

La configuration des centres d'asymétrie de la molécule est représentée sur la Fig. 6. Cette figure montre également les angles de torsion des cycles C , C' et D (Klyne & Prelog, 1960). La conformation des cycles C et C' est proche de la conformation 'demi-chaise' du cyclohexène (Bucourt & Hainaut, 1965), et celle du cycle D proche de la conformation 'chaise' du cyclohexane.

Les distances intermoléculaires les plus courtes sont données dans le Tableau 5. Outre les liaisons hydrogène déjà mentionnées, il existe une interaction faible (3,10 Å) entre l'atome d'hydrogène de l'un des azotes indoliques [N(23)] et l'atome d'oxygène d'un groupement méthoxyle [O(17)] appartenant à une autre molécule. C'est ce groupement qui se trouve tourné à 20° par rapport au plan de l'indole (Fig. 4), tandis que le groupement méthoxyle appartenant à l'autre partie de la molécule se trouve situé exactement dans le plan de l'indole correspondant. Dans ce dernier cas, la conjugaison avec le système insaturé de l'indole provoque un net raccourcissement des liaisons comme, par exemple, dans le cas du 1-4 diméthoxybenzène (Goodwin, Przybylska & Robertson, 1950).

Les principaux programmes utilisés pour les calculs ont été les suivants:

- pour la résolution: *DEVIN* (Riche, 1972);
- pour l'affinement: une version locale de *ORFLS* (Busing, Martin & Levy, 1962);
- pour les calculs de distances et de plans moyens, la série des programmes N.R.C. (Ahmed, Hall, Pippy & Huber, 1966);
- pour certains dessins, *ORTEP* (Johnson, 1965).

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The Crystal Structure of α Sodium Triborate, α -Na₂O · 3B₂O₃

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The α -modification of sodium triborate, α -Na₂O · 3B₂O₃, is monoclinic with the following unit-cell dimensions (at 22°C): $a = 10.085 \pm 0.002$, $b = 11.363 \pm 0.002$, $c = 10.845 \pm 0.002$ Å, $\beta = 104.48 \pm 0.02^\circ$; $Z = 6$, calculated density 2.242 g cm⁻³; space group $P2_1/c$. Three-dimensional X-ray intensity data were measured with a Picker automatic single-crystal diffractometer using Mo $K\alpha$ radiation. The structure was determined by direct methods and refined by full-matrix least-squares calculations to an R value of 0.098. The borate anion in this structure forms two separate and interpenetrating infinite frameworks. Each framework consists of pentaborate groups and diborate groups in equal amounts. The boron-oxygen distances all fall within the normal ranges, and with the typical variations depending on the position within the groups. The three crystallographically different sodium atoms are coordinated by 5, 6 and 6 oxygen atoms respectively at distances ranging from 2.280 to 2.742 Å.

Introduction

Milman & Bouaziz (1968) reported three different crystalline triborate phases in the system sodium oxide-boron oxide. The crystal structure of one of these

phases, labelled β -Na₂O · 3B₂O₃, has been studied by Krogh-Moe (1972*b*). Before this the only anhydrous triborate structure known was that of the caesium compound (Krogh-Moe, 1960).

The structure of β sodium triborate is substantially

different from caesium triborate, and hence a structure determination of other triborate phases was considered of interest. The present paper is concerned with the structure of a second modification (denoted α) of sodium triborate. As will be shown, this modification has a borate anion structure not encountered previously. This serves to emphasize the several ways in which the borate anion groups may polymerize for a given stoichiometry. Such differences in structures have previously been noted for the diborates (Krogh-Moe, 1974). It is tempting to relate the evident range of possible structures to the strong glass-forming ability of these compounds.

Experimental

Crystalline α -Na₂O·3B₂O₃ was prepared by fusing borax (Na₂B₄O₇·10H₂O p.a. quality) with boric acid (H₃BO₃ p.a. quality) in stoichiometric ratio in a platinum crucible. The water was driven off and a glass melt was formed, which crystallized on further heating. The exact temperature at which crystallization took place was not known (because of strong temperature gradients in the muffle furnace used), but was approximately 725°C.

A single-crystal fragment of irregular shape was mounted for the X-ray work. The dimensions were approximately 0.18 × 0.14 × 0.08 mm. Intensity data were collected with an on-line Picker single-crystal automatic diffractometer. Mo K α radiation was used, and the X-ray reflexions were measured at 2770 reciprocal lattice points.

