The structures of the ditellurides of niobium and tantalum which have been worked out as part of this project, have similar metal–metal chains parallel to the *b* axis except that the chains are made of three rows of metal atoms rather than two. Details of this work are presented in a companion paper (Brown, 1966).

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The Crystal Structure of the Strontium and Lead Tetraborates, $SrO \cdot 2B_2O_3$ and $PbO \cdot 2B_2O_3$

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$SrO \cdot 2B_2O_3$ and $PbO \cdot 2B_2O_3$ are isostructural. These compounds crystallize in the orthorhombic system, $P2_1nm$, with two formula units in a cell of dimensions $a=4{\cdot}237$, $b=4{\cdot}431$, $c=10{\cdot}706$ Å for $SrO \cdot 2B_2O_3$, and $a=4{\cdot}244$, $b=4{\cdot}457$, $c=10{\cdot}840$ Å for $PbO \cdot 2B_2O_3$. A detailed structure analysis was carried out for the strontium compound only.

The structure was solved by conventional Patterson and electron-density syntheses utilizing the heavy atom to establish the initial phases. Least-squares refinement on three-dimensional data yielded a residual $R=7{\cdot}1\%$.

The results reveal an unusual type of borate framework. All boron atoms are tetrahedrally coordinated. The unusual feature is the occurrence of an oxygen atom common to three tetrahedra.

Although the tetrahedra form a three-dimensional network by corner sharing, the borate network gives the appearance of a layer-like structure because there are comparatively few links in the **c** direction. The layers can be described in terms of chains (parallel to **a**) of six-membered rings having B–O edges in common. These chains are joined by non-ring oxygens to form layers parallel to the *a b* plane.

The Sr coordination is not clearly defined. There are nine nearest-neighbor oxygen atoms at distances ranging from 2·52 to 2·84 Å. There are six more oxygen atoms at 3·04 to 3·20 Å which could conceivably be considered as part of the Sr coordination sphere.

Introduction

In previous studies (Weir & Schroeder, 1964; Block, Perloff & Weir, 1964) on a series of $M^{II}O \cdot 2B_2O_3$ compounds (M = Ca, Ba, Zn, Sr, Pb) it has been observed that the Sr and Pb borates are isostructural with each other, but apparently of a structure type completely different from the others. It was also noted that the Sr and Pb borates had an unusually dense structure compared with most known borates, which suggested the possibility of a new borate structure type. A single-crystal X-ray structure analysis was undertaken on $SrO \cdot 2B_2O_3$ to investigate this possibility. Only the Sr compound is reported in detail, but the structure is valid for the Pb compound.

Cell and space group

The cell dimensions and diffraction aspect have been previously reported by Block, Perloff & Weir (1964) with axial designations interchanged from those used in this work. The present structure is based on the following orthorhombic cell data* for $SrO \cdot 2B_2O_3$:

$a=4{\cdot}237\pm0{\cdot}004$ Å	Space group $P2_1nm$
$b=4{\cdot}431\pm0{\cdot}004$	$Z=2$
$c=10{\cdot}706\pm0{\cdot}010$	ϱ (calc.) $=4{\cdot}01$
Systematic extinctions: $h0l$, $h+l=2n+1$	

* The uncertainties quoted on the cell dimensions are standard deviations based on least-squares refinement of powder pattern data.

The isostructural $PbO \cdot 2B_2O_3$ has the cell dimensions:

$a = 4{\cdot}244 \pm 0{\cdot}004$ Å
$b = 4{\cdot}457 \pm 0{\cdot}004$
$c = 10{\cdot}840 \pm 0{\cdot}010$

The observed diffraction aspect P^*n^* allows $Pmnm$ and $Pmn2_1$ (alternatively $P2_1nm$) as possible space groups. A complete lack of unobserved reflections, other than space group extinctions, favored the non-centrosymmetric choice, because the normal intensity distribution (Wilson, 1949) predicts a greater proportion of very weak reflections, which implies a greater proportion of unobserved reflections, for centrosymmetric space groups than for non-centrosymmetric ones.

