$	3.25(3)	$Rb(0)-O_D(mi)$	3.06(3)	$Rb(0)-O_C(0i)$	3.07(3)
$Rb(0)-O_D(0i)$	3.07(3)	$Rb(0)-O_A(2i)$	3.10(3)	$Rb(0)-O_A(2)$	3.11(3)
$Rb(0)-O_A(1i)$	3.11(3)	$Rb(0)-O_B(0)$	3.17(3)	$Rb(0)-O_C(mi)$	3.17(3)
$Rb(0)-O_B(m)$	3.22(3)	$Rb(0)-O_A(1)$	3.46(3)	$T_1(m)-O_B(m)$	1.61(3)
$T_1(m)-O_C(m)$	1.72(3)	$T_1(m)-O_A(2)$	1.77(3)	$T_1(m)-O_D(m)$	1.79(2)
$T_1(m)-T_2(0)$	2.99(2)	$T_1(m)-T_2(0i)$	3.18(2)	$T_1(m)-T_2(m)$	3.18(2)
$T_1(m)-T_1(0i)$	3.22(2)	$T_1(m)-O_B(0)$	1.66(3)	$T_1(0)-O_D(0)$	1.71(3)
$T_1(0)-O_C(0)$	1.72(3)	$T_1(0)-O_A(2i)$	1.80(3)	$T_1(0)-T_2(m)$	3.03(1)
$T_1(0)-T_1(mi)$	3.17(1)	$T_1(0)-T_2(mi)$	3.22(1)	$T_1(0)-T_2(0)$	3.26(1)
$T_2(m)-O_D(0i)$	1.56(3)	$T_2(m)-O_C(0)$	1.61(3)	$T_2(m)-O_B(m)$	1.66(3)
$T_2(m)-O_A(1)$	1.70(3)	$T_2(m)-T_2(0)$	3.14(2)	$T_2(m)-T_1(0i)$	3.16(2)
$T_2(0)-O_C(m)$	1.55(3)	$T_2(0)-O_A(1)$	1.62(3)	$T_2(0)-O_D(mi)$	1.70(2)
$T_2(0)-O_B(0)$	1.71(3)	$T_2(0)-T_1(mi)$	3.20(1)	$T_1(mi)-O_A(2i)$	1.58(3)
$T_1(mi)-O_D(mi)$	1.68(2)	$T_1(mi)-O_C(mi)$	1.68(3)	$T_1(mi)-O_B(mi)$	1.80(3)
$T_1(mi)-T_2(0i)$	3.03(2)	$T_1(mi)-T_2(mi)$	3.24(1)	$T_1(0i)-O_A(2)$	1.64(3)
$T_1(0i)-O_C(0i)$	1.74(3)	$T_1(0i)-O_D(0i)$	1.80(3)	$T_1(0i)-O_B(0i)$	1.81(3)
$T_1(0i)-T_2(mi)$	3.06(1)	$T_1(0i)-T_2(0i)$	3.21(1)	$T_2(mi)-O_B(mi)$	1.56(3)
$T_2(mi)-O_A(1i)$	1.57(3)	$T_2(mi)-O_C(0i)$	1.62(3)	$T_2(mi)-O_D(0)$	1.68(3)
$T_2(mi)-T_2(0i)$	3.08(2)	$T_2(0i)-O_B(0i)$	1.50(3)	$T_2(0i)-O_D(m)$	1.55(2)
$T_2(0i)-O_A(1i)$	1.66(3)	$T_2(0i)-O_C(mi)$	1.71(3)	$O_A(2)-O_B(m)$	2.75(3)
$O_A(2)-O_B(0i)$	2.77(3)	$O_A(2)-O_D(0i)$	2.78(4)	$O_A(2)-O_D(m)$	2.85(4)
$O_A(2)-O_C(m)$	2.87(4)	$O_A(2)-O_C(0i)$	2.88(3)	$O_A(1)-O_C(m)$	2.47(4)
$O_A(1)-O_D(0i)$	2.57(4)	$O_A(1)-O_C(0)$	2.58(4)	$O_A(1)-O_B(m)$	2.66(3)
$O_A(1)-O_B(0)$	2.76(4)	$O_A(1)-O_B(m)$	2.77(4)	$O_B(m)-O_D(0i)$	2.69(3)
$O_B(m)-O_C(0)$	2.72(4)	$O_B(m)-O_D(m)$	2.77(4)	$O_B(m)-O_C(m)$	2.83(4)
$O_B(0)-O_C(m)$	2.65(3)	$O_B(0)-O_D(0)$	2.76(4)	$O_B(0)-O_A(2i)$	2.80(3)
$O_B(0)-O_D(mi)$	2.81(3)	$O_B(0)-O_C(0)$	2.83(4)	$O_C(m)-O_D(mi)$	2.76(4)
$O_C(m)-O_D(m)$	2.82(3)	$O_C(0)-O_D(0i)$	2.65(3)	$O_C(0)-O_D(0)$	2.76(4)
$O_C(0)-O_A(2i)$	2.94(4)	$O_D(m)-O_B(0i)$	2.54(3)	$O_D(m)-O_C(mi)$	2.61(4)
$O_D(m)-O_A(1i)$	2.64(3)	$O_D(0)-O_B(m)$	2.68(3)	$O_D(0)-O_A(1i)$	2.70(4)
$O_D(0)-O_C(0i)$	2.72(3)	$O_D(0)-O_A(2i)$	2.76(4)	$O_A(2i)-O_D(mi)$	2.59(4)
$O_A(2i)-O_B(mi)$	2.72(3)	$O_A(2i)-O_C(mi)$	2.83(4)	$O_A(1i)-O_B(mi)$	2.52(4)
$O_A(1i)-O_B(0i)$	2.55(4)	$O_A(1i)-O_C(0i)$	2.60(4)	$O_A(1i)-O_C(mi)$	2.72(4)
$O_B(mi)-O_C(0i)$	2.55(4)	$O_B(mi)-O_C(mi)$	2.77(4)	$O_B(mi)-O_D(mi)$	2.85(3)
$O_B(0i)-O_C(mi)$	2.66(3)	$O_B(0i)-O_C(0i)$	2.92(4)	$O_B(0i)-O_D(0i)$	2.95(4)
$O_C(mi)-O_D(mi)$	2.73(3)	$O_C(0i)-O_D(0i)$	2.85(4)		

