positional rigid-body parameters are explicitly evaluated in order to permit a simultaneous refinement of these parameters. It is also possible to apply only geometrical or only thermal constraints and treat a part of the structure in an unconstrained manner in which cases the derivatives are appropriately modified.

The general organization of the program was based in part on an *ORFLS* version modified by Professor F. L. Hirshfeld for a purpose similar to ours, but with some constraints different.

References

ABRAHAMS, S. C., ROBERTSON, J. M. & WHITE, J. G. (1949). *Acta Cryst.* **2**, 233–244.

BERNSTEIN, J. & TRUEBLOOD, K. N. (1971). *Acta Cryst.* B**27**, 2078–2089.

CRUICKSHANK, D. W. J. (1957). *Acta Cryst.* **10**, 504–508.

HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040–1044.

HERBSTEIN, F. H. & SNYMAN, J. A. (1969). *Phil. Trans. Roy. Soc.* A**264**, 635–662.

KURODA, H., AMANO, T., IKEMOTO, I. & AKAMATU, H. (1967). *J. Amer. Chem. Soc.* **89**, 6056–6063.

PAULING, L. (1960). *The Nature of the Chemical Bond.* New York: Cornell Univ. Press.

PAWLEY, G. S. (1963). *Acta Cryst.* **16**, 1204–1208.

PAWLEY, G. S. (1971). *Advances in Structure Research by Diffraction Methods*, Vol. 4, pp. 1–64. Edited by W. HOPPE & R. MASON. Oxford: Pergamon Press.

SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B**24**, 63–76.

SHMUELI, U. & GOLDBERG, I. (1973). *Acta Cryst.* B**29**, 2466–2471.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

WALLWORK, S. C. (1973). Private communication.

WILLIAMS, D. E. (1972). *Acta Cryst.* A**28**, 629–635.

WILLIAMS, R. M. & WALLWORK, S. C. (1967). *Acta Cryst.* **22**, 899–906.

Acta Cryst. (1974). B**30**, 578

The Crystal Structure of Sodium Diborate $Na_2O.2B_2O_3$

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Sodium diborate, $Na_2O.2B_2O_3$, is triclinic, space group $P\bar{1}$, with unit-cell dimensions at 22°C: $a=6\cdot5445\pm0\cdot0004$, $b=8\cdot6205\pm0\cdot0004$, $c=10\cdot4855\pm0\cdot0006$ Å, $\alpha=93\cdot279\pm0\cdot006$, $\beta=94\cdot870\pm0\cdot006$, $\gamma=90\cdot843\pm0\cdot006°$. The calculated density is 2·272 g cm^{-3} with four formula units in the cell. The structure was determined from three-dimensional X-ray data by direct methods. A full-matrix least-squares refinement resulted in an R index of 0·035 (0·040 for the weighted R index). The anion borate polymer in this structure forms layers composed of di-pentaborate groups and triborate groups with one non-bridging oxygen. This is a novel feature in diborates. Boron–oxygen bond lengths are normal, but with significant differences depending on the location within the groups. The sodium atoms are coordinated by six or seven oxygen atoms at distances ranging from 2·260 to 2·872 Å.

Introduction

Several of the anhydrous diborates have polymer structures built up from diborate groups. This is the case for lithium diborate, $Li_2O.2B_2O_3$ (Krogh-Moe, 1968), and cadmium diborate, $CdO.2B_2O_3$ (Ihara & Krogh-Moe, 1966). The structure of this cadmium compound is the first determined structure in an isomorphous series including the Mg, Mn, Fe, Co, Ni, Zn, Hg compounds (Ecker, 1966). Diborates containing groups other than the diborate group are known, however. Potassium diborate (Krogh-Moe, 1972*a*) has, in addition to diborate groups, di-triborate groups. Barium diborate (Block & Perloff, 1965) has a single framework based on di-triborate and di-pentaborate groups. The structure reported for sodium diborate in the present paper provides yet another example of a diborate with a novel combination of groups.

Sodium diborate exists in more than one modification (Jenkel, 1936; Milman & Bouaziz, 1968). The phase studied here crystallizes from the melt (or glass) below the melting point 742°C down to about 650°C. It appears to be the stable phase, at least in the temperature range mentioned. Vila & Font-Altaba (1955) have reported unit-cell dimensions for what appears to be the same phase, though their values differ appreciably from those obtained by us.

Experimental

Anhydrous sodium diborate was prepared by dehydrating and melting borax, $Na_2O.2B_2O_3.10H_2O$, (p.a.

Table 1. *Final observed and calculated structure factors*

The columns are *l*, $10F_o$ and $10F_c$. The reflexions marked by asterisks are affected by extinction and were omitted from the final cycles of least-square refinement.