The Howells, Phillips & Rogers (1950) $N(z)$ test on the $0kl$ data also indicated a non-centrosymmetric intensity distribution, but this was more misleading than helpful inasmuch as that projection, according to the final structure, should have had a center of symmetry. Presumably this discrepancy is due to the presence of a special position heavy atom which violates the 'random' electron-density distribution on which the statistical methods are based. The result of the N(z) test was accepted for the indexing of the data with the intention of obtaining the space group in the conventional orientation (*International Tables for X-ray Crystallography*, 1952). The conventional orientation was not obtained because of the erroneous indication of the $N(z)$ test.

The final choice of the space group was dictated by the impossibility of getting a chemically satisfactory structure consistent with the Patterson map while retaining a mirror plane normal to the very short a axis.

Experimental

A few small, needle-shaped, single crystals were obtained by partially fusing a sample of the proper stoichiometry. Intensity data were obtained from a crystal 0·135 mm long and with minimum and maximum cross-sectional dimensions of 0·034 and 0·054 mm. Three-dimensional data (hkl, $h=0$, 1, 2, 3) about the needle axis, which coincides with the a axis, were collected with an integrating Weissenberg camera using the usual multiple-film techniques. Copper $K\alpha$ ($\lambda = 1{\cdot}542$ Å) radiation was used. All of the 225 possible independent reflections were observed. Most of the intensities were measured with a densitometer and the very weak reflections were estimated visually. Lorentz and polarization corrections were applied. The crystal was small enough for absorption effects to be no worse than 15% which was the estimated accuracy of the data, so no absorption corrections were made. Neglecting the absorption corrections did not introduce any serious errors in the results since in the final refinement the strongest intensities were omitted to reduce the influence of extinction and, indirectly, absorption, since all of the very strong low order reflections which would have the largest absorption errors were included in the omitted group.

Structure determination

In either of the possible space groups the Sr atom must occupy a twofold special position on a mirror plane which has, effectively, only one variable parameter y. In the centrosymmetric space group, x and z are fixed by symmetry, while in the non-centrosymmetric versions one is fixed by symmetry and the other must be arbitrarily fixed to establish an origin. Thus, the Sr position could be established before deciding on the true space group. Once the correct space group was established the structure determination reduced to a simple heavy atom problem.

The $0kl$ Patterson projection provided an initial y parameter of 0·2126 and the x and z parameters were fixed at zero. A single superposition on the Sr–Sr peak yielded four new peaks which could be sensibly interpreted as oxygen positions only if the space group were $P2_1nm$. In an attempt to confirm this space group the Howells, Phillips & Rogers (1950) $N(z)$ test was made on the $0kl$ data. Unfortunately, this indicated a non-centric projection which implied the space group $Pmn2_1$. At this point it was felt that $Pmnm$ could be safely discarded from consideration. To resolve the dilemma between the two settings of the non-centrosymmetric space group, all the data were first put on a common scale by setting $\Sigma F_o = \Sigma F_c$ (Sr only) for each level, and then three-dimensional electron-density maps, based on Sr phases, were made for both settings.

Only the map using $P2_1nm$ symmetry contained the correct number of prominent peaks and reasonable distances between the peaks. $P2_1nm$ was then accepted as the correct space group. Oxygen positions were obtained from the electron density map. A subsequent difference Fourier map unambiguously established the boron positions.

Positional parameters and individual isotropic temperature factors for all the atoms were refined with a modified version of the Busing, Martin & Levy (1962), full-matrix least-squares program. The program minimizes the quantity $R' = \Sigma w(F_o - F_c)^2$. The weighting scheme was based on the estimated errors of the uncorrected relative intensities. The original intensity scale ranged from 1 to 823. Most reflections were estimated to have an error of 15%. The weighting factor (w) was taken as unity for all reflections with an estimated error, $\sigma(I_o) \leq 18$ (which was equivalent to an $I_o = 120$) and as $18/0{\cdot}15\ I_o$ for all reflections with an estimated $\sigma(I_o) > 18$ (equivalent to $I_o > 120$). Refinement was continued until all final shifts were less than one-tenth of the standard deviation. Individual level scale factors were obtained by setting $\Sigma F_o = \Sigma F_c$ for each level between successive least-squares cycles. The scale factors could not be included as parameters in the least-squares refinement because there were large interactions between the various scale factors and the Sr temperature

Table 1. *Final atomic coordinates and standard deviations for* $SrO.2B_2O_3$ *from least-squares refinement*

