The modified expression for the Fourier transform after inclusion of the line density correction term becomes:

$$C(R,\psi,l/c)=\sum_m \sum_p \sum_d \sum_q \sum_s \varrho_m \,.\, J_p(2\pi Rr_0)\,.\,J_d(2\pi R\Delta)\,. \times J_q(2\pi R\bar{r}_1)\,.\,J_s(2\pi r_1 \sin\alpha\,.\,l/c)E\,, \quad (3)$$

where

$$E=\exp[i(p(\psi-\varphi_0+\tfrac{1}{2}\pi)+q(-\psi+\varphi_0+\varphi_1+\tfrac{1}{2}\pi) +d(\psi+\varphi_1-\varphi_0+\tfrac{1}{2}\pi)+s(\pi+\varphi_1)+2\pi z_0 l/c)\,,$$

subject to

$$N_0p+(N_1-N_0)q+(N_1+N_0)d+N_1s=l+m$$

where

$$\varrho_0=A'\,.\,\varrho_1=\varrho_{-1}=B'$$

and all ϱ_i for $|i|>1$ are zero.

The theory has been checked by comparing results obtained from the expression of equation (3) with the optical diffraction pattern obtained from a mask which corresponded to a projection of a coiled-coil of constant line density and also, following a suggestion by Dr A. R. Stokes, by computing the Fourier transform obtained by a direct substitution of the coordinates of points (x_i,y_i,z_i), spaced at small equal increments in Δs along the coiled-coil, into the expression

$$C(R,\psi,l/c)=\sum_{i=1}^{N} \exp[2\pi i(Xx_i+Yy_i+Zz_i)]\,.$$

In both cases a good agreement was obtained (Pardon, 1966).

Discussion

It has been shown that for models in which the original helix suffers appreciable distortion in forming a coiled-coil, the filament generated with the cartesian coordinate system adopted by Crick (1953) does not approximate to a filament of constant line density. The modified theory outlined above represents the Fourier transform of a model with much less variation in line density; this has been used in calculations for groups consisting of various numbers of coiled-coils packed coaxially. The theory has been found to be very satisfactory for models in which $P=120$ Å, $r_0=50$ Å, 30 Å $\le r_1 \le$ 40 Å and where N_0/N_1 varied from 1 to 3.

If the original simple helix undergoes little distortion in forming the coiled-coil, as in the case considered by Crick (1953*a,b*), then the differences between the Fourier transforms calculated from the original theory and those from the modified theory are small. It seems probable that the original theory is sufficiently accurate for a model such as has been studied for α-keratin.

The proposed super-helical model for nucleohistone and supporting experimental evidence has been recently described by Pardon, Wilkins & Richards (1967).

I should like to thank Professor M. H. F. Wilkins, Dr W. Fuller and Dr A. R. Stokes for helpful discussion, and to acknowledge the award of a Medical Research Council Scholarship.

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The Structure of the *M'*- Phase of YTaO$_4$, a Third Fergusonite Polymorph

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The *M'* phase of fergusonite is closely related to the other known monoclinic form (*M* phase), except that it has a halved *b* axis and its space group is *P*2/*a* instead of *I*2. The structure of the *M'* phase has been determined for YTaO$_4$ from three-dimensional diffractometer data, and its relationship to the other polymorphs is discussed. The lattice parameters of *M'*-YTaO$_4$ are: $a=5{\cdot}292$, $b=5{\cdot}451$, $c=5{\cdot}110$ Å, $\beta=96{\cdot}44°$, $Z=2$.

Introduction

Using a natural fergusonite (impure YNbO$_4$), Komkov (1959) determined the structure of the high-temperature form to be that of scheelite, space group $I4_1/a$, with oxygen coordinates close to those of CaWO$_4$ (Kay, Frazer & Almodovar, 1964), and that of the low-temperature form to be a distorted scheelite, space group *I*2. The two forms, which will be referred to as the *T* phase and the *M* phase, respectively, are related by a second-order (continuous) phase transformation (Gingerich & Bair, 1964); Fig. 1 illustrates how small the actual change is. Wolten & Chase (1967) give details of their discovery that the M phase can be obtained only as a transformation product of the T phase. If crystals are grown below the M–T transformation temperature, a new monoclinic structure, the M' phase, is obtained. On heating, the M' phase is converted by a very sluggish first-order phase transformation to the tetragonal form which, when cooled, distorts slightly to give M.

Crystals of the M' phase have been grown for $YTaO_4$ and for the lanthanon tantalates from Sm through Yb. $YTaO_4$ was chosen for the structure determination reported here, because in this compound the difference between the scattering powers of the two kinds of metal atom is a maximum. The objective of the work was to ascertain the structure type of the new polymorph and to compare it with the other two known forms.

Experimental

Table 1 gives the lattice parameters of $YTaO_4$ in its two monoclinic forms. Those for the M' phase were determined from single-crystal diffractometer data under high-resolution conditions, using copper radiation, $K\alpha_1 = 1{\cdot}54051$ Å. Compared with the M phase, the newly discovered M' phase, apart from belonging to a different space group, has a halved b axis and a, c, and β parameters that are about 1% larger.

