

- GREEN, M. L. H. (1968). In *Organometallic Compounds*, Vol. 2, 3rd. ed. pp. 70–74. London: Methuen.
- HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502.
- HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040.
- HUBER, P. J. (1961). *Helv. Chim. Acta*, **44**, 2032.
- JOHNSON, C. K. (1965). ORTEP. ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521.
- PAUL, I. C., JOHNSON, S. M., PAQUETTE, L. A., BARRETT, J. H., HALUSKA, R. J. (1968). *J. Amer. Chem. Soc.* **90**, 5023.
- ROBSON, A. & TRUTER, M. R. (1968). *J. Chem. Soc. (A)*, p. 794.
- SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* **B24**, 63.
- SMITH, D. L. & DAHL, L. F. (1962). *J. Amer. Chem. Soc.* **84**, 1743.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175.
- SUTTON, L. E. (1965). *Tables of Interatomic Distances and Configurations in Molecules and Ions*. Special Publication No. 18. London: The Chemical Society.

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The Crystal Structure of the High-Temperature Modification of Potassium Pentaborate

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The high-temperature modification of potassium pentaborate, $\alpha\text{-K}_2\text{O}\cdot 5\text{B}_2\text{O}_3$, is orthorhombic with 4 formula units in the unit cell of dimensions $a=8\cdot 383 \pm 0\cdot 001$, $b=8\cdot 418 \pm 0\cdot 001$, $c=21\cdot 540 \pm 0\cdot 003$ Å. The space group is *Pbca*. The calculated density of the phase, $1\cdot 93$ g.cm $^{-3}$, is much less than the density of the low-temperature modification, $2\cdot 29$ g.cm $^{-3}$, for which the structure was reported previously. The structure in this study was determined and refined by least-squares methods to an *R* value of 0·062 with three-dimensional diffractometer data. The weighted *R* value was 0·038. The structure contains the characteristic double-ring pentaborate groups connected in two separate interpenetrating networks. The bond lengths of the boron–oxygen bond in the planar BO_3 triangle are about 1·320 Å if the adjacent bond to the oxygen atom is from a fourfold coordinated boron atom, and 1·385 Å if the adjacent bond to the oxygen atom is from a threefold coordinated boron atom. The standard deviation for the boron–oxygen bond is 0·006 Å. The potassium ion is surrounded by 8 close oxygen neighbours at distances ranging from 2·770 to 3·139 Å.

Introduction

The crystal structure of the low-temperature modification of potassium pentaborate, $\beta\text{-K}_2\text{O}\cdot 5\text{B}_2\text{O}_3$, was determined previously by Krogh-Moe (1959a, 1965). Unit-cell data for the high-temperature modification were also reported (Krogh-Moe, 1959b). Because of the great difference in density of these two phases (the ratio of the densities is $\varrho_\beta/\varrho_\alpha = 1\cdot 19$), it was assumed that there might be interesting structural differences between the two phases; therefore, a complete structure determination of the high-temperature modification was undertaken.

Experimental

Crystalline $\alpha\text{-K}_2\text{O}\cdot 5\text{B}_2\text{O}_3$ was prepared by fusing potassium carbonate with boric acid in the stoichiometric ratio in a platinum crucible and crystallizing the mixture at 760°C. A single crystal in the shape of a prism bounded by the (100), (010), and (001) faces was used for collecting data. Dimensions of the crystal were $0\cdot 0090 \times 0\cdot 0095 \times 0\cdot 0080$ cm along the *a*, *b*, and *c* axes respectively; the *a* axis was aligned with the goniometer head axis. Data were collected with an on-line

Picker single-crystal automatic diffractometer. Unit-cell dimensions and standard errors $a=8\cdot 383 \pm 0\cdot 001$, $b=8\cdot 418 \pm 0\cdot 001$, $c=21\cdot 540 \pm 0\cdot 003$ Å were obtained by the method of least-squares from angle data recorded at 22°C for 12 high-angle reflections (based on the wavelength 1·5405 Å for $\text{Cu } K\alpha_1$). For four formula units of $\text{K}_2\text{O}\cdot 5\text{B}_2\text{O}_3$ in the cell, the calculated density is $1\cdot 93$ g.cm $^{-3}$. Krogh-Moe (1959b) obtained an experimental value of $1\cdot 95$ g.cm $^{-3}$ for this phase.

