THE CRYSTAL STRUCTURE OF $MgO.3\,B_2O_3.5\,H_2O$

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Dedicated to Professor S. Škramovský on the occasion of his 70th birthday.

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The crystal structure of $MgO.3\,B_2O_3.5\,H_2O$ was determined by the symbolic addition procedure from the three-dimensional photographic X-ray data. The space group is Pbca and the cell dimensions are: $a = 24{\cdot}376(4)$, $b = 7{\cdot}523(2)$, $c = 12{\cdot}627(3)$ Å. Isolated polyanions $(B_6O_7(OH)_6)^{2-}$ are connected by cations Mg^{2+} and by intermolecular hydrogen bonds of the type O—H···O. Polyanion $(B_6O_7(OH)_6)^{2-}$ is composed of three tetrahedral BO_4 groups and three planar BO_3 groups. These coordination polyhedra are joined by sharing corners so that one oxygen is common to all three tetrahedra BO_4. In the polyanion, six terminal oxygen atoms of the boron–oxygen triangles and tetrahedra attach hydroxyl groups. Three hydroxyl groups from three boron–oxygen tetrahedra of the same $(B_6O_7(OH)_6)^{2-}$ polyanion coordinate each Mg^{2+} cation. The octahedral environment around Mg^{2+} is completed by a hydroxyl group from another $(B_6O_7(OH)_6)^{2-}$ polyanion and by two water molecules. The mean interatomic distance B—O in the tetrahedral BO_4 groups is 1·48 Å, in the triangular BO_3 groups is 1·38 Å. The mean B—O distance of the oxygen atom linked to three boron atoms is 1·53 Å. The mean bond angle in the boron–oxygen tetrahedra is 109·5°, in the BO_3 groups is 120°. The Mg—O distances in the MgO_6 octahedron lie between 2·03 and 2·13 Å. The estimated standard deviations for bond lengths are 0·01—0·03 Å, for bond angles are 1°.

The polynuclear borate anions are composed of triangularly or tetrahedrally coordinated boron atoms combined in such a manner that the polyhedral groups have common vertices[1]. The existence of two different coordination numbers in many isopolyborates and the fact that hydroxyl ions may constitute part of the coordination group around a boron atom is associated with the complicated classification of the boron–oxygen compounds. Compared with the silicon–oxygen compounds, in which the silicon atoms are uniformly tetrahedrally coordinated with the oxygen atoms only, the isopolyborate structures cannot be classified in terms of their B : O ratio. However, in all the known borate structures the only cyclical group found is the six-membered ring with the three boron atoms alternating with the three oxygen atoms. In the hydrated borates, the positions in the coordination polyhedra around each boron atom which are not common to two polyhedral groups are predominantly occupied by hydroxyl ions.

Several considerations have been proposed about the ratio of tetrahedral boron to total boron. The postulate stated by Edwards and Ross[2] requires that the ratio of tetrahedral to total boron be

equivalent to the ratio of cation charge to total boron (2 : 6 in the $MgO.3\,B_2O_3.5\,H_2O$ structure). However, coordination of one oxygen to three boron atoms invalidates this postulate. Such a linkage was reported by Clark[3] for tunellite, $SrO.3\,B_2O_3.4\,H_2O$. The structure consists from infinite sheets of composition $n(B_6H_9(OH)_2)^{2n-}$ with a ratio of tetrahedral boron to total boron 3 : 6. The idealized view of the isolated polyanion of composition $(B_6O_7(OH)_6)^{2-}$ with all terminal oxygen atoms attaching the protons is shown in Fig. 2. Circles denote hydroxyl oxygens; all other oxygen atoms are linked to more than one boron. The elimination of a water molecule from two adjacent OH groups of each polyanion $(B_6O_7(OH)_6)^{2-}$ would lead to a complex anion composed of infinite chains of composition $n(B_6O_8(OH)_4)^{2n-}$. The following scheme can be designed in relation to the stepwise dehydration of $(B_6O_7(OH)_6)^{2-}$:

$$(B_6O_7(OH)_6)^{2-} \xrightarrow[-H_2O]{} (B_6O_8(OH)_4)^{2-} \xrightarrow[-H_2O]{} (B_6O_9(OH)_2)^{2-} \xrightarrow[-H_2O]{} (B_6O_{10})^{2-}$$