Table 1. *Lattice parameters of monoclinic* $YTaO_4$

	M' phase (this work)	M phase (Keller, 1962)
Space group	$P2/a$	I2
a_0	5·292 Å	5·239 Å
b_0	5·451	10·893
c_0	5·110	5·056
β	96·44°	95·52°
Z	2	4

All single crystals of the M' phase are thin (010) laths, elongated parallel to $\mathbf{a}_0$. No equant crystals were found, and the crystals could not be shaped. The one chosen for this investigation had the typical dimensions 0·330 × 0·115 × 0·023 mm. The absorption coefficient of $YTaO_4$ is 962·23 cm^{-1} for Cu radiation and 319·5 cm^{-1} for Ag radiation. It was therefore essential to calculate absorption corrections accurately and to do this from the actual shape of the crystal rather than an approximation. This was accomplished with the aid of a recent version of the computer program ORABS (Wehe, Busing & Levy, 1962). The limiting factor in these calculations is the precision with which one can measure and describe to the program the shape and size of the crystal. To minimize this source of error, a low absorption coefficient is desirable, leading to the choice of Ag radiation, $K\alpha_{av} = 0{\cdot}5608$ Å. The transmission factors calculated for the crystal employed in this work covered a range of 40:1, from a high of 0·1859 for the 091 reflection to a low of 0·0047 for the $\bar{2}01$ reflection.

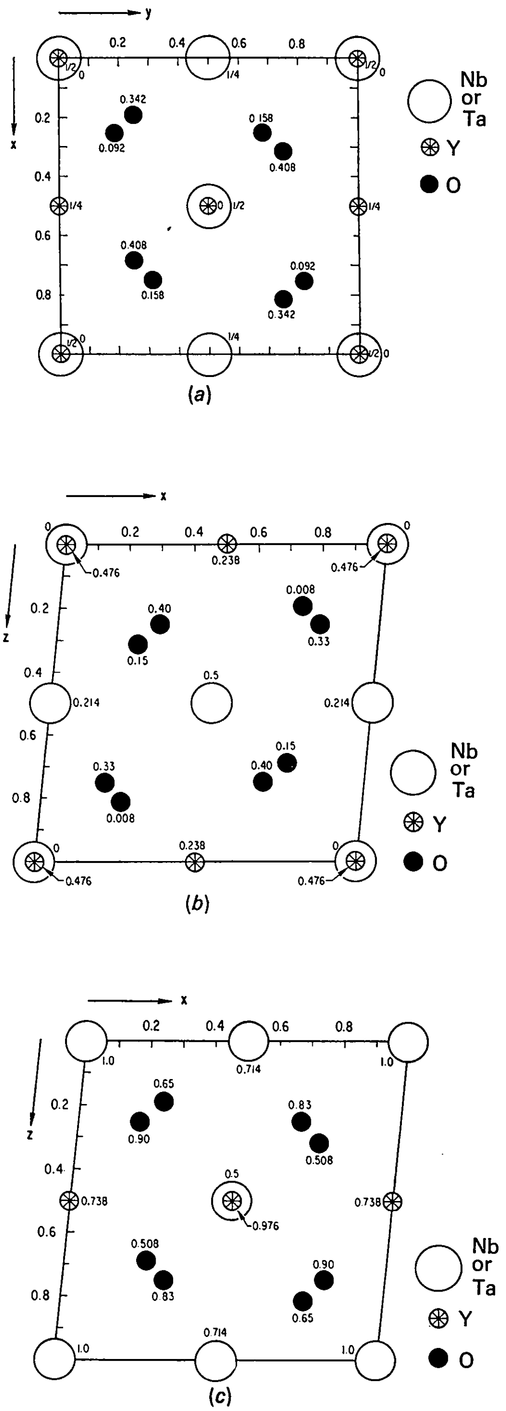


Fig. 1. Comparison of M and T fergusonite structures. (a) Tetragonal fergusonite: unique(c)-axis projection of tetragonal (scheelite-type) $YTaO_4$. One half of the unit cell (z from 0 to 0·51) is shown; the other half is rotated 90° about c. Data from Komkov. (b) Monoclinic fergusonite, M Type: unique(b)-axis projection of one half (y from 0 to 0·5) of M-type fergusonite cell. Data from Komkov 00$\bar{1}$/010/100. (c) Monoclinic fergusonite, M type: unique (b)-axis projection of upper half (y from 0·5 to 10) of M-type fergusonite cell.

The magnitude of the absorption corrections makes them a major factor in the conversion of observed intensities to structure factors. Since they have been calculated from dimensional measurements some of which are only accurate to two significant figures, it is obvious that they limit the possible accuracy of the structure factors. The final list of F_o *versus* F_c shows a correlation between large absorption corrections and large values of F_o–F_c.

