# The Crystal Structure of RbTh<sub>3</sub>F<sub>13</sub>

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The complex fluoride RbTh<sub>3</sub>F<sub>13</sub> crystallizes with space group  $P2_1ma$ ;  $a_0 = 8.6490$  (5),  $b_0 = 8.176$  (2) and  $c_0 = 7.4453$  (4) Å. There are two formula weights per unit cell and the calculated density is 6.488 g.cm<sup>-3</sup>. There are two thorium ions in the asymmetrical unit and each of these ions has nine nearest neighbor fluorine ions at the corners of capped trigonal prisms. The Th-F distances range from 2.32 (3) to 2.48 (2) Å. The Rb ion has eleven nearest neighbor fluorine ions in the range 2.79 (3) to 3.37 (2) Å. The final discrepancy index is 0.0710 for 2134 Ag K $\alpha$  reflections >  $\sigma$ .

Single crystals of RbTh<sub>3</sub>F<sub>13</sub> were grown as the primary phase from a melt having the composition; 40 mole % RbF and 60 mole % ThF<sub>4</sub>. A single crystal ellipsoid  $0.312 \times 0.234 \times 0.312$  mm was mounted on a computer controlled Picker four-circle goniostat equipped with a scintillation counter detector.

The conditions for reflection, hk0, h=2n, and the diffraction symmetry *mmm* are consistent with space groups *Pmma*, *P*2<sub>1</sub>*ma* and *Pm*2*a*. The final choice for the structure is *P*2<sub>1</sub>*ma*. The more conventional space group *Pmc*2<sub>1</sub> may be derived by a change of axes, *bca*, from *P*2<sub>1</sub>*ma* with corresponding appropriate changes in Tables 1 and 3. Independent reflections hkl, h, k and  $l \ge 0$  were measured by the  $\theta-2\theta$  scan technique using unfiltered Ag K $\alpha$  radiation to  $2\theta=70^{\circ}$ . The 2576 accessible reflections were corrected for Lorentz and polarization effects and absorption ( $\mu = 466.95 \text{ cm}^{-1}$ ), but only the 2134 reflections  $>\sigma$  were used in the least-squares adjustment for the final parameters.

The unit-cell parameters were determined by a leastsquares adjustment of 33 high angle  $(120-145^{\circ} 2\theta)$ Cu  $K\alpha_1$  reflections (Cu  $K\alpha_1 = 1.54050$  Å,  $T = 24^{\circ}$ C). The cell parameters are  $a_0 = 8.6490$  (5),  $b_0 = 8.176$  (2) and  $c_0 = 7.4453$  (4) Å. There are two formula weights per unit cell and the calculated density is 6.488 g.cm<sup>-3</sup>.

The initial positions of the Th ions were determined from a three-dimensional plot of the Patterson function. These Th positions were tried in each of the three possible space groups and good results were obtained with only  $P2_1ma$ . The positions of the Rb and F ions were determined from Fourier and difference Fourier syntheses. The structure was refined by iterative leastsquares adjustment using a modification of the Busing, Martin & Levy (1962) computer program. The quantity minimized by the least-squares program was  $\sum w ||F_o^2| - |F_c^2||$  with weights, w, equal to the reciprocals of the variances estimated from the empirical equation

$$\sigma^2(F_o^2) = \{ [T + B + (0.03(T - B))^2] / [A(Lp)^2] \}$$

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Table 1. Atomic parameters (
$$\times 10^3$$
) and temperature factors ( $\times 10^4$ ) for RbTh<sub>3</sub>F<sub>13</sub>

Standard errors in parentheses, corresponding to the last significant digit, are given by the variance-covariance matrix Temperature factor = exp  $\left[-\left(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl\right)\right]$ . The *F* temperature factors are constrained to be isotropic;  $\beta_{22} = \beta_{11} (b^{*2}/a^{*2}), \beta_{33} = \beta_{11} (c^{*2}/a^{*2}), \beta_{12} = \beta_{13} = \beta_{23} = 0$ .