Structure determination

The systematic extinction corresponds to that of space group *Pbca*. The experimental intensities were converted in the usual manner to observed structure factors. No correction for absorption was made. With a linear absorption coefficient of $62\cdot 9$ cm $^{-1}$, the absorption error in the relative structure factors from a crystal of the above mentioned size and shape will not be large. (A sphere of about the same size would require a maximum relative correction of 2% in the intensities between the high-angle and low-angle limit of the observations.) Preliminary signs for the largest structure factors were determined by the symbolic-

Table 1. Final observed and calculated structure factors

The columns are I , $10F_O$, and $10F_C$ respectively. Five reflexions marked by an asterisk are affected by extinction and were omitted from the least-squares refinement.

0,0,L	6	727	750	7	560	575	5	142	119	10	186	207	18	559	-538	11	112	-131	2	521	-514	16	114	108	2	73	-17		
2 76	75	8	352	-341	10	879	878	7	116	115	12	73	-51	9	176	-163	3	138	-147	15	122	-93	3	111	-77				
4 298	-264	9	178	-157	11	112	68	8	232	-219	13	61	-27	21	151	127	7	99	90	5	481	-481	12	456	-466	5	145	150	
8 156	144	10	127	-148	13	569	-557	9	221	203	3,6,L	23	61	17	6	208	227	6	466	454	11	167	-174	6	137	126			
12 554	-562	11	203	-197	14	615	-614	10	170	151	15	96	7	115	-154	18	352	-357	3,0,L	4	173	-178	8	290	-290	9	231	-237	
16 671	675	12	151	-141	15	96	7	115	159	154	18	352	-357	3,0,L	2	123	17	187	187	9	117	-120	7	207	-207	9	163	-175	
18 65	-15	14	941	343	17	118	98	12	213	127	17	187	-187	16	98	-79	22	352	-355	1	524	-527	10	281	-293	6	149	-142	
20 403	-398	15	168	-154	16	460	-450	14	155	86	10	109	125	20	271	270	0	94	68	11	264	-262	5	273	-258				
24 519	507	16	84	89	19	343	-334	15	115	-93	14	267	276	18	158	162	13	222	215	4	712	-716	12	163	-167				
0,2,L	17	65	-75	20	100	103	16	127	-110	13	100	-106	16	541	533	4,6,L	14	348	336	3	193	-195	16	57	26				
* 10 172	-1619	1,6,L	21	455	-451	17	85	20	12	165	-142	14	770	-760	0	424	421	1	686	-672	2	229	-221	17	115	-100			
1 131	1243	19	82	-84	23	276	277	22	61	19	17	85	-90	12	428	-423	1	752	-748	10	161	-145	8	81	63				