The reciprocal sphere for Ag radiation contains over 5000 independent reflections for this crystal. An automated diffractometer not being available, reflections were measured only at values of $2\theta < 60°$, except for nine additional $0k0$ reflections to $2\theta = 135°$. Omitting space group absences and a few of the strongest reflections suspected of extinction, 729 reflections, including 16 unobserved ones, entered into the refinement. For the first 100 reflections, both hkl and $\bar{h}k\bar{l}$ were measured and averaged. The mean reproducibility was 2% for strong and 5% for weak reflections.

The intensities were measured by means of θ–2θ scans; backgrounds were measured by stationary counts on the low and high angle sides of each reflections. The detector was a scintillation counter. Two standard reflections were measured at frequent intervals to check for variations of the primary beam intensity, and scale factors were applied when indicated.

Since the diffractometer was operated manually, each reflection was first observed on the strip-chart recorder to determine an appropriate scanning range for the intensity count. Both components of the $K\alpha$ doublets were always included.

The structure determination

From qualitative intensity considerations it was clear that the yttrium and tantalum atoms could be placed only in equipoints 2(*e*) and 2(*f*), with coordinates $\frac{1}{4},y,0; \frac{3}{4},\bar{y},0$ and $\frac{1}{4},y',\frac{1}{2}; \frac{3}{4},\bar{y}',\frac{1}{2}$. Several possible values of the two y parameters were derived from selected reflections, and a particular combination of two of these was confirmed by two appropriate Patterson sections. The coordinates of the metal atoms were then used in a three-dimensional Fourier synthesis which located the oxygen atoms.

The coordinates were refined by full-matrix least squares, using anisotropic temperature factors for Y and Ta and isotropic ones for oxygen. The final R value was 0·149, which is considerably higher than the reproducibility of the intensity measurements had led us to believe.

Absorption errors, already discussed, may have contributed to the high value of R. In addition, inaccuracies in the data may have arisen from one or both of the following factors.

1. There may have been some disorder in the crystal. This point was also raised by a referee who suggested substitutional disorder. Substitutional disorder is quite unlikely in this compound, but the existence of two other polymorphs suggests the possibility of random displacements of some atoms towards positions they would occupy in one of the other polymorphic modifications. Such displacements would occur mostly – if indeed they do – in the xz plane, and the final list of F_o *versus* F_c shows the worst fits for $h0l$ reflections. Our first batch of crystals, before we learned to grow them better, actually had some extra X-ray reflections which were tentatively interpreted as due to nuclei of the tetragonal phase upon which the crystals of the M' phase had grown. Even later batches, which furnished the crystal used here, were never perfect enough to give recognizable interference figures in the conoscope. It is true that all the crystals were quite small, but a really good crystal will give a recognizable optic figure even if very small. These observations lend a measure of support to the suggestion of some degree of disorder.

2. While a silver target X-ray tube will give the optimum ratio of characteristic to continuous radiation at potentials from 75 to 90 kV, it had to be operated at 50 kV because of equipment limitations. At the same time, the recommended filter thickness (Rh) of 3·5 mils was reduced empirically to about 2 mils to obtain reasonable intensities. While no β lines were observable, the monochromaticity of the beam may not have been as good as desirable. A number of reflections had pronounced 'tails', and while these may be interpreted as due to disorder, they are more likely – in retrospect – to be due to background radiation, corresponding to the general radiation streaks visible on precession photographs. It may well be that in a number of cases the existence of these 'tails' caused errors in apportioning the intensity properly between reflection and background.

Table 2. *Atomic coordinates and temperature factors*

The numbers in parentheses are the standard deviations in the last digits given.

Atom Equipoint	Yttrium 2(*e*)	Tantalum 2(*f*)	Oxygen 1 4(*g*)	Oxygen 2 4(*g*)
x	$\frac{1}{4}$	$\frac{1}{4}$	0·490 (6)	0·081 (12)
y	0·7694 (6)	0·3056 (3)	0·462 (4)	0·069 (8)
z	0	$\frac{1}{2}$	0·266 (4)	0·258 (7)
B	—	—	0·59 (30)	3·12 (75)
B_{11}	0·00024 (108)	0·0024 (6)	—	—
B_{22}	0·0033 (7)	0·0047 (4)	—	—
B_{33}	0·0049 (12)	0·0068 (6)	—	—
B_{13}	−0·001 (2)	0·0064 (10)	—	—

Results

It has been noted that much of the responsibility for the relatively high R value rests with reflections of the type $h0l$. Since the yttrium and tantalum atoms are in special positions with only the y parameter variable, it is believed that they have been determined quite well. The oxygen positions may not be better than 0·05 Å. This is sufficient to establish the nature of the coordination polyhedra but not the degree of distortion of the latter from cubes and tetrahedra respectively.

Most of the temperature factors have reasonable values but a few do not. With an R value barely below 15%, it is questionable how much importance can be attached to their exact numerical values, hence they are quoted without further comment.