Unit-cell dimensions and standard errors: $a = 10.085 \pm 0.002$, $b = 11.363 \pm 0.002$, $c = 10.845 \pm 0.002$ Å, $\beta = 104.48 \pm 0.02^\circ$ were obtained by the method of least squares from angle data recorded at 22°C for 12 high-angle reflexions (based on the wavelength 0.7093 Å for Mo K α). With 6 formula units of Na₂O·3B₂O₃ in the cell, the calculated density is 2.242 g cm⁻³. This is close to the value 2.246 g cm⁻³ obtained for the β -modification (Krogh-Moe, 1972b).

A calculated X-ray powder pattern based on the unit-cell dimensions given above, did not agree entirely with any of the powder patterns published by Milman & Bouaziz (1968) for the sodium triborates. There was sufficient resemblance with the pattern of their α -phase, however, to make it a reasonable assumption that the phase studied in the present paper is identical with the α -phase of Milman & Bouaziz.

Structure determination

The systematic extinctions correspond to those of the space group $P2_1/c$. A statistical test of the distribution of normalized structure factors indicated a centre of symmetry as expected for $P2_1/c$. The experimental intensities were converted in the usual manner to observed structure factors. No correction for absorption was applied because of the rather irregular shape of

the crystal fragment. Since Mo K α radiation was used, neglect of absorption corrections should not lead to appreciable errors in the present case.

The structure was determined by direct methods, using the programs of Germain, Main & Woolfson (1971). The set of signs with the highest 'figure of merit' gave a sensible structure. The structure was refined by the method of least squares, utilizing the 2270 reflexions with intensities above the background. (Thus about 500 reflexions with negative intensities were removed from the data set.) The atomic scattering factors for O, B and Na⁺, applied for obtaining the calculated structure factors, were taken from *International Tables for X-ray Crystallography* (1962). The refinement was carried out with a weighting scheme based on the statistical counting errors compounded with errors assumed to be 1% of the observed intensity. The structure refined to an R index of 0.098 (or 0.097 for the weighted R index). This is somewhat high for diffractometer data, but can be explained by the large fraction of the measured intensities which are small compared with their statistical counting errors. (Thus the 1756 reflexions with structure factors greater than three times their standard errors gave an R index of 0.073 for the structure reported here.) During the last cycle of refinement, the largest shift to error ratio was 0.42. The observed and calculated structure factors are given in Table 1, and the final atomic coordinates and the thermal parameters are given in Table 2.

Discussion of the structure

The anion in α sodium triborate forms two separate interpenetrating twin frameworks, built from the well-known pentaborate and diborate groups. The latter two groups can be seen in Fig. 1 projected approximately along the b axis. The manner in which these groups combine to form one of the two three-dimensional frameworks can be studied in Fig. 2. Finally in Fig. 3 the complete structure is shown, both frameworks being included in the stereo-pair.

Interpenetrating double frameworks occur frequently in the anhydrous borates. The first case was established by Krogh-Moe (1959) for potassium pentaborate. Since then examples have been found for enneaborates (Krogh-Moe & Ihara, 1967), tetraborates (Krogh-Moe, 1965) and diborates (Krogh-Moe, 1968). The present work represents the first known case for a triborate.

Though the principle of double frameworks built up from borate groups is familiar, the combination of diborate and pentaborate groups found here is new. Each diborate group is bonded to four pentaborate groups and *vice versa*. The four bonds emerging from the diborate group are not all of the same character (two involve fourfold and two involve threefold coordinated borons of the diborate group). This results in an asymmetry of bonding in each ring of the double ring pentaborate group. Krogh-Moe (1972a) has in-

Table 1. Final observed and calculated structure factors

The columns are I, IOF_o and IOF_c.

Table with multiple columns containing numerical data for structure factors. The data is organized into rows and columns, with some cells containing codes like '100-100', '110-110', etc., and others containing numerical values. The table is very dense and spans most of the page.

Table 1 (cont.)

