nucleoside residue in the axial position on it. The principal difference, apart from the base, lies in the disposition of the tetrahydropyranyl ring with respect to the O(2′)–C(2″) bond. This amounts to an interchange of the C(3″) and O(1″) atoms and a rotation of 90° about C(2″) → O(2′). We may assume that the tetrahydropyranyl ring in the more laevorotatory isomer of ThPU will have a similar disposition to that in ThPA(−) so that ThPU(−) would have the molecular structure shown in Fig. 6. A comparison of the pictures of the two isomers (Fig. 6) shows that ThPU(+) can form an internal hydrogen bond [O(3′)–H(O3′)–O(1″)] with very little distortion of the molecule, the distance O(3′)–O(1″) in the crystal being only 3·4 Å. By contrast, in ThPU(−), a large rotation would be necessary to bring O(3′) and O(1″) within hydrogen-bonding distance, and such rotation would lead to unacceptably short atomic contact distances, especially between H(12″) and H(11′) or O(3′). Thus in a non-polar solvent, ThPU(+) is able to present a less polar exterior than ThPU(−) with the resultant observed differences in their physical properties.

We wish to thank Mr K. V. Deugau for crystallizing the compound and the National Research Council of Canada for financial support.

References

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Green, E. A., Shiono, R. & Rosenstein, R. D. (1971). *Chem. Commun.* pp. 53–54.

Griffin, B. E., Jarman, M. & Reese, C. B. (1968). *Tetrahedron*, **24**, 639–662.

Hawkinson, S. W. & Coulter, C. L. (1971). *Acta Cryst.* B**27**, 34–42.

Hoog, A. J. de, Buys, H. R., Altona, C. & Havinga, E. (1969). *Tetrahedron*, **25**, 3365–3375.

Iball, J., Morgan, C. H. & Wilson, H. R. (1966). *Proc. Roy. Soc.* A**295**. 320–333.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.

Karle, J. & Karle, I. L. (1966). *Acta Cryst.* **21**, 849–859.

Kennard, O., Motherwell, W. D. S., Coppola, J. C. Griffin, B. E., Reese, C. B. & Larson, A. C. (1971). *J. Chem. Soc.* (*B*), pp. 1940–1946.

Klyne, W., Mose, W. P., Scopes, P. M., Holder, G. M. & Whalley, W. B. (1967). *J. Chem. Soc.* (*C*), pp. 1273–1276.

Lin, G. H.-Y. & Sundaralingam, M. (1971). *Acta Cryst.* B**27**, 961–969.

Rahman, A. & Wilson, H. R. (1972). *Acta Cryst.* B**28**, 2260–2270.

Saenger, W. & Scheit, K. H. (1970). *J. Mol. Biol.* **50**, 153–169.

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

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Stothart, P. H. (1973). M. Sc. Thesis, McMaster Univ.

Sundaralingam, M. (1969). *Biopolymers*, **7**, 821–860.

Sundaralingam, M. & Jensen, L. H. (1965). *J. Mol. Biol.* **13**, 930–943.

Acta Cryst. (1973). B**29**, 2242

The Crystal Structure of Hexalead Pentaborate, 6PbO.5B$_2$O$_3$

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(*Received* 2 *April* 1973; *accepted* 30 *May* 1973)

Hexalead pentaborate, 6PbO.5B$_2$O$_3$, is triclinic with the following unit-cell dimensions at 22°C: $a = 6{\cdot}833 \pm 0{\cdot}002$, $b = 7{\cdot}010 \pm 0{\cdot}002$, $c = 11{\cdot}125 \pm 0{\cdot}002$ Å, $\alpha = 85{\cdot}89 \pm 0{\cdot}03$, $\beta = 102{\cdot}00 \pm 0{\cdot}02$, $\gamma = 119{\cdot}56 \pm 0{\cdot}02°$. The space group is $P\bar{1}$ with one formula unit in the cell and a calculated density of 6·183 g cm^{-3}. Three-dimensional X-ray intensity data were measured with a Picker automatic single-crystal diffractometer and Mo $K\alpha$ radiation. The structure was determined by the heavy-atom method. A full-matrix least-squares refinement resulted in an R value of 0·065. The structure contains isolated $B_{10}O_{21}^{12-}$ polyions. These ions are built up from two diborate groups linked by two BO$_3$ triangles. The lead atoms are covalently bonded to oxygen, as evidenced by the short lead–oxygen distances (2·23 Å and upwards) as well as the asymmetric coordination of the lead atoms.