This scheme shows that a compound of composition $MgO.3\,B_2O_3.5\,H_2O$ might represent at least four different structural types: $Mg(B_6O_7(OH)_6).2\,H_2O$ (*I*), $Mg(B_6O_8(OH)_4).3\,H_2O$ (*II*), $Mg(B_6O_9(OH)_2).4\,H_2O$ (*III*) or $Mg(B_6O_{10}).5\,H_2O$ (*IV*).

The purpose of this work was the explanation of the structural features of the hydrated magnesium triborate $MgO.B_2O_3.5\,H_2O$.

EXPERIMENTAL

Crystals of the composition $MgO.3\,B_2O_3.5\,H_2O$ occur as mineral aksait[4]. The compound[5] has been prepared by Lehmann and Rietz by fusing the stoichiometric amounts of MgO (1 g) and H_3BO_3 (9·3 g) with $MgCl_2.6\,H_2O$ (100 g) in a sealed glass tube and annealing the mixture for eight weeks at 120°C. The clear prismatic crystals grew from melt. They could be separated from magnesium chloride due to different solubility of both compouds in water. The compound also could be separated as a stable phase from the heated mixture of MgO (1 g), H_3BO_3 (9·3 g), $MgCl_2$ (50 g) and H_2O (16·5 g) after annealing for one half year in a sealed glass tube at 100°C.

Crystals of $MgO.3B_2O_3.5\,H_2O$ are orthorhombic with the following cell dimensions: $a = 24{\cdot}376(4)$, $b = 7{\cdot}523(2)$, $c = 12{\cdot}627(3)$ Å.

Space group symmetry is Pbca. Cell content $Z = 8(MgO.3\,B_2O_3.5\,H_2O)$. The intensities of reflections were measured from the equiinclination photographs using microdensitometer Zeiss made in the Institute of Physical Chemistry DAW, by means of the apparatus of our construction. The intensities of 706 independent reflections were collected for the layers h01

TABLE I

The Statistical Distribution and Some Statistics of the E_s Values

Value	Observed	Calculated	Value	Observed	Calculated
$\|E_s\| > 3$	0·38%	0·27%	$\langle\|E_s\|\rangle$	0·729	0·798
$\|E_s\| > 2$	5·79%	4·55%	$\langle\|E_s^2 - 1\|\rangle$	1·026	0·968
$\|E_s\| > 1$	31·03%	31·73%	$\langle\|E_s^2\|\rangle$	0·922	1·000

through h41. In the same region of the reciprocal lattice, where observed reflections have been recorded, 653 unobserved reflections were found. However, high number of unobserved reflections indicated that only a part of these reflections are in fact of zero value. The rest of unobserved reflections are weak spectra with unmeasurable intensities owing to the low reflection power of the small crystal. CuKα radiation has been used. No absorption corrections were applied as X-ray absorption was negligible. Lorentz and polarization corrections were made in the usual way with a IBM 360/65 computer. The different layers of the b — axis data were brought to an approximately common scale by comparison with 0k1 and hk0 spectra.