3,5:1L	9 58 87	7,9:1L	9,9:1L	2 59 19	7 208 271	8,1:1L	8,0:1L	1 50 134	2 211 196	8,2:1L	1 95 97	5 41 85	10,4:1L
1 212 202	10 177 205	6 688 688	8 225 249	1 157 161	8 148 190	0 379 338	2 32 28	2 32 28	0 334 237	0 86 84	0 71 27	3 24 78	1 35 10
2 821 807	1 239 251	2 220 243	4 88 24	2 239 251	1 156 193	1 169 200	1 460 468	1 156 193	1 156 193	1 156 193	2 156 193	2 156 193	2 161 186
4 607 597	3,7:1L	3 132 132	4 27 38	5 84 10	12 132 132	10 122 122	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258
8 237 226	10 132 132	7 187 187	3 85 109	4 27 38	10 122 122	10 122 122	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258
7 231 218	8 189 212	6 298 305	5 114 127	6 84 10	10 122 122	10 122 122	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258	2 258 258
4 218 225	2 78 41	3 320 366	9 237 249	3 320 366	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249
9 169 158	2 78 41	3 208 191	1 140 150	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249	2 237 249
11 58 95	10 76 18	6 228 244	U 20 ⁰ 172	6 235 250	3 238 267	1 292 287	6,7:1L	7,9:1L	6,3:1L	6,3:1L	6,3:1L	6,3:1L	6,3:1L
3,7:1L	2 42 ⁰ 382	9,5:1L	3 193 190	7 132 89	2 128 134	0 98 51	8 101 92	7 93 22	6 208 201	7 128 111	4 43 24	4 113 107	0 88 120
11 200 189	8 276 288	10 52 70	1 348 361	8 95 120	3 240 178	3 240 178	0 98 50	8 90 74	8 90 74	8 90 74	8 90 74	8 90 74	8 90 74
10 227 250	8 358 348	9 196 96	2 362 312	6 215 168	7 140 79	1 114 83	5 107 85	2 80 74	7,7:1L	4 138 121	3 119 79	3 132 130	0 117 130
9 43 21	8 252 247	7 297 277	3 165 164	5 121 138	9 122 118	4 96 100	1 133 150	5 99 89	8 103 35	3 390 365	2 82 78	6 290 300	0 137 130
6 189 183	2 98 7	4 258 251	5 82 57	6 215 168	10 98 58	4 96 100	1 133 150	7,8:1L	7 103 86	1 651 673	0 128 107	4 128 176	0 137 130
7 80 88	4 138 121	5 119 83	6 171 114	3 182 165	6 215 168	6 215 168	6 215 168	6 215 168	6 215 168	6 215 168	6 215 168	6 215 168	6 215 168
6 252 228	5 119 83	6 171 114	7 98 32	2 370 370	6,7:1L	6 181 183	6,8:1L	6 138 12	5 222 211	6 138 12	5 222 211	6 138 12	0 137 128
2 223 223	10 192 118	5 119 83	6 171 114	1 227 176	10 186 174	8 93 73	0 152 137	1 121 139	1 227 176	1 121 139	1 227 176	1 121 139	1 90 25
3 284 283	9 122 117	2 378 388	6,11:1L	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
2 223 229	8 853 588	1 378 388	6,11:1L	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
1,796 797	7 926 923	0 138 119	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
0 914 420	6 193 181	4 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
6 273 273	0 138 119	6 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
0 192 192	0 988 981	6 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
2 929 421	2 157 187	2 293 256	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
3 226 227	0 149 176	3 245 276	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
6 88 63	6 230 230	6 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
6 200 208	0 173 208	6 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
9 240 246	1 243 211	6 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
10 71 108	6 163 114	6 230 230	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
3,7:1L	6 77 25	6 194 187	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
11 165 158	6 77 25	6 194 187	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
10 140 158	6 77 25	6 194 187	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
9 226 205	8 527 638	6 85 88	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
6 184 172	9 283 378	6 85 88	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
7 133 122	10 110 146	6 173 150	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
6 76 107	8 111 100	6 111 100	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
5 71 54	3 209 221	3 87 169	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
4 72 9	2 398 402	1 152 113	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
3 780 759	11 180 162	10 115 58	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L
2 310 369	10 115 58	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L	
1 81 100	8 286 284	6 149 22	6 238 218	7 248 218	6,7:1L	2 153 188	2 0 30	0 155 187	1 236 215	1 236 215	2 208 225	1,1:1L	
0 80 106	6 129 121	2 185 172	6 74 96	6 57 95	0 42 27	2 180 177	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125
1 117 87	4 101 94	3 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125	6 119 125
2 208 259	3 249 249	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291
6 634 614	2 97 75	5 228 197	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291
5 81 88	1 105 106	6 105 106	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291	6 238 291
6 113 96	0 165 164	7 25 14	5 124 125	10 135 110	8 67 30	1 383 372	1 81 78	6 223 219	5 46 5	2 189 218	6,9:1L	9,2:1L	12,2:1L
6 526 508	9 198 168	3 83 33	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110	8 117 110

