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The crystal structure of kaliborite

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Cristallografia. — *The crystal structure of kaliborite* (*). Nota di EGIZIO CORAZZA e CESARE SABELLI, presentata (**) dal Socio G. CAROBBI.

RIASSUNTO. — La kaliborite, $HKMg_2B_{12}O_{16}(OH)_{10} \cdot 4\,H_2O$, è monoclina, con quattro molecole nella cella unitaria avente le dimensioni $a = 18{,}53$, $b = 8{,}43$, $c = 14{,}665$ Å, $\beta = 100{,}13°$; il gruppo spaziale è il $C\,2/c$. La struttura è stata determinata partendo da dati ottenuti col metodo Weissenberg equiinclinazione, radiazione CuK_α, sia sfruttando metodi diretti che la funzione di Patterson tridimensionale. Le coordinate atomiche ed i parametri termici di tutti gli atomi fuorché gli idrogeni furono raffinati per mezzo di alcuni cicli di minimi quadrati; le posizioni approssimate degli idrogeni furono ottenute per mezzo di una Fourier delle differenze tridimensionale. L'indice R finale è 0,095.

La struttura cristallina della kaliborite può essere descritta come una serie di catene indipendenti, parallele all'asse y. Ciascun elemento della catena è un polione $[B_6O_8(OH)_5]_n^{-3n}$ formato da tre triangoli e tre tetraedri, disposti in un doppio anello con un'appendice. Gli ottaedri regolari del Mg ed i cubi schiacciati del K, legati ai poliedri del Mg per uno spigolo, riempiono i vuoti fra le catene.

Molto probabilmente uno dei ponti idrogeno è centrato ($O \cdots H \cdots O = 2{,}41$ Å).

INTRODUCTION.

Kaliborite is a potassium–magnesium borate hydrate. The chemical formula of this mineral, as resulted from a crystal–chemical point of view in the course of this investigation, is $HK\,Mg_2B_{12}O_{16}(OH)_{10} \cdot 4\,H_2O$.

In a previous paper [1] we discussed the results obtained by other authors. Pardillo Vaquer [2] assigned to the mineral the formula $KMg_2B_{11}O_9 \cdot 15\,H_2O$. This formula is different from the best known one, usually referred to by many authors, only on account of the number of water molecules; indeed Van't Hoff [3] on the basis of his chemical analysis reported the formula $KMg_2B_{11}O_{19} \cdot 9\,H_2O$.

Recalculating the oxide percentages *a posteriori* from the formula found by us, one may note the good agreement with Van't Hoff's old analysis. Pardillo Vaquer raised the number of the water molecules from 9 to 15 in order to obtain a greater molecular weight so that, keeping 4 the number of molecules in the unit cell, it might fit the volume resulting from the lattice constants found by him:

$$a = 19.00 \quad , \quad b = 8.62 \quad , \quad c = 15.00\ \text{Å} \quad , \quad \beta = 99°54'$$

(*) This work was performed in the Sezione di Firenze del Centro Nazionale di Cristallografia del C.N.R., Istituto di Mineralogia di Firenze, and the complete paper was presented at the 22nd Congress of Società Mineralogica Italiana, Forte dei Marmi, Lucca, Italy, September 28 – October 1, 1965.

(**) Nella seduta del 10 dicembre 1966.

In our previous work [1] we have reported the diffractometric data, which allowed us to determine and refine the lattice constants. Moreover, to decide on the presence or absence of a centre of symmetry, we used the statistical test of Howells, Phillips and Rogers [4] which did not allow us to make any direct deduction about the presence of a centre. As a further check, a test of piezoelectricity was carried out, but with negative results; the structure therefore was initially assumed to be centrosymmetric, space group $C\,2/c$. Further work showed no evidence of any departure from centrosymmetry.

Experimental.

The specimen used for the present investigation comes from Sallent (Barcelona). A regularly shaped crystal, suitable for X–ray analysis, approximately 0.6 mm × 0.3 mm × 0.4 mm was chosen, and because of its round shape it was considered as spherical (radius 0.215 mm) for the absorption correction.

Crystal data

$$(\lambda\,(\mathrm{Cu}K_\alpha) = 1.5418\ \text{Å})$$

Chemical formula: $HKMg_2B_{12}O_{16}(OH)_{10}\cdot 4\,H_2O$
Monoclinic,

$$a = 18.53 \pm 0.03\ , \quad b = 8.43 \pm 0.02\ , \quad c = 14.665 \pm 0.007\ \text{Å},$$

$$\beta = 100.13^\circ \pm 0.12^\circ\ , \quad U = 2255\ \text{Å}^3$$

$$D_m = 2.116\ \text{g}\cdot\text{cm}^{-3}\ , \quad Z = 4\ , \quad D_x = 2.110\ \text{g}\cdot\text{cm}^{-3}$$

$$F\,(000) = 1448\,.$$

Space group: $C\,2/c$
Absorption coefficient for X–rays:

$$\mu = 39.14\ \text{cm}^{-1}\ , \quad \mu R = 0.86$$

Density of 2.116 $\text{g}\cdot\text{cm}^{-3}$ was measured by the pycnometer method by Pardillo Vaquer.

Intensity data were collected at room temperature on an equi-inclination Weissenberg integrating camera around the b axis, the multiple-film technique being used; layer lines zero through six were recorded. On the total of 1950 independent reflections recorded, 439 were below the observational limit. Intensities were measured by the aid of a microdensitometer. Lorentz-polarization and absorption corrections were applied, and the relative structure factors derived. The F_o^2's were converted to a common absolute scale by Wilson's statistical method.

STRUCTURE DETERMINATION.

Since the general position in $C\,2/c$ is eightfold, the K atoms must lie in special positions, namely symmetry centres or twofold axes. A three-dimensional Patterson synthesis was computed with the actually observed reflections; because in this synthesis the peaks in positions 1/2 0 1/2 and 1/2 1/2 1/2 are feeble, it is unlikely that K atoms lie on symmetry centres. Therefore the K atoms were located on twofold axes, so that the potassium–potassium vectors were attributable to the highest peak of the series of unresolved maxima in position $1/2\ y\ 1/2$. Potassium–magnesium and magnesium–magnesium vector peaks were also recognized in this synthesis. Some feeble peaks surrounding the Mg—Mg and K—Mg maxima were assigned to the six oxygens of Mg octahedron. In spite of the presence of many other weak peaks, no further information was obtained from the interpretation of the Patterson.

At the same time a statistical test for attributing the signs to the structure factors, according to the method of Cochran and Woolfson [5] was carried out. The sign was so given to nearly the 13% of the recorded reflections. A three-dimensional Fourier synthesis, obtained using the statistical signs, showed a great number of maxima, two of which were prominent for their intensities. They were attributed to the K atom on the twofold axis and to the Mg atom in general position; surrounding the latter, six more maxima in octahedral position were outstanding at distances characteristic of oxygens in coordination with the Mg atom.

The agreement between the locations of eight atoms (Mg octahedron and K atom) obtained from statistical signs and the ones attained by the Patterson synthesis interpretation, is very remarkable. On the basis of the contribution of K, Mg and six O, which reaches only 20% of the total of electrons, a three-dimensional Fourier synthesis was computed. At this stage the reliability index R was 0.57. By means of three subsequent three-dimensional electron-density maps, three more oxygens, then five oxygens and finally the remaining oxygen and the boron atoms could be located. The R index dropped to 0.15.

Scattering factors were calculated assuming that K and Mg atoms are ionized and the six oxygens of Mg octahedron have a charge of —1/2. The scattering factor curve of $O^{-1/2}$ was constructed by interpolation between O and O^{-1} as reported in the International Tables [6]. For the remaining atoms the values given by Cromer and Waber [7] were used.

REFINEMENT.

The refinement of the structure was carried out by the least-squares method using all 1511 observed reflections, assigning isotropic temperature factors of 1 Å^2 for Mg and B atoms, and 1.5 Å^2 for K and O atoms. The

program, for the I.B.M. 1620 computer, applies the following weighting scheme, according to Cruickshank et al. [8]:

$$w\,(h\,k\,l) = 1/(a + F_o + c\,F_o^2)$$

where a is twice the minimum observed F_o and c is twice the reciprocal of the maximum observed F_o. The structure-factor least-squares calculations were carried out using a block-diagonal approximation to the least-squares matrix which refined atomic coordinates and thermal parameters.

Several rounds of least-squares were completed. In the later stages of refinement the correction for the secondary extinction was introduced according to Hall and Maslen's [9] formula:

$$F_o^2 = F_{o1}^2 \exp(\varepsilon \cdot I_c)$$

in which F_o and F_{o1} are respectively the corrected and uncorrected observed structure factors, and I_c is the calculated intensity. The coefficient of secondary extinction, ε, is 1.99×10^{-6}.

Towards the end of the refinement the hydrogen atoms were included in the calculation of the structure factors and they were given a temperature factor of 5.5 Å^2, but their parameters were not refined.

The final R value was 0.095 for all the observed reflections and 0.11 including the non-observed reflections, which were given an intensity one half the minimum observable in each level.

The parameters as resulting from the last cycle of refinement are given in Table I. The values of the observed and calculated structure factors are given in Table II.

New formula determination.

In the last electron-density map boron atoms were attributed to the residual peaks in such positions as to be at the centre of triangularly or tetrahedrally grouped oxygens, at distances proper for boron-oxygen bonds. During this attribution we established that in the asymmetric unit there were six boron atoms, all in general positions, instead of five in general positions and one in special position, as Van't Hoff's formula showed. Moreover, the oxygen atoms were found to be 15 instead of 14.

The unexpected presence of these two atoms caused a break in the electrostatic balance of charges. Since neither K nor Mg atoms could be subjected to numerical variations, the half negative charge, due to the increase of one oxygen and half a boron, had to be balanced necessarily by a hydrogen atom in special position. It was not conceivable that the new equilibrium was obtained by the contribution of a different cation, because there was no peak (however little) in the last Fourier map, but chiefly because the voids around the centres of symmetry and around twofold axes are too small to receive any cation other than hydrogen.

TABLE I.

Atomic coordinates and isotropic temperature parameters with their standard deviations.