Structure Determination and Refinement

The structure was solved using the symbolic addition method[6]. Normalized structure factors, E_s, were calculated by the formula

$$E_s = F_s/(\varepsilon|F_s^2|^{1/2}), \qquad (1)$$

where F_s is the structure factor on the absolute scale, $|F_s^2|$ is the average of absolute intensities, ε is a factor correcting for space group dependent extinctions and s represents triplet of integers hkl. The structure factors are put on absolute scale using Wilson's method[7]. The statistical distri-

TABLE II

The Basic Set in the Symbolic Addition Procedure

s	E_s	Symbol	s	E_s	Symbol
20 1 3	3·866	*A*	6 3 11	2·853	*F*
9 1 4	3·184	*B*	5 4 8	2·807	*G*
23 4 3	3·063	*C*	22 1 3	2·797	*H*
5 4 7	3·052	*D*	15 4 4	2·757	*I*
10 3 6	3·008	*E*	13 2 7	2·751	*J*

TABLE III

Frequency of the Relative Peak Heights D_{max} Larger than a Threshold Value D_t in the Independent Part of the $D(xyz)$ Function

D_t	No of peaks with $D_{max} > D_t$	D_t	No of peaks with $D_{max} > D_t$
600	1	300	18
500	5	250	20
450	8	200	29
400	14	150	63
350	16	100	123

bution and some statistics of the E_s values are given in Table I together with the theoretical values. The structure determination was done automatically using the program system Gaasa[8,9]. Ten of the greatest normalized structure factors were chosen by the program as the basic set. The symbols for their signs are given in Table II. The probability limit for accepting the indicated combinations of symbols for new phases was chosen 0·95. Equations between symbols were solved if their probabilities were greater than 0·99. After ten cycles of the symbolic addition procedure it was seen that the signs of 198 structure factors were determined by the three original symbols H, I, J. Now, the value +1 was assigned to all three symbols. After a few cycles of refinement using the Σ_2-relation[10]:

$$sgE_s = sg(\Sigma_t E_t E_{s-t}) \qquad (2)$$

where $sg(\ldots)$ means "sign of", 208 signs were determined, 95 of which were positive, 101 negative and 12 were inconsistent, *i.e.*, they had probabilities between 0·95 and 0·05. Symbols s and t represent integers h_1, k_1, l_1 and h_2, k_2, l_2, respectively.

An E-map was calculated on the 196 consistent signs. Table III gives frequency of the relative peak heights D_{max} larger than a treshold value D_t in the independent part of the $D(xyz)$ function. The full line in Fig. 1 represents this peak density distribution, the dotted line is related to the increase Δ of number of peaks with D_{max} depending on D_t. In case of correct solution, a minimum or a "plateau" in Δ distribution is expected to indicate the significant D_t value at the correct number of atoms. The borate compound contains 22 "heavy" atoms in the formula unit (1 Mg + 15 O + 6 B). The plateau in Δ distribution ends at D_t around 240 corresponding to the correct number of atoms. In fact, all peaks with $D_{max} > 240$ gave the correct positions of atoms. After the final least squares refinement it was confirmed that all the 196 signs were correctly determined.

The coordinates of all 22 atoms as obtained from the E-map were subjected to the least squares refinement on an IBM 360/65 computer using the block diagonal approximation. The scattering factors for the atoms were taken from *International Tables for X-ray Crystallography*[11]. The coordinates and the individual isotropic temperature factors of 22 atoms, together with the scale factor were refined using the weighting scheme proposed by Hughes[12]. The unobserved reflections were not included into the refinement. During 5 cycles of refinement the initial relia-

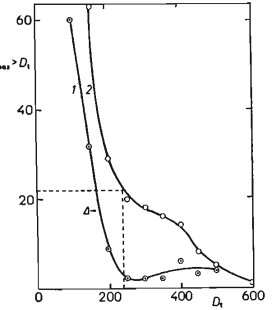

FIG. 1

Frequency of the Relative Electron Density Peak Heights D_{max}

The full line represents the distribution function for $D_{max} > D_t$, where D_t is a selected threshold value. The dotted line is related to the increase Δ of number of peaks with thevalues $D_{max} > D_t$.

bility index $R = \sum||F_o| - |F_c||/\sum|F_o|$ decreased from 23% to 10·9% for 706 observed reflections. The over — all R — factor for 1359 reflections was 21·7%. Final atomic coordinates and corresponding individual anisotropic temperature factors are given in Table IV. Observed and calculated structure factors obtained from the final parameters in Table IV are given in Table V. The unobserved reflections are indicated by asterisks. One third of average intensity of 20 weakest reflections has been ascribed to each of unobserved reflections during calculations.

