Acta Cryst. (1965). **18**, 1088

Least-squares refinement of the crystal structure of potassium pentaborate.

By J. KROGH-MOE*, *Svenska Silikatforskningsinstitutet, Göteborg, Sweden*

(*Received* 26 *November* 1964)

The approximate structure of anhydrous potassium pentaborate, $K_2O \cdot 5B_2O_3$, has previously been determined by Krogh-Moe (1959). Awaiting the appropriate computing facilities, the structure was not refined as far as the observed intensity material would permit. Thus the reliability index R was as high as 27% for the three-dimensional data in the 1959 report. The essential correctness of the earlier structure proposal is beyond doubt, however, since a majority of the signs were uniquely given by a study of the isomorphous replacement of potassium with rubidium. A three-dimensional Fourier synthesis based on these signs gave large peaks only at the atomic positions. A least-squares refinement of the structure is nevertheless desirable, permitting the determination of a more accurate set of bond lengths with standard deviations. The present note reports such a refinement.

As a first step in the refinement, the unit-cell dimensions were redetermined with greater precision from an X-ray powder pattern obtained with a Hägg–Guinier camera. A least-squares fit of 15 indexed powder lines was carried out. Lead nitrate was used for internal standard. The following cell dimensions were found:

$$a = 7{\cdot}418 \pm 0{\cdot}002,\ b = 11{\cdot}702 \pm 0{\cdot}004,$$
$$c = 14{\cdot}745 \pm 0{\cdot}005\ \text{Å}.$$

Space group: *Pbca*

The set of three-dimensional intensity data, observed in the earlier work, was obtained from Weissenberg exposures of the zero to fourth layer line about the *a* axis. These intensity data were extended for the present study with corresponding measurements about the *b* axis, including the fourth layer line. The experimental methods were the same as before. Average values were taken for measurements from the overlapping region of the reciprocal space. The structure factors used for the present refinement are only slightly different from those given in Table 1 of the earlier work. Structure factors for 404 points of the reciprocal lattice were used for the refinement computations.

The least-squares refinement was carried out with a program written for the Facit EDB computer by Åsbrink & Brändén. The complete set of three-dimensional data were subjected to 12 cycles of the refinement. During the last 4 cycles the reliability index R remained at 11·1%. (Only observed structure factors are included in this index).

The final list of positional parameters and isotropic temperature factors is given in Table 1. This set of parameters is not very different from the original structure proposal, which is thus substantiated. The structure consists of two separate interlocking three-dimensional boron-oxygen networks as shown in Fig. 1. The basic unit of these networks is a pentaborate group. This pentaborate group is similar to the boron-oxygen skeleton of the isolated anion found in potassium pentaborate tetrahydrate (Zachariasen & Plettinger, 1963).

Table 1. *Atomic position parameters, with standard deviations*

Values are given as fractions, multiplied by 10^4, of the unit-cell edge. The last two columns give the parameter B (together with its standard deviation) of the temperature factor $\exp\{-B(\sin\theta/\lambda)^2\}$.

Atom	x/a	$\sigma_{x/a}$	y/b	$\sigma_{y/b}$	z/c	$\sigma_{z/c}$	B(Å^2)	σ_B
K	720	7	2943	4	1207	3	2·90	0·08
O(1)	3098	17	252	9	255	7	1·49	0·22
O(2)	4364	18	2051	11	709	8	2·07	0·22
O(3)	6391	18	2120	10	1969	8	1·66	0·23
O(4)	4459	19	3670	11	1682	8	1·96	0·24
O(5)	6910	19	3256	10	653	8	2·29	0·25
O(6)	1179	19	636	12	1977	8	2·23	0·25
O(7)	4987	20	353	11	1536	8	2·07	0·23
O(8)	9045	18	164	11	879	8	2·32	0·25
B(1)	5578	37	2750	19	1271	15	2·74	0·49
B(2)	4139	32	904	15	806	12	1·02	0·32
B(3)	4770	32	4791	17	1548	13	1·59	0·39
B(4)	7045	35	4355	18	435	14	2·21	0·43
B(5)	5857	37	1034	18	2136	13	1·74	0·37

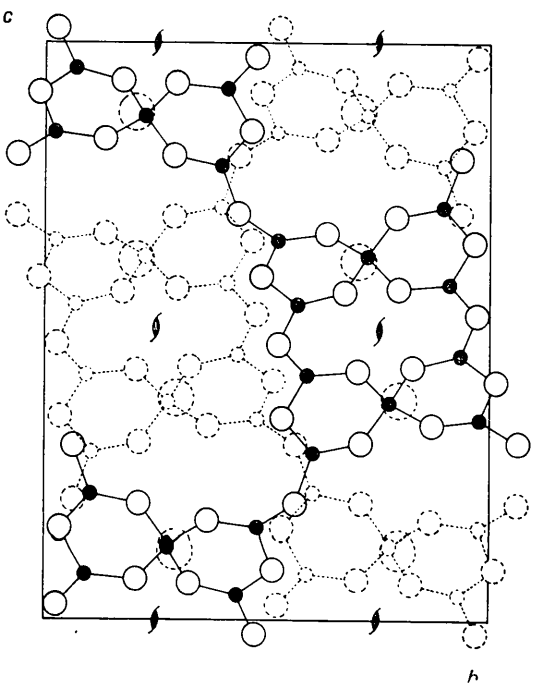

Fig. 1. A projection of the structure along the *a* axis, showing the manner in which the structural units are interlinked to a three-dimensional network. Black circles represent boron atoms, open circles represent oxygen and the large dashed circles represent the potassium atoms. The network indicated by dashed lines is a separate interlocking twin of the first network. Unit-cell dimensions are indicated by the rectangle.

