the F–F distance is 3·03±0·04 Å. The tetrahedra are therefore elongated in the direction of the *c* axis. The Br–F distance is 1·81±0·04 Å, close to the value cited for BrF_3. (The sum of the tetrahedral radii is 1·75 Å.) All potassium atoms are bonded to 8 equidistant fluorines with K–8F = 2·84±0·03 Å. The coordination of the fluorines involves 4 fluorines at the corners of a square in a plane below the potassium and 4 fluorines similarily disposed in a plane above.

The structure is similar to $BeSO_4.4H_2O$ (Beevers & Lipson, 1932). The BrF_4^- ions are replaced by tetrahedral SO_4^{2-} ions while the K^+ ions are replaced by tetrahedral $(Be4H_2O)^{2+}$ groups. The space group is D_{2d}^{10} and no symmetry planes exist. Thus, the sulphurs do not lie on symmetry planes, as do the potassiums, and the sulphate ions can therefore be rotated from the position occupied by BrF_4^-. The rotation which is observed is slight. Otherwise the distribution of SO_4^{2-} ions about the $(Be4H_2O)^{2+}$ ions is similar to that observed for the BrF_4^- groups around K^+.

I am indebted to several members of the staff for the samples used in this study. Chemical preparations of the $KBrF_4$ samples and the filling of the capillaries were carried out by Dr I. Sheft and Dr A. Martin of the Chemistry Division. The density measurements, as well as the preparation of the narrow quartz capillary and the sample, were made by Mr G. Schnizlein of the Chemical Engineering Division.

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The Crystal Structure of Barium Dititanate, $BaO.2TiO_2$*

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The crystal structure of $BaO.2TiO_2$ has been determined by means of two-dimensional Patterson and Fourier syntheses. The unit cell is monoclinic with $a = 9{\cdot}410 \pm 0{\cdot}004$, $b = 3{\cdot}930 \pm 0{\cdot}001$, $c = 16{\cdot}892 \pm 0{\cdot}013$ Å, $\beta = 103° 2' \pm 3'$, space group $A2/m$, and contains six formula units. The structural arrangement is discussed in relation to other baria–titania compounds.

Introduction

In the course of investigations of the phase diagram of the system $BaO–TiO_2$, Rase & Roy (1955) have observed the formation, in addition to the polymorphic forms of $BaTiO_3$, of the compounds $2BaO.TiO_2$, $BaO.3TiO_2$, $BaO.4TiO_2$, and $BaO.2TiO_2$ (or $3BaO.7TiO_2$).

The present paper is concerned with the structure of the last compound.

The compound is formed in mixtures of TiO_2 and $BaCO_3$ containing between 50 and 75 molar% of TiO_2 which have undergone prolonged heating at temperatures above 1210° C. Acicular crystals some 10 mm. long and 0·5 mm. in width grow readily on cooling a melt containing $62\frac{2}{3}$ molar% TiO_2.

Experimental

Crystals grown by Rase & Roy were examined by oscillation, rotation and Weissenberg photographs, using Cu *Kα* radiation. They were found to be monoclinic with the *b* axis parallel to the needle length.

Accurate measurements of the cell dimensions were obtained by the method of Weisz, Cochran & Cole (1948). The values *a*, *c*, and *β* were found from X-ray photographs in which the *b* axis was rotation axis, and the value of *b* from photographs in which the *a* axis was rotation axis. The space group was derived from systematic absences observed on the zero, first, second and third *b*-axis Weissenberg photographs.

0*kl* and *h*0*l* intensities, recorded on zero-layer *a*- and *b*-axis Weissenberg photographs respectively, using Cu *Kα* radiation, were measured by visual estimation. No correction for absorption was made in the latter case since the needle-shaped crystal was mounted with its length along the rotation axis and its cross-section was small. However, owing to the

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high absorption coefficient it was considered that the *h0l* intensities would not be completely free from absorption errors. For the *0kl* intensities, in which the needle length was at right angles to the rotation axis, absorption corrections were calculated by the method of Howells (1950) for a long needle-shaped crystal. These values were found to be adequate for the reflexions which emerged from the side of the needle through which the incident ray entered, but inexact for reflexions transmitted completely through the crystal which were more strongly absorbed. For the latter reflexions the more accurate graphical method of Howells (1950) was used.