	$x(\sigma_x)$	$y(\sigma_y)$	$z(\sigma_z)$	$B(\sigma_B)$
Sr	0	0·2119(0·0003)	0	0·41(0·02)
O(1)	0·585(0·005)	0·772(0·003)	0	0·75(0·28)
O(2)	0·547(0·004)	0·357(0·002)	0·141(0·001)	0·63(0·16)
O(3)	0·144(0·004)	0·725(0·002)	0·135(0·001)	0·70(0·19)
O(4)	0·147(0·003)	0·132(0·002)	0·277(0·001)	0·53(0·17)
B(1)	−0·041(0·009)	0·327(0·003)	0·378(0·001)	0·68(0·25)
B(2)	−0·006(0·012)	0·823(0·003)	0·248(0·001)	0·76(0·24)

factor. As the refinement proceeded it was noted that the strongest intensities had calculated values appreciably higher than observed ones. This was attributed to extinction effects and the strongest twenty-one reflections (based on the uncorrected intensities) were arbitrarily eliminated from the final refinement. The final R value, for the 204 reflections used in the final refinement, is 7·1% (for all 225 reflections $R=10·1\%$).

All of the computations up to this point were done with the X-ray 63 system of programs developed at the University of Maryland (1964) and at the University of Washington, Atomic scattering factors used in the refinements were O^- and neutral B (*International Tables for X-Ray Crystallography*, 1962) and Sr^{2+} (Thomas & Umeda, 1957). The final distances, angles, and associated errors were obtained with the use of the Busing & Levy Function and Error Program (1959).

Results and discussion

The final least-squares results for the atomic parameters of the seven atoms in the asymmetric unit are given in Table 1. Sr and O(1) are in the twofold special position (x, y, 0; $\frac{1}{2}+x$, $\bar{y}$, $\frac{1}{2}$). The remaining three oxygen atoms and the two boron atoms are in the fourfold general position (x, y, z; $x, y, \bar{z}$; $\frac{1}{2}+x, y, \frac{1}{2}-z$; $\frac{1}{2}+x, \bar{y}, \frac{1}{2}+z$). All pertinent interatomic distances and angles are listed in Table 2.

Table 2. $SrO.2B_2O_3$ *bond distances* (Å), *angles* (°) *and their standard deviations**

Values in parenthesis have been calculated from the results of Krogh-Moe (1964)

Sr–O		
nearest neighbors		
Sr^1–$O(1)^3$	2·626 ± 0·020	
Sr^1–$\underline{O(2)^3}$ & $O(2)^4$	2·523 ± 0·016	
Sr^1–$O(2)^1$ & $O(2)^2$	2·836 ± 0·017	
Sr^1–$O(3)^1$ & $O(3)^2$	2·666 ± 0·012	
Sr^1–$\underline{O(3)^1}$ & $\underline{O(3)^2}$	2·764 ± 0·012	
second nearest neighbors		
Sr^1–$O(1)^3$	3·042 ± 0·018	
Sr^1–$O(1)^1$	3·152 ± 0·021	
Sr^1–$O(4)^1$ & $O(4)^2$	3·050 ± 0·011	
Sr^1–$O(4)^5$ & $O(4)^6$	3·204 ± 0·012	
B–O distances		
$B(1)^1$–$O(1)$	1·474 ± 0·023	(1·37)
–$O(2)$	1·466 ± 0·020	(1·47)
–$O(3)$	1·365 ± 0·044	(1·55)
–$O(4)^1$	1·597 ± 0·027	(1·50)
$B(2)^7$–$O(2)^1$	1·455 ± 0·019	(1·53)
–$O(3)^7$	1·430 ± 0·033	(1·38)
–$O(4)^1$	1·507 ± 0·059	(1·48)
–$O(4)^8$	1·546 ± 0·029	(1·61)

Table 2 (*cont.*)