Table 2. Final atomic parameters

The anisotropic temperature factors are of the form

$$\exp [-2\pi^2(U_{11}(ha^*)^2 + U_{22}(kb^*)^2 + U_{33}(lc^*)^2 + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}kbc^*)].$$

Positional parameters are expressed as fractions of the cell edges. All values are multiplied by 10⁴.

	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Na(1)	707	1350	3774	181	279	192	7	122	29
Na(2)	4098	3681	858	181	260	175	70	90	25
Na(3)	3613	6504	3901	708	228	220	-83	119	-89
O(1)	2929	3565	2425	160	164	49	89	-4	-21
O(2)	2425	9938	4865	115	100	119	16	27	-16
O(3)	4816	2372	3049	131	197	58	76	25	12
O(4)	3614	9610	3158	98	120	78	14	8	29
O(5)	1659	1818	202	182	129	116	39	54	29
O(6)	3200	6248	1744	115	162	78	-52	43	-20
O(7)	1274	5013	1634	96	97	134	23	75	81
O(8)	3405	301	1038	160	187	61	64	62	45
O(9)	979	6636	250	139	142	134	64	41	33
O(10)	4032	8343	4915	200	149	79	50	76	48
O(11)	2119	4743	3872	163	139	78	54	48	40
O(12)	1828	8303	3412	114	189	100	-29	76	-42
O(13)	1881	951	2240	122	282	101	104	68	71
O(14)	179	2289	1585	185	175	118	58	67	77
O(15)	3810	3209	4640	165	164	64	60	27	9
B(1)	4123	6748	1172	65	117	150	-9	29	28
B(2)	2922	9093	4088	167	97	90	57	-5	2
B(3)	3873	3049	3420	119	149	122	-38	21	11
B(4)	2058	4454	2648	78	173				

indicated that the coordination number of external borons bonded to pentaborate groups may affect the internal boron-oxygen bond lengths in a systematic manner. The present structure has the novel feature of asymmetric bonding of the same ring unit of the double ring pentaborate group. This asymmetry, apparently, does not lead to any significant asymmetry within the pentaborate ring system, however. The boron-oxygen bond distances within the pentaborate group (see Table 3) are rather similar to those observed with external borons fourfold coordinated (Krogh-Moe, 1972*b*).

The diborate groups also show some characteristic bond-length variations within the group. The bonds to the central oxygen O(2) of this group are shorter than the other bonds from the same boron atom, 1.45 Å *vs.* values ranging from 1.46 to 1.51 Å (Table 3). The bond angle involving the central oxygen, B(2)-O(2)-B(9), is 108.5° (Table 3). These values are in good agreement with values found for other diborates (Krogh-Moe, 1972*c*).

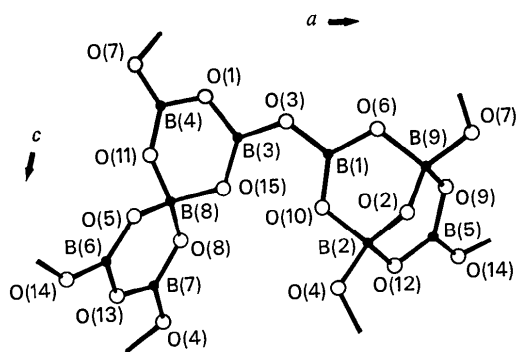


Fig. 1. Projection approximately along the *b* axis showing the two borate groups included in the asymmetric unit. The directions of the two other axes are indicated. Open circles represent oxygen and filled circles represent boron. The numbering of atoms is as given in the tables.

The B-O-B bond angles for the oxygens linking the diborate groups with the pentaborate groups, show the values 117.4, 124.8, 126.9 and 137.2°. This broad range of values for intergroup bond angles is often observed (Krogh-Moe, 1972*c*).