The positional parameters, temperature factors and standard deviations derived from the least squares procedures are listed in Table 2. Fig. 2 is a b-axis projection of the electron density, Table 3 gives some distances and angles, and Table 4 is the list of F_o *versus* F_c.

Table 3. *Distances and angles**

The first column lists all the distinct metal–oxygen distances, the second column the angle subtended at the metal atom by the equidistant oxygen pair having the M–O separation to the left.

One oxygen atom in the yttrium coordination sphere, the one at a distance of 2·40 Å, approaches a tantalum atom to within 2·14 Å.

Y–O	O–Y–O
2·25 Å	113·6°
2·34	71·5
2·40	95·2
2·42	76·4
Ta–O	**O–Ta–O**
1·94 Å	74·6°
2·03	105·0

* As is to be expected from the similarity of the lattice parameters, the Y–O distances are almost identical with those in the M phase, which range from 2·26 to 2·45 according to Komkov. In Y_2O_3, where the oxygen cubes are less distorted, the Y–O distances are 2·26 to 2·354 (Paton & Maslen, 1965) The Ta–O distances in $NaTaO_3$ and $KTaO_3$ are 1·93 and 1·99 respectively (Vousden, 1951). This would seem to justify the conclusion that the metal-oxygen distances are more accurate than the oxygen positional parameters are estimated to be.

Computational details

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The quantity minimized by least squares was the sum of the weighted and squared differences between F_o^2 and F_c^2, with weights essentially inversely proportional to the observed intensities. The equations are given in the CRYRM manual (Duchamp, 1964).

During the final cycle of least squares, all of the positional parameters shifted by about 0·2 standard deviations, except x for O(2) which moved 0·9 s.d. All temperature factors shifted between 0·01 and 0·05 s.d., except B_{13} for Ta which changed by 0·09 and B for O(2) which changed by 0·08 s.d.

Discussion of the structure and phase transformation

The yttrium atoms are surrounded by distorted cubes of eight oxygen atoms, and the tantalum atoms by distorted tetrahedra of four oxygen atoms. Because of