O–B–O angles		
$O(1)$–$B(1)^1$–$O(2)$	108·3 ± 1·7	(117·3)
$O(1)$–$B(1)^1$–$O(3)$	113·3 ± 2·0	(96·4)
$O(1)$–$B(1)^1$–$O(4)^1$	105·2 ± 2·0	(117·8)
$O(2)$–$B(1)^1$–$O(3)$	113·2 ± 2·0	(83·0)
$O(2)$–$B(1)^1$–$O(4)^1$	107·3 ± 1·8	(123·2)
$O(3)$–$B(1)^1$–$O(4)^1$	109·0 ± 1·6	(103·7)
$O(2)^1$–$B(2)^7$–$O(3)^7$	117·3 ± 2·5	(127·9)
$O(2)^1$–$B(2)^7$–$O(4)$	111·4 ± 2·9	(113·5)
$O(2)^1$–$B(2)^7$–$O(4)^8$	104·7 ± 1·9	(85·2)
$O(3)^7$–$B(2)^7$–$O(4)^1$	109·0 ± 2·2	(111·0)
$O(3)^7$–$B(2)^7$–$O(4)^8$	104·7 ± 2·8	(106·1)
$O(4)^1$–$B(2)^7$–$O(4)^8$	109·2 ± 1·9	(107·3)
O–O intratetrahedral distances		
$O(1)$–$O(2)$	2·383 ± 0·015	(2·42)
$O(1)$–$O(3)$	2·371 ± 0·026	(2·18)
$O(1)$–$O(4)^1$	2·440 ± 0·012	(2·46)
$O(2)$–$O(3)$	2·365 ± 0·022	(2·00)
$O(2)$–$O(4)^1$	2·469 ± 0·014	(2·61)
$O(3)$–$O(4)^1$	2·415 ± 0·023	(2·39)
$O(2)^1$–$O(3)^7$	2·464 ± 0·016	(2·61)
$O(2)^1$–$O(4)^1$	2·447 ± 0·021	(2·52)
$O(2)^1$–$O(4)^8$	2·377 ± 0·014	(2·12)
$O(3)^7$–$O(4)^1$	2·391 ± 0·023	(2·37)
$O(3)^7$–$O(4)^8$	2·356 ± 0·015	(2·39)
$O(4)^1$–$O(4)^8$	2·488 ± 0·012	(2·49)

* The atomic designations have been chosen so that this listing corresponds to distances shown in Figs. 3 & 4. Unsuperscripted atoms have parameters as given in Table 1. The superscripts refer to the following operations: (1) $\frac{1}{2}+x, 1-y, \frac{1}{2}-z$; (2) $\frac{1}{2}+x, 1-y, \frac{1}{2}+z$; (3) $-\frac{1}{2}+x, 1-y, \frac{1}{2}-z$; (4) $-\frac{1}{2}+x, 1-y, \frac{1}{2}+z$; (5) $x, 1+y, z$; (6) $x, 1+y, 1-z$; (7) $1+x, y, z$; (8) $1+x, 1+y, z$. For the underlined atoms, substitute $2-y$ for $1-y$ in the transformation indicated by the superscript.

The structure consists of a three-dimensional borate network, which contains channels parallel to **b**. The Sr ions fit into these channels. This can most clearly be seen in the (010) projection (Fig. 1). All boron atoms are tetrahedrally coordinated and all tetrahedra share all corners. The structure contains the unusual feature of an oxygen atom, O(4), coordinated to three boron atoms. Such a coordination for oxygen has been reported in only two previous structures, the hydrated strontium borate mineral tunellite (Clark, 1963) and B_2O_3 (Berger, 1953).

The borate structure could be described in terms of cross-linked chains of tetrahedra, but in view of the common occurrence of six-membered B–O rings in structures rich in B_2O_3 it seems more significant to consider it as built up of chains, parallel to the a axis, of six-membered rings that share edges. These chains

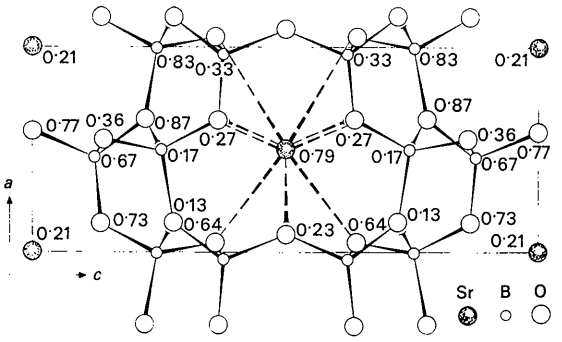

Fig. 1. (010) projection. The numbers are the y parameters. Sr–O nearest neighbor coordination shown by dashed lines. B–O bonding shown by solid lines. Lines ending in short arcs indicate bonding to atoms in the cell above.