12 244	-1300	19	98	-111	22	352	-317	19	71	-17	10	85	-90	9	276	-256	10	508	-501	3	302	-291	16	235	220				
# 1571	1744	1,0,L	8	266	256	8	266	256	8	266	256	8	266	256	8	266	256	8	266	256	8	266	256	8	266	256			
4 1149	-1180	16	96	126	28	99	-97	19	173	186	6	558	-555	4	92	57	5	74	38	20	84	-90	16	346	339				
5 240	-240	15	101	116	22	385	-379	17	174	-179	5	189	-180	2	1607	1766	6	209	191	1	343	-349	10	173	-152				
6 140	-132	14	248	251	20	178	181	16	218	-231	4	354	-359	4,0,L	7	299	-279	20	201	-195	2	98	-113	8	422	434			
7 137	-1394	13	128	-140	18	479	472	15	339	348	2	435	-434	8	164	175	18	80	70	3	268	-273	6	715	746				
8 566	566	12	142	-158	16	526	526	14	222	242	2	435	-434	9	148	159	17	149	137	4	91	57	4	101	76				
9 151	1573	11	161	-143	12	528	-511	11	254	250	1	82	25	11	537	536	11	152	156	9	309	308	2	642	-666				
10 608	-602	10	143	143	12	558	-537	10	182	-182	3,5,L	8	779	-788	12	253	-239	14	161	-131	6	427	441	8,0,L	8,0,L				
11 533	541	9	143	-143	10	143	143	10	160	-160	3,5,L	8	779	-788	12	253	-239	14	161	-131	6	427	441	8,0,L	8,0,L				
12 618	-607	7	98	-79	8	297	-293	9	389	-382	1	223	227	10	392	388	13	170	-157	13	87	36	8	207	204				
13 98	-58	6	333	323	6	836	-836	8	324	-320	2	197	-209	12	417	420	14	193	-192	9	115	-115	2	350	362				
14 854	4	390	-395	4	701	-733	7	367	371	3	136	-122	14	336	-344	15	122	-120	11	224	-212	11	218	-213	6	274	269		
15 242	252	2	558	-553	2	520	515	6	373	-360	4	85	16	130	-135	16	438	-455	9	201	192	12	288	-314	8	263	258		
16 475	-473	1	62	47	2,0,L	5	380	377	6	159	180	18	149	-121	17	78	43	8	153	143	13	320	326	10	88	76			
17 138	-137	1,5,L	5	592	-589	7	191	184	20	265	267	4	7,L	7	192	-187	12	167	167	16	144	138	14	181	-196				
20 294	296	1	195	-184	8	211a	2755	3	202	200	2	100	-64	2	130	139	14	118	-121	4	410	410	31	6,5,L	8,1,L				
21 134	-103	2	320	-299	4	241	230	1	723	-712	10	98	-97	23	149	148	13	58	-63	3	94	31	8,1,L	8,1,L					
22 208	-207	3	576	568	6	209	223	0	130	119	11	64	35	12	69	42	19	285	264	11	171	-175	1	84	-94				
0,4,L	6	521	520	8	447	456	2,6,L	10	244	-231	13	102	83	17	107	-129	10	250	255	5,1,L	5,1,L	5,1,L	5,1,L	5,1,L	5,1,L				
0 163	143	7	247	-239	14	191	191	15	281	-274	14	153	-116	17	300	300	8	363	354	1,1,L	1,1,L	1,1,L	1,1,L	1,1,L	1,1,L				
1 130	132	15	95	-88	16	433	414	1	109	-84	15	152	-147	16	300	300	8	363	354	1,1,L	1,1,L	1,1,L	1,1,L	1,1,L	1,1,L				