The three different sodium atoms of the asymmetric unit are coordinated in one case by five and for the other two by six oxygen atoms. Distances range from 2.280 to 2.742 Å. Only distances below 3 Å are included in this coordination shell, however. If this arbitrary limit to the sodium coordination is applied to β sodium triborate, all the three non-equivalent sodium

Table 3. *Interatomic distances and bond angles*

Boron-oxygen bond lengths (standard deviation 0.008 Å), sodium-oxygen bond lengths (standard deviation 0.005 Å) below 3 Å, and oxygen-boron-oxygen and boron-oxygen-boron bond angles (standard deviation 0.5°).

B(1)-O(3)	1.382 Å	B(8)-O(11)	1.476 Å
B(1)-O(6)	1.364	B(8)-O(15)	1.512
B(1)-O(10)	1.347	B(9)-O(2)	1.453
B(2)-O(2)	1.447	B(9)-O(6)	1.508
B(2)-O(4)	1.484	B(9)-O(7)	1.458
B(2)-O(10)	1.510	B(9)-O(9)	1.494
B(2)-O(12)	1.468		
B(3)-O(1)	1.378	Na(1)-O(9)	2.240
B(3)-O(3)	1.360	Na(1)-O(13)	2.316
B(3)-O(15)	1.353	Na(1)-O(2)	2.440
B(4)-O(1)	1.399	Na(1)-O(7)	2.460
B(4)-O(7)	1.343	Na(1)-O(14)	2.535
B(4)-O(11)	1.353	Na(1)-O(5)	2.630
B(5)-O(9)	1.359	Na(2)-O(10)	2.280
B(5)-O(12)	1.337	Na(2)-O(1)	2.302
B(5)-O(14)	1.385	Na(2)-O(2)	2.358
B(6)-O(5)	1.358	Na(2)-O(15)	2.500
B(6)-O(13)	1.375	Na(2)-O(4)	2.519
B(6)-O(14)	1.357	Na(2)-O(3)	2.742
B(7)-O(4)	1.354	Na(3)-O(6)	2.291
B(7)-O(8)	1.359	Na(3)-O(10)	2.349
B(7)-O(13)	1.389	Na(3)-O(11)	2.501
B(8)-O(5)	1.476	Na(3)-O(12)	2.688
B(8)-O(8)	1.443	Na(3)-O(15)	2.703

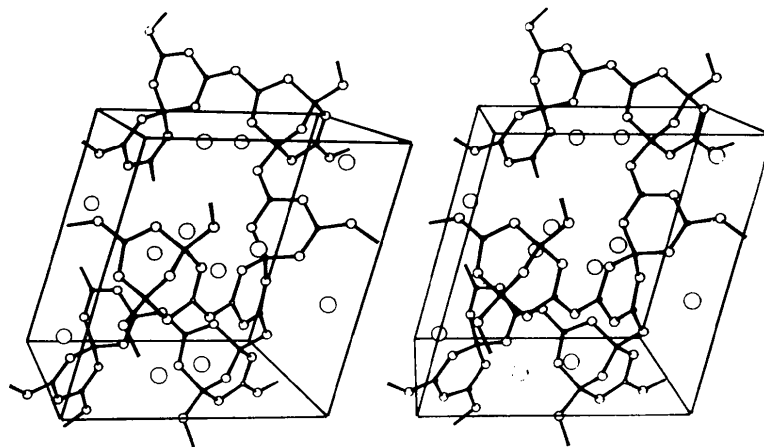


Fig. 2. Stereo-pair showing one of the two double frameworks in α sodium triborate. Unit-cell edges are also shown. The *b* axis is approximately perpendicular to the paper plane, the *a* axis and *c* axis are approximately horizontal and vertical respectively. Large open circles represent the sodium atoms.