-7,-3,L		
4	50	46
3	43	41
2	41	43
1	35	36
0	16	23
-1	33	32
-7,-2,L		
-3	19	3
-2	25	25
-1	41	38
0	155	152
1	115	116
2	103	104
3	138	140
4	104	110
5	58	53
-7,-1,L		
5	147	143
4	124	127
3	18	20
2	42	48
1	79	65
0	317	323
-1	55	54
-2	103	105
-3	18	12
-7,0,L		
-3	209	210
-1	61	62
0	24	22
1	169	171
2	7	3
3	23	26
4	46	44
5	73	75
-6,0,L		
8	18	14
7	42	45
6	68	92
5	57	53
4	16	18
3	370	377
2	24	20
1	202	203
0	176	173
-1	177	171
-2	243	248
-3	192	196
-4	151	148
-5	66	70
-6	60	79
-6,-1,L		
-6	16	6
-5	6	7
-4	28	30
-3	63	67
-2	137	146
-1	359	356
0	98	95
1	31	34
2	176	162
3	284	285
4	145	148
5	85	90
6	79	82
7	102	100
8	27	21
-6,-2,L		
8	44	46
7	105	174
6	69	75
5	40	58
4	77	79
3	236	247
2	79	63
1	116	110
0	104	104
-1	16	16
-2	11	5
-3	114	115
-4	15	13
-5	215	210
-6	17	9
-6,-3,L		
-5	30	33
-4	40	50
-3	196	196
-2	11	8
-1	225	227
0	56	53
1	78	67
2	117	122
3	233	247
4	37	23
5	127	116
6	137	139
7	13	22
-6,-4,L		
6	73	81
5	85	89
4	26	25
3	33	23
2	95	99
1	232	236
0	195	187
-1	29	24
-2	65	66
-3	22	20
-4	22	21
-6,-5,L		
-3	98	98
-2	27	24
-1	74	62
0	21	28
1	93	94
2	61	68
3	64	61
4	8	3
5	90	91
-6,-6,L		
2	64	58
1	50	27
0	61	67
-5,-7,L		
4	30	35
3	169	166
2	16	0
1	35	33
0	72	64
-1	61	65
-2	61	61
-5,-6,L		
-4	124	116
-3	208	216
-2	13	11
-1	65	69
0	91	94
1	155	157
2	140	157
3	16	0
4	132	143
5	40	52
6	52	45
-5,-5,L		
7	33	21
6	129	135
5	237	237
4	48	50
3	16	15
2	238	234
1	58	57
0	164	175
-1	31	25
-2	344	359
-3	91	91
-4	8	9
-5	121	122
-5,-4,L		
-6	64	64
-5	46	45
-4	99	96
-3	28	28
-2	171	166
-1	63	62
0	69	64
1	91	49
2	306	310
3	247	241
4	160	148
5	28	25
6	113	112
7	165	173
8	77	76
-5,-3,L		
9	32	44
8	90	96
7	158	156
6	91	92
5	152	151
4	195	195
3	225	225
2	83	86
1	28	20
0	108	102
-1	216	216
-2	314	320
-3	172	182
-4	82	81
-5	200	194
-6	135	135
-7	100	100
-5,-2,L		
-8	17	16
-7	95	89
-6	208	211
-5	142	138
-4	159	151
-3	362	370
-2	89	88
-1	10	4
0	136	132
1	154	149
2	406	410
3	221	220
4	254	250
5	297	294
6	307	313
7	164	160
9	28	30
-5,-1,L		
9	111	113
8	222	219
7	35	42
6	370	373
5	251	253
4	34	46
3	65	72
2	370	376
1	131	129
0	61	55
-1	20	21
-2	159	155
-3	163	157
-4	193	195
-5	25	15
-6	19	6
-7	23	25
-8	58	58
-5,0,L		
-8	110	113
-7	101	97
-6	110	108
-5	64	59
-4	68	67
-3	145	146
-2	314	312
-1	197	193
0	111	113
1	249	240
2	197	192
3	388	394
4	150	150
5	21	19
6	211	207
7	94	95
8	69	64
9	117	118
-4,0,L		
10	91	89
9	125	125
8	30	31
7	29	30
6	62	62
5	136	134
4	198	190
3	24	12
2	19	23
1	167	158
0	21	23
-1	153	149
-2	28	30
-3	60	54
-5	12	6
-6	227	227
-7	414	419
-8	104	104
-9	119	114
-4,-1,L		
-9	10	3
-8	90	89
-7	123	112
-6	133	130
-5	214	209
-4	8	7
-3	23	27
-2	165	173
-1	75	72
0	202	197
1	303	303
2	361	353
3	190	182
4	226	225
5	267	262
6	243	235
7	147	155