	$T_1(m)$	$T_1(0)$	$T_2(m)$	$T_2(0)$
$O(A)-T-O(B)$	116(2)	110(2)	113(2)	102(2)
$O(A)-T-O(C)$	109(2)	114(2)	113(2)	116(2)
$O(A)-T-O(D)$	109(2)	108(2)	104(2)	108(2)
$O(B)-T-O(C)$	110(2)	107(2)	112(2)	107(2)
$O(B)-T-O(D)$	107(2)	104(2)	102(2)	112(2)
$O(C)-T-O(D)$	106(2)	113(2)	111(2)	111(2)

	$T_1(mi)$	$T_1(0i)$	$T_2(mi)$	$T_2(0i)$
$O(A)-T-O(B)$	106(2)	117(2)	107(2)	113(2)
$O(A)-T-O(C)$	120(2)	107(2)	106(2)	108(2)
$O(A)-T-O(D)$	107(2)	107(2)	111(2)	112(2)
$O(B)-T-O(C)$	109(2)	107(2)	109(2)	111(2)
$O(B)-T-O(D)$	110(2)	110(2)	112(2)	106(2)
$O(C)-T-O(D)$	105(2)	109(2)	111(2)	108(2)

$T_1(0i)-O_A(2)-T_1(m)$	142(2)	$T_2(0)-O_A(1)-T_2(m)$	143(2)
$T_1(m)-O_B(m)-T_2(m)$	153(2)	$T_1(0)-O_B(0)-T_2(0)$	150(2)
$T_2(0)-O_C(m)-T_1(m)$	132(2)	$T_2(m)-O_C(0)-T_1(0)$	131(2)
$T_2(0i)-O_D(m)-T_1(m)$	144(2)	$T_2(mi)-O_D(0)-T_1(0)$	144(2)
$T_1(mi)-O_A(2i)-T_1(0)$	140(2)	$T_2(mi)-O_A(1i)-T_2(0i)$	145(2)
$T_2(mi)-O_B(mi)-T_1(mi)$	149(2)	$T_2(0i)-O_B(0i)-T_1(0i)$	151(2)
$T_1(mi)-O_C(mi)-T_2(0i)$	127(2)	$T_2(mi)-O_C(0i)-T_1(0i)$	131(2)
$T_1(mi)-O_D(mi)-T_2(0)$	142(2)	$T_1(0)-O_D(0i)-T_1(0i)$	140(2)

pseudo–center of symmetry in $C1$, the temperature factors of atoms related by a center of symmetry [e.g., $O_A(2)$ and $O_A(2i)$] have large correlation coefficients, >0.9; therefore, the temperature factors of pseudo–centrosymmetrical atoms in $C1$ were constrained to be equal (Table 2). The multiplicities of the eight tetrahedral sites were adjusted simultaneously with the adjustment of anisotropic temperature factors for each of the eight sites. Finally, the multiplicities were fixed and anisotropic temperature factors were determined with the constraints discussed above.