Introduction

The system lead oxide–boron oxide exhibits two phases of unusual stoichiometry. Geller & Bunting (1937) reported the existence of 1:2, 5:4, 2:1 and 4:1 phases in this system. Bauer & Plötscher (1967) claimed to have found in addition 1:1 and 9:8 phases Crystalline compounds with the 5:4 and 9:8 stoichiometric ratios of metal oxide to boron oxide have not been found for other borate systems. The present investigation was undertaken to study the nature of the phase which Bauer & Plötscher believed to be a 9PbO.8B$_2$O$_3$ com-

Table 1. *Final observed and calculated structure factors*

The columns are l, $10F_o$ and $10F_c$ respectively.

-9,2,L		
6	299	224
5	1451	1503
4	604	673
2	143	180
1	516	570
-9,3,L		
-1	274	99
0	963	968
1	498	491
2	960	896
3	727	829
4	554	629
5	162	126
6	342	243
7	948	965
-9,4,L		
8	426	391
7	440	448
6	749	744
5	874	829
4	947	986
3	772	728
1	1377	1420
0	324	112
-1	554	544
-2	394	365
-9,5,L		
-2	566	612
-1	529	591
0	531	569
1	134	37
2	590	489
3	218	109
4	1344	1380
5	256	261
6	374	327
7	1323	1315
8	624	627
-9,6,L		
7	1107	1158
5	326	290
4	475	445
3	510	439
2	945	925
1	974	923
0	642	651
-1	1282	1337
-9,7,L		
0	306	206
1	289	252
2	864	915
3	628	660
4	1126	1130
5	1119	1083
6	313	454
-8,8,L		
-2	475	413
-1	678	600
0	619	625
1	361	426
2	450	315
3	1275	1253
4	872	889
5	354	210
6	294	314
7	1183	1224
-8,7,L		
9	228	165
8	693	702
7	287	14
6	536	565
5	482	465
4	1297	1290
3	216	102
2	283	251
1	1104	1134
0	1163	1148
-2	283	139
-3	741	759
-4	775	656
-8,6,L		
-4	406	358
-3	922	928
-2	1164	1213
-1	677	621
1	1108	1121
2	554	476
3	476	480
4	733	669
5	611	533
6	261	264
7	1534	1506
8	455	324
9	891	891
10	773	786
-8,5,L		
11	298	251
10	468	473
9	304	313
8	281	195
7	501	534
6	757	735
5	421	392
4	1876	1801
3	387	360
2	294	300
1	1181	1102
-1	201	242
-2	791	844
-4	586	569
-5	1174	1216
-8,4,L		
-5	316	293
-3	159	123
-2	1536	1535
-1	133	78
0	147	182
1	1772	1784
2	595	512
3	253	259
5	317	155
6	894	927
7	923	880
8	582	534
9	1544	1580
10	513	427
11	583	558
-8,3,L		
11	533	504
10	560	556
9	328	222
8	853	878
7	251	157
6	1353	1371
5	709	710
4	1177	1120
3	1005	995
2	258	219
1	378	339
0	230	204
-1	542	574
-2	1300	1366
-3	221	36
-4	218	59
-5	1547	1608
-8,2,L		
-4	641	650
-3	517	489
-2	652	709
-1	70	28
0	824	828
1	1119	1173
2	989	1012
3	869	876
4	208	71
5	1090	1153
7	538	492
8	436	466
9	1228	1300
10	367	343
-8,1,L		
9	607	631
8	558	662
7	569	577
6	1204	1243
5	24	58
4	385	414
3	446	479
2	1148	1267
1	147	75
0	335	362
-1	1290	1350
-2	518	964
-3	597	550
-8,0,L		
-1	837	874
0	530	541
1	272	306
2	676	677
3	907	982
4	597	703
5	1001	1104
6	327	401
7	1025	1134
-7,-2,L		
6	632	667
5	289	378
4	1350	1503
2	585	659
1	469	405
0	623	617
-7,-1,L		
-4	228	84
-3	1311	1392
-2	677	666
-1	241	259
0	435	455
1	1199	1226
2	543	600
3	647	712
4	102	22
5	953	986
6	335	366
7	1134	1190
8	625	646
9	589	699
-7,0,L		
11	800	870
10	626	627
9	541	527
8	1054	1140
7	222	329
6	636	594
5	456	424
4	1272	1266
3	867	867
2	280	307
1	684	682
0	1741	1787
-1	266	234
-2	413	373
-3	547	516
-4	497	515
-5	227	112
-7,1,L		
-6	914	993
-5	852	893
-4	604	642
-3	1507	1559
-2	173	85
-1	405	384
0	163	45
1	304	302
2	283	363
3	1889	1929
4	533	483
5	621	635
6	1281	1336
7	1023	1037
8	255	52
9	366	407
10	345	395
11	1000	1083
12	532	507
-7,2,L		
12	168	62
11	598	608
10	751	725
9	1356	1394
8	1003	1019
7	359	302
6	1526	1515
5	339	280
4	234	173
3	623	603
2	148	107
1	232	309
0	2145	2213
-1	766	725
-2	825	824
-3	1030	1053
-4	352	365
-6	432	402
-7	139	206
-7,3,L		
-8	1018	1122
-7	583	572
-6	458	424
-5	1412	1504
-4	119	252
-3	755	751
-2	424	465
-1	548	569
0	883	837
1	527	539
2	759	732
3	2264	2290
4	548	506
6	1251	1234
7	98	32
8	168	57
9	947	975
10	423	411
11	991	1042
12	1279	1363
-7,4,L		
12	335	322
11	192	191
10	187	187
9	1485	1495