	x	у	z	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Th(1)	0*	0	108.2(2)	11.7 (6)	18.6 (8)	19(1)	0	5 (2)	0
Th(2)	834.3 (2)	252.83 (9)	602.01 (8)	14·2 (4)	16.4 (4)	26.5 (6)	+13(5)	11 (1)	-0.4(9)
Rb	695.9 (5)	1*2	104.1 (8)	52 (5)	121 (7)	60 (6)	0	10 (6)	0
	x	у	Z	$\beta_{11}$					
F(1)	848 (4)	47	453 (3)	30 (8)					
F(2)	596 (2)	302 (2)	452 (3)	37 (7)					
F(3)	144(2)	744 (2)	173 (3)	27 (5)					
F(4)	457 (2)	803 (2)	111 (3)	46 (7)					
F(5)	865 (2)	176 (2)	304 (3)	35 (6)					
F(6)	366 (3)	<del>1</del> 7†	230 (3)	23 (8)					
F(7)	737 (3)	Õ	037 (3)	38 (9)					
F(8)	179 (3)	0	364 (3)	26 (8)					
F(9)	460 (3)	0	409 (4)	31 (8)					

\* Arbitrary value to establish origin on  $2_1$  axis.

† These values are not multiplied by 103.

Table 2. Interatomic distances for RbTh<sub>3</sub>F<sub>13</sub>

Th(1) - F(7)	2·32 (3) Å	2(Th(1)-F(4))	2·32 (2) Å	Th(1) - F(7)	2·33 (3) Å
2(Th(1)-F(5))	2.36 (3)	2(Th(1) - F(4))	2.45 (2)	Th(1) - F(7)	2.48 (3)
Th(2) - F(5)	2.32(2)	Th(2) - F(9)	2.34(1)	Th(2) - F(2)	2.34 (2)
Th(2) - F(3)	2.35 (2)	Th(2) - F(1)	2.38(1)	Th(2) - F(2)	2.38 (2)
Th(2) - F(6)	2.39(2)	Th(2)—F(4)	2.43 (2)	Th(2) - F(8)	2.48 (2)
Rb - F(1)	2.79(3)	Rb	2.89 (2)	2(Rb - F(3))	2.91 (2)
Rb——F(6)	3.00(2)	2(Rb - F(2))	3.17 (2)	2(Rb - F(4))	3.22 (2)
2(Rb - F(5))	3.37 (2)	2(RbF(4))	3.72 (2)	F(1) - F(6)	2.50 (3)
2(F(1) - F(2))	2.72 (3)	2(F(1) - F(2))	2.82(3)	2(F(1) - F(5))	2.83 (2)
F(2) - F(9)	2.75 (2)	F(2) - F(5)	2.77 (3)	F(2)F(3)	2.85 (2)
F(2) - F(5)	2.90 (3)	F(2) - F(8)	2.91(2)	F(2)F(4)	2.94 (3)
F(2) - F(6)	3.05 (3)	F(3) - F(8)	2.54(2)	F(3)F(5)	2.68 (2)
F(3) - F(4)	2.70 (3)	F(3) - F(7)	2.73 (2)	F(3)F(4)	2.80 (2)
F(3) - F(6)	2.81(2)	F(4) - F(7)	2.72(3)	F(4)F(9)	2.74 (3)
F(4) - F(6)	2.75 (2)	F(4) - F(7)	2.96 (3)	F(5) - F(7)	2.69 (3)
F(5) - F(9)	2.70 (3)	F(5) - F(5)	2.88 (3)	F(8) - F(9)	2.45 (3)
F(8)F(9)	2.54(3)				

corrections (Brown & Levy, 1964). The scattering factors for the ions were taken from Cromer & Waber (1965) and the anomalous dispersion terms for Rb and Th for Ag  $K\alpha$  radiation were taken from Cromer (1965). The values of The standard deviation of an observation of unit weight  $[\sum w(F_o - F_c)^2/(n_o - n_v)]^{1/2}$  is 1.387 where  $n_o$  is the number of reflections, 2134, and  $n_v$  the number of variables, 53. The atomic parameters and temperature factors are listed in Table 1, the interatomic distances in Table 2

 $R_1 = \sum ||F_o^2| - |F_c^2|| / \sum |F_o^2| = 0.0910 \text{ for } 2134 \text{ reflections } > \sigma$ 

and

 $R_2 = \sum ||F_o| - |F_c|| / \sum |F_o|$ = 0.0710 for 2134 reflections >  $\sigma$ .