2 304	271	9	659	-640	18	314	310	-1	104	-98	15	152	-147	16	300	300	8	363	354	1,1,L	1,1,L	1,1,L	1,1,L	1,1,L	1,1,L				
3 427	-406	11	598	590	12	209	198	11	121	-120	13	121	309	12	209	209	11	121	-120	5	149	149	1,1,L	1,1,L	1,1,L				
4 690	680	12	410	414	24	160	175	5	166	-162	20	50	-5	11	72	72	1	287	-285	5	479	486	6,5,L	3,0,L	3,0,L				
5 575	-555	13	611	681	14	361	-346	2,1,L	7	362	362	3,4,L	9	297	298	8	161	-162	4,8,L	8	161	162	8,2,L	8,2,L					
6 104	340	14	188	224	18	618	-622	15	92	87	14	306	329	3,4,L	9	297	298	8	161	-162	4,8,L	8	161	162	8,2,L	8,2,L			
7 161	151	21	170	182	16	488	-475	16	169	-139	13	99	91	14	306	329	4,2,L	9	161	-162	4,2,L	9	161	162	8,2,L	8,2,L			
8 173	511	22	170	182	16	488	-475	16	169	-139	13	99	91	14	306	329	4,2,L	9	161	-162	4,2,L	9	161	162	8,2,L	8,2,L			
9 349	343	21	114	98	15	714	-700	17	61	-31	11	471	471	17	61	82	1	451	-446	5,8,L	8	163	-164	5,8,L	8	163	164	8,2,L	8,2,L
10 249	249	20	174	-174	12	211	218	18	84	84	14	306	329	17	61	82	1	451	-446	5,8,L	8	163	-164	5,8,L	8	163	164	8,2,L	8,2,L
21 62	-81	19	85	-14	11	331	322	19	148	-141	9	452	-440	8	281	301	8	214	222	6,7,L	10	113	-113	6,7,L	10	113	113	8,2,L	8,2,L
22 151	152	15	254	-253	16	373	370	10	208	-207	12	114	120	7	138	140	8	214	222	7,7,L	10	113	-113	7,7,L	10	113	113	8,2,L	8,2,L
0,6,L	15	68	-25	10	170	-187	16	262	260	6	460	455	9	297	298	6	161	-162	5,0,L	8	161	-162	5,0,L	8	161	-162	5,0,L	5,0,L	
0 371	-346	13	331	335	16	63	-158	14	110	-62	4	340	331	8	161	-162	5,0,L	8	161	-162	5,0,L	8	161	-162	5,0,L	5,0,L			
1 359	-243	12	103	103	5	309	-315	15	163	-163	3	452	441	8	161	-162	5,0,L	8	161	-162	5,0,L	8	161	-162	5,0,L	5,0,L			
2 82	-26	10	124	124	4	167	179	15	181	-181	16	309	315	9	297	298	5,7,L	10	113	-113	5,7,L	10	113	-113	5,7,L	5,7,L			
3 150	-174	10	124	124	3	365	-356	9	297	298	17	464	-454	8	161	-162	5,7,L	10	113	-113	5,7,L	10	113	-113	5,7,L	5,7,L			
4 197	166	9	244	-238	6	283	270	10	127	-122	1	316	-316	17	464	-454	5,7,L	10	113	-113	5,7,L	10	113	-113	5,7,L	5,7,L			
5 100	78	7	171	-172	17	428	402	7	285	291	3,2,L	12	91	91	17	464	-454	5,7,L	10	113	-113	5,7,L	10	113	-113	5,7,L	5,7,L		
6 129	-143	13	63	93	19	111	-83	9	62	62	23	56	37	9	240	245	13	174	-174	6,1,L	12	96							