8	240	237
9	110	109
10	135	134
-4,-2,L		
10	48	52
9	144	151
8	184	191
7	434	447
6	256	253
5	28	14
4	35	30
3	148	145
2	147	140
1	310	310
0	103	96
-1	289	284
-2	6	6
-3	235	237
-4	58	59
-5	333	337
-6	364	366
-7	57	64
-8	100	94
-9	81	75
-4,-3,L		
-8	28	23
-7	130	132
-6	35	10
-5	29	30
-4	98	87
-3	187	176
-2	325	324
-1	123	119
0	227	222
1	133	127
3	115	114
4	101	103
5	94	98
7	247	234
8	44	40
9	202	207
10	108	106
-4,-4,L		
9	169	162
8	234	234
7	75	83
6	132	131
5	54	60
4	62	68
3	501	504
2	67	67
1	144	138
0	207	210
-1	32	25
-2	54	49
-3	54	51
-4	238	238
-5	136	138
-6	21	16
-7	38	35
-8	11	5
-4,-5,L		
-7	30	24
-6	46	48
-5	23	17
-4	121	123
-3	176	176
-2	32	26
-1	234	230
0	102	97
1	22	16
2	392	387
3	16	6
4	63	71
5	147	158
6	119	115
7	30	26
8	57	63
9	20	9
-4,-6,L		
8	156	151
7	155	160
6	115	117
5	75	80
4	20	17
3	329	337
2	165	155
1	77	76
0	363	362
-1	49	45
-2	122	119
-3	89	87
-4	149	153
-5	109	111
-6	181	183
-4,-7,L		
-4	25	25
-2	272	270
-1	76	76
0	91	89
1	19	19
2	140	143
3	158	157
4	215	206
5	101	99
6	22	27
-4,-8,L		
4	63	57
3	130	126
2	43	27
1	116	118
0	68	56
-1	70	75
-2	25	16
-3,-9,L		
3	33	32
2	107	102
1	83	83
0	128	125
-1	56	52
-3,-8,L		
-4	20	17
-3	149	146
-2	7	5
-1	43	42
0	8	7
1	68	71
2	196	202
3	63	61
4	85	81
5	14	6
6	28	20
-3,-7,L		
8	23	18
7	14	14
6	62	55
5	182	178
4	97	86
3	102	96
2	174	162
1	205	201
0	301	305
-1	179	175
-2	16	7
-3	122	125
-4	153	152
-5	134	134
-6	268	271
-3,-6,L		
-7	30	38
-6	211	210
-5	290	292
-4	161	159
-3	130	134
-2	364	370
-1	260	262
0	181	178
1	277	271
2	180	192
3	245	241
4	351	354
5	170	166
6	70	84
8	127	125
9	16	5
-3,-5,L		
10	135	134
9	40	41
8	402	411
7	331	326
6	160	171
5	76	74
4	100	94
3	40	53
2	198	192
1	243	238
0	103	100
-1	17	13
-2	149	143
-3	217	214
-4	239	237
-5	177	177
-6	65	61
-7	130	141
-8	117	117
-3,-4,L		
-9	12	15
-8	214	212
-7	362	366
-6	173	179
-5	257	251
-4	111	104
-3	96	97
-2	247	240
-1	82	76
0	440	448
1	177	171
2	63	56
3	85	79
4	280	286
5	31	35
6	208	209
7	56	45
8	40	30
9	120	132
10	20	25
-3,-3,L		
11	126	126
10	46	46
9	140	130
8	133	127
7	121	115
6	91	89
5	266	235
4	40	33
3	33	35
2	470	478
1	573	570
0	16	8
-1	376	360
-2	121	117
-3	300	310
-4	227	224
-5	142	145
-6	35	36
-7	231	230
-8	110	100
-9	26	26
-3,-2,L		
-10	255	265
-9	83	84
-8	136	139
-7	144	144
-6	84	78
-5	403	405
-4	33	40
-3	87	87
-2	163	161
-1	146	143
0	174	158
1	191	165
2	444	422
3	30	30
4	221	210
5	45	43
6	190	191
7	48	44
8	66	66
9	261	257
10	110	113
11	70	78
-3,-1,L		
11	64	60
10	71	69
8	453	459
7	332	325
6	63	50
5	283	265
4	75	53
2	234	206
1	74	73
0	310	292
*-1	965	1108
-2	15	21
-3	101	93
-4	297	291
-5	54	52
-6	277	273
-7	101	97
-8	12	13
-9	282	281
-10	180	185
-3,0,L		
-8	0	6
-7	90	86
-6	266	261
-5	10	11