# Table 3. Observed and calculated structure factors and phase angles for $RbTh_3F_{13}$

ALPHA = Phase angle  $\alpha \times 10$ 

## Table 3 (cont.)

L FORS FER, RUMA L FORS FER,	RUPHA L FOSS FLAL ALFHA
· 231 249 422 5 46 41 1506 14 33 35 966 3 13 L 11 88 90 571 0 92 94	957 3 141 144 97
2 85 88 577 7 19 8 285 1 8 17 821 2 83 77 873 13 72 75 1551 2 9 26	784 5 28 23 1320
3 64 67 1054 8 38 30 141 2 101 105 835 3 27 24 871 14 53 49 962 3 46 56 4 204 214 1353 9 38 34 150 3 61 58 1097 4 38 32 919 4 5L 4 5L 4 68 72	903 7 126 117 1786
5 126 138 843 2 14 L 4 27 33 622 5 14 10 907 0 131 138 120 5 137 136 6 147 156 1330 0 130 129 419 5 11 19 1321 6 62 55 931 1 81 87 318 6 67 54	966 9 55 56 1430
7 39 33 327 1 75 70 924 6 61 66 883 7 15 20 925 2 35 42 233 7 20 24 8 90 92 446 2 28 34 466 7 46 45 1015 8 42 46 975 3 56 56 1706 8 22 28	991 10 36 35 1378 717 11 69 67 164
9 87 88 855 3 36 37 1016 8 46 51 971 9 0 1 179 4 87 86 1789 9 97 98	348 (2 115 115 1343 975 (13 54 65 182
11 63 59 1103 5 74 71 897 10 0 28 966 1 29 23 909 6 70 63 1718 4 13 1	14 14 26 1284
13 35 36 1256 7 14 20 451 12 59 54 906 3 56 51 894 8 50 48 33 1 30 40	341 1 73 84 1075
2 7 L 2 15 L 14 24 5 474 5 30 7 1272 10 44 51 28 3 25 23	1460 3 107 108 924
0 97 98 49 0 42 38 278 3 6 L 6 89 90 875 11 22 18 189 4 52 54 I 77 82 0 1 54 48 197 3 85 89 991 7 50 38 908 12 14 14 1742 5 50 42	1420 5 33 39 846
2 23 22 177 2 0 12 663 4 130 135 923 3 15 L 13 36 36 1726 6 36 38 3 54 57 1635 3 21 26 1683 5 0 16 1251 1 49 38 994 14 27 45 1780 7 30 4	1569 6 65 66 887 372 7 104 103 829
4 80 83 1620 4 50 36 1505 6 165 169 879 2 0 17 888 4 6 L 8 44 30 5 95 96 1779 5 50 55 1610 7 53 59 791 3 54 59 912 0 263 224 439 4 14 L	65 8 57 57 1015 9 14 19 731
6 57 53 1615 6 12 24 1375 8 180 124 893 4 23 7 802 1 106 114 821 0 136 133	402 10 0 29 996
8 25 42 115 0 77 70 909 10 46 45 1108 6 37 21 635 3 56 56 1077 2 41 33	494 12 55 50 751
10 42 53 245 2 20 19 993 12 137 137 914 1 77 70 930 5 125 126 852 4 109 106	1413 5 6 L BOI 1 152 157 1382
12 23 14 1590 4 52 53 951 3 7 1 3 7 1 3 108, 104 907 7 38 26 620 6 80 75	1322 2 145 152 54
2 8 L 1 244 219 1001 2 59 59 606 0 197 170 1097 9 80 79 915 4 15 1	86 89 213
1 134 140 1003 2 67 97 1355 3 43 34 641 1 356 327 463 10 143 145 442 0 31 39 1 188 191 498 3 334 339 946 4 30 17 885 2 58 58 635 11 71 57 976 1 41 43	179 6 73 74 1710
- 2 50 55 1067 4 58 60 1045 5 24 15 819 3 114 118 1386 12 32 21 1226 2 0 8 - 3 77 76 1388 5 30 34 464 6 41 52 1045 4 77 95 985 13 14 28 1261 3 37 23	1650 8 113 107 1719