DESCRIPTION OF THE STRUCTURE AND DISCUSSION

The crystal structure of $MgO.3\,B_2O_3.5\,H_2O$ consists of isolated polyanions $(B_6O_7.(OH)_6)^{2-}$ connected by cations Mg^{2+} and intermolecular hydrogen bonds of the type O—H···O into a threedimensional network. Polyanion $(B_6O_7(OH)_6)^{2-}$ is composed of three tetrahedral BO_4 groups and three planar BO_3 triangles (Fig. 2). These coordination polyhedra are joined by sharing corners so that one oxygen is common to all three tetrahedra BO_4. Similar polyanion has been found in the crystal structure of the mineral tunellite $SrO.3\,B_2O_3.4\,H_2O$, but in tunellite individual polyanions are linked at four corners to form infinite sheets of composition[3] $n(B_6O_9(OH)_2)^{2n-}$. In the polyanion $(B_6O_7(OH)_6)^{2-}$ all six terminal oxygens of the boron–oxygen tetrahedra and triangles attach hydroxyl groups. Three hydroxyl groups from three boron–oxygen tetrahedra of the same $(B_6O_7(OH)_6)^{2-}$polyanion coordinate each Mg^{2+} cation. The octahedral environment around Mg^{2+} is completed by a hydroxyl group from another polyanion and by two water molecules (Fig. 3).

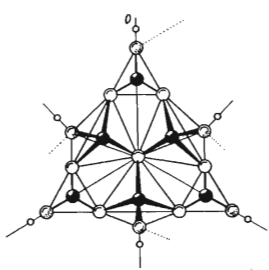

FIG. 2

Schematic Representation of Atomic Arrangement in the Polyanion $(B_6O_7(OH)_6{}^{-2}$.

The boron atoms are indicated by filled circles, the oxygen atoms by open circles. Oxygens of the hydroxyl groups are represented by dotted circles.

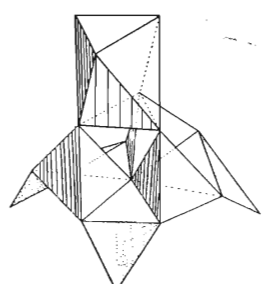

FIG. 3

Arrangement of the Polyhedra BO_3, BO_4 and MgO_6 in the Crystal Structure of $Mg(B_6.O_7(OH)_6).2\,H_2O$

The bond lengths and angles obtained from the coordinates in Table IV are given in Table VI together with their standard deviations. The labelling of atoms is in agreement with Fig. 4. The average of the boron–oxygen bond lengths in the BO_4 tetrahedra is 1·48 Å as opposed to 1·38 Å for the BO_3 triangles. This compares well with the corresponding mean bond lengths observed in potassium diborate tetrahydrate $K_2O.2\,B_2O_3.4\,H_2O$ (1·480 Å in BO_4, 1·368 Å in BO_3) (ref.[13]), in cadmium diborate, $CdO.2\,B_2O_3$ (1·47 Å in BO_4, 1·37 Å in BO_3) (ref.[14]), in lithium diborate $Li_2O.2\,B_3O_3$ (1·477 Å in BO_4, 1·373 Å in BO_3) (ref.[15]) and in numerous other borate structures. The higher order of the boron-oxygen bonds in BO_3 probable influences the significant bond length reduction compared with the B—O distance in BO_4. The mean bond angle in the boron–oxygen tetrahedra is 109·5°, in the BO_3 triangles is 120°. One of the factors determining the coordination number of boron in the polyborates may be assigned to considerable energetic stability of the six-membered boron-oxygen rings constructed from two BO_4 tetrahedra and one BO_3 triangle. The mean O—B—O bond angle in the sixmembered boron–oxygen rings is 113°, the mean B—O—B bond angle is 120°.