8	308	297
7	159	142
6	2081	2085
5	686	644
4	304	107
3	379	312
2	566	557
1	798	825
0	1203	1223
-1	529	563
-2	1433	1523
-3	457	458
-4	970	970
-5	720	792
-6	358	410
-7	621	603
-8	608	667
-7,5,L		
-7	151	31
-5	775	802
-4	630	626
-3	661	663
-2	421	408
-1	1351	1346
0	406	354
1	1104	1115
2	933	905
3	1299	1241
4	1030	1021
5	514	514
6	233	123
7	557	505
8	852	829
9	1568	1560
10	309	243
11	131	262
12	1388	1432
-7,6,L		
11	791	786
10	642	629
9	478	479
8	385	407
7	704	664
6	1122	1134
5	1345	1342
4	574	565
3	229	200
2	1662	1592
1	304	105
0	644	617
-1	374	269
-2	1059	1055
-3	339	351
-4	593	569
-5	866	943
-6	1135	1188
-7	555	491
-7,7,L		
-6	332	342
-4	607	606
-3	1248	1264
-2	747	754
-1	1123	1078
0	677	651
1	911	870
2	233	70
3	402	405
4	384	330
5	1424	1448
6	77	87
8	1530	1551
9	882	872
10	68	256
-7,8,L		
8	1002	1018
7	394	304
6	398	287
5	580	623
4	599	536
3	744	758
2	1304	1309
1	516	497
0	1106	1110
-1	558	575
-2	136	55
-7,9,L		
-2	235	81
-1	422	351
0	421	455
1	315	166
2	411	415
3	666	724
4	442	417
5	1302	1334
6	740	717
-6,9,L		
7	473	489
6	329	358
5	1320	1414
4	203	102
3	160	58
2	1194	1200
1	1025	1043
0	195	218
-1	199	135
-2	652	637
-3	844	756
-6,8,L		
-6	451	469
-5	751	730
-4	169	119
-3	230	222
-2	939	920
-1	1540	1501
0	423	356
1	70	118
2	1263	1314
3	372	453
4	365	416
5	649	615
6	631	620
7	573	522
8	1253	1296
9	484	551
-6,7,L		
11	511	489
10	427	448
9	214	206
8	420	454
7	806	799
6	613	617
5	1768	1740
4	761	731
3	419	391
2	1055	1076
1	166	101
0	115	194
-1	965	942
-3	430	450
-4	1659	1597
-5	716	729
-6	610	647
-7	411	409
-6,6,L		
-8	669	697
-7	1277	1269
-6	706	664
-5	248	235
-4	289	259
-1	1706	1763
0	115	247
1	522	502
2	2088	2068
3	862	804
4	293	309
5	318	36
6	371	346
7	1027	1079
8	799	821
9	327	351
10	1714	1771
11	324	112
12	542	530
-6,5,L		
13	1112	1154
12	500	470
11	725	744
10	348	212
9	984	947
8	271	121
7	1766	1753
6	615	577
5	1211	1190
4	1204	1175
3	626	567
2	859	740
0	821	820
-1	1660	1665
-2	251	214
-3	219	221
-4	1927	1989
-5	185	60
-6	440	416
-7	234	145
-8	304	288
-9	441	341
-6,4,L		
-9	542	549
-8	10	23
-7	1618	1722
-6	278	276
-5	94	170
-4	1177	1217
-3	1059	1035
-2	520	514
-1	1101	1100
0	330	274
1	1393	1387
2	1345	1290
3	1332	1285
4	1582	1507
5	470	494
6	1384	1340
7	249	285
8	781	791
9	598	625
10	1312	1313
11	564	596
12	207	181
13	777	776
-6,3,L		
14	915	393
13	421	400
12	530	485
11	1559	1590
10	610	605
9	438	405
8	1018	940
7	1407	1378
6	287	145
5	772	698
4	591	528
3	1587	1520
2	562	538
1	896	820
0	1747	1717
-1	979	992
-2	1222	1233
-3	309	151
-4	1049	1073
-5	627	650
-6	980	984
-7	607	644
-8	431	469
-9	355	393
-6,2,L		
-9	693	683
-8	514	470
-7	927	980
-6	537	498
-5	471	423
-4	800	709
-3	1821	1806
-2	185	185
-1	733	678
0	1185	1217
1	583	549
2	322	240
3	522	495
4	1373	1281
5	1498	1431
6	1179	1078
7	560	501
8	1590	1563
9	520	556
10	318	219
11	152	63
12	298	298
-6,1,L		
13	380	430
12	147	283
11	1835	1910
8	642	608
6	351	291
5	1468	1497
4	650	653
3	1501	1415
2	1677	1644
1	871	862
0	962	939
-1	279	279
-2	332	313
-3	850	843
-4	458	455
-6	1680	1769
-7	542	495
-8	133	89
-9	801	893
-6,0,L		
-7	218	115
-6	176	259
-5	313	209
-4	385	353
-3	1592	1657
-2	807	790
-1	1321	1309
0	1443	1470
1	616	676
2	803	767
4	299	155
5	1316	1346
6	756	292
7	88	12
8	2171	2260
9	250	370
10	80	82
11	635	667
12	554	470
-6,-1,L		
11	1094	1126
10	670	758
9	619	610
8	295	187
7	244	242
6	606	681
5	1853	1983
4	279	242
3	398	443
2	1486	1595
0	462	470
-2	700	761
-3	1046	1111
-4	895	922
-5	643	659
-6	1571	1650
-7	321	395
-6,-2,L		
-5	776	797
-4	330	256
-3	639	641
-2	63	127
-1	1469	1629
0	373	370
1	630	674
2	1326	1428
3	699	731
4	690	670
5	252	272
6	421	449
7	797	808
8	1134	1278
9	704	755
10	542	698
-6,-3,L		
7	353	233
6	1073	1126
5	1184	1243
4	441	506
2	415	508
1	778	857
0	762	774
-1	841	893
-2	926	1011
-5,-4,L		
8	158	125