ATOM	x	$10^3\,\sigma(x)$	y	$10^3\,\sigma(y)$	z	$10^3\,\sigma(z)$	B (Å^2)	σ (B)
K	.0000	.0	.1185	.3	.2500	.0	2.15	.04
Mg	.1382	.1	.4235	.3	.1671	.1	1.14	.03
O_1	.0134	.2	.7312	.7	.0241	.3	1.35	.07
O_2	.0361	.2	.7349	.6	.1891	.3	.94	.07
O_3	.1170	.2	.8729	.6	.1055	.3	1.12	.07
O_4	.3765	.2	.4001	.7	.2265	.3	1.04	.07
O_5	.1656	.2	.6617	.7	.2083	.3	1.17	.07
O_6	.2944	.2	.6093	.7	.2377	.3	1.69	.08
O_7	.2461	.2	.3453	.7	.2104	.3	.98	.07
O_8	.2989	.2	.4859	.7	.0915	.3	1.26	.07
O_9(OH)	.4217	.2	.1359	.7	.1070	.3	1.23	.07
O_{10}(OH)	.4276	.3	.5920	.8	.0567	.3	1.98	.09
O_{11}(OH)	.0325	.2	.4884	.7	.1124	.3	1.37	.07
O_{12}(OH)	.2554	.3	.9587	.8	.0642	.3	2.06	.09
O_{13}(OH)	.1709	.2	.4626	.7	.0409	.3	1.51	.08
$O_{14}(H_2O)$	.0985	.3	.2052	.8	.1205	.3	2.00	.09
$O_{15}(H_2O)$	.3893	.3	.8760	.7	.2045	.3	1.81	.08
B_1	.5003	.3	.1490	1.1	.1079	.4	.97	.10
B_2	.0688	.3	.8387	1.2	.0261	.4	1.25	.11
B_3	.1105	.3	.7950	1.1	.1960	.4	.97	.10
B_4	.2629	.3	.2013	1.0	.2551	.5	1.13	.11
B_5	.3050	.3	.4598	1.1	.1929	.4	1.10	.10
B_6	.2399	.4	.4957	1.2	.0234	.5	1.49	.12

TABLE II.

Observed and calculated structure factors.

The calculations have been made with the atomic coordinates given in Table I.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
0	0	2	182.8	—175.4	—6	0	2	145.4	—152.8	12	0	2	54.5	53.0
		4	84.0	99.4	6	0	0	38.4	—36.4			4	25.0	—20.4
		6	146.2	—156.0			2	22.3	13.8			6		.6
		8		—4.2			4	99.8	102.8			8	34.5	34.4
		10	64.7	67.4			6	33.4	—37.6			10	69.1	—59.2
		12	37.7	—38.6			8	21.4	19.0			12	52.1	—46.6
		14	90.2	—97.2			10	94.8	—99.4			14	51.4	40.4
		16		—2.8			12	42.4	39.2	—14	0	16		3.6
		18		5.4			14	28.9	—19.0			14	30.4	—30.6
—2	0	18		—1.6			16	44.9	—36.6			12	90.8	97.4
		16	11.1	13.4	—8	0	18		5.4			10	9.9	—13.6
		14	68.1	—74.0			16	23.7	—25.8			8	18.9	14.6
		12	57.8	59.0			14		—4.2			6	64.4	69.0
		10	14.0	10.6			12	58.0	56.2			4	16.0	—10.2
		8	178.7	207.8			10	30.8	—29.2			2	43.2	46.0
		6	15.7	—20.0			8	27.9	29.8	14	0	0	23.7	—18.4
		4	133.2	—134.2			6	104.0	104.0			2	19.3	—13.6
		2	220.8	200.0			4	34.7	31.4			4	17.0	17.4
2	0	0	29.7	33.0			2	37.8	27.2			6	71.7	—74.8
		2	44.5	—49.8	8	0	0	172.9	188.8			8	70.3	68.0
		4	52.0	—59.6			2	270.4	—287.6			10	115.3	—113.6
		6	71.1	71.4			4	83.6	88.6			12	76.9	70.8
		8	18.8	—17.4			6	89.4	—85.2	—16	0	16		—6.0
		10	29.0	28.0			8	54.1	51.4			14	24.2	26.6
		12	70.0	—74.0			10		8.0			12		—.8
		14	63.7	—59.8			12		8.2			10	58.1	—60.4
		16	45.9	41.2			14	11.4	—13.6			8	79.7	86.4
		18	15.9	11.2			16	21.4	15.8			6	50.8	—49.0
—4	0	18	25.2	—22.4	—10	0	18	21.2	—18.6			4	30.3	24.0
		16	61.3	53.0			16	18.7	—13.6			2	64.7	—63.0
		14		—5.2			14	40.9	—44.2	16	0	0	78.6	83.0
		12	81.8	84.2			12	80.6	85.4			2	38.8	35.4
		10	37.6	—29.2			10	10.8	—10.6			4	81.0	77.8
		8	15.8	13.2			8	124.1	133.8			6	25.6	—25.8
		6	161.3	—181.6			6	176.6	—196.4			8	68.4	—59.8
		4	123.2	138.0			4	10.5	8.6			10	14.6	10.2
		2	100.1	106.6			2	81.9	87.8	—18	0	14	46.6	47.8
4	0	0	20.3	20.2	10	0	0	56.8	53.8			12		—8.6
		2	25.2	19.8			2	125.1	—134.8			10	26.6	22.4
		4	163.6	171.0			4	24.0	—15.4			8	59.5	62.2
		6	32.0	—38.6			6	23.0	—22.6			6	21.8	—12.8
		8	93.6	98.4			8	73.7	69.2			4	65.7	68.8
		10	44.7	—44.8			10	71.1	65.0			2	23.0	—25.0
		12	24.8	—20.8			12	60.5	—55.2	18	0	0	31.1	—25.6
		14	33.2	—29.8			14	31.0	—28.0			2		2.6
		16	18.5	20.8	—12	0	16		—4.6			4	33.0	24.4
—6	0	18		—3.0			14	45.2	—48.4			6	50.0	—46.8
		16	16.1	17.8			12	38.3	—32.2			8		4.8
		14	64.9	—67.4			10	24.5	—13.0	—20	0	12	44.3	42.2
		12	51.3	50.6			8		1.6			10	24.1	25.6
		10	48.0	45.4			6	83.4	—86.4			8		—.2
		8	35.1	25.8			4	153.9	174.2			6	20.0	—12.6
		6	152.0	158.4			2	101.1	106.8			4	57.5	52.8
		4	35.0	44.4	12	0	0	40.0	38.8			2	56.1	—56.8

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
20	0	0	20.4	18.6	—3	1	7	153.0	164.6	5	1	15	15.8	—17.2
		2	27.7	—20.2			6	83.0	90.0			16	32.4	28.2
		4	40.9	—37.8			5	170.9	—187.2			17	31.6	27.4
		6	20.5	—17.2			4	58.8	61.8	—7	1	17		—6.2
—22	0	8	33.0	31.8			3	80.8	73.4			16	26.5	28.8
		6	19.0	20.4			2	27.5	27.2			15	21.6	16.4
		4	20.0	—16.8			1	34.4	—24.2			14	51.8	53.8
		2	21.4	—21.4	3	1	0	12.4	—12.2			13	43.0	—39.6
22	0	0	39.3	—35.0			1	32.0	32.2			12	50.9	—53.2
		2		—5.8			2	49.8	—56.4			11	61.1	63.2
—1	1	18	15.2	—19.6			3	79.2	79.6			10		6.2
		17		3.4			4	141.5	161.4			9	48.6	—47.2
		16	40.3	37.8			5	77.4	89.8			8	122.2	132.6
		15		1.8			6	102.6	111.0			7	11.5	8.0
		14	31.0	—30.4			7	23.8	21.2			6	55.0	—54.2
		13	34.7	32.8			8	8.3	4.6			5	28.1	—27.8
		12	19.3	23.4			9	17.6	—1.0			4	41.7	—34.4
		11	90.8	99.0			10	132.7	—148.8			3	82.6	85.4
		10	69.1	—69.0			11	45.8	44.4			2	191.3	—203.0
		9	42.3	—46.6			12	58.7	—52.8			1	34.7	—32.8
		8	18.6	19.6			13		—9.6	7	1	0	115.3	—119.8
		7	88.8	96.4			14	16.5	—9.0			1	45.7	43.6
		6	202.0	—218.2			15	33.2	32.8			2	116.4	123.2
		5	42.4	—48.8			16	28.5	—26.2			3	42.4	—33.4
		4	196.8	190.6			17	46.8	—44.8			4	100.5	102.2
		3	21.9	18.0	—5	1	18	32.4	—31.4			5	92.2	98.6
		2	135.6	—128.4			17	18.8	—21.0			6	53.0	—58.4
		1	59.6	47.6			16	13.7	—12.4			7		—2.6
1	1	0	50.3	—25.2			15	39.7	38.6			8	24.1	24.2
		1	62.2	60.0			14	16.2	16.0			9	23.9	18.6
		2	75.9	71.2			13	37.2	—40.4			10		1.6
		3	60.7	62.4			12	44.3	45.6			11	29.5	26.6
		4	42.0	40.6			11	38.0	39.2			12	18.4	20.4
		5	37.9	35.6			10	43.5	—48.6			13	43.7	—39.0
		6	15.1	12.0			9	59.4	64.2			14	36.3	—30.6
		7		—12.8			8	55.9	60.4			15	13.1	13.2
		8	34.9	35.4			7	60.1	54.8			16		5.6
		9	33.4	—34.4			6	33.3	32.8	—9	1	17	20.3	—21.2
		10	31.0	—30.2			5	93.1	—97.6			16	21.5	23.2
		11	94.5	98.4			4	15.6	12.2			15		—11.8
		12	24.4	20.6			3	9.9	8.2			14	62.7	—63.4
		13	21.5	20.2			2	113.3	—115.4			13	60.9	66.6
		14	54.8	56.2			1	52.5	—44.4			12	27.5	—28.2
		15	34.6	29.8	5	1	0	11.0	7.0			11	30.7	—31.6
		16		3.6			1	43.5	44.8			10	50.6	—48.4
		17		—6.6			2	157.5	—175.0			9	12.0	—16.8
		18	41.6	—35.2			3	103.9	112.8			8	27.3	24.2
—3	1	18	27.5	—24.0			4	43.0	48.0			7	19.3	—16.4
		17	35.6	39.8			5	10.9	—12.6			6	35.8	—33.0
		16	26.5	24.4			6	55.9	—57.0			5	60.1	61.4
		15	34.4	—35.6			7	106.5	114.8			4	115.8	120.8
		14		8.2			8	45.8	—46.8			3	76.6	80.2
		13	67.9	71.0			9	88.1	—97.2			2	92.2	—99.6
		12	83.7	92.2			10	47.0	41.8			1	99.8	—104.4
		11		—2.2			11	35.6	37.0	9	1	0	94.0	98.4
		10	26.8	—27.8			12	85.4	90.2			1	22.0	—19.8
		9	37.3	34.8			13		—5.2			2	13.9	—10.8
		8	120.9	—136.2			14	29.6	—29.0			3	77.0	76.6