4 100 100 892 6 43 41 1084 7 72 75 957 5,282 297 1361 4 7 L 4 34 30 5 196 200 1334 7 290 268 845 8 16 30 1160 6 106 104 897 0 89 88 234 5 42 52	1495 9 70 59 366 1609 10 0 31 585
6 101 103 804 8 63 58 881 9 24 16 962 7 52 52 1034 1 83 81 34 4 16 1 7 33 27 1173 9 87 80 941 10 25 27 887 8 46 39 436 2 18 20 356 0 70 65	. 11 97 90 1343 954 12 81 73 31
8 47 45 678 10 32 25 869 11 57 52 881 9 184 178 380 3 45 40 1664 1 87 84 9 121 124 404 11 145 144 957 12 34 34 843 10 92 86 908 4 67 66 1550 2 27 19	409 13 84 81 1360 871 5 7 L
10 76 78 919 12 57 42 926 13 36 29 881 11 101 97 570 5 79 79 1740 5 0 1 11 70 76 510 13 116 112 923 3 8 1 12 7 22 1386 6 45 47 1557 1 129 119	1 56 62 746
12 0 23 1536 14 36 39 920 1 157 167 957 13 85 86 1537 7 13 7 846 2 254 245 13 48 56 1438 15 42 41 781 2 54 61 873 14 43 56 966 8 52 40 155 3 152 158	1396 3 91 89 682
2 9 L 3 1 L 3 224 226 920 4 1 L 9 63 65 8 4 140 140 0 108 109 133 1 26 26 616 4 37 23 880 0 128 154 16 10 56 45 232 5 24 22	1476 5 0 22 930
1 58 52 274 2 100 107 779 5 2 25 908 1 122 114 297 11 16 17 152 6 174 174 3 17 21 73 1 78 1 98 1 980 5 18 19 197 19 10 12 114 129 11 16 17 152 6 174 174	385 7 65 70 998 1760 8 68 66 843
3 33 42 1547 4 32 29 596 7 202 203 905 3 63 64 1533 13 40 37 1792 8 177 169	459 9 22 8 1375
5 71 69 1602 6 75 69 973 9 58 66 942 5 96 94 1509 0 119 121 919 10 45 39	1420 11 46 47 816
7 9 10 257 8 47 46 1079 11 108 115 935 7 4 4 278 2 31 36 944 12 122 122 8 43 43 65 9 0 12 12 12 12 12 12 12 12 12 12 12 12 12	1337 13 0 20 675
9 50 53 135 10 34 31 928 13 78 87 889 9 73 75 111 4 104 98 891 14 0 29	1312 1 93 101 8
11 31 17 477 12 46 50 871 1 27 29 888 11 22 20 149 6 89 87 855 1 109 99 12 22 9 1655 1 24 23 825 2 87 42 881 12 21 12 178 7 88 30 908 2 153 147	972 3 121 118 79 968 u 91 96 1292
2 10 L 14 0 8 555 3 34 96 933 13 43 41 1738 8 40 38 786 3 135 133 0 189 191 415 3 21 4 51 41 085 14 43 45 1738 9 129 133 366 4 61 63	824 5 27 29 1269
1 98 105 940 1 28 23 1252 5 0 14 1063 4 2 L 10 88 78 907 5 37 42 5 5 5 5 5 5 1 2 36 23 1252 5 0 14 1063 4 2 L 10 88 78 907 5 37 42	966 7 107 101 1765 985 8 132 127 #65
3 48 51 1055 3 108 113 1012 7 37 30 1007 1 171 167 890 12 8 22 1398 7 114 112	880 9 35 47 1486
5 105 104 888 5 26 37 1414 9 0 4 836 3 73 75 991 0 103 108 139 9 30 17	671 11 52 61 184
7 36 28 409 7 63 64 902 11 27 31 905 5 144 146 890 2 3 17 217 11 54 64	932 5 9 L
8 71 67 993 6 222 216 890 12 43 50 861 6 100 177 1277 3 56 52 1503 12 53 6 9 67 71 820 9 43 47 845 5 10 L 7 49 40 519 4 69 70 1716 13 41 33	2 697 2 72 69 778
11 43 46 107 11 10 10 1 37 32 32 1 10 1 10 10 10 10 1 10 10 10 10 10 10	L 4 31 35 531