The mean B—O distance of the oxygen atom O(6) linked to three boron atoms is 1·53 Å, slightly longer than the average tetrahedral B—O distance. The average of three B—O—B bond angles around this oxygen is 117·3°. These values can be compared with the corresponding average values in the tunellite structure: 1·51 Å and 119·7°. respectively[3]. The Mg—O distances in the MgO_6 octahedron lie between 2·03 and 2·13 Å, the O—Mg—O bond angles being: 83°, 87°, 87°, 88°, 89°, 89°, 91°, 93°, 91°, 94°, 96°. The octahedral coordination around Mg^{2+} may be suggested as a tri-

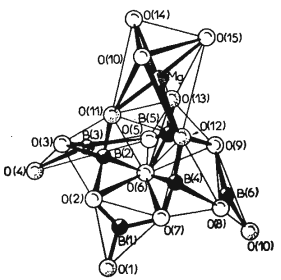

FIG. 4

Atomic Arrangement in the Structure of Mg. $.(B_6O_7(OH)_6).2\,H_2O$

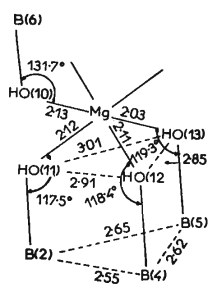

FIG. 5

Coordination of the Mg^{2+} Cation by the Hydroxyl Groups

gonal antiprism. One oxygen triangle of the polyhedron is created by three hydroxyl groups of the same polyanion (Fig. 5). The average oxygen–oxygen separation is 2·92 Å, in the second oxygen triangle of the antiprism is 2·94 Å. The three hydroxyl groups of the polyanion $(B_6O_7(OH)_6)^{2-}$ coordinate the Mg^{2+} cation in such way that the three B—O bonds are oriented approximately parallelly. The oxygen base of the B_3O_3 prism is extended owing to repulsive forces in comparison with the boron base. The average boron–boron separation is 2·61 Å (Fig. 5). The three Mg—O—B bond angles are: 117·5°, 118·4° and 119·5°. The Mg—O(10)—B(6) bond angle to the hydroxyl group of the neighbouring polyanion is significantly larger: 131·7°.

TABLE IV

Final Atomic Coordinates and the Corresponding Anisotropic Thermal Vibration Matrix Elements (Å^2), with Standard Deviations

A thermal factor of the form $\exp[-2\pi(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + \ldots + 2U_{12}hka^*b^* + \ldots)]$ was employed.

Atom	$x.10^4$	$y.10^4$	$z.10^4$	$U_{11}.10^4$	$U_{22}.10^4$	$U_{33}.10^4$	$U_{12}.10^4$	$U_{13}.10^4$	$U_{23}.10^4$
Mg	1 284	530	4 315	100	169	109	185	−57	−50
σ	(2)	(12)	(4)	(1)	(26)	(3)	(9)	(3)	(17)
0(1)	5 048	1 523	2 869	122	233	286	16	−122	55
σ	(5)	(24)	(10)	(2)	(49)	(8)	(17)	(7)	(34)
0(2)	4 130	963	2 328	158	362	160	−507	125	10
σ	(5)	(24)	(9)	(2)	(51)	(7)	(18)	(6)	(35)
0(3)	3 178	1 681	2 149	170	235	152	204	10	272
σ	(5)	(23)	(9)	(2)	(48)	(6)	(17)	(6)	(32)
0(4)	2 239	2 528	1 985	83	440	280	329	164	280
σ	(4)	(26)	(9)	(1)	(56)	(8)	(18)	(6)	(41)
0(5)	2 851	3 215	596	114	181	200	308	5	82
σ	(4)	(24)	(9)	(2)	(48)	(7)	(16)	(6)	(34)
0(6)	3 813	2 518	767	213	275	135	−148	215	549
σ	(5)	(24)	(9)	(2)	(49)	(6)	(19)	(6)	(35)
0(7)	4 783	2 396	1 186	140	106	141	18	17	208
σ	(4)	(23)	(8)	(2)	(47)	(7)	(19)	(6)	(34)
0(8)	4 467	4 167	−251	62	232	233	103	−102	67
σ	(5)	(23)	(9)	(2)	(50)	(7)	(16)	(6)	(35)
0(9)	3 507	4 945	−337	182	1	213	−352	−115	388
σ	(5)	(22)	(9)	(2)	(47)	(7)	(17)	(6)	(32)