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
9	1	4	72.2	—74.6	—13	1	3	35.3	36.6	—17	1	3	65.0	—68.8
		5	44.2	—41.8			2	45.1	48.0			2	54.4	—57.4
		6	91.9	—90.0			1	37.1	—36.8			1		9.0
		7	26.2	23.6	13	1	0	64.8	—65.6	17	1	0		1.6
		8	66.7	69.4			1	21.3	—21.2			1	31.4	28.0
		9	27.5	27.0			2	42.9	—41.2			2		—.6
		10	31.1	24.4			3	16.0	13.8			3	71.2	79.6
		11	27.5	26.6			4	55.1	51.2			4	66.0	—63.0
		12	31.3	30.4			5	48.6	42.0			5	41.6	—39.0
		13	22.8	—20.6			6	20.2	17.0			6	26.4	24.2
		14		3.2			7	30.0	—22.8			7	18.9	16.4
		15		6.4			8	13.5	11.8			8	27.0	22.4
—11	1	17	17.0	—19.4			9		4.8			9		.4
		16	30.5	36.4			10	17.9	9.2			10	19.5	—18.2
		15	33.4	37.0			11	43.0	38.2	—19	1	13		—1.2
		14	46.7	—52.2			12	21.0	18.2			12	78.8	76.6
		13	36.8	34.6			13	20.3	12.0			11		8.6
		12	61.2	57.8	—15	1	16	9.0	13.4			10	25.8	—26.0
		11		—9.4			15	32.4	23.6			9	50.6	—51.8
		10	21.8	—22.4			14		—3.0			8		10.8
		9	52.4	—51.0			13	35.0	—35.0			7	38.7	37.6
		8	32.5	34.2			12	20.3	—21.0			6	34.7	—33.6
		7	56.1	55.2			11	82.2	93.6			5	41.7	—39.2
		6	79.1	79.0			10	35.0	35.8			4	43.4	—43.8
		5	70.3	—69.6			9		8.0			3	35.0	32.8
		4	49.1	—50.0			8		1.0			2	24.4	26.2
		3	83.4	86.6			7	15.6	—16.0			1	58.0	—58.6
		2	58.0	—56.6			6	68.2	—74.8	19	1	0	58.4	54.4
		1	53.5	—54.4			5	48.9	—54.4			1	19.6	20.0
11	1	0	67.6	75.0			4	65.0	64.6			2	45.4	—44.4
		1		5.0			3	37.4	33.2			3	12.2	12.8
		2	18.8	—22.6			2	29.2	—27.6			4	25.7	22.2
		3		5.0			1	42.1	—39.4			5	10.2	—10.4
		4	16.5	15.8	15	1	0	97.3	109.2			6	14.4	8.2
		5		—4.8			1	50.1	—50.2			7	39.6	30.8
		6	16.5	—15.0			2	23.8	22.2			8	34.9	30.4
		7	28.4	24.4			3	98.2	107.4	—21	1	11	10.1	—14.8
		8	42.9	—39.2			4	70.5	69.6			10		7.6
		9		1.8			5	20.4	19.6			9	14.8	—9.2
		10	35.1	—28.6			6	74.7	—69.8			8	33.8	27.8
		11	45.9	45.2			7	22.7	—18.4			7		—3.2
		12	43.4	39.8			8	10.9	12.6			6		—6.8
		13	18.9	—20.4			9	11.3	9.8			5		.0
		14	13.1	6.6			10	29.5	—25.2			4		—.6
—13	1	17	10.4	—6.4			11	18.7	16.8			3		—3.2
		16		5.2			12	29.2	—24.0			2		4.6
		15	26.1	26.0	—17	1	15	14.7	12.6			1	55.0	—54.0
		14	33.2	35.6			14	35.6	—32.4	21	1	0	25.1	—24.4
		13		—6.4			13		10.4			1	15.7	—12.8
		12	35.2	—36.0			12	20.7	21.6			2	23.3	—22.6
		11	16.8	8.0			11	32.5	31.6			3	11.2	10.0
		10	94.1	—101.6			10		10.4			4	8.3	10.0
		9	56.8	56.0			9	32.7	32.4			5	8.6	6.2
		8		—.6			8	27.3	24.0	—23	1	7	10.9	—6.6
		7	19.2	—18.8			7	59.8	—67.2			6		2.8
		6		—5.2			6	15.6	—15.8			5		—6.0
		5	49.4	—51.6			5	37.9	38.6			4	38.2	36.8
		4		—2.4			4	23.8	19.8			3	19.1	—18.2

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—23	1	2	19.0	—18.0	—4	2	18	12.4	—13.4	6	2	4	79.4	—84.2
		1	6.4	7.6			17	18.1	21.4			5	32.0	—30.2
23	1	0	18.4	—13.8			16	24.7	25.2			6	44.1	48.8
		1	13.7	—11.6			15	15.8	17.2			7	37.9	—39.2
0	2	1	29.6	20.8			14	34.2	31.8			8	53.5	46.8
		2	142.6	135.8			13	56.6	—58.0			9	65.2	68.2
		3	8.6	7.6			12	30.0	—30.2			10	55.2	—57.6
		4	60.3	—64.8			11	22.8	24.6			11	89.4	—101.0
		5	76.3	—79.0			10	27.3	26.2			12	13.8	12.6
		6	72.0	—70.8			9	66.8	—71.2			13	23.1	24.8
		7	124.4	151.4			8	75.5	—78.0			14		—3.0
		8	39.5	—40.4			7	68.4	75.2			15	8.9	—13.2
		9	77.9	—82.4			6	146.1	—154.8			16	8.0	9.4
		10	52.8	—52.8			5	20.4	14.0	—8	2	18	15.4	18.2
		11	33.3	36.0			4	96.1	—99.4			17	57.5	—54.2
		12	38.1	42.4			3	105.3	103.8			16	30.7	—36.6
		13	11.8	—17.6			2	63.8	54.8			15	25.0	27.8
		14		—8.8			1	358.0	—252.6			14	13.7	11.0
		15	49.3	54.0	4	2	0		—5.0			13	52.6	—56.4
		16	18.0	—20.2			1	101.8	—106.2			12	16.0	15.4
		17	40.1	—34.4			2	52.0	54.8			11	16.0	14.0
		18	33.7	32.2			3	107.9	124.0			10	28.3	—24.2
—2	2	18	24.6	21.0			4	81.6	—79.2			9	39.8	—35.2
		17	24.3	—19.6			5	68.4	—72.8			8	34.5	—36.0
		16	20.8	—23.0			6	7.1	—6.8			7	14.1	—15.6
		15	75.6	80.6			7	16.7	7.0			6	25.0	—21.0
		14	43.2	45.0			8	101.1	109.0			5	14.7	—14.6
		13		7.4			9	30.4	—28.6			4	39.9	36.8
		12	20.3	—18.6			10	38.6	36.2			3		12.2
		11	31.6	—34.4			11	45.1	49.2			2	72.5	78.2
		10	36.6	—34.4			12	24.3	—24.0			1	146.3	151.4
		9	73.1	82.2			13	88.9	—102.0	8	2	0	41.5	—40.6
		8	118.4	139.2			14		5.0			1	111.1	123.8
		7	62.5	—60.2			15	36.3	39.0			2	97.7	—101.0
		6	74.7	—81.2			16		12.4			3	16.8	—14.8
		5	70.2	67.8			17		—1.8			4	53.2	—53.2
		4	9.4	—6.2	—6	2	18		.4			5	13.1	—13.4
		3	128.3	—125.0			17	16.6	—20.4			6	73.0	74.2
		2	213.7	—186.4			16		—2.2			7	57.9	60.8
		1	365.0	—233.6			15	36.5	—39.4			8		4.4
2	2	0	271.7	—199.6			14	51.7	—57.4			9	95.6	—109.2
		1	23.3	22.8			13		—4.4			10	24.2	—26.4
		2	59.4	—63.2			12	20.0	—18.0			11	20.3	23.0
		3	34.1	—31.4			11	34.5	39.2			12	28.7	27.2
		4	54.5	—57.8			10	63.1	66.4			13	33.7	—31.0
		5	132.7	—140.6			9	52.8	—55.6			14	22.3	15.0
		6		—8.0			8		7.6			15	15.7	17.8
		7	63.6	72.8			7	81.5	91.2	—10	2	17	14.5	12.8
		8	14.1	—13.4			6	73.8	—76.4			16		—6.5
		9	54.9	—56.2			5	118.5	—127.6			15	20.6	17.4
		10		4.0			4	22.1	—21.0			14	15.9	17.4
		11	36.5	37.2			3	138.2	131.0			13	47.1	—46.0
		12	9.5	10.2			2	109.7	—107.2			12	15.3	14.8
		13	13.4	—15.0			1	79.8	—83.2			11		5.0
		14		—5.6	6	2	0	38.5	36.0			10	25.2	23.4
		15	27.9	—31.0			1	100.3	—104.0			9	57.8	—60.8
		16	41.3	38.8			2	18.9	—17.6			8	9.2	—23.0
		17	35.2	29.0			3	148.7	166.2			7		6.4