An investigation of the probability distribution of the *0kl* and *h0l* intensities (Howells, Phillips & Rogers, 1950) showed that the two zones corresponded with centric projections.

A Patterson synthesis of the *h0l* reflexions was used to determine approximately the x and z atomic parameters. Successive *h0l* Fourier and difference syntheses resulted in a refinement of these parameters.

The application of limitations required by the space group, together with coordination principles, resulted in a unique set of y atomic parameters which were compatible with the observed *0kl* intensities.

Unit-cell dimensions and space group

The dimensions of the unit cell are:

$$a=9\cdot410\pm0\cdot004,\ b=3\cdot930\pm0\cdot001,\ c=16\cdot892\pm0\cdot013\ \text{Å},\ \beta=103°\ 2'\pm3'\ .$$

Systematic absences of the type $(k+l)$ odd for general *hkl* reflexions indicate lattice centring on the A face. There are no other systematic absences.

Investigation of the probability distribution of the *0kl* and *h0l* intensities shows that the two zones correspond to centric projections. The former of these zones showed some departure from the centric distribution curve at the high end, due, as was seen when the structure was determined, to the a-axis projection having a non-random arrangement, the atoms occurring in layers. However, since the calculated intensities show a very similar variation, the indication of a centric *0kl* zone is fairly conclusive. These results require the space group to be centrosymmetric, which is therefore $A2/m$.

The unit-cell content

Rase & Roy (1955) were unable to distinguish unequivocally between the alternative compositions of $BaO.2TiO_2$ or $3BaO.7TiO_2$, although the evidence favoured the former, which was nominally called the dititanate. This was due to it being impossible to produce complete conversion of any mixture to this phase either after prolonged solid-state reaction or by melting, thus rendering accurate chemical analyses difficult. Three separate chemical analyses of groups of the small crystals separated from the melt gave indefinite results of 69·4, 63·6, and 64·4 molar% TiO_2 respectively, the last two being microchemical analyses and possibly less accurate. From a study of the phase diagram, Rase & Roy indicated that two compositions only were likely: those having $66\frac{2}{3}$ and 70 molar% of TiO_2. Of these the former was to be preferred.

Their attempts to measure the density by pycnometer gave inconsistent values owing to the limited quantities of crystals available. Measurements of the density by sink–float methods using Thoulet solutions gave a value of 4·4 g.cm.$^{-3}$, which was very approximate owing to difficulties associated with solutions of such high supersaturation. It was hoped that this value of the density, used in conjunction with the unit-cell dimensions, would result in further evidence for the composition.

From the unit-cell dimensions given above it was found that the measured density required a unit-cell content of either 5·16 formula units of $BaO.2TiO_2$ or 1·58 formula units of $3BaO.7TiO_2$. This result appears to favour the former composition rather than the latter. However, the space group $A2/m$ requires an even number of equivalent positions, special as well as general, within the unit cell. The only cell contents reasonably possible are therefore:

4 formula units of $BaO.2TiO_2$, resulting density 3·4;
6 formula units of $BaO.2TiO_2$, resulting density 5·1;
2 formula units of $3BaO.7TiO_2$, resulting density 5·5.

These values suggest that the most likely cell content is $6(BaO.2TiO_2)$.

The structure determination

(*a*) *The h0l Patterson synthesis*

A two-dimensional Patterson projection, using relative values of F^2 obtained from the *h0l* intensities, was carried out on X-RAC in the Department of Physics, The Pennsylvania State University, and is shown in Fig. 1(*a*). The repeat unit for this projection is contained in an area $a\times\frac{1}{2}c$.

An interpretation was carried out as follows:

Whether the unit cell content is $6(BaO.2TiO_2)$ or $2(3BaO.7TiO_2)$, there will be 3 Ba atoms per repeat unit of the b-axis projection, one of which lies at a centre of symmetry with the other two symmetrically arranged with respect to it, thus:

○ ○ ○

This arrangement will give rise to the following peaks in the Patterson projection with the relative weights shown:

○	○	○	○	○
1	2	3	2	1

It will be seen that the peak at the origin, plus the peaks of double weight, give the relative positions of the Ba atoms.