-4	200	197
-3	282	279
-2	188	180
-1	67	68
0	737	770
1	131	129
2	90	92
3	383	374
4	20	22
5	103	98
6	90	93
7	60	65
8	337	335
9	112	112
10	118	116
11	13	13
-2,0,L		
12	16	19
11	143	146
10	133	135
9	330	341
8	103	97
7	242	236
6	227	220
5	28	25
4	61	53
3	264	262
2	73	70
1	148	129
0	133	131
-1	44	45
*-2	928	1114
-3	483	484
-4	356	340
-5	276	270
-6	63	59
-7	125	124
-8	12	7
-9	19	15
-10	170	176
-11	44	45
-2,-1,L		
-11	228	226
-10	21	13
-9	14	14
-8	321	328
-7	76	64
-6	515	525
-5	190	182
-4	348	336
-3	148	140
-2	185	192
-1	450	452
0	440	449
1	17	31
2	530	537
3	438	443
4	77	83
5	458	440
7	64	62
8	240	240
9	298	297
10	161	156
11	28	22
12	83	77
-2,-2,L		
11	98	102
10	297	306
9	226	231
8	325	326
7	185	178
6	238	234
5	412	404
4	856	889
3	117	128
2	127	98
1	137	121
0	72	75
-1	139	142
-2	234	222
-3	129	132
-4	573	590
-5	265	265
-6	125	125
-7	135	132
-8	65	64
-9	45	45
-10	43	44
-11	71	73
-2,-3,L		
-10	30	31
-9	95	92
-8	101	97
-7	46	60
-6	176	181
-5	360	362
-4	14	6
-3	73	74
-2	235	234
-1	121	134
0	478	479
1	143	149
2	487	464
3	459	435
4	28	25
5	52	39
6	30	27
7	205	201
8	60	57
9	33	30
10	42	37
11	32	35
-2,-4,L		
11	49	45
10	32	25
9	113	121
8	86	84
7	185	185
6	164	152
5	33	25
4	144	129
3	350	342
2	166	161
1	91	78
0	284	281
-1	103	105
-2	124	124
-3	76	65
-4	201	195
-5	48	41
-6	32	27
-7	245	244
-8	108	111
-9	90	90
-10	89	83
-2,-5,L		
-9	23	23
-8	93	91
-7	60	57
-6	78	77
-5	28	29
-4	169	171
-3	253	252
-2	88	85
-1	198	206
0	222	215
1	40	42
2	140	131
3	190	183
4	219	204
5	194	192
6	111	110
7	84	91
8	177	177
9	82	85
10	80	80
-2,-6,L		
9	163	164
8	129	135
7	226	234
6	173	166
5	96	97
4	161	152
3	28	25
2	196	186
1	88	81
0	256	252
-1	92	87
-2	314	324
-3	10	3
-4	209	207
-5	35	29
-6	131	130
-7	141	139
-8	33	29
-2,-7,L		
-7	44	47
-6	125	129
-5	189	189
-4	21	18
-3	40	52
-2	177	179
-1	75	75
0	72	61
1	26	19
2	148	135
3	385	385
4	55	51
5	130	125
6	38	45
7	127	115
8	109	112
-2,-8,L		
7	99	100
6	35	26
5	51	55
4	17	19
3	12	5
2	104	105
1	187	188
0	126	123
-1	340	365
-2	27	32
-3	95	97
-4	40	49
-5	32	30
-2,-9,L		
-3	95	97
-2	101	104
-1	110	119
0	13	3
1	60	65
2	124	123
3	33	27
4	62	68
5	82	85
-1,-9,L		
5	25	24
4	30	43
3	47	49
2	100	87
1	71	77
0	60	53
-1	90	93
-2	31	32
-3	27	27
-4	26	18
-1,-8,L		
-6	32	27
-5	44	45
-4	87	91
-3	38	41
-2	74	76
-1	151	159
0	222	218
1	23	17
2	53	59
3	241	230
4	89	80
5	17	4
6	94	108
7	128	142
-1,-7,L		
9	21	28
8	10	11
7	33	24
6	18	26
5	35	36
4	172	163
3	234	239
2	354	347
1	228	224
0	37	32
-1	19	11
-2	193	184
-3	142	147
-4	167	173
-5	31	22
-6	165	165
-7	175	182
-1,-6,L		
-9	90	96
-8	230	234
-7	10	9
-6	81	83
-5	14	13
-4	136	127
-3	60	57
-2	111	111
-1	167	165
0	177	176
1	81	80
2	81	73
3	251	245
4	456	455
5	208	203
6	303	306
7	126	121