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—10	2	6	84.7	—95.0	—14	2	11	15.7	—17.6	—18	2	6	15.7	—14.2
		5	32.4	—33.4			10		8.2			5	24.9	26.6
		4	18.5	22.6			9		—1.0			4		14.2
		3	78.8	80.8			8	52.9	—56.2			3	34.8	32.6
		2	69.0	—69.2			7	81.6	88.8			2	17.9	17.8
		1	38.4	—40.6			6	26.8	29.6			1	61.3	—70.6
10	2	0	35.8	—35.0			5	76.7	—90.2	18	2	0	49.1	—50.8
		1	104.1	—112.6			4		3.8			1		—6.4
		2	80.4	—81.0			3		—5.8			2		—5.8
		3	44.7	—46.0			2	67.2	—75.2			3	29.1	28.6
		4	36.9	36.0			1	56.0	—57.8			4	40.2	37.8
		5	20.6	—20.4	14	2	0	26.0	24.6			5	11.2	—11.4
		6	19.6	15.0			1	77.1	—81.2			6		5.2
		7	55.9	54.4			2	84.2	—85.0			7		—1.2
		8	16.1	—15.6			3	18.9	14.0			8	13.3	12.6
		9	58.9	—61.2			4	20.8	—18.4			9	6.6	—8.4
		10	29.8	30.0			5	70.9	—67.4	—20	2	12	9.5	8.0
		11	64.2	65.2			6	18.9	18.0			11	19.4	15.8
		12	13.5	13.0			7	47.7	44.2			10		—10.0
		13	25.2	—27.6			8		—11.4			9	52.2	—51.2
		14		2.0			9	14.2	12.8			8	28.3	—32.2
		15	16.8	15.6			10	15.0	14.6			7	36.9	39.8
—12	2	17	13.3	17.2			11	24.5	—27.0			6		1.6
		16		12.4			12		3.6			5		6.8
		15	20.8	20.8	—16	2	15	39.3	44.8			4		3.6
		14		—5.8			14		—11.0			3	14.5	—16.0
		13	34.4	—39.6			13	36.2	—43.0			2	19.7	—17.6
		12	54.6	—54.6			12	14.1	—13.8			1	15.7	—14.8
		11		—8.2			11	44.2	49.0	20	2	0		1.4
		10	22.1	—27.0			10	35.3	—32.8			1	17.6	—15.4
		9	12.6	—13.4			9	23.7	—22.0			2	17.3	16.6
		8	69.0	—74.6			8	51.3	—51.0			3	47.2	45.2
		7	61.3	66.0			7	43.7	39.6			4		6.6
		6		10.4			6		—4.2			5	47.3	—40.0
		5	50.4	51.4			5	36.2	—35.2			6		—1.0
		4	77.6	80.2			4	59.5	—68.0	—22	2	9		3.6
		3	46.9	—51.4			3	36.7	35.6			8		—2.8
		2		—6.8			2		—7.2			7	40.9	40.0
		1	54.9	—57.4			1		6.8			6	41.4	38.6
12	2	0	46.6	—46.4	16	2	0	35.0	33.0			5	59.8	—55.0
		1	124.2	—151.0			1	67.3	67.4			4	13.2	—12.6
		2	74.0	79.6			2	21.2	—21.6			3	14.8	13.2
		3	27.5	27.4			3	46.6	—46.2			2	40.8	—35.6
		4	16.2	—12.4			4	61.1	65.8			1		2.6
		5	26.8	—30.4			5	15.9	14.0	22	2	0		—.6
		6	29.5	32.2			6	23.0	21.2			1	42.6	—39.4
		7	46.0	48.6			7	11.8	13.2			2		5.4
		8	30.4	—27.8			8	15.2	—14.8			3	14.9	—11.6
		9	25.8	22.6			9	28.5	—29.0	—1	3	17	35.3	—33.0
		10	36.6	—34.2			10	27.0	24.0			16		—4.6
		11	17.1	—15.2			11	23.0	23.0			15		—10.6
		12	23.5	22.8	—18	2	13	22.2	22.8			14	17.4	18.8
		13	24.2	—22.6			12		5.8			13	25.0	—29.2
—14	2	16	14.4	14.4			11	15.3	12.8			12	77.2	—84.4
		15	24.8	22.0			10	33.4	—34.2			11	63.2	67.8
		14		5.0			9		.4			10	45.8	50.4
		13	18.9	16.8			8		5.0			9	40.2	—43.4
		12	37.2	37.8			7	21.1	—18.8			8	88.0	—88.2

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—1	3	7	96.8	—101.0	3	3	16	20.8	—19.0	7	3	5	75.3	76.0
		6	97.2	104.2			17	47.8	—45.0			6	54.8	54.2
		5	16.7	17.8	—5	3	17	31.4	—28.6			7	34.8	—37.2
		4	95.3	—92.2			16		—15.0			8		—11.4
		3		—3.2			15	34.0	39.6			9		—2.0
		2	122.8	—105.2			14		—14.8			10	20.4	—15.6
		1	146.8	—108.0			13	78.1	—85.4			11		—14.4
1	3	0	86.1	—58.6			12	42.0	—44.4			12	55.4	—59.8
		1	151.2	—123.4			11		18.4			13		—5.4
		2	79.5	67.4			10	20.3	25.2			14	23.4	22.4
		3	117.9	113.2			9		—11.0			15	17.8	—13.0
		4	90.8	83.8			8	19.8	—16.6	—9	3	17		—12.6
		5	129.7	—133.8			7	102.0	112.4			16		—1.8
		6	40.5	—44.6			6	73.0	78.4			15	35.3	—39.4
		7	138.1	—148.8			5	97.2	—107.0			14		—14.4
		8	18.0	—22.4			4	86.1	—76.4			13		—3.8
		9		9.0			3	18.6	21.4			12		1.6
		10	48.9	50.0			2	39.0	32.4			11		8.2
		11		16.0			1	144.9	—133.6			10		—8.8
		12	65.3	70.8	5	3	0	68.4	65.6			9	48.2	—49.6
		13	26.6	—25.2			1	43.1	—34.6			8	58.6	—63.8
		14	31.2	32.6			2	20.5	16.2			7		12.6
		15	47.2	49.0			3	31.0	31.0			6	67.6	69.8
		16	24.8	—24.2			4	47.7	—48.4			5	53.8	57.4
		17	24.5	—21.0			5	69.0	—71.0			4		1.4
—3	3	17	21.5	—19.2			6	44.0	—42.0			3	21.5	19.8
		16		6.4			7	80.5	82.0			2	120.4	134.4
		15	21.0	—19.6			8	25.8	—24.8			1	24.5	28.0
		14		6.6			9	144.3	—160.0	9	3	0	38.6	—30.8
		13		21.4			10	89.8	92.4			1	72.0	—75.8
		12	24.3	—22.8			11	33.0	—34.0			2		9.8
		11	25.0	—27.4			12	18.6	17.8			3	121.7	137.4
		10	63.3	—74.6			13		2.8			4		—4.4
		9	23.2	—22.4			14	17.8	16.4			5	101.1	—108.6
		8	67.4	70.8			15		—3.2			6	25.0	—21.2
		7	55.0	56.0			16		—9.8			7		—7.4
		6	52.4	51.0	—7	3	17		—6.8			8	23.7	20.8
		5		—13.2			16		8.8			9		3.0
		4		4.8			15	18.8	22.4			10		—8.4
		3	34.2	—24.6			14	19.6	15.8			11	24.1	24.2
		2	70.2	—54.0			13	102.3	—117.8			12		—12.8
		1	46.4	—41.0			12		—1.8			13	33.7	—32.4
3	3	0	39.3	—33.8			11	24.9	24.4			14		—.8
		1	33.2	—33.0			10		10.8	—11	3	16	49.0	—42.2
		2	241.7	224.0			9	22.4	—26.8			15		10.4
		3	47.9	52.8			8		—14.2			14	36.6	39.2
		4	24.0	—25.6			7	56.9	—57.4			13	20.2	19.2
		5	110.9	—113.2			6	48.8	43.0			12		—16.4
		6	26.9	30.6			5	56.9	49.0			11		—2.4
		7	41.9	49.6			4	23.0	—20.8			10	71.5	71.6
		8	65.2	—61.8			3	22.6	—16.4			9	77.0	—84.2
		9		9.4			2	38.5	36.0			8	90.0	92.0
		10	16.3	15.4			1	57.9	56.0			7		10.0
		11	52.1	—54.2	7	3	0	39.6	—35.2			6		—2.6
		12		5.4			1	129.4	—140.4			5	62.2	—71.2
		13	27.5	—26.4			2	30.5	33.4			4		—7.2
		14	43.1	—43.0			3	19.4	—14.4			3	53.5	57.8
		15	24.6	27.2			4	80.8	86.8			2	55.7	—53.0

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—11	3	1	64.1	67.6	15	3	0		14.0	—21	3	3	61.6	54.2
11	3	0	25.8	28.2			1	39.4	—36.6			2		9.6
		1	18.5	13.8			2	69.7	75.6			1	49.2	—41.2
		2	94.4	101.6			3		4.2	21	3	0	13.5	13.2
		3		1.8			4	70.4	—73.2			1		—14.8
		4	78.8	—79.4			5	28.0	27.8			2		3.4
		5	103.5	—114.0			6		—14.4			3	9.5	—9.2
		6		11.4			7	20.2	—18.6	0	4	1	191.8	145.6
		7	51.4	51.4			8		—9.2			2	238.3	190.0
		8	85.4	—84.0			9	29.4	—29.8			3	69.1	64.8
		9		—9.2			10	28.7	26.4			4	72.5	—64.0
		10	28.6	27.4	—17	3	13	24.0	20.2			5	36.8	—32.6
		11		—9.0			12	38.7	—37.0			6	95.6	92.8
		12		—5.4			11		8.8			7	17.7	19.2
		13	15.7	15.8			10		14.4			8	45.3	—48.8
—13	3	16	14.0	22.4			9		10.2			9		—1.0
		15		6.8			8		10.2			10		12.8
		14		—4.0			7	21.9	28.4			11		—3.6
		13	39.7	—39.8			6		12.6			12		—7.8
		12	24.4	—29.0			5	20.9	—15.0			13	29.7	31.4
		11	24.9	23.8			4		—8.4			14		—4.4
		10		18.4			3	41.4	—39.8			15	16.0	—17.8
		9		—19.0			2	27.6	—30.0			16	11.2	14.2
		8		19.0			1	32.1	28.2			17	16.0	—19.4
		7		5.2	17	3	0	20.2	—20.0	—2	4	17	29.3	—30.6
		6		7.0			1		—3.2			16	9.5	—13.0
		5	46.5	—47.8			2	29.7	—30.4			15	25.1	26.4
		4	66.7	—72.4			3	29.9	31.2			14		—.2
		3	60.6	61.2			4	17.1	—11.6			13		—2.8
		2		—6.2			5	44.4	—46.8			12	52.0	—53.6
		1	27.4	—28.0			6		—4.0			11	51.0	56.8
13	3	0	22.1	22.8			7	17.9	—12.8			10	73.3	81.4
		1	24.6	—23.4			8	17.0	15.6			9	48.2	52.4
		2	28.6	—24.8			9	27.6	—24.8			8	41.1	—38.2
		3	67.0	—69.0	—19	3	11	53.4	—50.0			7	61.8	—58.8
		4	31.0	—30.0			10		13.4			6	126.8	136.2
		5	47.1	—49.2			9		11.6			5	67.7	—67.2
		6	55.0	—54.6			8	25.0	—25.6			4		—2.6
		7	28.1	23.4			7	32.2	32.2			3	28.0	14.6
		8	55.0	52.6			6		—9.4			2	39.5	—33.8
		9	52.4	—54.2			5	35.0	—33.6			1	109.0	—86.6
		10	50.5	45.2			4	19.5	—17.0	2	4	0	16.9	—24.0
		11	48.6	49.2			3	74.5	85.2			1	83.5	61.8
		12	17.5	—17.6			2	34.2	36.8			2	110.8	—92.6
—15	3	14	22.9	—26.4			1		—9.6			3	71.7	—62.8
		13	28.6	—29.4	19	3	0		2.6			4	27.2	17.4
		12		4.6			1		3.0			5	48.2	—43.6
		11	73.4	82.0			2	25.7	27.4			6	24.6	23.2
		10	37.2	39.6			3		—7.2			7		—9.0
		9	45.2	—52.8			4		—9.8			8		4.2
		8		22.2			5	22.6	—19.6			9	50.0	—50.8
		7	31.5	33.0			6	36.5	31.2			10		—8.4
		6		1.8			7	27.4	22.0			11	31.5	34.8
		5		—13.4	—21	3	8		2.0			12	21.7	—19.8
		4	75.3	—85.8			7	28.1	26.2			13		1.6
		3		6.6			6	12.6	—11.8			14	46.0	48.6
		2	67.8	74.0			5		—1.2			15		—8.2
		1		19.6			4		4.8			16		5.0