7	1195	1273
6	409	465
5	636	608
3	867	965
2	256	63
1	978	1037
0	728	767
-1	1270	1307
-2	713	815
-5,-3,L		
-5	61	51
-4	1536	1571
-3	402	406
-1	199	127
0	301	39
1	891	910
2	1424	1438
3	715	753
4	1435	1512
5	427	447
6	760	803
7	343	343
8	488	424
9	484	439
10	1137	1228
-5,-2,L		
12	387	309
11	364	317
10	899	929
9	470	437
8	501	561
7	1703	1778
6	556	520
5	962	952
4	785	768
2	842	557
1	605	609
0	472	485
-1	2098	2177
-2	280	198
-3	305	279
-4	1360	1423
-5	448	389
-6	204	129
-7	475	548
-8	158	135
-5,-1,L		
-9	739	793
-8	952	985
-7	1178	1199
-6	715	715
-5	75	68
-4	1099	1091
-3	864	534
-2	437	633
-1	1114	1138
0	628	628
1	171	57
2	2345	2439
3	238	211
4	1043	1015
5	477	926
6	346	254
8	509	493
9	491	507
10	1714	1757
11	524	480
12	224	151
13	1081	1183
-5,0,L		
14	304	330
13	1378	1405
12	604	582
11	199	90
10	286	316
9	348	328
8	1236	1247
7	1362	1294
6	472	444
5	2190	2174
4	242	56
3	1380	1332
2	697	627
1	938	901
0	897	862
-1	1748	1756
-2	381	315
-3	770	696
-4	1555	1567
-5	1732	1719
-6	377	319
-8	925	950
-9	732	737
-10	536	551
-5,1,L		
-11	816	895
-10	544	561
-9	1060	1149
-8	754	761
-7	721	741
-6	917	934
-5	498	920
-4	329	275
-3	336	234
-2	2113	2199
-1	1486	1473
0	291	205
1	701	647
2	2143	2117
3	474	682
4	1032	954
5	360	255
6	1666	1624
8	1687	1657
9	1118	1109
10	1327	1337
11	1202	1238
12	266	225
13	425	603
14	438	469
-5,2,L		
14	862	815
13	987	990
12	1163	1164
11	760	760
10	425	380
9	1382	1331
8	426	604
7	1402	940
6	279	226
5	1997	1923
4	1140	1946
3	772	707
2	1261	1202
1	2336	2314
0	582	544
-1	427	783
-2	492	478
-3	910	914
-4	474	492
-5	1346	1403
-6	1141	1165
-7	847	820
-8	1308	1344
-9	353	252
-10	673	673
-11	355	301
-5,3,L		
-11	175	294
-10	1039	1061
-9	340	381
-8	150	54
-7	485	437
-6	228	206
-5	1513	1615
-4	808	765
-3	930	991
-2	2105	2263
-1	197	48
0	912	822
1	237	173
2	478	469
3	933	848
4	2196	2101
5	950	878
6	1184	1130
7	1718	1693
8	1412	1570
9	275	262
10	484	472
11	694	634
12	1049	1031
13	432	383
-5,4,L		
14	596	578
12	852	834
11	967	962
10	1325	1308
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0	831	833
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-2	1687	1671
-3	2539	2501
-4	[illegible]	[illegible]
-5	[illegible]	313
-6	[illegible]	[illegible]
-7	[illegible]	[illegible]
-8	[illegible]	[illegible]
-9	[illegible]	[illegible]
-10	[illegible]	[illegible]
-11	[illegible]	[illegible]
-12	1451	[illegible]
-13	[illegible]	[illegible]
-14	[illegible]	[illegible]
0,-4,L		
-13	902	[illegible]
-12	481	415
-11	692	644
-10	[illegible]	[illegible]
-9	[illegible]	[illegible]
-8	241	[illegible]
-7	241	[illegible]
-6	[illegible]	[illegible]
-5	[illegible]	[illegible]
-4	[illegible]	[illegible]
-3	[illegible]	[illegible]
-2	[illegible]	[illegible]
-1	[illegible]	[illegible]
0	[illegible]	[illegible]
1	[illegible]	[illegible]
2	[illegible]	[illegible]
3	221	152
4	[illegible]	[illegible]
5	[illegible]	[illegible]
6	[illegible]	[illegible]
7	[illegible]	[illegible]
8	81	121
9	[illegible]	[illegible]
10	312	[illegible]
11	[illegible]	[illegible]
12	[illegible]	[illegible]
13	224	[illegible]
0,-5,L		
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11	[illegible]	[illegible]
10	[illegible]	[illegible]
9	[illegible]	[illegible]
8	399	[illegible]
7	[illegible]	[illegible]
6	[illegible]	[illegible]
4	[illegible]	[illegible]
3	[illegible]	[illegible]
2	[illegible]	[illegible]
1	[illegible]	[illegible]
0	[illegible]	[illegible]
-1	[illegible]	[illegible]
-2	[illegible]	[illegible]
-3	[illegible]	[illegible]
-4	[illegible]	[illegible]
-5	[illegible]	[illegible]
-6	[illegible]	[illegible]
-7	[illegible]	[illegible]
-8	[illegible]	[illegible]
-9	[illegible]	[illegible]
-10	[illegible]	[illegible]
-11	[illegible]	[illegible]
-12	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]