2 11 L 13 34 40 908 4 100 100 937 11 66 52 984 8 48 36 8 2 190 19	69 6 54 51 1059
0 66 64 237 14 9 54 754 5 0 9 1100 12 25 25 1335 9 49 48 162 3 241 24 1 72 67 138 3 3 L -6 125 126 882 13 35 31 1154 10 58 51 106 4 130 13	0 134 8 54 40 1105
2 15 14 3/4 1 68 /4 //8 / 5/ 5/ 5/ 62/14 88 10/14511 0 14 448 5 4/ 3 3 45 40 1556 2 76 62 688 8 140 136 904 .4 3 L 12 0 13 1667 6 106 9	6 1782 10 27 22 911
4 51 56 1585 3 94 96 746 9 36 37 847 0 138 139 150 4 10 1 7 197 19 5 65 74 1734 4 30 38 993 10 24 35 1106 1 105 104 216 0 199 196 419 8 123 12	2 459 11 35 48 984 4.1677 12 32 38 697
5 30 35 1523 5 29 17 833 11 24 14 869 2 15 23 693 1 87 86 851 9 83 7 7 0 8 626 6 84 78 1043 311 L 3 47 47 1341 2 55 50 493 10 27 3	5 406 5 10 L 7 528 1 112 112 1337
8 36 32 79 7 70 68 1075 1 48 44 882 4 85 84 1579 3 40 42 974 11 96 9 9 54 59 118 8 32 40 1234 2 35 36 890 5 107 102 1612 4 165 161 1396 12 78 7	6 1314 2 106 107 26 9 0 3 168 161 1348
10 - 33 59 507 9 0 18 884 3 77 78 929 6 59 57 1562 5 100 97 881 13 89 9 11 22 22 230 10 39 33 895 4 28 8 619 7 31 7 222 6 111 111 1310 14 35 4	1 1361 4 72 67 176 0 459 5 29 19 363
C 105 108 951 12 29 42 801 6 31 30 927 9 73 72 118 8 78 72 323 1 100 10	0 715 7 140 132 459
1 131 132 453 13 38 25 855 7 50 58 925 10 64 58 159 9 52 66 921 2 128 13 2 35 31 1034 14 22 7 600 8 21 17 1006 11 13 15 138 10 116 116 429 3 128 12	1 916 8 84 82 1747 7 750 9 51 52 401
3 59 59 1382 3 4 L 9 5 15 1019 12 0 16 1706 11 42 49 994 4 49 5 4 72 74 930 1 211 221 939 10 18 20 900 14 24 41 1654 41 11 L 5 29 2	8 726 10 33 26 603 9 918 11 77 73 1325
5 135 138 1374 2 78 76 1083 11 39 43 901 4 4 L 0 55 57 186 6 98 5 101 7 001 1 57 57 57 58 27 3 12 L 0 147 145 972 1 57 51 100 7 101 5	17 961 511 L 33 915 1 31 34 750
7 27 23 1164 4 36 38 1010 1 110 106 930 1 249 261 449 2 17 16 129 8 77 7 8 41 32 622 5 28 29 694 2 42 46 889 2 55 49 801 3 34 35 1710 9 9 1	18 901 2 81 76 1030 16 1252 3 44 52 621
9 82 94 388 6 37 26 1018 3 165 159 916 3 87 93 1387 4 76 45 1566 10 24 0 52 64 909 7 263 258 874 4 41 33 824 4 102 104 922 5 61 62 1700 11 65 6	31 976 4 37 37 1074 50 827 5 8 19 453
2 13 L 8 58 59 901 5 12 17 875 5 262 265 1382 6 28 34 1571 12 61 1 0 83 76 219 9 80 80 90 6 29 23 1027 6 108 102 867 7 0 6 917 12 27	0 863 6 50 52 722 26 816 7 59 40 141
1 43 37 394 10 37 25 637 7 148 147 696 7 44 42 951 6 24 30 140 14 0 2 20 17 233 11 137 136 950 8 40 40 911 8 42 39 552 9 53 57 116 5 1	6 49 8 48 55 75
3 26 24 1602 12 37 43 891 9 50 45 970 9 160 164 371 10 12 32 32 1 120 1	27 พ.เกิวนิวถึงบีรี