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—4	4	17	25.6	26.0	6	4	7	46.8	47.6	10	4	2	45.2	47.6
		16	20.1	—22.0			8	61.8	—61.4			3		6.4
		15	46.9	—43.0			9	13.1	15.4			4	29.3	28.4
		14	28.3	—34.2			10	35.0	38.4			5		—5.8
		13		9.0			11		11.4			6		—3.2
		12	23.6	27.4			12	64.8	—67.6			7	25.9	22.8
		11		—5.4			13	12.2	—10.0			8	29.1	—32.4
		10	33.6	36.0			14		1.0			9		—13.0
		9		—11.4			15	24.9	26.6			10		—9.8
		8	88.0	—95.2	—8	4	16		7.0			11	61.9	77.0
		7		—6.8			15		5.4			12	17.7	—20.4
		6	26.4	26.6			14		9.2			13		—8.6
		5	102.1	109.0			13	42.9	46.8	—12	4	15	16.0	—16.8
		4	59.9	—53.2			12	26.3	23.6			14		9.2
		3	70.5	65.2			11	70.1	—81.0			13		5.8
		2	211.9	182.8			10	37.2	—43.0			12		9.8
		1	90.3	—70.8			9	68.9	68.4			11	34.8	41.2
4	4	0	164.4	141.4			8		6.6			10	25.3	—29.8
		1	76.1	—61.4			7	30.7	31.4			9	23.2	—22.2
		2	80.8	—69.2			6	23.9	27.6			8	20.4	22.6
		3	56.2	55.8			5	53.7	—50.6			7	32.8	—29.0
		4	25.0	—23.6			4	50.1	55.2			6	61.5	60.4
		5		6.6			3	73.4	73.4			5	63.6	62.0
		6	42.8	38.0			2	128.9	127.6			4		—7.2
		7	17.5	—16.8			1	29.4	—26.6			3	21.8	—22.0
		8	25.9	21.2	8	4	0	80.4	—74.0			2	46.5	51.0
		9		—8.6			1	73.9	75.2			1	19.2	—18.6
		10	66.8	72.0			2	83.8	86.2	12	4	0	37.5	—36.0
		11	20.3	24.6			3		—7.4			1		—9.0
		12	47.8	—52.2			4	24.0	—24.2			2		10.0
		13		—4.6			5	65.6	64.0			3	69.5	69.0
		14	16.3	19.0			6	50.0	46.6			4	66.1	69.0
		15	10.6	8.0			7		—8.2			5		—2.0
		16	25.5	—28.6			8	15.9	—16.2			6		—5.8
—6	4	17	22.8	—25.6			9	21.8	22.6			7		—7.6
		16	64.5	—64.6			10	17.2	—16.6			8		—9.0
		15		4.8			11	22.3	—23.6			9	38.0	42.4
		14		5.4			12	33.7	—40.2			10	37.5	—50.8
		13		.0			13	14.5	12.6			11		—7.4
		12	20.7	—18.6			14	12.0	14.2			12		—1.8
		11	66.7	74.2	—10	4	16		—2.0	—14	4	15	14.4	14.6
		10	46.9	54.0			15	41.5	41.0			14	69.7	69.4
		9	59.3	—63.0			14	37.6	44.0			13	31.3	40.8
		8		—1.4			13	18.9	—19.2			12	26.1	35.6
		7	122.0	136.2			12		—4.2			11		10.6
		6	17.1	—15.0			11		20.4			10	33.1	35.8
		5		.4			10	65.0	66.4			9	63.3	—59.4
		4		—10.8			9		8.8			8		—1.0
		3	54.3	—46.0			8	57.0	—56.8			7	32.4	33.2
		2	127.5	119.6			7	83.9	—93.8			6		—14.0
		1	79.4	74.2			6	112.7	119.2			5	73.6	—80.4
6	4	0	49.4	43.6			5		—4.4			4	56.9	56.6
		1	40.9	34.8			4	99.5	—103.8			3	17.4	—20.4
		2	24.6	22.2			3	62.0	55.4			2	43.4	43.2
		3	29.6	29.2			2	31.7	30.4			1	14.8	—15.2
		4	39.8	—38.6			1	30.7	—28.4	14	4	0	83.3	—87.0
		5	25.7	20.0	10	4	0	49.0	—46.2			1	29.0	30.6
		6	52.4	47.4			1	28.9	—30.8			2	39.6	—39.4

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
14	4	3	19.7	—18.4	—20	4	1		—2.0	3	5	3		—3.4
		4	41.6	—46.4	20	4	0		7.0			4	44.8	40.6
		5	32.7	—37.2			1		—10.4			5		.4
		6	38.0	42.2			2	28.7	35.0			6	63.1	—61.6
		7		—6.2			3	40.9	54.8			7	69.4	76.4
		8	28.6	—37.4			4		—9.4			8		—15.6
		9		1.4	—1	5	16	31.8	—34.2			9	20.6	28.0
		10	13.8	23.4			15	17.7	—21.4			10		—8.0
		11		—9.8			14	18.5	20.8			11		2.4
—16	4	13		—8.8			13	18.8	—21.4			12		—14.2
		12		—6.4			12	43.9	—46.0			13		3.0
		11	39.4	—37.0			11		—5.4			14	17.1	14.0
		10		9.8			10		17.0			15		—4.6
		9	29.5	32.8			9		—4.2	—5	5	16		—10.6
		8	47.6	—46.4			8		—9.6			15		—13.0
		7		—9.4			7	43.8	46.2			14		1.6
		6		10.8			6	70.6	77.2			13	57.4	65.6
		5	17.4	—16.0			5	65.8	64.2			12		—16.8
		4	27.9	31.6			4	123.4	—116.8			11		—12.8
		3	17.9	21.4			3	144.9	—134.2			10		11.6
		2	30.0	29.0			2		2.8			9	25.7	30.2
		1		—11.2			1	41.5	24.0			8	48.2	—50.8
16	4	0	66.9	—73.6	1	5	0	104.9	—69.2			7	41.6	44.2
		1		1.8			1	73.4	—45.8			6		—3.4
		2	38.4	42.0			2		8.8			5	49.9	—50.4
		3	22.7	—21.4			3	73.0	—67.2			4		15.4
		4	12.5	—19.2			4	30.2	—25.8			3	92.3	86.6
		5	14.1	15.6			5		3.2			2	23.6	26.6
		6	28.4	40.8			6	69.2	61.8			1	40.3	—38.4
		7		3.6			7	22.5	15.6	5	5	0	118.7	—103.2
		8		—8.4			8	55.5	—62.2			1	151.1	143.6
		9	15.4	—28.2			9		3.2			2	118.6	106.4
—18	4	11	24.7	21.4			10	42.0	41.4			3	66.9	—59.2
		10	15.0	—16.4			11	26.0	—29.2			4	98.9	—101.2
		9	40.6	37.0			12	42.2	44.2			5	64.6	61.0
		8		—4.2			13		8.0			6	22.8	—24.2
		7	26.6	—25.2			14		11.6			7	59.6	—66.4
		6	42.4	48.0			15	32.1	31.0			8	27.6	—29.4
		5	30.8	33.2			16		10.6			9		—.2
		4	21.1	24.0	—3	5	16	14.4	—12.0			10	21.5	19.6
		3	24.0	—23.4			15		1.4			11		3.6
		2	16.6	13.6			14	33.9	39.8			12	22.6	24.0
		1	17.5	—21.0			13	31.6	29.2			13	27.7	30.4
18	4	0	37.3	—45.8			12	39.1	—44.2			14	61.8	57.0
		1	23.5	—26.6			11	25.9	—31.8			15		—4.4
		2	18.7	21.0			10		—9.4	—7	5	16	12.9	16.4
		3		12.2			9	27.7	25.6			15		14.6
		4	12.9	—22.6			8	41.9	42.6			14	26.4	28.2
		5	16.1	24.0			7	40.4	43.2			13		5.8
		6		13.0			6		5.4			12		6.8
—20	4	9	21.7	—21.0			5	47.6	45.6			11	19.9	—22.6
		8		—1.2			4	53.2	46.2			10	32.8	—41.8
		7	27.6	24.4			3	47.7	—37.6			9	47.6	47.4
		6	28.4	27.8			2	46.2	39.8			8	33.1	—40.8
		5		—3.8			1	111.3	88.6			7	58.0	—66.0
		4	44.2	—38.8	3	5	0	102.8	—80.4			6	48.9	52.2
		3		5.4			1	46.4	32.2			5	59.3	55.2
		2	13.2	16.4			2	35.2	—24.8			4		5.6