pound. The result of our structure determination however, shows the suggested chemical composition to be in error. The compound actually has the formula $6PbO.5B_2O_3$. This is still a stoichiometry not previously encountered in crystalline binary borates.

Experimental

Hexalead pentaborate, $6PbO.5B_2O_3$, was obtained by devitrifying at 460–500°C a lead borate glass containing a small amount of lead oxide in excess of the metaborate composition. The glass was prepared as described by Bauer & Plötscher (1967). An X-ray powder diagram indicated the crystalline phase obtained to be identical with the phase believed by Bauer & Plötscher (1967) to be $9PbO.8B_2O_3$.

A single crystal ground to a sphere of diameter 0·013 cm, was used for the data collection. The maximum deviation from the average diameter was less than 5%. The intensities were measured with a Picker on-line single-crystal automatic diffractometer. Mo $K\alpha$ radiation was used and the X-ray reflexions were measured at 2874 independent reciprocal lattice points.

Unit-cell dimensions and standard errors, $a=6{\cdot}833 \pm 0.002$, $b=7{\cdot}010 \pm 0{\cdot}002$, $c=11{\cdot}125 \pm 0{\cdot}002$ Å, $\alpha=85{\cdot}89 \pm 0{\cdot}03$, $\beta=102{\cdot}00 \pm 0{\cdot}02$, $\gamma=119{\cdot}56 \pm 0{\cdot}02°$, 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 $K\alpha_1$). With one formula unit of $6PbO.5B_2O_3$ the calculated density is $6{\cdot}183$ g cm^{-3}. A crude experiment showed the material to have a density of $6{\cdot}5 \pm 0{\cdot}5$ g cm^{-3} in fair agreement with the calculated value.