Fig. 1. The crystal structure of  $RbTh_3F_{13}$ .

and the observed and calculated structure factors in Table 3. An extinction correction was applied to  $F_c$  by the method suggested by Zachariasen (1967, 1968). The absolute configuration of the crystal was deter-

mined by Hamilton's (1965) test on the discrepancy factor to be such that the  $\Delta f''$ s are positive  $[R_2(-\Delta f'')]$ =0.0734]. The ratio  $\frac{0.0734}{0.0710}$  is 1.034 which is significant

# Table 3 (cont.)

L F	085_F	CAL ALPHA	FORS	FCAL ALPHA	L FORS	FCAL ALPHA (	FORS	FCAL ALPHA L	FOBS F	CAL ALPHA L P	OBS FEAL AUP	HA L FOBS #	CR. R.PH
ţ	.67	61 11	7 45	35 280	2 37	41 28	46	43 1158 5	60	54 706 0	81 77 8	N 7 75	69 997
ŝ	89	87 32	9 39	36 1728	¥ 136	135 1774	53	22 698 1 46 747		32 1296 1	83 85 10 31 21 32	8 8 41 5 9 36	50 1087
5	28	20 1231 1	1 166	16-2 11 NS 1737	5 48 6 101	100 1800 4	7 74 9 49	72 850 0	515	205 943 3	40 42 165 72 66 175	910 0	31 926 44 834
5	92 84	68 433 1 78 1740 1	2 13	21 833	7 27	23 166 9	27 1 10	28 1043 3	68	66 1193 5 80 1323 6	93 B8 170 50 36 163	13 12 25 16 13 37	45 834 23 754
ş	20	95 486 33 1458 1	J 76 <sup>6</sup>	5 L 73 191	9 29	23 1779 1	64	52 924 4 35 1082 9	133	129 844 7	14 1 43	5 9	້
1	43 <sup>5</sup>	13 L 44 1056	65	66 170	0 20	21 24 1	i o	, 26 775 6	120	120 713 9	66 62 12	N 2 200	242 957
Ś.	48	42 639		41 1582	2 0	2 345	140	143 538 8	68	52 659 11	31 25 6	j ų 108	117 952
ý S	0	19 392 1	5 95 5 34	89 1712	4 35 5 67	28 1764	212	213 513 10	100	97 958 79 88 0	8 8 L	, 6 156	152 790
5	26	36 1132	2 0	6 733	5 0	9 1572	¥.	30 850 12	19	35 1749 1	142 143 41	1 8 163	157 881
8	ΨŌ	29 1246	9 72 1 1u	66 108	á xố	23 0	185	177 1287		1 1 3	70 69 13	4 10 43	49 1316
ļ	72	ີ່ຮູ້ເໝຼິ	i ii	28 99	0 48	125 1800	59	66 1535	104	98 47 5	159 154 134	12 12 126	120 920
ž	117	111 1342 1	3 32	35 1782	ş ii	15 1792 1	103	104 635 3	43	N9 1674 7	0 23 150	រុំស្រ្ល័	55 813
ŝ	37	14 484	0 81 1 207	78 1603	1 26	30 85		1 7 L	100	104 1766 9	108 105 43	9 3 70	76 784
7	۶Ś	96 476	35	36 1690	6 47	33 0	78	78 972	ũ	6 277 11	71 67 43	0 5 26	18 921
1	. 8 <sup>°</sup>	23 575	ŝ	51 29	ຸ້ນັ	ំទុំ ត	ເຮັ	30 687 9	69	20 59 0	75 80 17	7 7 76.	74 1060