* On leave to Institutt for uorganisk kjemi, NTH, Trondheim, Norway.

Table 2. *Boron–oxygen bond lengths (standard deviation ±0·03 Å) and potassium–oxygen separations shorter than 3·25 Å (standard deviation ±0·01 Å)*

Tetrahedron			
B(1)–O(2)	1·47 Å	K–O(1′)	3·17 Å
B(1)–O(3)	1·40	K–O(2)	2·99
B(1)–O(4)	1·49	K–O(2′)	3·00
B(1)–O(5)	1·47	K–(O3′)	2·90
		K–O(4)	2·98
Triangles		K–O(5)	2·96
B(2)–O(1)	1·36 Å	K–O(5′)	3·20
B(2)–O(2)	1·36	K–O(6)	2·95
B(2)–O(7)	1·40	K–O(7′)	2·91
B(3)–O(4)	1·35		
B(3)–O(6′)	1·37		
B(3)–O(8′)	1·39		
B(4)–O(1′)	1·36		
B(4)–O(5)	1·33		
B(4)–O(8′)	1·41		
B(5)–O(3)	1·35		
B(5)–O(6″)	1·41		
B(3)–O(7)	1·35		

Boron–oxygen bond lengths are given in Table 2. The standard deviation of these bond lengths is 0·03 Å. The average bond length is 1·46 Å for the tetrahedrally coordinated boron and 1·37 Å for the triangularly coordinated borons. These values agree with average values obtained recently for a number of borate structures. The individual bond lengths fall in a much closer range around the average values than they did before the refinement.

Potassium–oxygen separations shorter than 3·25 Å are also given in Table 2. It is seen that the cation has seven closest oxygen neighbours in the range from 2·90 to 3·00 Å. The coordination sphere, however, is apparently determined to a considerable extent from the demands of the boron–oxygen network.

References

KROGH-MOE, J. (1959). *Ark. Kemi*, **14**, 439.
ZACHARIASEN, W. H. & PLETTINGER, H. A. (1963). *Acta Cryst.* **16**, 376.

Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the General Secretary of the International Union of Crystallography (D. W. Smits, Rekencentrum der Rijksuniversiteit, Grote Appelstraat 11, Groningen, The Netherlands). Publication of an item in a particular issue cannot be guaranteed unless the draft is received 8 weeks before the date of publication.

International Union of Crystallography

Acta Crystallographica

Readers of the journal will have noticed the considerably larger size of the 1964 volume as compared with previous volumes, and that the total number of pages of the six parts printed in 1965 already amounts to 1090 pages, compared with 794 pages for the similar parts in 1964. This considerable increase in the size of the journal is due partly to the great efforts being made by the editorial board to reduce the delay in publication, and partly to the greater capacity of the new printing house where *Acta Crystallographica* has been produced since the April 1965 issue. It is expected that as from the current year more than 2000 pages will be printed annually. As volumes of such a size will be rather inconvenient, it has been decided that as from 1965 two semi-annual volumes should be published. The last part for the current Vol. 18 will therefore be the June 1965 issue, and the July 1965 issue will be the first part of Vol. 19.

The considerable increase in size has given rise to financial problems. The present subscription prices were set in 1963 when the size of the annual volumes was about 1300 pages, and in addition the printers' charges have increased by about 15 per cent since that time. A substantial increase in the subscription prices is therefore unavoidable.

After due consideration of the financial position of the journal, the Executive Committee has decided to postpone the change in the subscription prices until 1966, since the accumulated balance in the *Acta Crystallographica*, account is considered sufficient to meet a large deficit in 1965. The present subscribers and those who subscribe to the journal before the end of December 1965 will therefore obtain both Vols. 18 and 19 for a single subscription at the the current price of 240 Danish kroner (or 120 Danish kroner for personal subscribers). As from Vol. 20 (1966) the price of each semi-annual volume will be 200 Danish kroner (or 100 Danish kroner for personal subscribers), and these prices will then also apply for Vols. 18 and 19 purchased as back numbers. The price of single parts of the semi-annual volumes will be increased to 50 Danish kroner per part. For the earlier volumes the prices will not be changed, except for slight corrections in some dollar and sterling prices.

At the present rates of exchange the following prices will therefore be valid as from 1 January 1966:

Complete volumes, regular price per volume

Vols. 1–4 D.kr. 100 ($15 or £ 5.5.0)
Vols. 5–13 D.kr. 180 ($27 or £ 9.9.0)
Vols. 14–17 D.kr. 240 ($36 or £12.12.0)
*Vols. 18→ D.kr. 200 ($30 or £10.10.0)

Complete volumes, reduced prices for individuals

Vols. 1–4 D.kr. 60 ($ 9 or £3.3.0)
Vols. 5–13 D.kr. 100 ($15 or £5.5.0)
Vols. 14–17 D.kr. 120 ($18 or £6.6.0)
*Vols. 18→ D.kr. 100 ($15 or £5.5.0)

Single parts

Vols. 1–4 D.kr. 20 ($3 or £1.1.0)
Vol. 5 D.kr. 40 ($6 or £2.2.0)
Vols. 6–13 D.kr. 20 ($3 or £1.1.0)
Vols. 14–17 D.kr. 30 ($4.50 or £1.12.0)
*Vols. 18→ D.kr. 50 ($7.50 or £2.13.0)

No reduction for personal subscribers

* Semi-annual volumes