Structure determination

The observed intensities were converted to structure factors in the usual manner. The linear absorption coefficient is 547 cm^{-1} and a correction for absorption was applied, assuming the crystal to be a perfect sphere. A three-dimensional Patterson map was used to locate the lead atoms and the remaining atoms were clearly seen in an electron-density difference map. The resulting preliminary structure indicated a centre of symmetry.

Refinement of the structure was then carried out with a full-matrix least-squares program from the X-RAY System (Stewart, Kruger, Kundell & Baldwin, 1971). All 2775 independent reflexions observed as larger than the background, were included in the refinement. The atomic scattering factors for O and B (neutral atoms), used for obtaining the calculated structure

Table 2. *Final atomic parameters*

The isotropic and anisotropic temperature-factors are of the form:

$$\exp\left[-U8\pi^2(\sin\theta/\lambda)^2\right] \text{ and } \exp\left[-2\pi^2[U_{11}(ha^*)^2+U_{22}(kb^*)^2+U_{33}(lc^*)^2+2U_{12}hka^*b^*+2U_{13}hla^*c^*+2U_{23}klb^*c^*]\right]$$

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}
Pb(1)	4831 (1)	1405 (1)	3022 (1)	51 (3)	60 (3)	92 (4)	11 (3)	15 (3)	−11 (3)
Pb(2)	−2069 (1)	7618 (1)	4096 (1)	55 (4)	61 (3)	82 (3)	13 (3)	−1 (3)	−21 (3)
Pb(3)	4886 (1)	7326 (1)	615 (1)	73 (4)	64 (3)	89 (3)	21 (3)	41 (3)	−3 (3)

Table 2 (*cont.*)

	x	y	z	U
O(1)	−1679 (31)	3211 (29)	2012 (16)	112 (32)
O(2)	−2780 (31)	−447 (29)	2354 (16)	125 (32)
O(3)	1187 (25)	2324 (23)	2944 (13)	17 (24)
O(4)	5121 (28)	4806 (25)	2888 (14)	64 (27)
O(5)	3257 (30)	5683 (27)	4196 (15)	104 (31)
O(6)	2254 (32)	7804 (30)	5329 (17)	146 (34)
O(7)	321 (27)	6627 (24)	3234 (14)	47 (26)
O(8)	2273 (28)	5786 (26)	1981 (15)	78 (28)
O(9)	−590 (31)	6795 (28)	1080 (16)	112 (31)
O(10)	3042 (29)	9347 (27)	540 (15)	93 (30)
O(11)	0	0	0	361 (79)
B(1)	2992 (45)	4694 (41)	2976 (23)	88 (43)
B(2)	90 (44)	5657 (41)	2026 (23)	80 (42)
B(3)	−1059 (51)	1629 (47)	2417 (26)	141 (50)
B(4)	1873 (39)	6661 (36)	4262 (20)	31 (36)
B(5)	940 (37)	8737 (34)	563 (19)	12 (34)

factors, were those of *International Tables for X-ray Crystallography* (1962). The atomic scattering factors for neutral Pb were obtained from Hanson, Herman, Lea & Skillman (1964) and were corrected for dispersion. The imaginary dispersion terms (Cromer, 1965) were also used in the refinement. The vibrations of boron and oxygen were assumed to be isotropic. A weighting scheme based on the statistical counting errors, compounded with errors assumed to be 1% of the observed intensity, was used. The structure was refined to an R value of 0·065 (0·081 for the weighted R value), assuming the presence of a centre of symmetry, *i.e.* space group $P\bar{1}$. The ratio of maximum shift to standard deviation during the last cycle was 0·08. (An attempt was made to refine the structure without the restriction of a centre of symmetry, but the refinement did not converge properly.) Observed and calculated structure factors are given in Table 1. The final atomic coordinates and thermal parameters are given in Table 2.