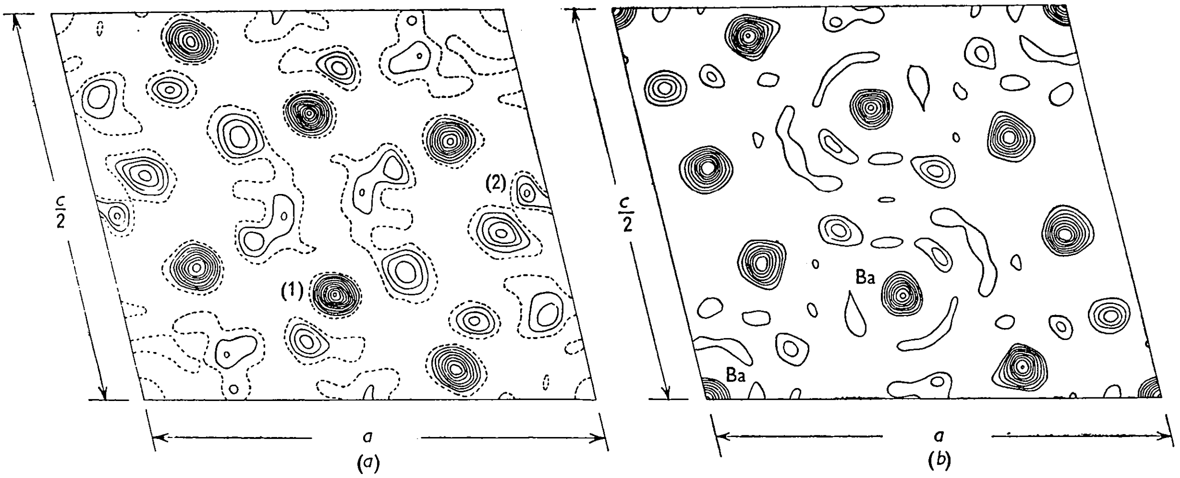

Fig. 1. (*a*) The *b*-axis Patterson projection. Contours at equal but arbitrary intervals; zero contour dashed; contours of the origin peak and negative contours not shown.

(*b*) The *b*-axis Fourier projection. Contours begin at 3 arbitrary units for the Ba atoms and continue at intervals of 3 units. For the remainder of the projection contours begin at 2 units, continuing at unit intervals.

Since, apart from chance superposition of other vector peaks, the double-weight Ba peak will be the largest peak of the projection, apart from that at the origin, it should be an easy matter to distinguish it. The peak labelled (1) in Fig. 1(*a*) fulfils the description and has an associated peak, labelled (2), of approximately half the height at a vector distance from the origin equal to twice that of (1).

The three Ba positions deduced in this way (hatched circles, Fig. 2) were then used as a basis for the vector coincidence method of Robertson & Beevers (1950). By this method eight other atom positions in the repeat unit were located (full circles and double circles, Fig. 2). To six of these, of heaviest weight, a Ti atom coincident with an O atom was assigned, and single O atoms to the other two. The arrangement of Ti coincident with O was adopted in view of the *b*-axis length of 3·930 Å. Comparison with the dimensions of Ti–O octahedra in other compounds, e.g. $BaTiO_3$ (Evans, 1953), shows that this distance must correspond to the distance between opposite apices of a Ti–O octahedron. Thus, an O atom at the apex and a Ti atom at the centre of an octahedron will appear superimposed in the *b*-axis projection. The occurrence of six heavy-weight atomic positions, in addition to the Ba positions, was further confirmation of the unit-cell content of $6\,(BaO.2TiO_2)$, rather than $2\,(3BaO.7TiO_2)$, since the latter would require seven Ti positions per repeat unit of the projection.