8	71	69
9	21	21
10	90	96
-1,-5,L		
10	175	170
9	349	349
8	335	340
7	7	6
6	47	48
5	115	118
4	44	35
3	277	263
2	16	14
1	198	184
0	395	394
-1	438	453
-2	116	120
-3	192	197
-4	196	186
-5	132	132
-6	10	17
-7	64	61
-8	280	292
-9	115	112
-1,-4,L		
-10	169	165
-9	11	4
-8	36	35
-7	167	165
-6	117	111
-5	162	159
-4	86	82
-3	240	235
-2	468	469
-1	22	25
0	540	535
1	234	221
2	176	179
3	575	570
4	242	232
5	237	230
6	22	16
7	46	47
8	240	246
9	73	79
10	99	94
11	28	19
-1,-3,L		
11	57	56
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-5	83	85
-6	50	48
-7	92	89
5,-4,L		
-8	52	51
-7	144	151
-6	44	48
-5	98	101
-4	85	83
-3	114	114
-2	55	55
-1	276	274
0	158	154
1	59	54
2	116	114
3	72	63
4	30	28
5	82	79
6	12	9
7	14	10
5,-3,L		
8	125	122
7	147	143
6	76	71
5	47	50
4	126	123
3	251	248
2	30	23
1	255	253
0	31	34
-1	102	105
-2	48	43
-3	146	139
-4	42	38
-5	12	7
-6	247	245
-7	252	258
-8	26	21
5,-2,L		
-9	22	20
-8	194	194
-7	398	408
-6	17	19
-5	161	158
-4	138	136
-3	229	226
-2	93	87
-1	58	55
0	71	66
1	74	65
2	183	180
3	424	431
4	23	14
5	157	159
6	14	9
7	84	81
8	35	32
5,-1,L		
8	99	100
7	58	53
6	192	190
5	212	214
4	33	35
3	48	15
2	180	173
1	98	97
0	67	62
-1	161	158
-2	188	185
-3	490	505
-4	440	454
-5	178	178
-6	260	262
-7	128	133
-8	121	123
-9	5	11
5,0,L		
-9	110	118
-8	69	64
-7	96	95
-6	213	207
-5	14	19
-4	151	150
-3	302	304
-2	197	192
-1	240	240
0	116	113
1	200	193
2	318	312
3	148	146
4	86	87
5	66	59
6	114	108
7	102	97
8	112	113
6,0,L		
6	81	79
5	67	70
4	154	148
3	195	196
2	245	248
1	177	171
0	176	175
-1	202	203
-2	21	20
-3	374	377
-4	16	18
-5	56	53
-6	90	92
-7	45	45
-8	13	14
6,-1,L		
-8	34	32
-7	73	71
-6	52	60
-5	64	65
-4	67	63
-3	15	18
-2	267	272
-1	398	402
0	10	8
1	245	249
2	25	4
3	234	239
4	50	40
5	96	101
6	73	65
6,-2,L		
6	117	115
5	71	71
4	79	79
3	42	41
2	341	336
1	133	135
-1	214	214
-2	51	54
-3	20	11
-4	107	110
-5	171	167
-6	124	124
-7	15	4
6,-3,L		
-7	219	219
-6	58	54
-6	58	54
-5	17	16
-5	17	16
-4	222	225
-3	40	47
-2	545	561
0	54	52
1	294	293
2	34	33
3	22	22
4	88	83
5	20	20
6	28	28
6,-4,L		
5	53	52
4	70	68
3	31	31
2	85	82
1	37	36
0	203	202
-1	24	23
-2	14	10
-3	169	164
-4	80	85
-5	26	26
-6	63	67
6,-5,L		
-5	14	15
-4	213	218
-3	35	31
-2	21	15
-1	47	47
0	194	191
1	50	49
2	24	20
3	256	262
4	165	163
6,-6,L		
2	51	54
1	120	124
0	13	11
-1	230	238
-2	102	103
7,-4,L		
-2	71	62
-1	126	124
0	100	100
7,-3,L		
2	31	34
1	54	54
0	37	40
-1	161	162
-2	173	175
-3	94	101
-4	250	257
7,-2,L		
-5	46	50
-4	76	70
-3	45	50
-2	65	56
-1	67	70
0	67	66
1	59	61
2	59	64
3	70	71
7,-1,L		
3	39	37
2	34	30
1	29	25
0	44	39
-1	182	181
-2	167	167
-3	75	81
-4	14	15
-5	57	57
7,0,L		
-5	74	75
-4	47	44
-3	27	26
-2	10	3
-1	170	171
0	24	22
1	62	62
2	6	2
3	208	210