Table 4. *Observed and calculated structure factors*

k	$10F_o$	$10F_c$
−8 K 1		
0	464	−323
1	267	246
2	169	128
3	598	−529
−8 K 2		
0	802	831
1	199	−125
2	652	−712
3	377	399
−8 K 3		
0	272	−285
1	205	204
2	267	217
3	580	−570
−8 K 4		
0	699	829
1	168	−194
−7 K 1		
1	1133	929
2	431	−334
3	540	−521
4	461	388
5	174	157
−7 K 2		
1	339	−314
2	577	492
3	45	21
4	622	−642
5	322	265
−7 K 3		
1	859	869
2	343	−325
3	639	−574
4	507	442
5	76	66
−7 K 4		
1	304	−310
2	369	428
3	41	75
4	679	−656
−7 K 5		
1	691	789
2	317	−311
3	432	−451
−6 K 1		
0	423	349
1	367	−257
2	265	−228
3	732	704
4	142	−172
5	729	−689
6	530	424
−6 K 2		
0	1142	1106
1	414	285
2	906	822
3	522	−419
4	452	−386
5	413	404
6	86	−85
−6 K 3		
0	533	427
1	277	−322
2	208	−196
3	643	635
4	181	−117
5	626	−686
6	421	417
−6 K 4		
0	835	−947
1	45	140
2	754	790
3	472	−467
4	343	−297
5	198	375
−6 K 5		
0	231	331
1	188	−202
2	220	−246
3	553	608
4	115	−126
−6 K 6		
0	678	−813
1	177	197
2	596	616
3	390	−355
−5 K 1		
1	1111	1129
2	493	404
3	860	765
4	612	−535
5	---	−76
6	332	248
7	246	−167
−5 K 2		
1	469	363
2	607	−589
3	---	−86
4	865	813
5	415	−371
6	648	−613
7	575	487
−5 K 3		
1	980	1104
2	420	407
3	577	595
4	561	−474
5	246	−164
6	381	323
7	121	−245
−5 K 4		
1	353	383
2	548	−528
3	---	−19
4	702	707
5	326	−262
6	614	−601
−5 K 5		
1	817	−882
2	370	335
3	677	574
4	493	−457
5	62	−59
−5 K 6		
1	264	296
2	398	−413
3	---	−90
4	610	618
−5 K 7		
1	733	−701
2	314	279
−4 K 1		
0	541	−468
1	605	472
2	350	265
3	864	−809
4	210	168
5	848	821
6	617	−514
7	589	−519
8	637	525
−4 K 2		
0	1959	1281
1	165	−145
2	1149	1115
3	747	616
4	465	388
5	655	−445
6	99	72
7	103	131
−4 K 3		
0	511	−468
1	250	246
2	260	270
3	835	−818
4	229	186
5	865	773
6	539	−450
7	440	−467
−4 K 4		
0	1291	1188
1	396	−290
2	942	−848
3	538	451
4	462	393
5	464	−433
6	20	91
7	265	139
−4 K 5		
0	372	−412
1	437	340
2	247	205
3	691	−609
4	133	103
5	701	661
6	483	−406
−4 K 6		
0	797	844
1	76	−139
2	805	−723
3	467	422
4	348	270
5	356	−339
−4 K 7		
0	364	−279
1	167	160
2	192	207
3	581	−523
4	128	113
−4 K 8		
0	708	661
−3 K 1		
1	1750	1521
2	738	−579
3	774	−790
4	647	528
5	270	218
6	469	−387
7	260	282
8	---	−72
−3 K 2		
1	712	−513
2	763	671
3	87	−40
4	1082	−954
5	396	338
6	750	720
7	632	−583
8	312	−313
−3 K 3		
1	1201	1269
2	564	−411
3	869	−794
4	684	618
5	123	88
6	345	−265
7	265	185
8	21	9
−3 K 4		
1	387	−363
2	714	602
3	150	112
4	779	−804
5	430	376
6	622	603
7	584	−474
−3 K 5		
1	1012	1002
2	422	−399
3	591	−572
4	467	420
5	183	140
6	366	−305
−3 K 6		
1	355	−326
2	444	453
3	59	7
4	645	−610
5	246	223
6	544	528
−3 K 7		
1	745	721
2	319	−257
3	581	−458
4	445	377
−3 K 8		
1	198	−213
2	367	313
−2 K 1		
0	1359	706
1	395	−290
2	383	−262
3	1131	1049
4	327	−243
5	1107	−947
6	561	531
7	530	538
8	668	−575
−2 K 2		
0	2200	1925
1	636	386
2	1301	1085
3	653	−563
4	576	−533
5	569	544
6	61	−83
7	247	−142
8	387	323
−2 K 3		
0	683	490
1	564	−510
2	330	−272
3	911	827
4	167	−162
5	753	−837
6	590	517
7	578	520
8	611	−528
−2 K 4		
0	1440	1131
1	275	196
2	954	1082
3	640	−568
4	460	−369
5	524	407
6	123	−83
7	158	−135
−2 K 5		
0	433	377
1	193	−221
2	231	−242
3	718	712
4	207	−167
5	753	−674
6	467	401
7	402	417
−2 K 6		
0	1080	−983
1	233	206
2	736	675
3	444	−367
4	356	−324
5	430	358
6	14	−63
−2 K 7		
0	430	313
1	343	−252
2	197	−133
3	548	466
4	105	−87
5	546	−521
−2 K 8		
0	1055	−607
1	176	106
2	701	539
3	383	−299
−1 K 1		
2	417	382
3	1064	971
4	977	−825
5	169	−130
6	416	270
7	223	−194
8	40	−6
9	347	347
−1 K 2		
1	488	462
2	697	−828
3	158	−97
4	989	1009
5	484	−451
6	709	−738
7	655	573
8	379	358
−1 K 3		
1	760	1409
2	662	615
3	762	813
4	462	−466
5	201	−186
6	413	383
7	299	−271
8	36	66
−1 K 4		
1	431	412
2	585	−623
3	61	3
4	809	808
5	323	−315
6	665	−657
7	576	529
8	312	284
−1 K 5		
1	992	1027
2	332	308
3	652	675
4	599	−517
5	121	−90
6	323	211
7	185	−151
−1 K 6		
1	325	270
2	522	−431
3	114	−54
4	679	642
5	346	−296
6	383	−472
−1 K 7		
1	879	−720
2	291	286
3	542	432
4	373	−292
5	121	−112
−1 K 8		
1	339	199
2	434	−323
3	22	5
0 K 0		
1	371	−408
2	865	1189
3	677	631
4	490	612
5	615	−611
6	49	71
7	233	137
8	401	−346
9	202	202
10	289	233
11	320	−305
12	89	−129
13	466	325
14	---	−33
15	311	−177
16	99	0
17	76	0
18	95	0
0 K 1		
0	335	−690
1	324	543
2	167	290
3	951	1060
4	239	230
5	942	974
6	592	−569
7	561	−568
8	657	583
9	210	179
0 K 2		
0	1138	1369
1	164	−290
3	714	724
4	614	460
5	529	−454
6	172	98
7	131	155
8	266	−276