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—7	5	3		21.2	—11	5	5	48.0	—58.0	15	5	2		8.6
		2	80.0	—69.4			4	58.6	—65.6			3		6.8
		1		—8.2			3	52.0	—57.2			4	27.3	—30.6
7	5	0	20.0	—17.2			2	24.3	—29.4			5	17.2	18.8
		1	59.9	—61.4			1	29.9	25.8			6	31.0	29.8
		2	51.6	51.8	11	5	0		11.6			7		—3.8
		3		—.2			1		—4.2			8	28.6	—24.6
		4		—11.6			2	34.4	37.0			9	23.2	26.0
		5	42.0	45.6			3	22.0	22.4	—17	5	11		6.8
		6	43.0	49.0			4	22.1	23.0			10		4.0
		7	30.7	30.2			5	34.5	40.4			9	18.4	—18.4
		8	51.0	—52.6			6	43.9	—43.4			8		—11.0
		9	33.6	36.0			7	43.1	44.8			7	15.8	22.6
		10	30.9	—35.2			8	26.4	23.4			6	38.0	44.0
		11	30.2	32.0			9		—10.8			5		—6.0
		12	27.3	23.2			10		9.8			4	21.5	—25.8
		13		2.8			11	44.0	—43.0			3	36.8	—45.0
		14		—9.0			12		—6.2			2	18.5	12.0
—9	5	16		1.4	—13	5	14	32.5	—35.6			1	20.6	—19.6
		15	20.6	—17.0			13		—12.4	17	5	0	35.6	—45.8
		14	53.5	51.8			12		2.6			1		15.4
		13		4.4			11	43.0	—46.4			2		—6.8
		12	48.9	—57.2			10		2.6			3	15.0	—14.2
		11	22.5	25.4			9	49.2	55.0			4		4.8
		10		—11.0			8		—5.8			5	15.6	—17.0
		9		19.2			7	39.6	—38.6			6		1.2
		8	68.6	—70.0			6	33.4	—35.6			7	28.5	—26.8
		7	78.4	87.2			5		—8.2	—19	5	9	28.2	27.0
		6	30.2	35.6			4		—19.0			8	27.2	—27.0
		5		5.6			3		5.6			7	28.5	—32.4
		4	88.3	—92.0			2		—20.2			6		5.8
		3	64.4	—62.8			1	64.4	—72.2			5	16.9	19.0
		2	27.7	26.4	13	5	0		—23.4			4		3.8
		1	26.0	24.0			1		—13.8			3		7.4
9	5	0		—11.2			2	22.9	24.8			2	15.5	11.6
		1	58.2	59.8			3	28.5	—30.2			1	20.6	23.4
		2	26.1	20.4			4	62.2	—68.2	19	5	0		17.4
		3	19.1	—16.0			5	26.5	24.8			1		.8
		4	23.7	—23.0			6	47.1	51.0			2	15.2	14.2
		5	98.9	104.4			7	24.1	—29.6			3		6.2
		6	43.3	44.2			8	42.4	33.6	0	6	1	39.5	33.2
		7	18.4	—15.4			9	23.0	23.8			2	61.1	40.8
		8	57.7	—59.6			10	27.0	28.2			3	27.3	29.0
		9	27.9	27.4	—15	5	13		7.2			4	44.1	—33.4
		10	77.0	78.4			12	13.3	16.0			5	40.0	43.8
		11		3.6			11	15.2	—11.6			6	56.2	—50.8
		12	28.5	—26.4			10	16.6	16.0			7		—3.4
		13	19.2	—19.0			9		—8.4			8	37.5	36.8
—11	5	15	15.0	15.8			8		—12.0			9		.6
		14		12.8			7	31.8	—33.8			10	69.4	77.2
		13	21.1	28.4			6	35.5	33.4			11	41.8	—42.0
		12	49.4	—62.0			5	58.3	63.2			12	46.3	—53.2
		11	31.9	—31.8			4	41.4	—40.6			13	16.1	17.2
		10	64.4	80.8			3	23.1	—24.0			14	8.6	6.2
		9	44.6	51.4			2	36.8	38.0			15	25.9	—30.0
		8	23.3	17.2			1	19.3	—19.8	—2	6	15	27.7	—29.0
		7	22.4	—24.6	15	5	0	22.1	20.6			14	40.0	—38.0
		6		—12.2			1		10.8			13		3.0

Continued: TABLE II.

h	k	l	Fo	Fc	h	k	l	Fo	Fc	h	k	l	Fo	Fc
—2	6	12	9.5	—12.8	—6	6	15	11.4	—13.8	—10	6	14	13.5	—14.6
		11		4.6			14	23.7	26.4			13	23.7	26.8
		10	44.7	51.4			13	19.5	22.8			12	20.0	26.6
		9	50.2	57.2			12	23.1	—24.8			11	25.4	—24.4
		8		1.4			11		5.8			10	29.8	36.6
		7	54.1	—51.6			10		1.8			9	10.5	—14.6
		6	36.7	37.0			9	26.5	23.8			8	70.6	—77.8
		5	93.7	—91.6			8	32.5	29.2			7	19.8	—28.2
		4	34.4	—32.2			7	80.7	—86.0			6	17.3	—24.2
		3	51.0	—46.8			6	54.7	—59.0			5	34.3	26.0
		2	9.2	—11.6			5	114.0	103.4			4	48.3	—55.4
		1	55.6	31.4			4	41.5	—39.0			3	32.1	—36.4
2	6	0	78.7	—62.2			3	91.8	—88.0			2	16.9	—17.0
		1	33.9	—23.8			2	30.1	25.6			1	47.2	44.2
		2	35.2	26.4			1	96.0	83.8	10	6	0		—2.6
		3	41.4	—38.4	6	6	0	46.1	—36.6			1	34.9	29.4
		4	95.1	—87.0			1	61.9	50.0			2	26.8	27.6
		5	84.6	75.4			2	17.6	—10.6			3	83.5	—94.4
		6	55.1	—52.6			3	7.1	—12.8			4	71.7	—76.4
		7	86.3	—90.8			4	22.0	—21.0			5	30.2	26.6
		8	74.0	78.6			5	60.2	—59.0			6	47.5	52.0
		9	9.8	—9.6			6	23.6	—23.2			7	23.4	—30.4
		10	64.9	—69.0			7	15.3	15.2			8	14.6	17.0
		11	33.7	—33.0			8	50.2	—54.4			9		9.8
		12	26.4	29.8			9	29.3	33.4			10	23.3	—23.2
		13	27.3	24.6			10	13.2	14.6			11	9.1	—6.6
		14		1.2			11		—6.2			12	27.3	36.0
		15	6.5	—6.2			12	12.5	—9.2	—12	6	14	6.7	6.0
—4	6	15	24.2	—27.0			13		3.8			13		—.2
		14	8.1	9.4	—8	6	15	9.9	17.4			12	20.9	—25.4
		13	19.7	—23.4			14		—.4			11	15.3	15.6
		12	14.0	12.4			13	7.3	8.4			10	9.0	—6.4
		11		—14.6			12	24.6	—28.0			9		4.2
		10	12.8	—17.6			11	29.3	—36.2			8	13.7	—14.0
		9		—3.2			10	27.3	25.0			7		11.4
		8	14.6	13.6			9		—10.2			6	13.5	—12.4
		7	62.1	—60.0			8	50.6	—56.2			5	16.7	17.6
		6	20.3	—19.2			7	11.4	11.0			4	9.4	—9.4
		5		9.2			6	56.9	—61.0			3	18.0	17.2
		4	18.3	17.6			5	37.1	43.2			2	47.5	—48.0
		3	34.3	34.4			4	33.6	32.8			1	35.1	33.4
		2		1.8			3		—3.2	12	6	0	64.0	64.0
		1	114.7	91.2			2		3.0			1	12.5	17.4
4	6	0	81.5	67.0			1	89.5	—84.8			2	57.1	—67.4
		1	14.5	11.0	8	6	0	42.4	—40.2			3	24.0	27.0
		2	66.8	—51.2			1		8.0			4	56.6	59.0
		3		—3.4			2	19.1	18.2			5	31.2	36.8
		4	80.0	—78.4			3	23.2	—21.6			6	47.0	54.8
		5	45.7	45.2			4	24.3	—21.6			7	30.9	—36.2
		6	39.8	33.6			5	59.9	—62.6			8	13.8	—11.4
		7	90.0	—100.0			6		12.2			9		1.6
		8	19.8	—19.6			7	15.3	13.6			10	31.6	34.2
		9	9.6	14.6			8	41.1	41.2	—14	6	12		5.4
		10	13.4	14.2			9	25.9	26.2			11	12.7	—13.2
		11		5.6			10		—6.6			10		5.6
		12	13.4	13.6			11	51.0	—54.2			9		8.8
		13	18.9	20.6			12	22.2	—25.0			8		3.4
		14	13.2	—13.6			13		1.0			7	44.0	—49.6

Continued: TABLE II.

h	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc	*h*	*k*	*l*	Fo	Fc
—14	6	6		—9.8			10	20.8	17.8			6	17.6	15.0
		5		7.2			9	11.7	11.0			7	17.8	22.0
		4	19.3	—19.4			8	11.8	—11.8	—18	6	9	18.2	20.0
		3	20.7	—27.2			7	20.7	—18.6			8	29.5	—29.0
		2	43.6	45.0			6		7.4			7	10.4	9.6
		1	60.9	60.0			5	21.5	27.6			6	58.2	59.4
14	6	0	51.3	—48.2			4	21.7	22.2			5		8.8
		1	52.8	56.6			3		5.6			4		—6.4
		2		—1.6			2	29.9	—30.6			3	17.3	—18.2
		3	19.0	—21.2			1	33.3	37.6			2		—2.6
		4		.6	16	6	0		—2.0			1		3.2
		5		—6.8			1	10.4	—9.2	18	6	0	13.4	16.0
		6	10.3	—9.4			2	39.8	45.2			1	11.7	—13.2
		7	52.0	—49.2			3		—1.2			2	26.7	—25.8
		8	6.2	8.8			4	16.1	—13.4			3	38.6	—38.0
—16	6	11	18.9	—20.8			5	27.7	—20.8			4	6.1	—6.2

TABLE III.

Positional parameters of the hydrogen atoms.

ATOM	*x*	*y*	*z*
H_1	.368	.065	.012
H_2	.380	.550	.062
H_3	.102	.474	.329
H_4	.353	.794	.218
H_5	.407	.223	.143
H_6	.204	.934	.071
H_7	.017	.420	.058
H_8	.063	.222	.063
H_9	.108	.092	.116
H_{10}	.000	.735	.250

LOCATION OF HYDROGEN ATOMS.

In the attempt to locate hydrogen atoms, a three-dimensional difference synthesis was computed on the basis of the coordinates obtained from the last cycle of refinement, as reported in Table I. A number of residual peaks, the most intense ones, were selected. Eight of these maxima fitted well with

eight hydrogen atoms (H_1 to H_8); H_9 was little evident in the map and it was given a reasonable position on the O_{14}—O_3 vector.