quality) in a platinum crucible at 800 °C. The viscous liquid was then crystallized at a temperature of about 700 °C. A single crystal with a prismatic shape and dimensions 0·008 × 0·009 × 0·014 cm was used for the X-ray work. Intensity data were collected with an on-line Picker single-crystal automatic diffractometer and Cu $K\alpha$ radiation. The X-ray reflexions were measured at 2106 different reciprocal lattice points. 2079 reflexions were observed larger than the background.

Unit-cell dimensions and standard errors are: $a = 6{\cdot}5445 \pm 0{\cdot}0004$, $b = 8{\cdot}6205 \pm 0{\cdot}0004$, $c = 10{\cdot}4855 \pm 0{\cdot}0006$ Å, $\alpha = 93{\cdot}279 \pm 0{\cdot}006$, $\beta = 94{\cdot}870 \pm 0{\cdot}006$, $\gamma = 90{\cdot}843 \pm 0{\cdot}006°$, and were obtained by the method of least squares from angle data recorded at 22 °C for 12

Table 2. *Final atomic parameters*

The anisotropic temperature factors are of the form $\exp[-2\pi^2(U_{11}(ha^*)^2 + U_{22}(kb^*)^2 + U_{33}(lc^*)^2 + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$ corresponding to the Cruickshank (1956) U notation. Positional parameters are expressed as fractions of the cell edges. All values are multiplied by 10^4.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Na(1)	1099	1331	6123	158	467	167	83	71	96
Na(2)	1751	3394	0176	143	257	234	28	−1	−27
Na(3)	4773	4643	2272	283	285	235	167	−41	−85
Na(4)	4393	9221	8562	206	184	104	−73	20	−3
O(1)	1481	0992	1548	128	65	103	−10	59	3
O(2)	1952	1369	3899	63	173	99	−16	7	35
O(3)	0992	3453	2499	88	65	156	−4	50	8
O(4)	1393	8652	7119	50	86	88	−19	10	−8
O(5)	1987	7772	4918	81	167	70	−1	14	11
O(6)	2162	5973	6594	104	85	164	13	76	28
O(7)	4719	7946	6649	53	178	83	−9	10	21
O(8)	3089	6936	2876	80	196	90	15	10	−30
O(9)	0347	8417	1884	111	68	166	12	70	24
O(10)	1859	6245	0754	120	79	76	−20	28	−11
O(11)	0480	4043	7574	93	82	178	10	58	37
O(12)	4509	2033	5527	87	323	88	−37	14	−9
O(13)	3039	8873	0559	132	90	132	−8	75	−5
O(14)	4746	6812	9551	150	137	154	35	82	21
B(1)	0709	1770	2713	71	92	94	0	23	−2
B(2)	2494	7605	6317	86	109	101	−11	14	26
B(3)	1178	6847	2005	94	89	83	−1	17	8
B(4)	3290	7225	0254	97	111	84	11	−16	3
B(5)	1555	9444	1335	107	120	81	2	0	11
B(6)	0512	5533	7202	100	104	84	−11	−5	−3
B(7)	3917	1807	4220	108	109	101	8	22	17
B(8)	3443	7561	4091	110	105	118	8	−1	32
Standard deviations									
Na	1·7	1·5	1	6	7	6	5	4	5
O	2·6	2	1·6	8	9	9	7	7	7
B	4·2	3·4	2·6	13	14	13	11	11	11

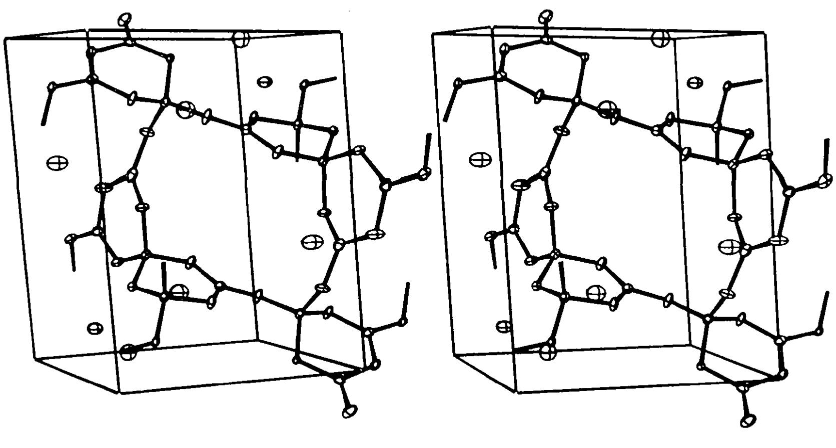

Fig. 1. The structure of sodium diborate, shown as a stereo-pair. The unit cell is indicated. The a axis is approximately perpendicular to the plane of the paper the b axis is horizontal and the c axis vertical in the plane of the paper. The boron–oxygen bonds are shown as solid lines. The isolated vibration ellipsoids represent the sodium atoms.

high-angle reflexions (based on the wavelength 1·5405 Å for Cu $K\alpha_1$). With four formula units of $Na_2O.2B_2O_3$ in the cell, the calculated density is 2·272 g cm^{-3}. Jenkel (1936) measured a density of 2·279 g cm^{-3} for this phase.

Structure determination

The observed intensities were converted to structure factors in the usual manner. A correction for absorption was also applied, the linear absorption coefficient being 31·3 cm^{-1} ($\lambda = 1{\cdot}54178$).