addition method, using a program written by Hjortås (1969). An electron-density map showed the potassium position clearly, and it was found that the potassium atom alone was heavy enough to dominate most signs.

With additional signs derived from potassium coordinates, the structure was clearly revealed by electron-density calculations. The structure was refined by the method of least-squares, using a version of the full-

Table 2. Final atomic parameters

Positional parameters are in fractions of the cell edge $\times 10^4$, and temperature factors are of the form:

$$\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)] \times 10^4$$
.

	<i>x</i>	<i>y</i>	<i>z</i>	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K	1055.6	4058.0	3764.6	88	85	7.9	-6	4	-2
O(1)	1716	3498	1024	66	110	7.2	-17	2	-15
O(2)	221	1966	1760	105	57	5.6	5	-2	-2
O(3)	4141	2358	4254	67	90	5.2	-9	-2	6
O(4)	4301	4568	3560	74	62	6.7	13	4	4
O(5)	3824	3415	321	47	122	8.3	-15	3	-15
O(6)	1275	2440	20	57	151	6.7	-4	0	-12
O(7)	357	4171	2453	147	42	7.7	3	-11	13
O(8)	718	1671	2858	146	47	7.6	-14	-11	5
B(1)	101	3093	1238	72	79	9.2	-4	5	-5
B(2)	2257	3119	468	84	58	8.7	12	-4	-1
B(3)	4696	2141	4822	63	66	10.3	9	2	0
B(4)	410	2537	2327	76	67	10.3	-8	-6	0
B(5)	342	119	2996	84	74	7.2	8	2	-2
Standard deviations:									
K	1.3	1.2	0.4	2	2	0.2	2	0.5	0.5
O	4	3	1	5	5	0.6	5	2	2
B	7	6	2	9	9	1.2	8	3	3

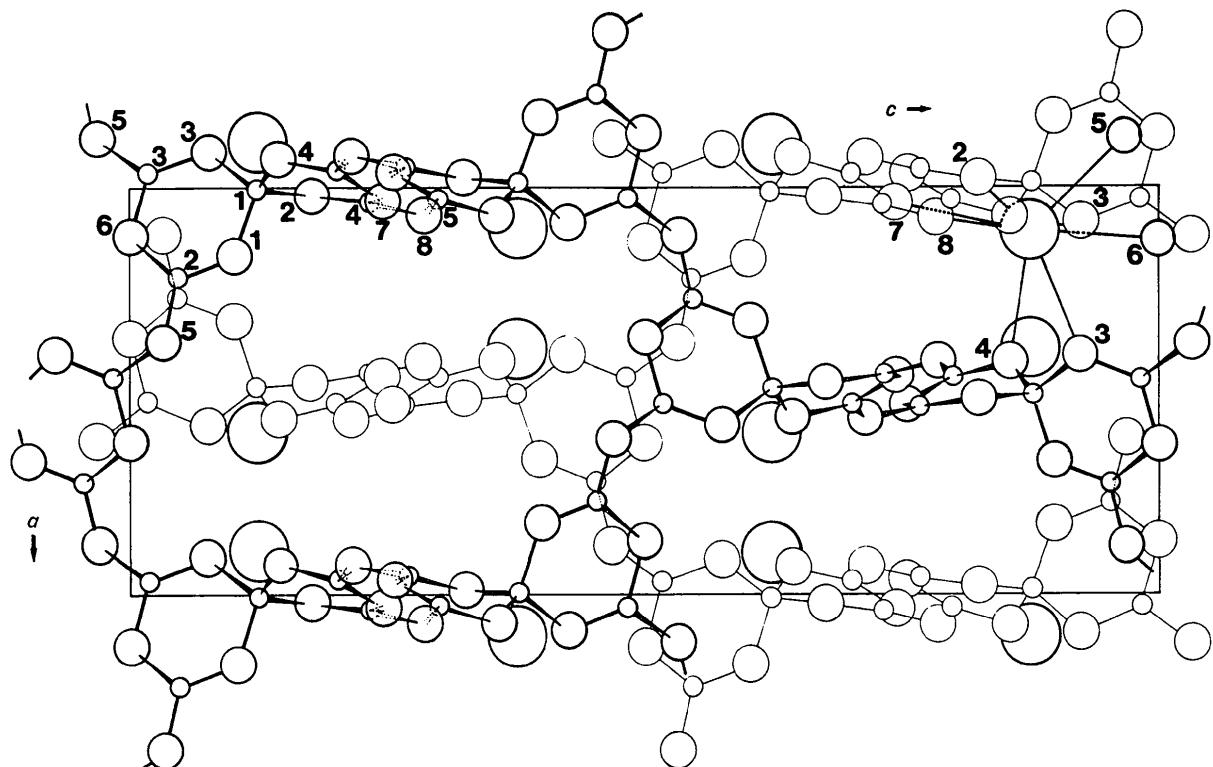


Fig. 1. Projection of the structure of α -K₂O·5B₂O₃ along the *b* axis. The small circles represent boron, intermediate circles represent oxygen, and large circles represent potassium. The numbering of atoms is consistent with Table 2. The structure has two identical interlocking boron–oxygen networks, of which one is drawn with thin lines. The networks connect through O(8) to unit cells directly above and below. The oxygen atoms surrounding one potassium ion are indicated in the upper right corner. Unit-cell dimensions are indicated by the rectangle.