The last hydrogen atom H_{10}, in special position, was still missing. It was evident that this atom could not be on a symmetry centre because the nearest oxygens around the centres have distances, each from its centro-

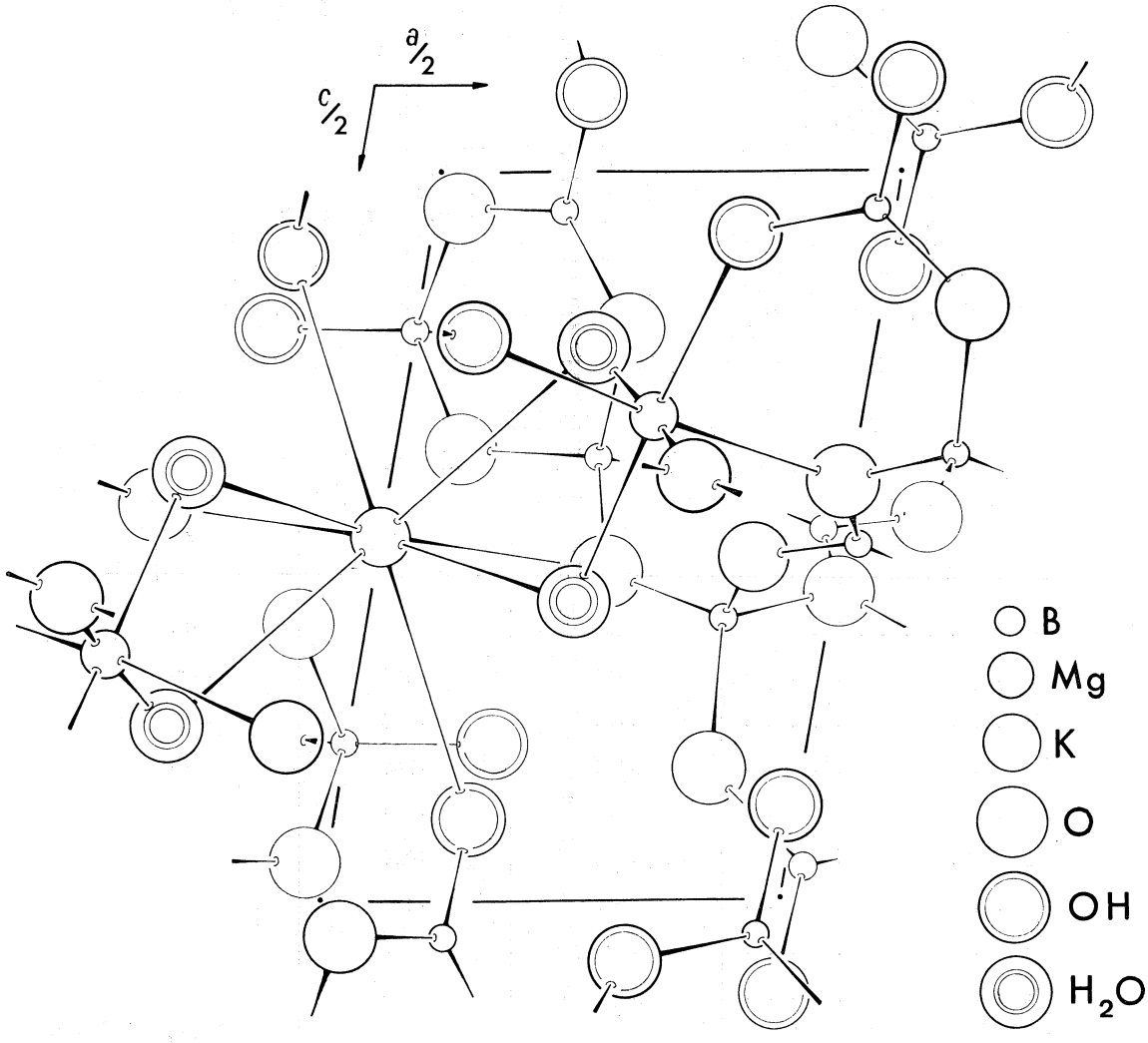

Fig. 1. – Projection of the structure of kaliborite on (010).

symmetrical one, of more than 3.3 Å, too great for hydrogen bonds. So the twofold axis was the only symmetry element which could receive H_{10}. The shortest distance around this axis is 2.4 Å from O_2 and its centrosymmetrical one; H_{10} was placed therefore at the centre of the O_2—O_2 bond, though in the (F_o—F_c) map there was no indication of its presence.

The coordinates of hydrogen atoms are reported in Table III; they were given a general isotropic temperature parameter of 5.5 Å^2. This value gives the best agreement between observed and calculated structure factors.

DISCUSSION.

The general arrangement of the atoms in the crystal is illustrated in fig. 1. The coordination polyhedron of Mg is close to a regular octahedron with an average Mg—O distance of 2.08 Å. A potassium atom binds eight oxygens (the average K—O distance is 2.94 Å) according to a distorted cube

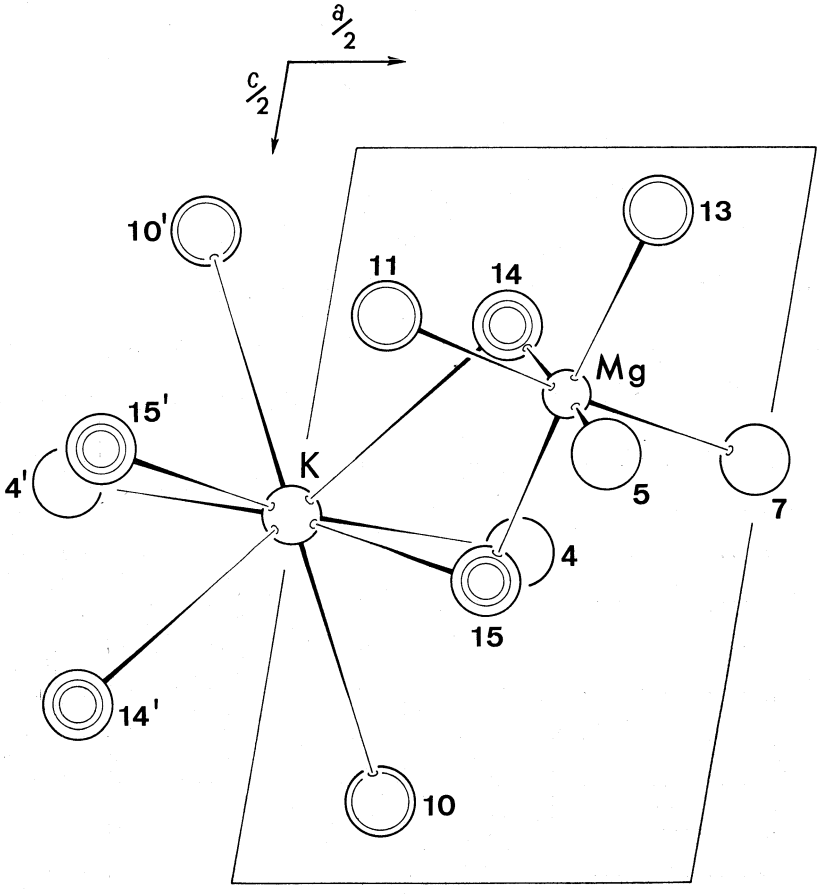

Fig. 2. - Coordination polyhedra for K and Mg.

The two polyhedra share one edge, formed by the two water molecules. The small circles are K and Mg atoms and the larger ones are oxygen atoms (double circles are OH and triple circles are H_2O). The numbering corresponds to the designation in Table I.

which shows a flattening parallel to (100). The K and Mg coordination polyhedra share one edge, the corners of which are the two water molecules of the asymmetric unit. The configuration of the K—O and Mg—O groups is shown in fig. 2; interatomic distances and bond angles are given in Tables IV and V.

TABLE IV.

K—O *and* Mg—O *bond lengths.*

ATOMS	Distance	$\sigma \times 10^3$	ATOMS	Distance	$\sigma \times 10^3$
K—O_{14}	2.948 Å	4 Å	Mg—O_{11}	2.055 Å	4 Å
O_4	2.909	4	O_{14}	2.053	6
O_{15}	2.980	5	O_{13}	2.072	4
O_{10}	2.923	3	O_7	2.094	4
			O_5	2.132	6
			O_{15}	2.074	3

TABLE V.

O—K—O *and* O—Mg—O *bond angles.*

Standard deviations are about 0.1° for the angles around K (0.3° for oxygens related by twofold axis) and 0.2° for the angles around Mg.

ATOMS	Angle	ATOMS	Angle
O_{14}—K—O_4	70.7°	O_{11}—Mg—O_{14}	81.2°
O_{14} O_{15}	58.9	O_{11} O_{13}	91.0
O_{14} O_{10}	114.4	O_{11} O_7	174.3
O_{14} O'_4	130.1	O_{11} O_5	91.3
O_{14} O'_{15}	98.9	O_{11} O_{15}	92.1
O_{14} O'_{10}	68.0	O_{14} O_{13}	89.4
O_{14} O'_{14}	151.3	O_{14} O_7	94.9
O_4 O_{15}	86.5	O_{14} O_5	172.6
O_4 O_{10}	67.6	O_{14} O_{15}	89.9
O_4 O'_{15}	169.5	O_{13} O_7	84.7
O_4 O'_{10}	106.5	O_{13} O_5	90.6
O_4 O'_4	101.5	O_{13} O_{15}	176.7
O_{15} O_{10}	70.0	O_7 O_5	92.5
O_{15} O'_{10}	116.9	O_7 O_{15}	92.2
O_{15} O'_{15}	86.5	O_5 O_{15}	90.5
O_{10} O'_{10}	171.2		

Kaliborite is built up of boron-oxygen chains running parallel to the *b* axis around the screw axis. Each chain element consists of three tetrahedrally coordinated and three triangularly coordinated boron atoms. The central tetrahedron BO_4 is linked by one side to a tetrahedron $BO_2(OH)_2$ and to a

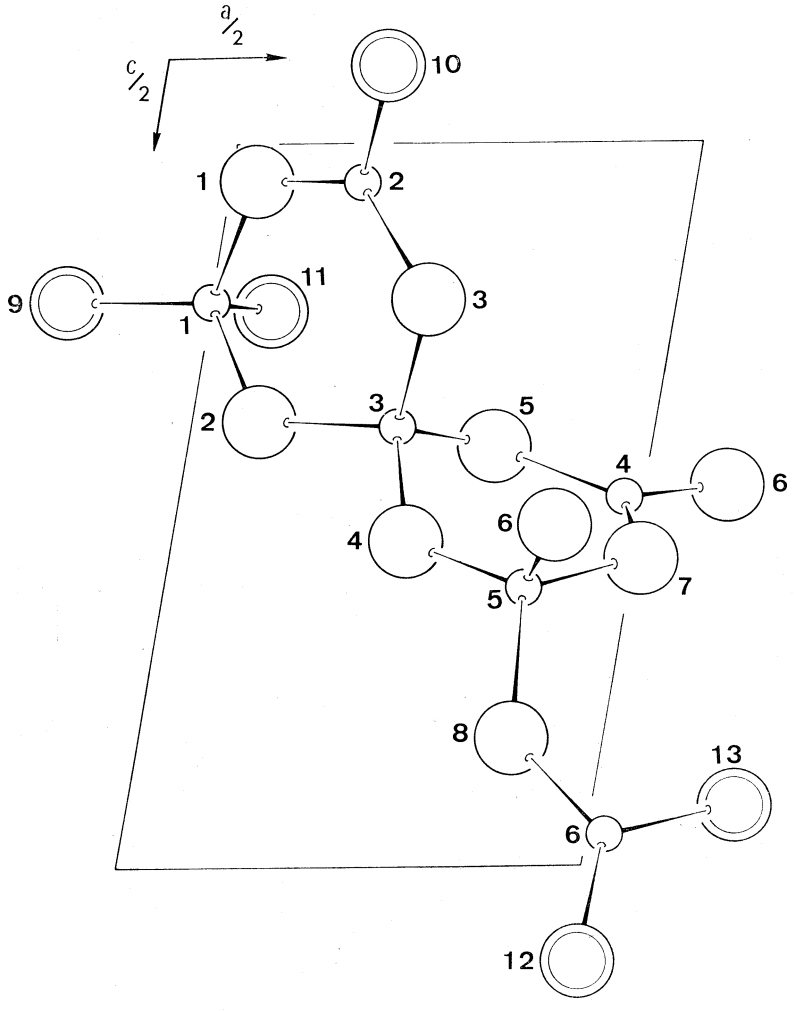

Fig. 3. – The basic borate repeat unit.