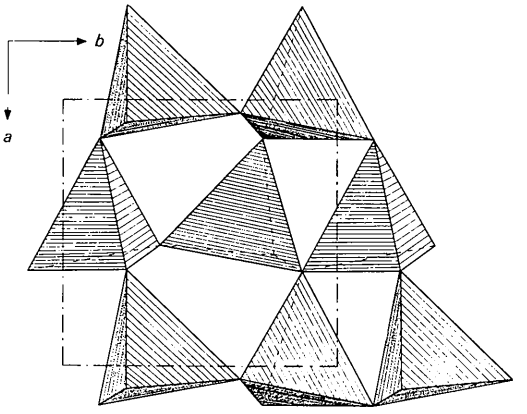

Fig. 2. Borate tetrahedra layer between $z=0$ and $z=\frac{1}{2}$ viewed down the c axis. Sr atoms have been omitted.

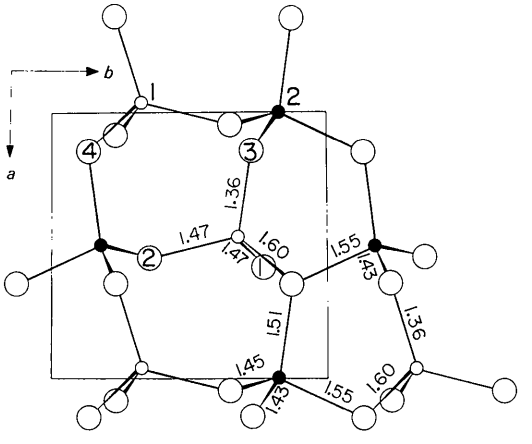

Fig. 3. (001) projection between $z=0$ and $z=\frac{1}{2}$ showing B–O distances (Å). Sr atoms have been omitted. Large circles are oxygen atoms, small open circles are B(1) and small solid circles are B(2) atoms. Some distances have been repeated to show the values around the six-membered rings as well as around each type of boron atom. The numbered atoms are those listed in Table 1.

are then connected through the non-ring general position oxygen, O(2), to form layers parallel to the (001) plane (Fig. 2). These layers are then linked through the special position oxygens to give the three-dimensional network.

An examination of the B–O distances shows a rather large range, 1·36 – 1·60 Å, although their average is 1·48 Å the normal tetrahedral value. As might be expected the bonds around the three-coordinated oxygen are significantly longer than the average with compensating shortening of other bonds. It should be noted (Fig. 3) that the very long and very short B–O distances are all involved in ring formation while the non-ring distances are more nearly normal. This suggests that the six-membered borate ring is a sufficiently favored configuration, energetically, to permit sizable distortions of the bond lengths from their typical values. The angles all lie within the range found in other borate structures.

The nearest neighbor Sr–O distances are given in Table 2 and Fig. 4. The 2·63 Å distance represents only one interaction while the other bonds all are repeated by the mirror plane to give a total coordination of nine. The actual coordination is not clearly defined because there are six next-nearest oxygen neighbors, at 3·04 – 3·20 Å distances, which probably have some significant part in the Sr coordination.

In summary, we have the first example of an anhydrous borate which has only tetrahedral boron and in which all the boron and oxygen atoms are involved in the borate network. It is the first anhydrous borate containing an oxygen linked to three boron atoms. Also, it is another example to show that the rules for deriving the number of tetrahedral and triangular borons from the bulk composition, derived by Edwards & Ross (1960) for hydrated borates and extrapolated by Krogh-Moe (1960, 1962) to anhydrous borates, are not universally valid. For $SrO \cdot 2B_2O_3$ these rules would predict that half of the boron atoms would be

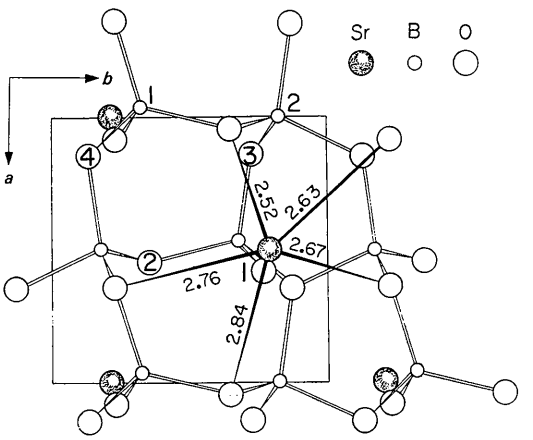

Fig. 4. (001) projection between $z=0$ and $z=\frac{1}{2}$ showing Sr–O nearest neighbor distances (Å). The 2·63 value represents a single distance lying in the mirror plane. The other values are repeated by the mirror plane. The numbered atoms are those listed in Table 1.

in tetrahedral and half in triangular coordination, whereas all of the borons are found to have tetrahedral coordination. The danger of relying on bulk composition to predict structure is well illustrated by the completely unrelated structures of $SrO.2B_2O_3$ and $BaO.2B_2O_3$ (Block & Perloff, 1965).