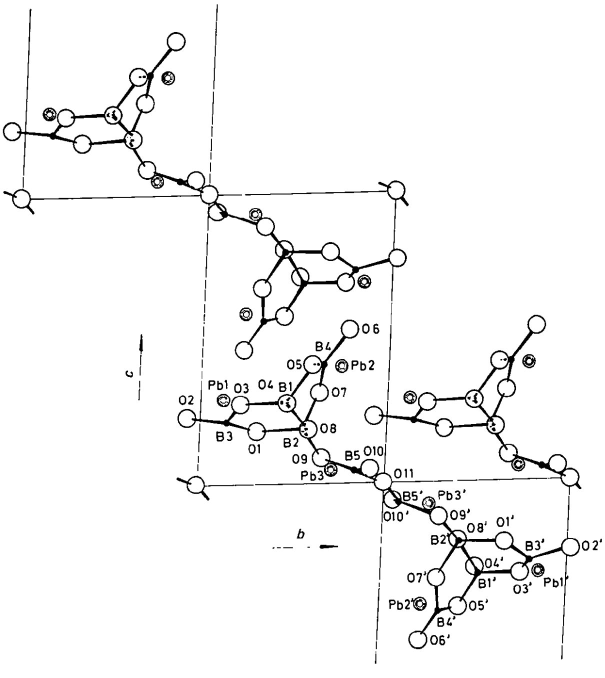

Fig. 1. View of the structure of $6PbO.5B_2O_3$ along the a axis. The concentric circles represent lead, the small filled circles boron and the remaining circles oxygen. The atom numbering corresponds to the numbering used in the text.

Discussion of the structure

The anion structure of hexalead pentaborate consists of $B_{10}O_{21}^{12-}$ isolated units (Figs. 1 and 2). This new anion type is the largest isolated borate anion found so far in an anhydrous borate. The anion may be described as two diborate groups connected to each other by a chain section consisting of two BO_3-triangles. The diborate group was first found by Morimoto (1956) in the mineral borax and has since been found in several borates, both anhydrous and hydrated. Each diborate subunit in the hexalead pentaborate anion is terminated by three oxygen atoms bonded only to one boron, not counting bonds to the lead atoms. Isolated diborate groups have previously only been found with hydroxyl units as terminal atoms. Boron–oxygen bond lengths and bond angles are given Table 3. The values are normal within the error limits. As usual the longest boron–oxygen distances are found for the tetrahedrally coordinated boron atoms.

The lead coordination has noteworthy features. The three crystallographically different lead atoms are all coordinated by three or four oxygens at a short distance (2·23–2·55 Å) and oxygens at longer distances, upwards of 2·76 Å. All the short lead–oxygen distances of a given lead atom fall within the same hemisphere around lead. Moreover the short lead–oxygen distances, with two exceptions [O(8) and O(7)], involve terminal oxygen atoms, *i.e.* oxygens bonded to only one boron. One exception is the lead–oxygen bond to the bridging O(8) atom. This atom, however, is engaged in the two relatively weak boron–oxygen bonds of a pair of BO_4-tetrahedra.

Table 3. *Interatomic distances and bond angles*

B(1)–O(3)	1·50 (3) Å	Pb(1)–O(6)	2·28 (2) Å
B(1)–O(4)	1·44 (4)	Pb(1)–O(4)	2·29 (2)
B(1)–O(5)	1·50 (3)	Pb(1)–O(1)	2·55 (2)
B(1)–O(8)	1·44 (3)	Pb(1)–O(2)	2·76 (3)
B(2)–O(1)	1·53 (3)	Pb(1)–O(3)	2·85 (2)
B(2)–O(7)	1·49 (3)	Pb(1)–O(10)	2·94 (2)
B(2)–O(8)	1·46 (4)	Pb(2)–O(4)	2·23 (1)
B(2)–O(9)	1·41 (3)	Pb(2)–O(2)	2·39 (2)
B(3)–O(1)	1·39 (4)	Pb(2)–O(7)	2·44 (2)
B(3)–O(2)	1·34 (3)	Pb(2)–O(5)	2·79 (2)
B(3)–O(3)	1·36 (3)	Pb(2)–O(5)	2·81 (2)
B(4)–O(5)	1·43 (4)	Pb(2)–O(6)	2·93 (2)
B(4)–O(6)	1·39 (3)	Pb(3)–O(10)	2·30 (2)
B(4)–O(7)	1·38 (3)	Pb(3)–O(2)	2·33 (2)
B(5)–O(9)	1·42 (3)	Pb(3)–O(8)	2·38 (2)
B(5)–O(10)	1·29 (3)	Pb(3)–O(10)	2·48 (2)
B(5)–O(11)	1·38 (3)	Pb(3)–O(4)	3·00 (2)
		Pb(3)–O(1)	3·19 (2)
		Pb(3)–O(9)	3·26 (1

Table 3 (*cont.*)