matrix program *ORFLS* (Busing, Martin & Levy, 1962), revised by Borgen & Mestvedt (1966). Structure factors corresponding to the five strongest experimental intensities were clearly affected by extinction and were omitted from the refinement, as were the unobserved reflexions. This left a total of 1087 observed structure factors (Table 1) in the refinement. Atomic scattering factors used for O, B, and K⁺ were taken from *International Tables for X-ray Crystallography* (1962). The refinement was carried out with a weighting scheme based on a combination of statistical counting errors with other errors assumed to be 1% of the

observed intensity. With anisotropic temperature factors the structure refined to an *R* value of 0.062 (or 0.038 for weighted *R*). Final atomic coordinates with standard deviations, and the parameters of the anisotropic temperature factors, are given in Table 2.

Discussion of the structure

A projection of the structure along the *b* axis is shown in Fig. 1. The structure consists, as in the case of the β -modification (Krogh-Moe, 1965), of two three-dimensional interpenetrating networks, built up from the double-ring pentaborate group. Fig. 2 shows a schematic picture of this group.

The present structure differs from the β -modification in the mutual arrangement of the pentaborate groups: a twofold screw axis runs close to one end of the pentaborate group. Groups attached to each other by a common oxygen atom, located in the neighbourhood of the twofold screw axis, extend from this atom in opposite directions (*trans* configuration). In the denser β -modification, however, a twofold screw axis runs near the centre of the pentaborate groups. The groups turn back in nearly the same direction around this screw (*cis* configuration). Obviously, this relates to the considerable difference in packing of these modifications.

The boron–oxygen bond lengths of the pentaborate group are listed in Table 3. They show some interesting systematic variations, apart from the well-known difference between boron coordinated by four oxygen atoms (average bond length 1.473 Å) and boron coordinated by three oxygen atoms (average bond length 1.365 Å). As seen in Fig. 2, the boron–oxygen bond length from a threefold coordinated boron (trigonal bond) depends on the second bond associated with the same oxygen atom. If this is a long tetrahedral bond, the trigonal bond becomes appreciably shortened, from ~1.385 Å to ~1.320 Å. This effect seems to be present also in the previously studied β -modification, where the average for the shorter trigonal bonds is 1.35 Å and for the longer trigonal bonds 1.38 Å. The accuracy of the structure determination of the β -modification, however, does not permit a detailed discussion of bond lengths in that phase.

Hyman, Perloff, Mauer & Block (1967) determined the structure of sodium tetraborate with the same level of accuracy as in the present work. This structure also has a pentaborate group in the network (together with a triborate group). The bond lengths of this pentaborate group are shown in Fig. 2 for comparison and reveal excellent agreement with the present results with respect to shortening of the trigonal bonds in identical surroundings (*i.e.* the left sides of the pentaborate groups in Fig. 2). However, when two trigonal bonds from the same boron atom are adjacent to tetrahedral bonds, the shortening for each bond is less, (the bonds are shortened from 1.38 to 1.36 Å instead of about 1.33 Å).