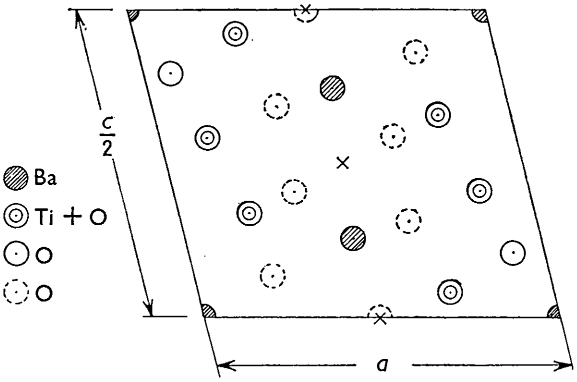

Fig. 2. Atom positions resulting from the Patterson synthesis.

Seven O atoms remained to be placed in the repeat unit. They were assigned to the structure on the basis of satisfying small peaks occurring in the Patterson projection, and also to provide acceptable anion grouping around the cations. They are indicated by broken circles in Fig. 2. Inaccuracies in these oxygen positions would be expected to have but small effect on the structure factors, to which the Ba and Ti atoms make the main contribution.

(*b*) *The hOl structure-factor calculations and Fourier syntheses*

Structure factors for the *hOl* intensities, calculated on the basis of the arrangement shown in Fig. 2, when compared with observed values, gave a value for $R[\equiv (\Sigma|F_o-F_c|)\div\Sigma|F_o|]$, the residual discrepancy, equal to 0·308 if all reflexions were considered, and 0·259 if those too weak to be observed were ignored. The James–Brindley atomic scattering factors for O^{2-} and Ti^{4+} were used, together with the Thomas–Fermi curve for Ba extrapolated smoothly from its value at $(\sin\theta)/\lambda = 0{\cdot}3$ to a value of 54 at $(\sin\theta)/\lambda = 0$.

The signs obtained from the calculated values were used in conjunction with the observed $|F|$'s to carry out an *hOl* Fourier synthesis on X-RAC. All oxygen atoms save one could be assigned to peaks in the synthesis. This last, the 15th, was required to be at a special position at the corners or centre of the projection, or the mid-points of the edges. It could not occur coincident with Ba at the corners, since the b dimension is not large enough to accommodate two such ions of larger diameter. However, there were no peaks which could significantly represent oxygen at the other possible positions.

Structure factors were calculated on the basis of the new parameters, omitting the last oxygen. These, when compared with the observed values, made R equal to 0·252 if all reflexions were considered and 0·227 if those too weak to be observed were ignored.

In order to locate the missing oxygen atom a difference synthesis was carried out on X-RAC with the new signs. Of the special positions at which the missing oxygen atom could be situated, peaks occurred only at the points shown by crosses in Fig. 2. It seemed most unlikely that the oxygen would lie at the centre of the cell, since this position is surrounded by four other oxygen atoms. The second position, at the centre of an edge, was reasonable, since it provided coordination for neighbouring Ba and Ti atoms.

Structure factors calculated with the new atomic parameters indicated by the difference synthesis and including the last oxygen atom gave values of R no smaller than those of the second $h0l$ calculation.

A second Fourier synthesis was therefore computed, using the signs of the last calculation. The result is shown in Fig. 1(b). Structure factors calculated from the new parameters (Table 1) gave values of R equal to 0·230 or 0·206 depending on whether or not reflexions too weak to be observed were ignored.

Table 1. *Final atomic parameters, expressed in terms of the Wyckoff notation for space group A2/m*

Coordinates of equivalent positions (0, 0, 0; 0, ½, ½)

		x/a	y/b	z/c
2: (a)	Ba_2	0	0	0
2: (d)	O_5	0·500	0·500	0
4: (i) ±(x, 0, z)	Ba_1	0·482	0	0·130
	Ti_1	0·195	0	0·669
	O_1	0·195	0	0·169
	Ti_2	0·127	0	0·293
	O_2	0·127	0	0·793
	Ti_3	0·286	0	0·462
	O_3	0·286	0	0·962
	O_4	0·416	0	0·322
	O_6	0·209	0	0·563
	O_7	0·072	0	0·396
	O_8	0·374	0	0·712

(c) *The y atomic parameters*

It had been noted when zero-, first-, second- and third-layer b-axis Weissenberg photographs were taken to determine the space-group extinctions, that when allowance had been made for the difference in scale, the zero- and second-level photographs were identical, as far as could be judged by inspection, as were the first and third; i.e. the $h0l$ reflexions as a group were identical in intensity with the $h2l$, and the $h1l$ reflexions were identical with the $h3l$ indicating that $F^2(h0l) = F^2(h2l)$ and $F^2(h1l) = F^2(h3l)$. These results required the y atomic parameters to have values of either 0, $\frac{1}{4}$, $\frac{1}{2}$ or $\frac{3}{4}$.