j.	ñ	31 544	ș și	57 8	2 20	16 47	្រំ	51 951 11	18	23 43 2	18 24	0 9 3	17 997
ŝ	ល័្	14 1005	32	12 1020	4 63	54 1786	÷ 43	46 1018 13	ំ ទ័រ	39 1772 4	69 68 167	5 14 46	46 874
1	45	42 41 1	52	46 1792	1 121	107 1598 1	3 34	26 983 0	ူးလို	215 465 6	57 46 16	<u> </u>	4 L -
ó	373	354 43 1	2 36	29 1632	3 150	154 1701 1	37	41 626	65	65 408 B	26 37 12	8 2 66	69 1014
ş	72	65 69	<u> </u>	71	\$ 0	10 790	66	66 1800	168	170 1315 10	51 47 10	รัฐัญ	49 937
ų.	274	271 1774	1 35	46 11	7 106	109 33	101	103 1771	1.57	136 1391 0	137 137 41	8 6 30	27 999
é	187	186 1784	រំរំ	23 1590	9 50	47 8	9	11 556 6	i in the second se	77 468 2	16 37 36	4 8 63	59 835
6	10	រប់ ប៉ូរី	\$ 50	49 1780	ii Ai	53 1695	56	87 14 10	135	126 444 4	119 116 13	9 10 0	20 428
10	180	179 23	7 0	3 133	13 145	56 1766	38	40 67 1	0	7 378 6	96 92 140	12 12 45	38 621
12	ő	N 821	9 28	27 81	1 26	រូវប្រទាំ	1 31	42 1786	ૺૢૢૢૢૢૢૢૢૢૢૢૢ	31 8	61 56 49	6 1 11	46 672
ių	9Š	108 1758 1	i Xi	10 107	3 84	75 1141	1 46	44 874	115	108 60 10	98 94 43	A 3 63	51 813 30 208
0	102	92 234	ົ້	່ຢີເັ້	5 3	19 753	3 58	64 838	64	56 1756 0	44 49 19	1 5 0 6 6 70	15 724
į	12	12 324	1 70	66 1716	7 64 8 57	72 774	5 19	14 804	100	103 1710 2	22 10 3	8 7 64	55 936
ų,	83	84 1631 85 1690	3 17	6 191	9 <u>19</u>	9 963	7 22	65 980 35 677	i ii	3 875 4	46 42 16	7 9 0	8 1144
ś	35	41 1415	5 66	67 51	11 39	Si 970	9 0 N	11 1523	1 13	72 84 6	20 22 14	76 II 32 16 IZ 44	37 842
8	49 63	48 107	7 42	30 248	13 41	25 858 1	ĩö	42 843 1	1 33	27 40 8	30 24	78 9 77 1 38	5 L 43 834
10	ü3 29	48 151	9 39 0 10	28 1730	1 173	163 462	1 102	100 537 1	ŝ X	421740	8121	2 205	215 954
12	24	19 1633 1	1 33	36 1728	3 261	264 487	3 162	156 530	0 176	168 939 1 177 µ35 2	108 106 4 30 25 11	7 4 103	103 871
14	ä	38 1742		9 L	5 34	38 799	5 18	21 870	2 62	56 1113 3 29 1322 W	54 51 13	13 6 132	132 805
0	99°	89 1471	1 68 2 14	68 21 8 180	7 219	214 1281	7 137 A 72	135 1277	4 119	118 883 5	117 116 13	73 8 146	139 872
ŝ	135	23 1617	3 37	36 1800	9 71	74 1571	9 30	51 1560	6 115	109 743 7 30 1421 B	12 17 14	24 10 42	44 1289
ŝ	61 278	57 134	5 83	78 1734	11 116	115 613 65 1769	1 49	7 11 1	8 50 9 115	55 624 117 440 D	60 13 L	85 1 75	2 807
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# Table 3 (cont.)