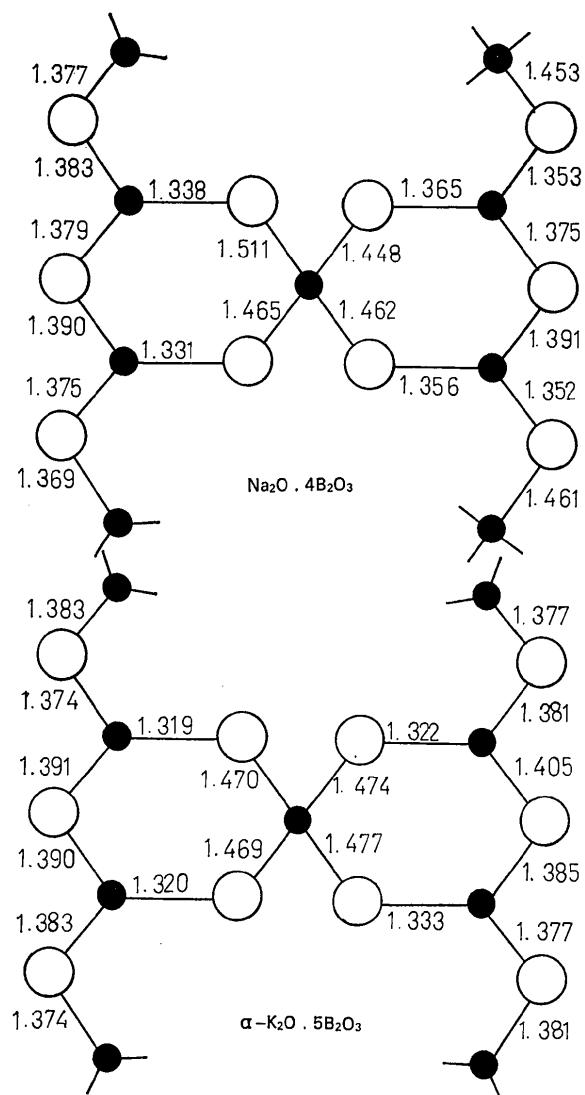


Fig. 2. Schematic drawing of the pentaborate group, including outside boron atoms joined to the group. The planes through the two ring units of the group are approximately perpendicular to each other. Bond distances in this group obtained from sodium tetraborate and from the present work are shown for comparison. For the sodium compound, the two outer boron atoms connected to the right-hand side ring are fourfold-coordinated.

Table 3. Interatomic distances and bond angles

Boron–oxygen bond lengths (standard deviation 0.006 Å). Oxygen–boron–oxygen bond angles (standard deviation 0.3°). Potassium–oxygen distances (standard deviation 0.003 Å) shorter than 3.80 Å, and potassium–potassium distances (standard deviation 0.002 Å).

B(1)–O(1)	1.470 Å	O(1)–B(1)–O(2)	108.7°
B(1)–O(2)	1.474	O(1)–B(1)–O(3)	112.0°
B(1)–O(3')	1.469	O(1)–B(1)–O(4)	108.4°
B(1)–O(4')	1.477	O(2)–B(1)–O(3)	108.4°
		O(2)–B(1)–O(4)	110.3°
		O(3)–B(1)–O(4)	108.4°
B(2)–O(1)	1.319	O(1)–B(2)–O(5)	119.6°
B(2)–O(5)	1.374	O(1)–B(2)–O(6)	121.8°
B(2)–O(6)	1.391	O(5)–B(2)–O(6)	118.7°
B(3)–O(3)	1.320	O(3)–B(3)–O(5)	125.5°
B(3)–O(5)	1.383	O(3)–B(3)–O(6)	122.6°
B(3)–O(6')	1.390	O(5)–B(3)–O(6)	111.9°
B(4)–O(2)	1.322	O(2)–B(4)–O(7)	122.0°
B(4)–O(7)	1.405	O(2)–B(4)–O(8)	126.5°
B(4)–O(8)	1.381	O(7)–B(4)–O(8)	111.1°
B(5)–O(4)	1.333	O(4)–B(5)–O(7)	122.1°
B(5)–O(7')	1.385	O(4)–B(5)–O(8)	118.4°
B(5)–O(8)	1.377	O(7)–B(5)–O(8)	119.4°
K–O(5'')	2.770	K–K'	4.856 Å
K–O(4)	2.789	K–K'	5.831
K–O(8)	2.816	K–K'	6.873
K–O(7)	2.886	K–K'	7.107
K–O(2')	2.901	K–K'	7.265
K–O(3'')	2.977		
K–O(6')	2.990		
K–O(3)	3.139		