Note added in revision

Shortly after this paper was submitted to the editor, a structure for $SrO.2B_2O_3$ was reported by Krogh-Moe (1964). His results agree with ours in the gross features of the structure, but are sufficiently different in detail to suggest the possibility of some error. To facilitate comparison our results have been converted to Krogh-Moe's origin and space group orientation ($Pmn2_1$) by the transformation $x'=z, y'=-y+\frac{1}{2}, z'=-x$ followed, where necessary, by the appropriate space group transformation. These are listed in Table 4. The agreement for all of the x' and y' parameters is very good, but the z' parameters for O(2) and B(1) differ by much larger amounts than can be accepted on the basis of the reported standard deviations. The source of the error is obvious. An electron-density map derived from phases based only on the Sr contribution contains false mirror planes at $z'=0$ and $\frac{1}{2}$. It seems clear that the positions chosen by Krogh-Moe for O(2) and B(1) belong to one enantiomorph while the rest of his positions belong to the other enantiomorph. Since these misplaced atoms lie quite close to the false mirrors, the errors on the B–O distances are not very large. However, the influence on the O–B–O angles is quite marked and the errors, also, show up in the abnormal O–O distances of a given borate tetrahedron. The distance and angles involving boron and oxygen as computed from Krogh-Moe's parameters and cell dimensions have been added to Table 2 for comparative purposes. The fact that our values all fall within the range typical of tetrahedral borate groups, while some of Krogh-Moe's are quite abnormal, strongly supports the correctness of the structure as reported in this paper.

As added confirmation we have refined, by least squares, our parameters with Krogh-Moe's observed data (case I) and his parameters with our observed data (case II) with the following results.

Case I: The cell dimensions, space group orientation, and the 189 observed structure factors as reported in the Krogh-Moe (1964) paper were used. One overall scale factor, all position parameters (except Sr x' and z', and O(1) x') and all individual isotropic temperature factors were varied for five cycles. Unit weights were used. The starting parameters were those given in Table 4. By allowing temperature factors to become negative (to compensate for the absorption problem) the structure reported in this paper refined to $R=11{\cdot}5\%$ (as opposed to Krogh-Moe's $12{\cdot}6\%$). All of the position parameters shifted less than one standard deviation during the refinement.

Case II: In an effort to test the two structures under identical refinement conditions, Krogh-Moe's parameters were converted to our origin and space group orientation and refined with our data and weighting scheme. This refinement was carried out in the same stepwise manner as our original refinement. Each step consisted of a structure factor calculation to adjust the four scale factors followed by four cycles of least-squares refinement of variable position parameters and

Table 3. *Observed and calculated structure factors*

Individual level scale factors are $s_1=24{\cdot}31$, $s_2=23{\cdot}58$, $s_3=23{\cdot}97$, $s_4=21{\cdot}48$. Reflections marked * were omitted from the final refinement.