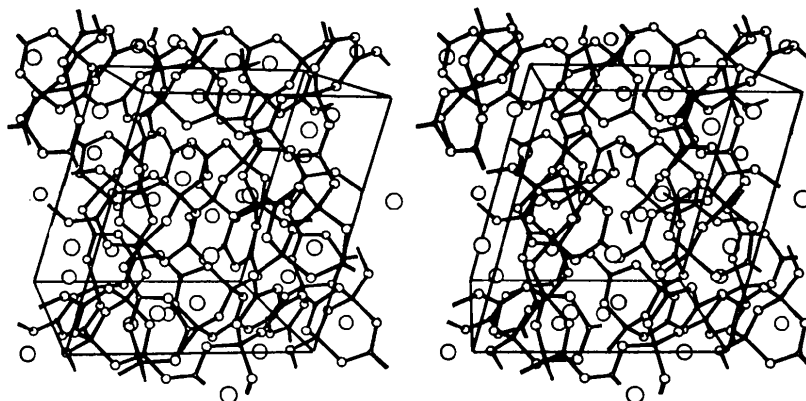


Fig. 3. Stereo-pair oriented as in Fig. 2, but including both the double frameworks. The manner in which the double frameworks interpenetrate can be studied by viewing this figure in a stereoscope.

Table 3 (cont.)

O(3)—B(1)—O(6)	116.6°	O(5)—B(8)—O(15)	106.6°
O(3)—B(1)—O(10)	120.3	O(8)—B(8)—O(11)	109.5
O(6)—B(1)—O(10)	123.1	O(8)—B(8)—O(15)	107.0
O(2)—B(2)—O(4)	115.0	O(11)—B(8)—O(15)	109.1
O(2)—B(2)—O(10)	110.0	O(2)—B(9)—O(6)	108.6
O(2)—B(2)—O(12)	111.7	O(2)—B(9)—O(7)	116.8
O(4)—B(2)—O(10)	102.9	O(2)—B(9)—O(9)	109.6
O(4)—B(2)—O(12)	109.0	O(6)—B(9)—O(7)	106.5
O(10)—B(2)—O(12)	107.8	O(6)—B(9)—O(9)	109.7
O(1)—B(3)—O(3)	114.0	O(7)—B(9)—O(9)	105.5
O(1)—B(3)—O(15)	121.1		
O(3)—B(3)—O(15)	124.9	B(3)—O(1)—B(4)	120.8
O(1)—B(4)—O(7)	117.8	B(2)—O(2)—B(9)	108.5
O(1)—B(4)—O(11)	117.9	B(1)—O(3)—B(3)	126.9
O(7)—B(4)—O(11)	124.2	B(2)—O(4)—B(7)	124.6
O(9)—B(5)—O(12)	122.1	B(6)—O(5)—B(8)	119.7
O(9)—B(5)—O(14)	120.1	B(1)—O(6)—B(9)	115.1
O(12)—B(5)—O(14)	117.6	B(4)—O(7)—B(9)	117.4
O(5)—B(6)—O(13)	120.9	B(7)—O(8)—B(8)	122.1
O(5)—B(6)—O(14)	126.6	B(5)—O(9)—B(9)	120.3
O(13)—B(6)—O(14)	112.4	B(1)—O(10)—B(2)	119.8
O(4)—B(7)—O(8)	123.4	B(4)—O(11)—B(8)	123.8
O(4)—B(7)—O(13)	117.5	B(2)—O(12)—B(5)	116.0
O(8)—B(7)—O(13)	119.1	B(6)—O(13)—B(7)	122.1
O(5)—B(8)—O(8)	115.5	B(5)—O(14)—B(6)	137.2
O(5)—B(8)—O(11)	109.0	B(3)—O(15)—B(8)	121.5

atoms are sixfold coordinated. The five-coordinated sodium atom, Na(3), reported in the present paper, seems to be anomalous. The U_{11} component of the anisotropic temperature factor is comparatively large (Table 2). Correspondingly the standard deviation for the positional x parameter is also somewhat large. The reason for this is not known at present.

Two other anhydrous sodium polyborates have been studied, and the coordinations of sodium in these compounds are as follows. In sodium diborate a sixfold coordination is found for three of the four non-equiv-

alent sodium atoms, the last being sevenfold coordinated (Krogh-Moe, 1974). As mentioned previously by Krogh-Moe (1972*b*), the table of sodium-oxygen distances reported by Hyman, Perloff, Mauer & Block (1967) for sodium tetraborate is not complete. One distance [Na(2)—O(3') of 2.610 Å] is missing. If this distance is included, both the two non-equivalent sodium atoms have eight oxygen neighbours below 3 Å. In all cases a similar wide range of bond lengths was observed. Thus sodium is apparently not coordinated in a strict and well defined manner in the anhydrous borates.

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