0 K 3		
0	255	−433
1	207	370
2	180	303
3	855	−902
4	116	214
5	788	831
6	371	−504
7	507	−519
8	377	519
0 K 4		
0	1655	1450
1	297	−268
2	845	−871
3	358	459
4	402	440
5	350	−450
6	26	60
7	123	112
8	361	−276
0 K 5		
0	607	−408
1	221	311
2	134	151
3	533	−634
4	95	139
5	681	672
6	279	−406
7	479	−416
0 K 6		
0	824	809
1	164	−136
2	666	−764
3	309	395
4	261	281
5	254	−290
6	140	50
0 K 7		
0	443	−202
1	159	167
2	152	160
3	594	−488
4	83	132
5	603	474
0 K 8		
0	585	645
1	93	−133
2	431	−457
3	251	230
1 K 0		
1	597	−611
2	624	834
3	33	−37
4	956	1119
5	342	420
6	625	783
7	740	−619
8	265	−356
9	460	498
1 K 1		
1	1648	1767
2	874	−785
3	998	−959
4	546	494
5	231	220
6	468	−423
7	290	295
8	82	−78
1 K 2		
1	498	−434
2	885	841
3	120	63
4	939	−935
5	442	421
6	589	747
7	656	−580
8	386	−336
1 K 3		
1	1596	1356
2	338	−352
3	740	−800
4	695	644
5	135	112
6	363	−229
7	199	161
8	---	0
1 K 4		
1	376	−350
2	477	536
3	---	6
4	780	−818
5	344	345
6	603	583
7	564	−471
1 K 5		
1	958	966
2	483	−378
3	627	−566
4	426	347
5	173	159
6	358	−273
7	236	199
1 K 6		
1	342	−231
2	541	434
3	---	−2
4	615	−541
5	289	243
6	553	488
1 K 7		
1	910	660
2	271	−201
3	498	−435
4	481	319
1 K 8		
1	323	−142
2	337	261
2 K 0		
0	1662	1503
1	235	215
2	1309	1568
3	745	−753
4	567	−465
5	606	475
6	158	−88
7	192	−149
8	301	286
2 K 1		
0	529	397
1	646	−586
2	430	−347
3	909	944
4	187	−212
5	847	−887
6	676	561
7	614	556
8	620	−548
2 K 2		
0	2124	1759
1	437	378
2	991	1030
3	613	−504
4	486	−519
5	562	496
6	45	−70
7	243	−125
8	372	297
2 K 3		
0	804	519
1	324	−300
2	229	−155
3	779	792
4	251	−192
5	835	−792
6	472	458
7	475	467
8	621	−501
2 K 4		
0	1433	1025
1	159	135
2	890	973
3	556	−492
4	430	−345
5	488	339
6	121	−47
7	166	−97
2 K 5		
0	341	229
1	289	−252
2	270	−184
3	646	598
4	122	−164
5	646	−585
6	525	381
2 K 6		
0	889	−852
1	259	185
2	713	591
3	442	−277
4	349	−310
5	412	278
2 K 7		
0	598	222
1	212	−186
2	143	−58
3	524	396
4	152	−107
3 K 0		
1	452	395
2	880	−809
3	164	−115
4	877	924
5	491	−446
6	748	−725
7	654	561
8	384	351
3 K 1		
1	1225	1384
2	525	422
3	914	889
4	705	−633
5	108	−96
6	372	247
7	211	−166
8	---	−2
3 K 2		
1	539	406
2	575	−589
3	---	41
4	762	894
5	354	−350
6	692	−648
7	558	526
8	270	315
3 K 3		
1	1101	1158
2	472	415
3	624	627
4	544	−407
5	234	−203
6	410	300
7	223	−222
3 K 4		
1	359	251
2	599	−524
3	86	−12
4	670	646
5	346	−310
6	636	−557
7	586	440
3 K 5		
1	802	−814
2	323	250
3	654	556
4	527	−374
5	99	−80
6	302	146
3 K 6		
1	267	171
2	320	−330
3	23	17
4	693	521
5	292	−247
3 K 7		
1	728	−592
2	310	199
3	420	347
4 K 0		
0	1651	1468
1	477	−393
2	980	1011
3	588	478
4	497	487
5	472	−465
6	5	86
7	---	137
8	343	−287
4 K 1		
0	756	−468
1	300	278
2	249	197
3	782	−814
4	212	204
5	835	791
6	521	−464
7	486	−476
4 K 2		
0	1349	1144
1	141	−100
2	935	1009
3	586	531
4	414	363
5	---	−370
6	---	38
7	28	93
4 K 3		
0	522	−277
1	313	305
2	295	177
3	689	−650
4	156	175
5	705	654
6	524	−422
7	472	−430
4 K 4		
0	1105	981
1	324	−240
2	740	−700
3	448	308
4	395	364
5	390	−309
6	56	40
4 K 5		
0	513	−238
1	222	206
2	164	75
3	567	−484
4	148	142
5	629	521
4 K 6		
0	914	636
1	66	−60
2	561	−579
3	382	280
4	274	232
4 K 7		
0	93	−102
1	31	130
5 K 0		
1	387	−368
2	586	577
3	---	−11
4	802	−795
5	367	321
6	659	639
7	577	−519
5 K 1		
1	1104	1124
2	421	−381
3	555	−594
4	546	427
5	221	202
6	373	−294
7	209	223
5 K 2		
1	379	−266
2	534	514
3	31	12
4	748	−697
5	345	329
6	638	560
7	561	−452
5 K 3		
1	859	876
2	380	−283
3	706	−615
4	502	393
5	56	81
6	286	−160
5 K 4		
1	283	−188
2	423	387
3	64	−26
4	670	−566
5	310	269
5 K 5		
1	745	699
2	286	−224
3	402	−398
4	405	237
5 K 6		
1	202	−112
2	338	293
6 K 0		
0	1056	1033
1	143	110
2	813	883
3	502	−481
4	366	−333
5	494	358
6	36	−42
6 K 1		
0	570	328
1	242	−284
2	199	−144
3	627	616
4	182	−156
5	695	−651
6	429	407
6 K 2		
0	1015	−971
1	307	240
2	727	712
3	340	−318
4	176	−366
5	212	315
6	80	−44
6 K 3		
0	85	218
1	63	−214
2	43	−166
3	574	522
4	134	−158
5	594	−543
6 K 4		
0	997	−706
1	39	64
2	602	636
3	350	−308
4	256	−257
6 K 5		
0	363	135
1	120	−157
2	167	−56
7 K 0		
1	342	260
2	401	−440
3	7	−18
4	678	663
5	301	−303
7 K 1		
1	694	−826
2	267	271
3	547	569
4	433	−382
5	71	−82
7 K 2		
1	256	193
2	439	−403
3	---	20
4	567	547
7 K 3		
1	692	−722
2	304	236
3	339	419
7 K 4		
1	242	122
2	286	−305
8 K 0		
0	1089	863
1	180	−194
2	578	−635
3	340	307
8 K 1		
0	347	−198
1	287	195
2	204	128
3	536	−503
8 K 2		
0	583	690
1	100	−79
2	639	−622