TABLE IV
(*continued*)

Atom	$x.10^4$	$y.10^4$	$z.10^4$	$U_{11}.10^4$	$U_{22}.10^4$	$U_{33}.10^4$	$U_{12}.10^4$	$U_{13}.10^4$	$U_{23}.10^4$
O(10)	4 180	7 081	−748	151	74	155	337	14	522
σ	(5)	(23)	(9)	(2)	(39)	(7)	(17)	(6)	(34)
O(11)	1 410	4 446	973	279	55	248	74	178	−185
σ	(5)	(23)	(9)	(2)	(49)	(8)	(18)	(7)	(32)
O(12)	4 432	4 037	4 610	164	50	231	36	−201	−29
σ	(5)	(23)	(9)	(2)	(48)	(7)	(16)	(6)	(32)
O(13)	3 323	3 170	4 217	203	11	372	−119	163	−217
σ	(5)	(24)	(10)	(2)	(46)	(9)	(18)	(7)	(35)
O(14)	1 992	2 049	4 146	123	69	125	1	−83	−157
σ	(4)	(23)	(8)	(2)	(45)	(6)	(16)	(6)	(31)
O(15)	1 129	500	2 649	167	326	337	−163	38	−142
σ	(5)	(25)	(10)	(2)	(54)	(8)	(18)	(7)	(40)
B(1)	4 628	1 584	2 152	122	1 073	74	−297	−361	−75
σ	(8)	(54)	(16)	(3)	(135)	(10)	(33)	(9)	(67)
B(2)	3 680	1 100	1 546	70	122	167	527	58	8
σ	(8)	(43)	(15)	(2)	(89)	(11)	(28)	(8)	(57)
B(3)	2 765	2 470	1 585	36	387	165	−336	−124	−516
σ	(7)	(44)	(14)	(3)	(8)	(9)	(30)	(9)	(54)
B(4)	4 395	2 482	321	203	103	69	−154	−120	119
σ	(8)	(44)	(14)	(3)	(88)	(8)	(29)	(10)	(51)
B(5)	3 360	3 232	−4	47	114	422	1 182	230	−146
σ	(7)	(40)	(18)	(2)	(80)	(15)	(29)	(9)	(68)
B(6)	4 054	5 322	−432	380	113	142	304	117	179
σ	(10)	(49)	(16)	(4)	(106)	(13)	(36)	(11)	(58)

The hydroxyl groups could not be detected directly from the X-ray data, since the errors in the final difference synthesis were of the same order as the expected heights of the electron densities at the positions of the hydrogen atoms. For allocation of protons, a procedure based on principle of local neutralization of charge was used[16]. This procedure allows to recognize O^{2-}, OH^- and H_2O in crystal structures determined by X-rays.