The small circles are boron atoms; other symbols as in fig. 2. The number designation is the same as in Table I.

triangle $BO_2(OH)$, to form a hexagonal ring; by the other side to a triangle BO_3 and to a tetrahedron BO_4, to form another ring having a triangle $BO(OH)_2$ as appendix linked to the latter BO_4 tetrahedron. This double ring repeat unit makes up a $[B_6O_8(OH)_5]_n^{-3n}$ polyion and is shown in fig. 3. With the exception of hydroxyls, the only non-ring oxygen O_6 alternatively shares a BO_4 tetrahedron of a polyion with a BO_3 triangle belonging to the following polyion to constitute an infinite chain in the *y* direction. Each polyion is

perpendicular to the chain direction. This configuration is reported in fig. 4. The individual distances, angles and standard deviations are given in Table VI.

Each Mg octahedron shares four of its oxygens with the boron atoms of a single chain, and it takes its place between two screw-axis related double rings. Potassium, irregularly cubic in its coordination, fills the voids between four boron-oxygen chains, linking them to each other. Besides, sharing one edge of its polyhedron with each surrounding Mg octahedron, it makes further bridges of groups Mg—2 H_2O—K—2 H_2O—Mg between pairs of neighbour boron–oxygen chains.

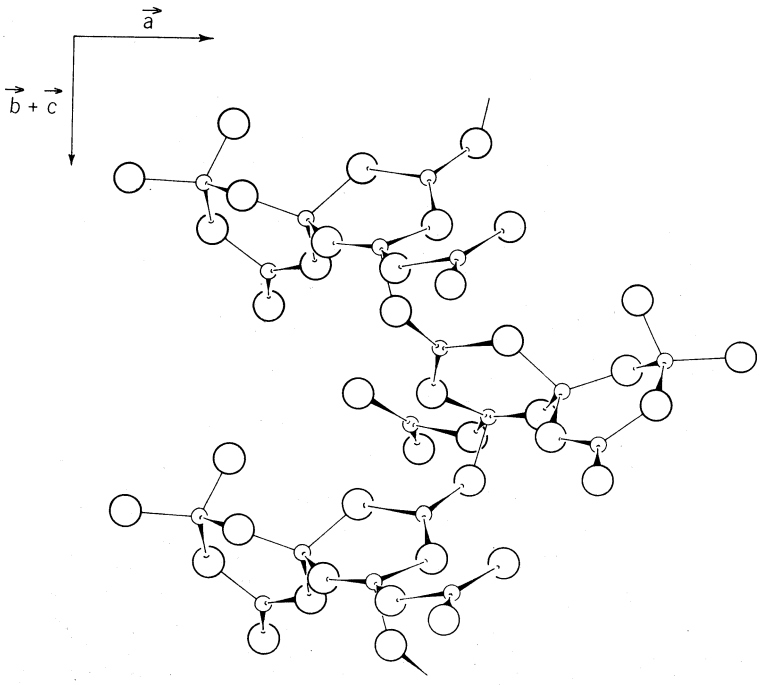

Fig. 4. – Perspective diagonal drawing of the boron–oxygen chains.

As we said above, the hydrogen atoms were located on the basis of the three-dimensional difference Fourier synthesis and of geometrical considerations. A later survey of O—O distances smaller than 3.3 Å revealed no more distances suitable for hydrogen bonds and confirmed all the hydrogen sites found in the difference map. Another argument which is consistent with hydrogen bridges localization is the fulfilment of Pauling's rule. Indeed, the balance of electrostatic charges is very satisfactory for all oxygens, as can be seen in Table VII. A further confirmation of these assumptions is the observance of Christ's [10] rule, which states "in the polyions of hydrated borates, those oxygens not shared by two borons always attach a proton and exist as hydroxyl groups". As a consequence of this rule it may be stated that the oxygens O_{14} and O_{15}, bound to K and Mg ions, belong to water molecules in respect to the electrostatic charges balance.

TABLE VI.

Boron–oxigen bond lengths and angles.

ATOMS	Distance	σ × 10³	ATOMS	Angle	σ
B_1—O_1	1.467 Å	7 Å	O_1—B_1—O_{11}	110.2°	.5°
O_{11}	1.476	20	O_1 O_2	109.4	.6
O_2	1.450	7	O_1 O_9	109.9	.4
O_9	1.458	6	O_{11} O_2	107.4	.4
			O_{11} O_9	109.0	.6
			O_2 O_9	110.9	.4
B_2—O_3	1.367	6			
O_1	1.365	9	O_3—B_2—O_1	122.3	.5
O_{10}	1.359	7	O_3 O_{10}	122.8	.7
			O_1 O_{10}	114.9	.5
			O_3—B_3—O_5	105.9	.4
B_3—O_3	1.504	7	O_3 O_4	113.8	.7
O_5	1.507	9	O_3 O_2	108.2	.4
O_4	1.427	8	O_5 O_4	110.6	.4
O_2	1.455	7	O_5 O_2	111.2	.6
			O_4 O_2	107.2	.4
B_4—O_5	1.380	6	O_5—B_4—O_6	123.5	.7
O_6	1.333	8	O_5 O_7	121.0	.6
O_7	1.388	9	O_6 O_7	115.5	.5
			O_8—B_5—O_4	107.5	.4
B_5—O_8	1.487	6	O_8 O_6	109.4	.6
O_4	1.421	7	O_8 O_7	109.5	.4
O_6	1.450	10	O_4 O_6	110.0	.5
O_7	1.512	8	O_4 O_7	111.8	.6
			O_6 O_7	108.6	.4
B_6—O_{12}	1.358	7	O_{12}—B_6—O_8	122.7	.6
O_8	1.347	7	O_{12} O_{13}	116.7	.6
O_{13}	1.376	8	O_8 O_{13}	120.6	.5

TABLE VII.

Electrostatic valency table.

ANION	Balancing cations	Charges of cations	Total charges surrounding anion
O_1	B_1, B_2, H_7, H_8	3/4 + 1 + 1/12 + 5/24	2.042
O_2	B_1, B_3, H_{10}	3/4 + 3/4 + 1/2	2.000
O_3(*)	B_2, B_3, H_6, H_9	1 + 3/4 + 1/4	2.000
O_4	K, B_3, B_5, H_5	1/8 + 3/4 + 3/4 + 7/24	1.917
O_5	Mg, B_3, B_4	1/3 + 3/4 + 1	2.083
O_6	B_4, B_5, H_4	1 + 3/4 + 1/4	2.000
O_7	Mg, B_4, B_5	1/3 + 3/4 + 1	2.083
O_8	B_5, B_6, H_2	3/4 + 1 + 1/8	1.875
O_9(OH)	B_1, H_1, H_3, H_5	3/4 + 1/3 + 5/24 + 17/24	2.000
O_{10}(OH).....	K, B_2, H_2	1/8 + 1 + 7/8	2.000
O_{11}(OH).....	Mg, B_1, H_7	1/3 + 3/4 + 11/12	2.000
O_{12}(OH).....	B_6, H_6	1 + 1	2.000
O_{13}(OH)	Mg, B_6, H_1	1/3 + 1 + 2/3	2.000
$O_{14}(H_2O)$	K, Mg, H_8, H_9	1/8 + 1/3 + 19/24 + 3/4	2.000
$O_{15}(H_2O)$	K, Mg, H_3, H_4	1/8 + 1/3 + 19/24 + 3/4	2.000

(*) The hydrogen H_6 though having the proper direction and distance for making a bridge O_{12}–$H_6\cdots O_3$, seems to give no charge contribution to O_3.

We think it of some interest to have found during the determination of this crystal structure an O···H····O bridge of 2.41 Å in which the hydrogen atom is most likely centered. In a recent work Ellison and Levy [11] established by neutron diffraction the existence of an O···H···O short bond (2.40 Å) with the hydrogen atom effectively centered. Earlier Zachariasen [12] in the determination of the crystal structure of the cubic metaboric acid found a distance of 2.49 Å between a hydroxyl and an oxygen. He reported an asymmetric configuration, but he pointed out that most likely there is a resonance, so that the H atom may occupy the middle position.

In order to have more information about the existence of the $O_2\cdots H_{10}\cdots O_2$ centered bridge, an investigation by the infrared technique was undertaken. Though up to now it has given no contradictory evidence with regard

to the centered hydrogen bond, at the same time it has not yet given any certain confirmation.

The hydrogen bonds are listed in Table VIII with the lengths between O—H and O—O.

Kaliborite (Palache et al. [13]) shows perfect {001} and {$\bar{1}$01}, and good {100} cleavages, all due to the feeble K—O bonds and to the hydrogen bridges between the boron–oxygen chains.

TABLE VIII.

Hydrogen bonds.

O—H···O	O—H	O···H	O—O
O_{13}—H_1···O_9	.99 Å	1.67 Å	2.65 Å
O_{10}—H_2···O_8	.97	1.72	2.68
O_{15}—H_3···O_9	.99	1.76	2.74
O_{15}—H_4···O_6	1.01	1.95	2.95
O_9—H_5···O_4	.97	2.07	3.04
O_{12}—H_6···O_3	.99	1.85	2.83
O_{11}—H_7···O_1	.99	1.78	2.75
O_{14}—H_8···O_1	.98	1.78	2.75
O_{14}—H_9···O_3	.97	1.86	2.83
O_2—H_{10}—O_2	1.206	1.206	2.412

On the basis of the results obtained from the present investigation of the crystal structure of kaliborite, the following considerations can be made with respect to its position in the systematic classification of borate minerals. Christ [10] with reference to the old formula postulated the existence of two kinds of polyions $[B_5O_6(OH)_4]^{-1}$ and $[B_3O_3(OH)_5]^{-2}$; in the asymmetric unit half a polyion of the former type and one of the latter would be present, totalling three triangles and two and a half tetrahedra. Still on the basis of the same formula Tennyson [14] placed kaliborite among the soroborates with heterogeneous structural motives.

At present kaliborite must be regarded as an inoborate with complexes made by three triangles and three tetrahedra. In Tennyson's classification it should be placed in the section of inoborates as a further type of boron complex, in addition to the seven complexes reported by this author.

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