0kℓ

k	ℓ	$\lvert S_1F_o\rvert$	$\lvert 10F_c\rvert$	$10A_c$	$10B_c$
*0	2	297	280	280	0
*	4	410	456	456	0
*	6	524	569	569	0
*	8	680	825	825	0
	10	266	254	254	0
	12	239	238	238	0
1	0	166	146	146	0
	1	285	319	0	319
	2	24	26	-26	0
*	3	495	660	0	660
*	4	449	500	500	0
	5	381	416	0	416
	6	18	18	-18	0
	7	384	418	0	418
	8	65	58	58	0
	9	174	180	0	180
	10	129	120	120	0
	11	375	386	0	386
	12	140	120	120	0
	13	261	254	0	254
*2	0	596	965	-965	0
	1	154	166	0	166
	2	401	474	-474	0
	3	352	367	0	367
	4	323	352	-352	0
	5	76	64	0	64
	6	417	461	-461	0
	7	240	238	0	238
	8	489	548	-548	0
	9	111	112	0	112
	10	260	254	-254	0
	11	235	229	0	229
	12	242	236	-236	0
3	0	247	255	-255	0
	1	167	179	0	-179
	2	182	186	-186	0
	3	349	379	0	-379
	4	544	634	-634	0
	5	344	364	0	-384
	6	108	119	-119	0
	7	91	75	- 0	-75
	8	171	174	-174	0
	9	246	240	0	-240
	10	248	232	-232	0
	11	241	232	0	-232
4	0	119	119	119	0
	1	325	325	0	-325
4	2	347	353	353	0
	3	243	243	0	-243
	4	189	181	181	0
	5	193	198	0	-198
	6	256	258	258	0
	7	292	288	0	-288
	8	174	160	160	0
	9	246	242	0	-242
5	0	249	242	242	0
	1	56	51	0	51
	2	313	307	307	0
	3	134	128	0	128
	4	384	356	356	0
	5	155	135	0	135
	6	268	257	257	0

1kℓ

k	ℓ	$\lvert S_2F_o\rvert$	$\lvert 10F_c\rvert$	$10A_c$	$10B_c$
*0	1	285	347	347	11
*	3	481	723	719	-80
	5	404	489	439	-216
	7	354	398	376	132
	9	264	278	269	-71
	11	355	384	384	-3
	13	251	256	246	-70
*1	0	376	476	64	476
	1	229	235	232	-33
*	2	478	739	43	738
	3	66	59	39	44
*	4	561	826	-335	755
	5	87	66	14	-65
	6	375	454	67	449
	7	239	227	218	61
	8	315	340	59	335
	9	103	89	63	-63
	10	439	515	-73	509
	11	48	62	52	35
	12	270	278	-90	273
	13	46	50	46	-20
2	0	446	522	-274	445
	1	359	408	-406	-44
	2	231	230	134	188
	3	340	384	-362	129
	4	168	156	-17	155
	5	340	361	-350	87
	6	296	297	16	296
2	7	322	333	-329	-52
	8	203	196	-72	182
	9	264	266	-266	6
	10	135	132	14	131
	11	235	240	-233	58
	12	122	101	42	92
3	0	321	332	-105	-315
	1	346	353	-350	45
	2	407	435	89	-426
	3	198	187	-182	-41
	4	312	318	45	-315
	5	165	157	-153	38
	6	340	345	27	-344
	7	336	333	-332	-28
	8	267	260	-31	-259
	9	222	208	-196	69
	10	266	254	29	-252
	11	160	146	-140	-42
4	0	301	307	89	-294
	1	296	279	276	40
	2	353	343	-9	-343
	3	144	116	43	-108
	4	257	249	-35	-246
	5	162	190	190	6
	6	362	350	17	-350
	7	230	214	213	-10
	8	223	217	51	-210
	9	212	190	186	39
5	0	168	144	43	137
	1	284	266	263	-36
	2	159	135	-57	123
	3	273	260	259	13
	4	150	133	15	132
	5	268	257	255	31

2kℓ

k	ℓ	$\lvert S_3F_o\rvert$	$\lvert 10F_c\rvert$	$10A_c$	$10B_c$
*0	2	423	533	519	-125
	4	372	420	420	-9
	6	397	465	459	79
	8	473	557	538	144
	10	254	269	266	-37
	12	328	306	303	-39
1	0	68	58	15	56
*	1	429	540	133	524
	2	159	155	136	-75
1	3	322	352	-29	351
	4	269	254	219	128
	5	390	437	107	424
	6	88	85	60	-60
	7	366	408	7	408
	8	63	59	59	-5
	9	288	310	92	296
	10	73	92	82	41
	11	233	240	-41	237
	12	146	119	119	-11
2	0	472	552	-518	-193
	1	200	188	79	170
	2	367	401	-400	-28
	3	264	264	-91	248
	4	379	419	-416	46
	5	218	207	69	196
	6	325	343	-333	-80
	7	105	98	-33	92
	8	371	389	-378	-88
	9	229	213	88	195
	10	261	263	-262	15
	11	182	169	-74	152
3	0	198	192	-190	-27
	1	278	280	-100	-262
	2	251	247	-244	35
	3	293	296	20	-295
	4	411	412	-395	-119
	5	257	251	41	-248
	6	193	178	-175	53
	7	276	272	-144	-231
	8	201	184	-184	7
	9	208	199	6	-198
	10	200	185	-174	-62
4	0	147	133	132	-16
	1	284	274	-20	-273
	2	243	234	214	95
	3	271	266	-13	-266
	4	294	283	278	-56
	5	228	228	-19	-227
	6	184	168	159	55
	7	235	221	1	-221
	8	123	116	110	35
5	0	326	311	309	-37
	1	119	111	69	87
	2	244	234	228	52
	3	137	116	-28	113
*	4	297	304	304	6