O(3)—B(1)-O(4)	108 (2)°	O(2)—Pb(3)-O(10′)	83 (1)°
O(3)—B(1)-O(5)	107 (2)	O(8)—Pb(3)-O(10′)	144 (1)
O(3)—B(1)-O(8)	109 (2)	O(10′)-Pb(3)-O(4)	146 (< 1)
O(4)—B(1)-O(5)	109 (2)		
O(4)—B(1)-O(8)	112 (2)	B(2)-O(1)-B(3)	122 (2)°
O(5)—B(1)-O(8)	111 (3)	B(1)-O(3)-B(3)	122 (2)
O(7)—B(2)-O(1)	107 (2)	B(1)-O(5)-B(4)	118 (2)
O(7)—B(2)-O(8)	108 (2)	B(4)-O(7)-B(2)	122 (2)
O(7)—B(2)-O(9)	108 (2)	B(1)-O(8)-B(2)	113 (2)
O(8)—B(2)-O(1)	107 (2)	B(2)-O(9)-B(5)	125 (2)
O(8)—B(2)-O(9)	113 (2)		
O(1)—B(2)-O(9)	113 (2)	O(6)—Pb(1)-O(4)	97 (1)
O(1)—B(3)-O(2)	116 (3)	O(6)—Pb(1)-O(1)	78 (1)
O(1)—B(3)-O(3)	118 (2)	O(4)—Pb(1)-O(1)	84 (1)
O(2)—B(3)-O(3)	126 (3)	O(4)—Pb(2)-O(2)	80 (1)
O(5)—B(4)-O(6)	119 (2)	O(4)—Pb(2)-O(7)	82 (< 1)
O(5)—B(4)-O(7)	120 (2)	O(2)—Pb(2)-O(7)	96 (1)
O(6)—B(4)-O(7)	121 (3)	O(10)-Pb(3)-O(2)	83 (1)
O(9)—B(5)-O(10)	126 (2)	O(10)-Pb(3)-O(8)	72 (1)
O(9)—B(5)-O(11)	115 (2)	O(10)-Pb(3)-O(10′)	73 (1)
O(10)-B(5)-O(11)	119 (2)	O(2)—Pb(3)-O(8)	81 (1)

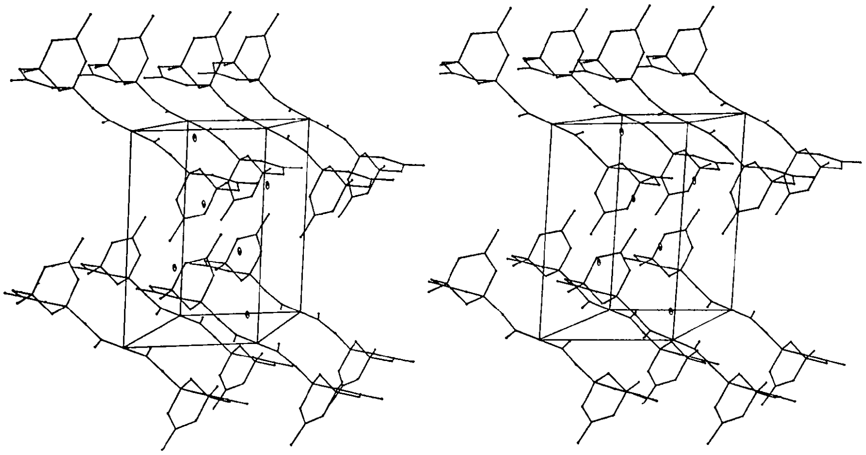

Fig. 2. A stereo pair showing the packing of the structure. The boron–oxygen bonding and the lead atoms are shown.

These characteristics point to the covalent nature of the lead–oxygen bonding. The shortest lead–oxygen distance of 2·23 Å has a value similar to the short lead–oxygen distance of 2·18 Å in lead oxide (Leciejewicz, 1961; Kay, 1961). An ionic lead–oxygen distance, in comparison, would be about 2·60 Å (Pauling, 1960). The asymmetric coordination around the lead atoms in lead oxide, caused by the two 6*s* electrons, is also retained in the borate compound studied here, emphasizing the similarity with lead oxide in the nature of the bonding.

References

Bauer, H. & Plötscher, G. (1967). *Z. anorg. allgem. Chem.* **350**, 271–280.

Cromer, D. T. (1965). *Acta Cryst.* **18**, 17–23.

Cruickshank, D. W. J. (1956). *Acta Cryst.* **9**, 747–753.

Geller, R. F. & Bunting, J. (1937). *J. Res. Natl. Bur. Stand.* **18**, 585–593.

Hanson, H. P., Herman, F., Lea, J. O. & Skillman, S. (1964). *Acta Cryst.* **17**, 1040–1044.

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

Kay, M. I. (1961). *Acta Cryst.* **14**, 80–81.

Leciejewicz, J. (1961). *Acta Cryst.* **14**, 66.

Morimoto, N. (1956). *Miner. J. Japan,* **2**, 1–18.

Pauling, L. (1960). *The Nature of the Chemical Bond,* 3rd ed., p. 518. New York: Cornell Univ. Press.

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