When the space group was found to be $A2/m$ it was seen that the short b axis, which resulted in no like atoms being superimposed in the ac projection, required all atoms to be situated on the mirror planes with y coordinates of 0 or $\frac{1}{2}$. This condition, with further space-group limitations, and the expectation that oxygen atoms would be grouped around Ba and Ti atoms approximately as in the cubic form of $BaTiO_3$ (namely Ba within a cubic arrangement of 12 O atoms, Ti within an octahedral arrangement of 6 O atoms) enabled a unique assignment of y parameters to be made, as in Table 1. The resulting structure is shown in Fig. 3. Structure factors calculated for the $0kl$ reflexions, on comparison with observed values, made R equal to 0·196. A comparison of the observed and calculated structure factors for the $0kl$ and $h0l$ reflexions is given in Table 2.

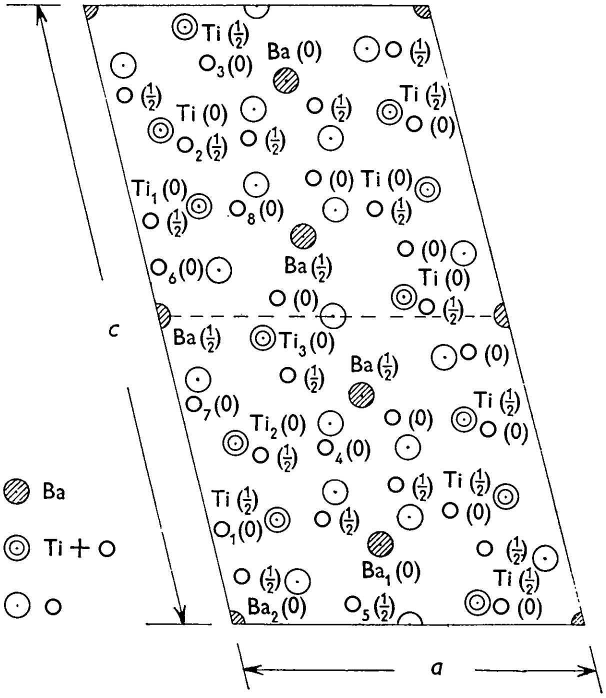

Fig. 3. Structural arrangement viewed along b; y parameters in brackets.

Discussion of the structure

The agreement between $|F_o|$ and F_c obtained for the $0kl$ and $h0l$ zones of reflexions shows that the structural parameters of the heavier atoms, Ba and Ti+O, given in Table 1 are fairly precise. However, series-termination effects have not been taken into account and these,

Table 2. *Comparison of F_c and $|F_o|$ for the* 0*kl and h*0*l reflexions*

(*a*) 0*kl*

0*kl*	F_c	$\|F_o\|$
002	9·0	7·2
4	−10·1	8·7
6	54·9	37·0
8	28·2	30·5
10	9·9	5·3
12	−27·0	24·3
14	19·9	20·4
16	27·1	16·5
18	7·6	6·5
011	27·8	22·0
3	−8·5	11·7
5	−16·6	35·6
7	69·3	84·2
9	36·4	43·8
11	8·4	8·7
13	10·1	13·0
15	48·1	40·1
17	26·2	25·4
19	−25·5	30·2
020	134·4	133·0
2	8·3	12·8
4	−9·7	12·1
6	43·3	53·4
8	27·2	42·5
10	8·0	<16
12	−23·9	23·0
14	18·8	21·7
16	25·4	17·8
18	7·1	5·1
031	21·9	18·2
3	−4·3	12·7
5	−14·2	20·4
7	51·3	48·7
9	30·5	30·6
11	−8·0	6·2
13	9·2	8·9
15	42·8	35·6
040	83·9	70·6
2	7·2	< 9
4	−7·8	8·6
6	31·0	27·1
8	23·0	21·9
10	6·1	3·0
12	−19·5	12·6
051	16·4	22·0