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Table 3 (cont.)

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4		67	70	848	0	87	8	1	910	s	45	39	1759	2	9	8	288	1	13	- 28	124	1	50	50	101	3	89	96 1315
- 5		23	13	1605	1	110	10	7	347	6	7	24	1747	3	29	- 20	1800	2	35	12	229	2	101	104	1344		17	3ι
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1		42	- 39	857	3	38	- 5	01	1686	0	127	133	370	5	41	41	1705	- 4	- 28	41	1718	4	58	51	1223	2	41	43 1034
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at the 0.005 significance level for a one-dimensional hypothesis and 2081 degrees of freedom.

### Results

The final structure of  $RbTh_3F_{13}$  is illustrated in Fig. 1. The two Th ions in the asymmetrical unit have nine nearest neighbor F ions at the corners of a trigonal prism with each of the prism faces capped by an F ion. The Th-F distances in both polyhedra range from 2.32 to 2.48 Å (Table 2) with minor differences in the standard errors. The Th(1) polyhedra form columns parallel to  $\mathbf{b}_0$  by sharing edges and the shared edge, F(1)-F(6), alternates successively parallel to  $\mathbf{a}_0$  at y=0.5and parallel to  $\mathbf{c}_0$  at y=0.0. The columns of Th(1) polyhedra are connected by corner sharing, F(2). The columns are intersected by layers of Rb ions and Th(2) polyhedra at y=0.5 and y=0.0 respectively. The layers are parallel to (010). Each Th(2) polyhedron shares an edge, F(3)-F(8), with each of two Th(1) polyhedra and a corner, F(4) and F(5), with each of four other Th(1) polyhedra. The F(1)-F(6) and F(3)-F(8) interatomic distances representing the shared edges are 2.50 (3) and 2.54 (2) Å respectively (Table 2). The Th(2) polyhedra do not touch one another. The Rb ions have eleven nearest neighbor F ions at distances of 2.79 (3) to 3.37 (2) Å.

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# Refinement of the Structure of Sodium β-Alumina

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An X-ray diffractometer re-investigation has been carried out on a clear crystal of fusion-cast sodium  $\beta$ -alumina, hexagonal, space group  $6_3/mmc$ ,  $a = 5\cdot594$ ,  $c = 22\cdot53$  Å,  $D_x = D_m = 3\cdot24 \pm 0\cdot02$  g.cm<sup>-3</sup>; probable unit-cell content = Na<sub>2.58</sub> Al<sub>21.81</sub> O<sub>34</sub>. Using Mo K $\alpha$  radiation, 1175 measured intensities were combined into 545 symmetry-independent values of significant magnitude above background. A least-squares refinement based on these 545 data yielded  $R_1 = 0.034$ . The crystal was apparently typical in that both neutron activation analysis and X-ray intensity analysis found 29% excess soda, relative to the classical formula Na<sub>2</sub>O . 11 Al<sub>2</sub>O<sub>3</sub>. In the averaged unit cell, sodium atoms are smeared out in a complex pattern in the basal mirror plane. About 0.75 sodium atoms are near each of two Beevers-Ross positions, but spread into a broad triangular pattern. The remaining sodium scattering matter is in elongated ellipsoids centered near the positions that are halfway between the oxygen atoms of the basal mirror planes. This is a sixfold set of positions, containing a total of about one sodium ion. Indications are that the counterion defects consist of aluminum vacancies distributed over the (single) 12-fold set of aluminum atoms. Speculations are offered concerning the structure of local defects that would average out to the mean unit cell found.

#### Introduction

The main features of the  $\beta$ -alumina structure were deduced by Bragg, Gottfried & West (1931). These

authors were troubled by the small percentage of sodium found by chemical analysis, which was inconsistent with the space-group symmetry and suggested a degree of randomness in the structure. Subse-