3kℓ

k	ℓ	$\lvert S_4F_o\rvert$	$\lvert 10F_c\rvert$	$10A_c$	$10B_c$
*0	1	227	315	308	-63
*	3	437	552	552	-26
	5	446	570	560	-104
	7	126	134	132	-21
	9	321	355	354	-22
*	11	345	322	322	-2
*1	0	336	373	63	367
	1	180	171	171	3
*	2	422	542	-38	541
	3	21	21	0	-21
	4	275	291	-210	201
	5	114	109	107	-21
	6	367	435	14	435
	7	105	92	85	37
	8	317	331	43	328
	9	138	138	133	-38
	10	326	322	-68	315
	11	14	12	-11	-5
2	0	191	182	-177	41
	1	255	271	-270	16
	2	303	304	40	301
	3	388	433	-427	73
	4	87	90	62	66
	5	358	385	-379	68
	6	270	260	-23	259
	7	204	202	-202	5
	8	121	118	-88	79
	9	253	245	-245	11
	10	151	130	29	127
3	0	372	394	11	-394
	1	322	321	-320	20
	2	231	220	49	-214
	3	76	106	-105	16
	4	195	196	29	-193
	5	208	205	-204	13
	6	264	253	40	-249
	7	245	226	-225	-20
	8	284	269	15	-269
4	0	176	175	94	-148
	1	183	166	165	12
	2	314	297	2	-297
	3	238	217	199	-86
	4	248	234	-39	-231
	5	141	122	121	-20
*	6	263	268	21	-267

Table 4. *Comparison between the results of Krogh-Moe* (1964) *and the present results, which have been converted to his cell and space group* $Pmn2_1$

All parameters have been multiplied by 1000

Atom designation							
This paper	From Krogh-Moe's paper	This paper x'	This paper y'	This paper z'	From Krogh-Moe's paper x	From Krogh-Moe's paper y	From Krogh-Moe's paper z
Sr	Sr	0	288	0	0	289	0
O(1)	O(1)	0	728	415	0	728	454
O(2)	O(2)	359	858	953	359	857	064
O(3)	O(4)	365	225	356	365	226	335
O(4)	O(3)	223	632	353	221	631	335
B(1)	B(1)	378	173	041	379	174	976
B(2)	B(2)	248	677	006	246	671	963

individual isotropic temperature factors. Convergence was initially quite slow, but after three steps (a total of three adjustments of the scale factors by structure factor calculation and twelve cycles of least squares) Krogh-Moe's model refined to a result identical with ours.

The authors feel that the combination of more reasonable interatomic distances and angles, a lower R value of our structure with Krogh-Moe's data, and the ultimate refinement of his structure into ours with our data provides ample proof of the correctness of the structure as reported in the present paper.

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The Crystal Structure of the Copper(I) Cyanide Hydrazine Complex, $CuCN.N_2H_4$*†

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$CuCN \cdot N_2H_4$ is orthorhombic, space group *Pbcm* with four formula units per unit cell. The lattice constants are $a=4{\cdot}684$, $b=9{\cdot}172$ and $c=7{\cdot}830$ Å. The CuCN portion forms planar zigzag infinite chains lying on the mirror. These chains are joined by the hydrazine molecules to form infinite puckered layers which nest together. The copper has four neighbors forming a quite distorted tetrahedron.

Introduction

Copper(I) cyanide forms addition compounds with a great many nitrogen-containing compounds. Because of the unusual structures shown by $KCu(CN)_2$ (Cromer, 1957) and $KCu_2(CN)_3 \cdot H_2O$ (Cromer & Larson, 1962) we have decided to investigate the structures of some of these addition compounds. The structure of $CuCN \cdot NH_3$ has recently been published (Cromer, Larson & Roof, 1965) and we now report on the hydrazine complex, $CuCN \cdot N_2H_4$.

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