(*b*) *h*0*l*

*h*0*l*	F_c	$\|F_o\|$
002	9·5	< 6
4	−10·5	< 8
6	54·6	40·0
8	28·2	36·1
10	10·4	<14
12	−27·1	33·0
14	36·7	31·1
16	27·1	26·0
18	7·9	<16
20	−7·2	<12
100	−12·8	< 6
2	6·6	< 8
4	80·8	45·2
6	37·5	29·0
8	−30·8	25·1
10	61·5	76·0
12	54·7	63·0
14	−6·2	<18
16	−8·3	<18
18	10·1	21·1
20	25·7	23·0
$\bar{1}02$	21·2	9·0
4	38·3	30·0
6	−17·7	16·0
8	−19·3	19·0
10	16·2	14·0
12	37·2	48·1
14	17·3	<18
16	−20·3	18·1
18	53·3	63·0
20	39·6	50·1
200	37·8	20·0
2	17·5	<10
4	−41·6	29·0
6	18·0	12·0
8	42·5	38·1
10	51·6	60·0
12	−12·7	<18
14	23·7	38·1
16	70·3	77·1
18	−11·1	14·0
20	4·3	< 8
$\bar{2}02$	4·1	< 8
4	−10·3	9·0
6	20·9	12·0
8	123·3	95·1
10	1·1	14·0
12	−5·7	<16
14	61·1	68·1
16	25·1	36·1
18	3·5	16
20	−8·0	<14
300	−43·1	25·0
2	55·2	38·1
4	41·2	37·0
6	8·2	<14
8	−19·9	28·1
10	−6·6	<18
12	38·7	42·0
14	5·0	<18
16	−14·1	23·0
18	14·1	24·1
$\bar{3}02$	45·3	40·0
4	100·5	68·1
6	−13·7	12·0
8	13·3	18·1
10	46·2	37·0
12	27·2	29·0
14	−17·2	18·1
16	−11·5	18·1
18	29·5	23·0
20	14·1	13·0
400	53·0	40·0
2	94·7	93·5
4	−13·8	21·0
6	23·6	31·1
8	74·6	89·0
10	−3·8	<18
12	−3·0	<18
14	5·7	<18
16	27·2	32·0
18	5·4	<10
$\bar{4}02$	9·3	<12
4	−25·4	16·0
6	36·6	33·0
8	35·4	20·0
10	−26·1	26·0
12	1·7	<18
14	18·0	18·1
16	51·0	54·1
18	−2·9	<16
20	−7·7	<14
500	−17·7	31·0
2	−11·9	7·0
4	44·1	44·1
6	36·7	42·0
8	−10·8	<18
10	1·6	<18
12	71·9	76·0
14	20·0	31·1
16	−15·6	26·0
$\bar{5}02$	20·6	25·0
4	20·2	21·0
6	10·9	14·1
8	−32·0	26·0
10	38·7	38·1
12	62·4	50·1
14	−18·7	18·1
16	22·3	28·1
18	51·2	54·1
20	14·6	11·0
600	48·3	53·0
2	6·5	<16
4	−0·2	<18
6	−22·1	18·0
8	35·5	36·1
10	21·3	17·1
12	−9·4	<16
14	15·3	21·1
$\bar{6}02$	17·3	21·0
4	−19·0	21·0
6	89·0	81·9
8	51·8	37·8
10	−8·9	17·1
12	8·0	<18
14	42·7	42·0
16	22·2	17·1
18	−23·6	33·1
20	11·1	<10
700	0·4	<18
2	−14·3	<18
4	69·6	83·0
6	23·9	33·0
8	−18·5	21·1
10	8·6	<16
12	13·5	27·0
$\bar{7}02$	59·8	60·0
4	38·5	38·1
6	−4·1	<18

Table 2 (*cont.*)