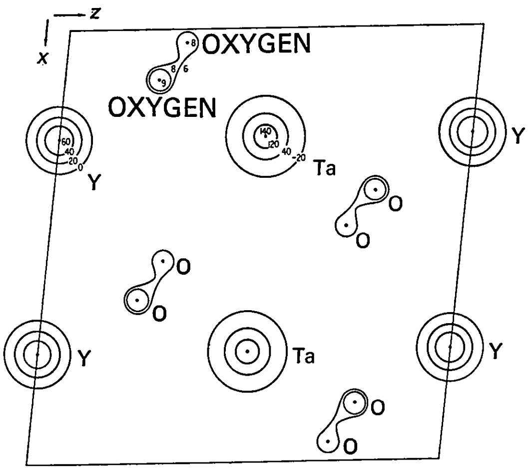


Fig. 2. $YTaO_4$, *M′* phase: *b*-axis projection of electron density. Contour lines labelled in e.Å^{-2}.

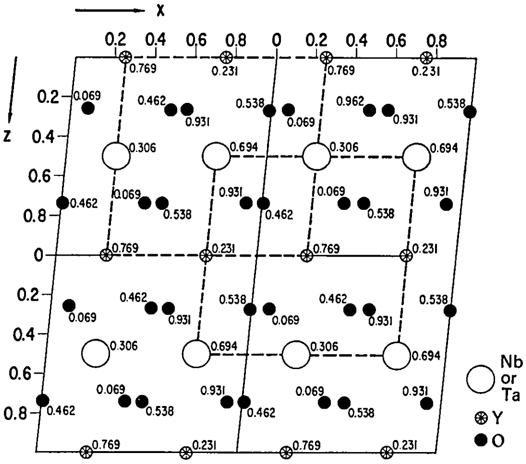


Fig. 3. Monoclinic fergusonite, *M′* type: unique(*b*)-axis projection of full cell. Four unit cells are shown side by side. Dashed cells have origins shifted in different ways to correspond to bottom and top halves of *M*-type or scheelite cells. Transformation involves shift of every second cell along *b* through *a*/2, *c*/2 and adjustment of oxygen atoms.

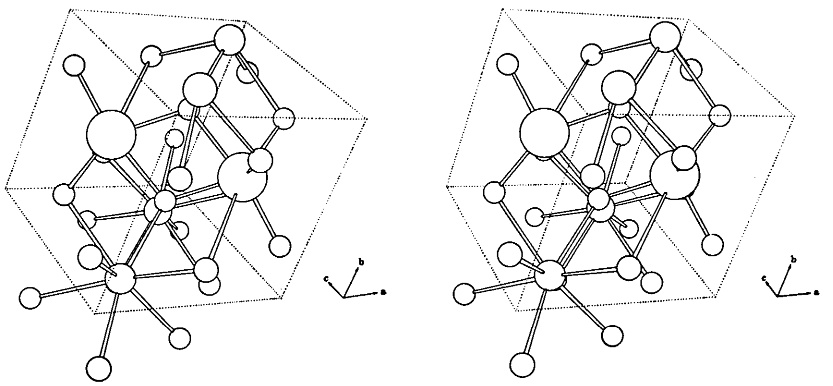
the location of the metal atoms on the twofold axes, all Y–O and Ta–O distances occur in pairs.