Three dimensional electron density maps were calculated for each tetrahedral position and the electron density was integrated numerically in a 0.59Å–radius sphere. The radius of the sphere was chosen arbitrarily to give a result for T-(O) in model $C\bar{1}$ nearly equal to the multiplicity for the same site. The amount of tetrahedral iron was also calculated assuming a linear variation in the site to oxygen distance of 1.60A for Si^{4+} to 1.88A for Fe^{3+} (Shannon and Prewitt, 1969). Figure 1 is a stereoscopic pair of drawings illustrating the contents of a unit cell of $RbFeSi_3O_8$.

Results

Structural models were calculated for both $C\bar{1}$ and $C1$ to determine if the distribution of the iron was noncentrosymmetrical even though the positional parameters were pseudo–centrosymmetrical. The atomic positional parameters, tetrahedral site multiplicities, and temperature factors for both structure models are listed in Table 2. The interatomic distances for $C\bar{1}$ are listed in Table 3 and the distances for $C1$ are in Table 4. The discrepancy indices for 2408 reflections $> \sigma^2(F_0^2)$

$$wR = [\Sigma w(F_0^2 - F_c^2)^2]^{1/2}/(\Sigma w(F_0^2)^2)^{1/2}$$

Table 5

Atomic Percent Iron at T-0 Positions

Position	$C\bar{1}$				$C1$			
	M	ED	T-0	A	M	ED	T-0	A
$T_1(m)$	38.9	42.4	35.6	38.9	26	33.2	43.7	34.3
$T_1(0)$	49.3	49.5	47.4	48.7	68	63.6	43.7	58.4
$T_2(m)$	10.4	13.1	7.2	10.2	4	9.2	11.7	8.3
$T_2(0)$	12.7	12.6	7.7	11.0	12	14.0	16.1	14.0
$T_1(mi)$					51	52.9	30.4	44.7
$T_1(0i)$					31	35.6	52.6	39.7
$T_2(mi)$					16	9.3	2.7	9.3
$T_2(0i)$					13	11.5	1.8	8.7

M - multiplicity   T-0 - average of T-0 distances

ED - electron density   A - average of M, ED and BA

Table 6

Average interatomic distances and angles

A - Intermediate Microcline, Bailey (1969)

B - $RbFeSi_3O_8$ - $C\bar{1}$

	A	B		distance - Angstroms	
				A	B
$T_1(m)$-0	1.643	1.702	0-0	2.682	2.762
$T_1(0)$-0	1.694	1.734		2.765	2.810
$T_2(m)$-0	1.616	1.620		2.637	2.645
$T_2(0)$-0	1.619	1.622		2.642	2.647
				Angles - degrees	
				A	B
			$0-T_1(m)$-0	109.49	109.43
			$0-T_1(0)$-0	109.45	109.43
			$0-T_2(m)$-0	109.41	109.43
			$0-T_2(0)$-0	109.43	109.45
			T-0-T	141.83	141.90

and

$$R(F_0{}^2) = \Sigma \, |F_0{}^2 - F_c{}^2|/\Sigma \, |F_0{}^2|$$

are respectively 0.0846 and 0.0755 for $C\bar{1}$ and 0.0830 and 0.0747 for $C1$. The results of Hamilton's (1965) significance tests on the R factors show that the ratios of $wR(C\bar{1})/wR(C1)$ and $R(C\bar{1})/R(C1)$ are not significant at the 0.01 level.

The $C\bar{1}$ structural model is considered to be correct because seven interatomic distances; $T_2(m)-O_D(0i)$, $T_2(0)-O_C(m)$, $T_1(mi)-O_A(2i)$, $T_2(mi)-O_B(mi)$, $T_2(mi)-O_A(1i)$, $T_2(0i)-O_B(0i)$ and $T_2(0i)-O_D(m)$ in the $C1$ model are shorter than the 1.60Å distance that is generally found for tetrahedral Si–O (Shannon and Prewitt, 1969) (Table 4), and the relative amounts of iron at the tetrahedral sites as calculated from average T–O distances are not consistent with the values given by the

multiplicities and electron densities (Table 5). The $N(z)$ test for center of symmetry does not clearly distinguish the choice of space group.

The $C\bar{1}$ structure of RbFe feldspar is that of an intermediate microcline similar to that described by Bailey and Taylor (1955). Table 6 summarizes the average of interatomic distances and angles for both structures for direct comparison. A more detailed comparison can be made by referring to Bailey (1969), page 1543. The T–O distances average respectively 0.059, 0.040, 0.004, and 0.003Å longer with Fe in place of Al. The O–O distances in the respective tetrahedral edges average respectively 0.080, 0.045, 0.008, and 0.005Å longer. The Rb–O distances range from 3.033(6) to 3.348(5)Å.

Table 5 summarizes the iron substitution at each tetrahedral position as estimated from positional multiplicity parameters, average T–O tetrahedral bond lengths, and electron density integration. Note the lack of consistency in the values for the $C1$ structure.

There is no systematic relationship between T–T distances and iron occupancy (Table 3) or between Rb–T distances and iron occupancy.

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