TABLE V
Observed and Calculated Structure Factors

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$	L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$	L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	0	0	L		5	0	L		10	0	L
2	132·38		173·48	2	52·69		−55·90	0	8·21	*	9·41
4	99·19		115·30	4	36·56		24·76	2	175·27		−183·60
6	220·37		−242·52	6	40·72		−41·84	4	9·61	*	−17·20
8	121·71		−115·18	8	41·55		−32·61	6	110·36		−98·82
10	12·94	*	−3·51	10	13·16	*	0·91	8	21·78		−20·88
12	13·53	*	−5·14	12	26·85		29·37	10	13·55	*	8·73
14	49·29		45·91	14	11·21	*	17·32	12	12·85	*	20·85
16	35·60		−39·06					14	31·03		35·20
					6	0	L				
	1	0	L						11	0	L
				0	51·56		31·53				
2	26·73		−37·53	2	6·70	*	−3·49	2	110·57		−101·98
4	194·43		217·54	4	95·82		−60·31	4	62·70		−54·06
6	9·07	*	1·04	6	9·83	*	0·11	6	32·21		−26·27
8	27·26		25·21	8	23·39		25·60	8	28·64		−27·45
10	25·90		−27·98	10	49·57		78·72	10	13·58	*	−13·70
12	13·52	*	7·76	12	13·37	*	−13·14	12	12·62	*	−16·02
14	11·60	*	8·97	14	29·16		20·49	14	16·23		14·47
16	6·46	*	9·42								
					7	0	L		12	0	L
	2	0	L								
				2	42·95		24·46	0	110·08		−94·22
0	39·21		38·90	4	88·89		79·75	2	101·98		−86·67
2	5·11	*	7·08	6	20·18		−15·58	4	59·31		50·10
4	83·07		84·99	8	50·45		43·28	6	33·24		34·55
6	9·14	*	8·04	10	44·22		−38·78	8	70·19		70·88
8	74·15		68·60	12	29·71		36·33	10	33·24		39·36
10	18·36		−19·38	14	40·38		38·71	12	12·33	*	−16·51
12	63·38		−52·89								
14	23·11		−23·25		8	0	L		13	0	L
	3	0	L	0	31·22		20·31	2	10·10	*	−2·44
				2	104·59		−96·92	4	10·94	*	−6·16
2	81·18		81·82	4	85·81		−85·63	6	46·93		−45·47
4	95·06		90·75	6	56·86		−53·58	8	18·72		20·29
6	145·70		161·58	8	27·07		−30·61	10	63·37		−50·48
8	80·43		73·72	10	13·42	*	−14·20	12	11·99	*	5·61
10	13·03	*	−24·41	12	26·35		27·21				
12	19·09		−22·20	14	27·83		34·98		14	0	L
14	30·36		−34·90								
					9	0	L	0	117·90		99·22
	4	0	L					2	89·49		79·85
				2	121·59		−112·98	4	11·40	*	−20·93
0	175·44		−187·37	4	39·01		−30·96	6	12·47	*	8·16
2	125·79		−154·46	6	103·72		106·58	8	56·88		−53·12
4	24·97		−17·93	8	46·16		42·25	10	13·39	*	−24·50
6	142·03		147·80	10	19·08		20·48	12	11·58	*	8·36
8	32·18		32·74	12	31·92		−37·19				
10	50·69		49·45	14	10·19	*	−12·02				
12	81·92		−74·94								
14	11·36	*	−19·33								

TABLE V
(*continued*)