$h0l$	F_c	$\|F_o\|$	$h0l$	F_c	$\|F_o\|$	$h0l$	F_c	$\|F_o\|$
8	− 3·8	18·1	14	64·7	54·1	$\overline{10}$,0,2	−12·0	16·0
10	35·2	36·1	16	26·9	25·1	4	27·3	35·1
12	4·8	<18	18	16·6	14·0	6	46·7	48·1
14	−13·9	17·1				8	23·1	23·0
16	− 5·2	16·0	900	− 6·4	<18	10	−19·6	26·0
18	23·6	26·0	2	−13·4	17·1	12	17·8	20·0
20	31·5	23·0	4	22·9	16·0	14	29·1	28·1
			6	35·8	37·0	16	− 3·4	< 8
800	32·7	36·1						
2	40·9	42·0	$\bar{9}$02	19·4	29·0	11,0,0	6·5	<12
4	8·6	18·0	4	42·4	51·0	2	− 7·5	<10
6	2·7	<18	6	−18·9	18·1	4	29·5	23·0
8	51·4	55·1	8	− 1·8	<18			
12	−25·0	26·0	10	59·5	54·1	$\overline{11}$,0,2	50·3	46·0
			12	24·7	23·0	4	8·7	<14
$\bar{8}$02	−30·5	36·1	14	− 2·9	<14	6	−20·6	30·0
4	9·1	<18	16	14·9	<14	8	11·2	13·0
6	18·8	22·0				10	10·9	16·0
8	17·0	<18	10,0,0	44·4	50·1	12	16·1	10·1
10	3·9	<18	2	44·5	49·0			
12	−13·4	<18	4	−23·7	27·0	$\overline{12}$,0,3	35·9	22·0

together with any errors in $|F_o|$ due to absorption, will affect the x and z coordinates of the oxygen positions. Diffraction ripples due to series termination are apparent around the Ba atoms in Fig. 1(*b*) and the peak representing atom O_4, which lies on such a ripple, will be considerably displaced from its true position. Atomic parameters in the neighbourhood of $x/a = 0{\cdot}325$, $y/b = 0{\cdot}359$ seem more appropriate on spatial grounds.

Comparison of bond lengths calculated from Fig. 1(*b*) with those occurring in other $BaO.TiO_2$ compounds, e.g. cubic and hexagonal $BaTiO_3$ (Burbank & Evans, 1948; Evans, 1953) indicates such discrepancies, as do the variations in peak heights among the oxygen positions.

However, these limitations do not affect the general structural scheme, which is clearly defined, and it is of interest to note that an idealized form of the structure can be constructed from regular groups of the appropriately coordinated Ti–O and Ba–O polyhedra. Thus, if a square lattice is drawn, as in Fig. 4, with a repeat distance of about 2·0 Å, and atoms are arranged at the intersections as shown, then the unit cell outlined by heavy lines gives the *b*-axis projection in idealized form. The value for the β angle so obtained is 102° 32′. The length of the repeat unit of the structure necessary to make a equal to 9·410 Å is 2·104 Å, whilst the value which makes c equal to 16·892 Å is 2·048 Å. In this arrangement the Ti atoms lie within regular octahedra of 6 O atoms whilst the Ba atoms lie within an arrangement of 12 O atoms of the type found in cubic $BaTiO_3$.

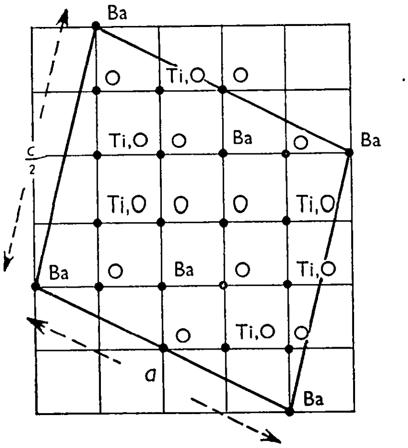

Fig. 4. Idealized structural arrangement, viewed along *b*.

I wish to express my indebtedness to Prof. R. Pepinsky for allowing me the use of X-RAC and to the members of the X-RAC Staff for their competent service.

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