A statistical test of the distribution of normalized structure factors gave a strong indication for the presence of a centre of symmetry. The structure was determined by direct methods, with the program system developed by Germain, Main & Woolfson (1971). The set of signs with the highest 'figure of merit' proved to give a sensible structure. The structure was refined by the method of least squares, with the X-RAY 71 program system edited by Stewart, Kruger, Kundell & Baldwin (1971). All the 2079 reflexions observed to be larger than the background were used in the refinement. The atomic scattering factors for O, B and Na^+ 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 0·045 (or 0·056 for the weighted R index). The 7 reflexions with the strongest intensities were systematically calculated larger than observed. These reflexions were assumed to be affected by extinction and removed from the data set. The data now refined to an R index of 0·035 (0·040 for the weighted R index). During the last cycle of refinement the largest shift to error ratio was 0·22. The observed and calculated structure factors are given in Table 1. (The reflexions affected by extinction are marked by an asterisk.) The final atomic coordinates and the thermal parameters are given in Table 2.

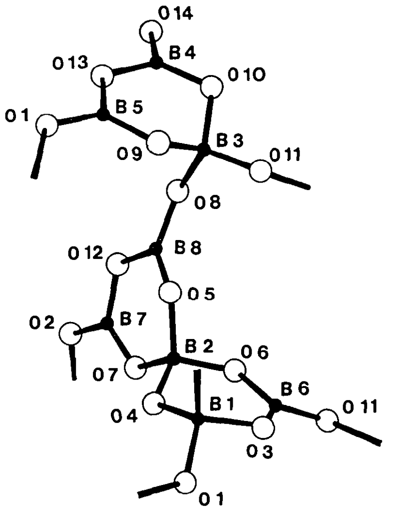

Fig. 2. The asymmetric unit of the boron–oxygen network, showing the triborate group and the di-pentaborate group. The atoms are numbered in accordance with Table 2.

Discussion of the structure

The anion network in sodium diborate may be described as infinitely extending layers approximately perpendicular to the c axis. A layer structure in anhydrous borates has previously only been found for β-sodium triborate (Krogh-Moe, 1972*b*). A section of the borate layer in sodium diborate can be seen in Fig. 1 as a stereo-pair. The layer is composed of two types of borate groups, di-pentaborate groups [notation: see Krogh-Moe (1972*a*)] and triborate groups. These groups can be seen in Fig. 2. The di-pentaborate group was found by Block & Perloff (1965) in barium diborate. The triborate group found here is new for the diborate composition. It has one non-bridging oxygen, O(14). Non-bridging oxygens have not previously been found either in crystalline anhydrous borates of the diborate composition or in more acid compositions. Because of the presence of non-bridging oxygens, the fraction of boron atoms in fourfold coordination, $\frac{3}{8}$, is lower than the value, $\frac{1}{2}$, expected for anhydrous diborates (Krogh-Moe, 1960).

Boron–oxygen bond lengths and bond angles are reported in Table 3. Systematic deviations of bond lengths from average values, depending on the local surroundings of the bond, can be seen. In particular

Table 3. *Interatomic distances and bond angles*

Boron–oxygen bond lengths (standard deviation 0·003 Å), sodium–oxygen bond lengths (standard deviation 0·002 Å) below 3Å and oxygen–boron–oxygen and boron–oxygen–boron bond angles (standard deviation 0·2°).

B(1)–O(1)	1·489 Å	Na(1)–O(9)	2·366 Å
B(1)–O(2)	1·488	Na(1)–O(5)	2·374
B(1)–O(3)	1·492	Na(1)–O(2)	2·445
B(1)–O(4)	1·446	Na(1)–O(12)	2·445
B(2)–O(4)	1·440	Na(1)–O(4)	2·592
B(2)–O(5)	1·492	Na(1)–O(11)	2·770
B(2)–O(6)	1·470	Na(2)–O(14)	2·297
B(2)–O(7)	1·489	Na(2)–O(10)	2·496
B(3)–O(8)	1·483	Na(2)–O(10′)	2·509
B(3)–O(9)	1·474	Na(2)–O(3)	2·525
B(3)–O(10)	1·489	Na(2)–O(1)	2·600
B(3)–O(11)	1·434	Na(2)–O(9)	2·835
B(4)–O(10)	1·406	Na(2)–O(11)	2·872
B(4)–O(13)	1·453	Na(3)–O(14)	2·273
B(4)–O(14)	1·295	Na(3)–O(6)	2·328
B(5)–O(1)	1·343	Na(3)–O(8)	2·358
B(5)–O(9)	1·363	Na(3)–O(7)	2·572
B(5)–O(13)	1·396	Na(3)–O(3)	2·699
B(6)–O(3)	1·371	Na(3)–O(10)	2·809
B(6)–O(6)	1·360	Na(4)–O(7)	2·260
B(6)–O(11)	1·364	Na(4)–O(13)	2·374
B(7)–O(2)	1·344	Na(4)–O(14)	2·379
B(7)–O(7)	1·351	Na(4)–O(4)	2·402
B(7)–O(12)	1·395	Na(4)–O(13′)	2·420
B(8)–O(5)	1·349	Na(4)–O(1)	2·722
B(8)–O(8)	1·355		
B(8)–O(12)	1·399,		