The drawing of Fig. 3 is a *b*-axis projection of four unit cells of the structure, based on the results of the least-squares refinement. The parameters derived from the Fourier synthesis differ by amounts ranging from 0·01 to 0·03, which is the right magnitude for series termination errors; hence, they have been so interpreted. This is the reason why two of the oxygen atoms which in the drawing appear on a line almost parallel ($\Delta x = 0{\cdot}01$) to the *z* axis are considerably more inclined to the *z* axis in the Fourier synthesis ($\Delta x = 0{\cdot}07$). The discrepancies in the *x* coordinates, assumed to be termination errors, are in opposite senses.

The dashed outlines indicate alternate unit cells with the origins translated in such a manner as to agree with the drawings of the *T*- and *M*-structures shown in Fig. 1. The two dashed cells are displaced with respect to one another by *a*/2, *c*/2. If they are brought into register, then one cell of the *M* structure is obtained, except for twisting the groups of oxygen atoms. Individual oxygen atoms, however, move only through small fractions of interatomic distances.

If the formal mechanism of the *M′*-to-*M* transformation just described, namely displacement of alternate unit cell through *a*/2, *c*/2 and adjustment of oxygen positions, is also the actual mechanism, then this is a very interesting transformation.

In the sense that there is no diffusion of individual atoms, but cooperative motion of groups of atoms, there is a startling resemblance to diffusionless shear transformations, as in ZrO_2 (Wolten, 1963, 1964). But unlike such transformations, not all the atoms move in unison, and the transformation is definitely not martensitic. Small groups of atoms, the building blocks of the structure, are sheared along certain planes and move as units through distances that exceed interatomic distances; this action would be analogous to the manner in which individual atoms diffuse in more conventional ('nucleation and growth' type) phase transformations. Since many strong bonds are broken in the process, the sluggish nature of the transforma-



FERGUSONITE **FERGUSONITE**

Fig. 4 Stereoscopic drawing of the structure of *M′*-type fergusonite.

tion is readily understood. Of course, in the absence of compelling evidence, translation of such large blocks *in toto* must be considered unlikely. The conservative view is to postulate the reorganization of the structure to be brought about by the diffusion of much smaller blocks, consisting of from one to perhaps three atoms each, which then reassemble in a pattern that recreates many of the features of the original structure.

A stereoscopic drawing of the structure of the *M′* phase is shown in Fig. 4.

The Patterson, Fourier, and least-squares calculations were performed at the California Institute of Technology under the CRYRM crystallographic computing system (Duchamp, 1964) through the generosity of Prof. R. E. Marsh, to whom the author is also greatly indebted for his expert advice on data handling. Dr W. R. Busing, Oak Ridge National Laboratory, was kind enough to furnish the computer program ORABS. Thanks are also due Mr W. Gehrer who patiently, over a period of many months, measured the intensities and backgrounds one by one.

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The Crystal Structure of a Double Oxalate of Yttrium and Ammonium, $NH_4Y(C_2O_4)_2.H_2O$

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A three-dimensional X-ray analysis of $NH_4Y(C_2O_4)_2.H_2O$ has yielded the positions of all the atoms except hydrogen with standard deviations of 0·0012 Å for Y, and less than 0·02 Å for the other atoms. The crystals are monoclinic with $a=9·18\pm0·01$, $b=6·09\pm0·01$, $c=7·89\pm0·01$ Å, $\beta=90·2°\pm0·1°$; space group $P2/n$; $Z=2$. There is ninefold coordination; eight oxygen atoms and the H_2O form a slightly distorted trigonal prism arrangement around the yttrium, at distances ranging from 2·343 Å (for the H_2O) to 2·412 Å. The two independent oxalate ions are planar and have crystallographic centres of symmetry. Their dimensions are in agreement with the results of previous work on oxalates.

Introduction

Hydrated oxalates of the rare earths having the composition $Ln_2(C_2O_4)_3.10H_2O$ (Ln = lanthanide) have been known for some time, and are thought to be interstitial hydrates though their detailed structures are not known (Gilpin & McCrone, 1952). Recently, Barrett, McDonald & Topp (1964) made a study of the precipitation of the rare earth oxalates from oxalic acid solution, in the course of which a new series of double salts was discovered with the composition $NH_4Ln(C_2O_4)_2.nH_2O$. Yttrium and the elements in the group Sm–Tm formed monohydrates which appeared to be isomorphous, while the remaining rare earths formed trihydrates exhibiting a number of different X-ray powder diffraction patterns. The monohydrated yttrium salt was obtained as single crystals and the present paper describes an X-ray analysis of the structure. The other members of the monohydrate series presumably also have this structure.

Experimental

$NH_4Y(C_2O_4)_2.H_2O$ is precipitated from strongly acidic solutions containing an excess of oxalate ion by the addition of ammonia. Prepared in this way, it is a fine powder. Crystals for the present work were grown hydrothermally. The prisms so obtained showed development of the forms {010} and {101} and were invariably twinned.

The intensity data were collected from three sets of equi-inclination Weissenberg photographs: (0–5,*k*,*l*), (*h*,*k*,0–6), and (*h*,0–4,*l*). Nickel-filtered Cu *Kα* radia-

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