Table 3 shows that the O–B–O bond angles involving the tetrahedrally coordinated boron atom B(1) are slightly larger ($\sim 111^\circ$) within the rings than adjacent to the rings ($\sim 108.5^\circ$). The average value, 109.4°, however, agrees with the ideal tetrahedral angle. The average B–O–B angle for all the triangularly coordinated boron atoms is 120.0°, also equal to the ideal value; however, bond angles involving B(3) and B(4) show interesting variations from 120°. The large (125.5°) bond angle O(5)–B(3)–O(3) and the adjacent small (111.9°) bond angle O(5)–B(3)–O(6) (as well as the analogous pair O(8)–B(4)–O(2) and O(8)–B(4)–O(7) in the other subunit of the pentaborate group) arise, perhaps, from the repulsion between pentaborate groups. The structure has two comparatively short oxygen–oxygen distances [O(2)–O(7), 2.940 Å and O(3)–O(6), 2.920 Å], where the oxygen atoms belong to different pentaborate groups. The larger angle in each pair of distorted bond angles is positioned in the

indirect O–B–O–B–O links between the two short ‘intergroup’ oxygen distances, so as to avoid the even shorter oxygen–oxygen distance following from the normal 120° bond angle. The B–O–B bond angle in the center of the indirect link is the oxygen bond angle between the pentaborate groups. This bond angle is 128.9 and 129.6° for B(2)–O(5)–B(3) and B(4)–O(8)–B(5), respectively, and is larger than the B–O–B bond angles in the rings (average 119.2°).

The β -modification has an even shorter ‘intergroup’ oxygen–oxygen distance of 2.73 Å, positioned analogous to the oxygen–oxygen distance discussed above for the α -modification. Also for this structure, comparatively large ($\sim 124^\circ$) O–B–O bond angles were observed in the O–B–O–B–O indirect linkage connecting the two closely spaced oxygen atoms. (The B–O–B angles for the oxygen atoms connecting the pentaborate groups were, in this case, 125.3 and 125.6°.)

The eight shortest potassium–oxygen distances are given in Table 3. The near surroundings of the potassium ions are indicated for one potassium atom in Fig. 1. Oxygen atoms are seen to surround potassium in an irregular eightfold coordination. The bond distances range from 2.770 to 3.139 Å, the next (9th) neighbour being at 3.817 Å. In the β -modification, the bond distances start at 2.91 Å and go to 3.20 Å for the nine closest neighbours. Apparently, as a rule, regular coordination polyhedra for the cations do not form in the polyborate network structures.

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References

- BORGEN, O. & MESTVEDT, B. (1966). *LSFIV4*. Technical Report 45. Institutt for Fysikalisk Kjemi, NTH, Trondheim, Norway.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS: A Fortran Crystallographic Least-Squares Program*. Report ORNL-TM-305. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
- HJORTÅS, J. (1969). *Symbol*. Institutt for Røntgenteknikk, NTH, Trondheim, Norway.
- HYMAN, A., PERLOFF, A., MAUER, F. & BLOCK, S. (1967). *Acta Cryst.* **22**, 815.
- INTERNATIONAL TABLES FOR X-RAY CRYSTALLOGRAPHY (1962). Vol. III. Birmingham: Kynoch Press.
- KROGH-MOE, J. (1959a). *Ark. Kemi*, **14**, 439.
- KROGH-MOE, J. (1959b). *Ark. Kemi*, **14**, 567.
- KROGH-MOE, J. (1965). *Acta Cryst.* **18**, 1088.