Table 3 (*cont.*)

O(1)—B(1)-O(2)	111·5°	O(3)—B(6)-O(6)	122·3°
O(1)—B(1)-O(3)	102·8	O(3)—B(6)-O(11)	121·0
O(1)—B(1)-O(4)	113·2	O(6)—B(6)-O(11)	116·6
O(2)—B(1)-O(3)	110·0	O(2)—B(7)-O(7)	123·5
O(2)—B(1)-O(4)	106·6	O(2)—B(7)-O(12)	116·4
O(3)—B(1)-O(4)	112·9	O(7)—B(7)-O(12)	120·1
O(4)—B(2)-O(5)	113·4	O(5)—B(8)-O(8)	124·5
O(4)—B(2)-O(6)	112·0	O(5)—B(8)-O(12)	120·3
O(4)—B(2)-O(7)	106·9	O(8)—B(8)-O(12)	115·2
O(5)—B(2)-O(6)	108·7		
O(5)—B(2)-O(7)	109·5	B(1)-O(1)—B(5)	123·9
O(6)—B(2)-O(7)	106·0	B(1)-O(2)—B(7)	125·3
O(8)—B(3)-O(9)	109·5	B(1)-O(3)—B(6)	119·2
O(8)—B(3)-O(10)	104·0	B(1)-O(4)—B(2)	117·0
O(8)—B(3)-O(11)	115·7	B(2)-O(5)—B(8)	120·2
O(9)—B(3)-O(10)	110·0	B(2)-O(6)—B(6)	120·9
O(9)—B(3)-O(11)	104·2	B(2)-O(7)—B(7)	124·3
O(10)-B(3)-O(11)	113·5	B(3)-O(8)—B(8)	130·2
O(10)-B(4)-O(13)	114·8	B(3)-O(9)—B(5)	115·5
O(10)-B(4)-O(14)	127·1	B(3)-O(10)-B(4)	113·7
O(13)-B(4)-O(14)	118·1	B(3)-O(11)-B(6)	129·6
O(1)—B(5)-O(9)	123·7	B(7)-O(12)-B(8)	118·5
O(1)—B(5)-O(13)	117·3	B(4)-O(13)-B(5)	122·9
O(9)—B(5)-O(13)	119·0		

attention is called to the boron–oxygen bond between B(4) and O(14). This bond, involving the non-bridging oxygen O(14) is quite short, 1·295 Å. However, a comparable short bond length of 1·280 Å to the non-bridging oxygen in sodium metaborate has been found by Marezio, Plettinger & Zachariasen (1963). The other two boron–oxygen bonds involving B(4) (especially the B(4)–O(13) bond of 1·453 Å) are comparatively long. This effect is apparently induced by the short B(4)–O(14) bond.

With the exception of Na(2), which is coordinated by seven oxygen atoms in the range 2·297 to 2·872 Å, the sodium ions are coordinated by six oxygen atoms in the range 2·260 to 2·809 Å. If sodium–oxygen distances above 3 Å are not counted in the coordination sphere, the coordination number six in the present case is the same as that observed in β-$Na_2O.3B_2O_3$ (Krogh-Moe, 1972*b*). Bond distances in these two compounds are also similar. The rather broad distribution in the sodium–oxygen bond distances is presumably typical of the borate network structures.

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References

Block, S. & Perloff, A. (1965). *Acta Cryst.* **19**, 297–300.

Cruickshank, D. W. J. (1965). *Acta Cryst.* **9**, 747–753.

Ecker, E. (1966). Thesis. Technische Hochschule, Karlsruhe, Germany.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Ihara, M. & Krogh-Moe, J. (1966). *Acta Cryst.* **20**, 132–134.

International Tables for X-ray Crystallography. (1962). Vol. III. Birmingham: Kynoch Press.

Jenkel, E. (1936). *Z. anorg. allgem. Chem.* **227**, 214–220.

Krogh-Moe, J. (1960). *Acta Cryst.* **13**, 889–892.

Krogh-Moe, J. (1968). *Acta Cryst.* B**24**, 179–181.

Krogh-Moe, J. (1972*a*). *Acta Cryst.* B**28**, 3089–3093.

Krogh-Moe, J. (1972*b*). *Acta Cryst.* B**28**, 1571–1576.

Marezio, M., Plettinger, H. A. & Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 594–595.

Milman, T. & Bouaziz, R. (1968). *Ann. Chim. Paris*, **3**, 311–321.

Stewart, J. M., Kruger, G. J., Kundell, F. A. & Baldwin, J. C. (1971). The X-RAY System, Computer Science Center, Univ. of Maryland, Maryland.

Vila, M. & Font-Altaba, M. (1955). *Publ. Dep. Crist. Min.* (*Barcelona*), **2**, 193–200.