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	15	0	L
2	73·02		−69·07
4	11·84	*	7·84
6	12·81	*	−13·64
8	13·53	*	−0·87
10	13·20	*	−24·10
12	11·11	*	5·50
	16	0	L
0	11·40	*	25·94
2	28·51		28·65
4	12·29	*	16·75
6	26·20		−33·87
8	33·26		−32·92
10	12·93	*	−10·93
12	10·55	*	13·46
	17	0	L
2	12·13	*	17·50
4	35·89		28·71
6	26·69		28·77
8	13·55	*	−18·47
10	68·84		−70·18
12	34·33		−30·62
	18	0	L
0	60·72		52·41
2	12·58	*	8·97
4	82·50		83·49
6	58·89		61·74
8	13·42	*	−6·80
10	12·12	*	−4·82
12	9·16	*	−16·70
	19	0	L
2	50·25		−45·98
4	13·33	*	−12·17
6	27·16		−22·46
8	26·37		−28·94
10	11·57	*	23·03
12	8·27	*	−10·64
	20	0	L
0	63·22		−53·84
2	35·16		35·54
4	67·57		59·95
6	33·15		30·36
8	31·42		38·04
10	10·91	*	1·93
12	25·72		−27·47
	21	0	L
2	30·19		24·17
4	67·90		−69·40
6	35·33		40·74
8	12·35	*	10·06
10	43·06		46·71
	22	0	L
0	57·57		51·68
2	57·62		−54·36
4	27·01		22·00
6	13·03	*	8·93
8	35·22		−38·46
10	9·25	*	−10·50
	23	0	L
2	40·51		−39·91
4	35·10		39·28
6	12·55	*	10·06
8	59·26		56·80
10	11·55		15·65
	24	0	L
0	13·35	*	34·97
2	13·25	*	16·48
4	12·86	*	1·45
6	60·80		−61·82
8	35·10		−38·10
	25	0	L
2	44·39		39·73
4	49·12		45·18
	26	0	L
0	12·39	*	−27·16
2	60·98		−66·73
4	11·54	*	21·33
	27	0	L
2	39·50		34·93
4	43·81		42·95
	28	0	L
0	53·55		−50·71
2	58·09		−60·26
4	9·54	*	−0·09
	30	0	L
0	16·42		17·71
2	7·80	*	3·36
4	17·72		−21·90
	1	1	L
1	25·93		−20·80
2	111·38		139·10
3	75·15		84·56
4	68·13		63·41
5	64·99		57·91
6	7·54	*	−12·62
7	75·04		76·69
8	46·17		−45·56
9	10·03	*	−6·35
10	10·70	*	−14·78
11	11·10	*	8·83
12	35·04		−37·76
13	40·76		−41·45
14	9·41	*	7·85
15	7·74	*	−3·69
16	4·94	*	7·83
	2	1	L
0	114·71		178·62
1	31·07		40·24
2	29·45		29·60
3	5·12	*	−6·90
4	53·53		55·08
5	33·17		−25·10
6	31·33		24·36
7	29·23		24·28
8	22·72		23·01
9	48·28		50·61
10	21·45		−11·02
11	58·79		62·84
12	11·07	*	−18·79
13	25·71		26·26
14	9·37	*	0·63
15	7·68	*	8·49
16	4·82	*	−22·69

TABLE V
(*continued*)

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	3	1	L
1	58·75		57·25
2	39·18		−39·42
3	96·66		−101·24
4	16·09		−14·20
5	51·94		−50·56
6	104·62		−107·31
7	85·62		−84·02
8	9·34	*	8·68
9	22·63		22·91
10	26·35		26·28
11	27·24		21·07
12	45·58		49·90
13	40·48		44·06
14	9·30	*	−20·53
15	7·59	*	12·46
16	11·25		−16·90
	4	1	L
0	35·70		28·30
1	4·30	*	17·68
2	4·85	*	6·19
3	31·29		28·25
4	18·79		17·81
5	28·11		27·53
6	20·69		20·77
7	8·63	*	−21·14
8	34·03		30·38
9	10·20	*	−12·95
10	10·81	*	12·58
11	22·27		−20·01
12	33·08		−27·09
13	10·39	*	4·01
14	37·95		−37·91
15	7·46	*	8·10
	5	1	L
1	35·25		32·39
2	15·61		−8·94
3	27·22		−21·70
4	19·45		13·37
5	26·00		22·97
6	67·70		−66·11
7	51·13		−48·23
8	33·11		31·56
9	10·29	*	0·04
10	26·61		24·60
11	22·29		−24·92
12	10·99	*	14·16
13	34·16		35·40
14	9·08	*	−10·13

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	6	1	L
0	93·09		100·67
1	31·61		26·19
2	54·04		50·74
3	80·56		−80·76
4	45·69		44·23
5	38·60		38·81
6	8·17	*	11·56
7	25·27		−21·17
8	23·75		26·04
9	20·79		−19·87
10	39·40		−38·30
11	22·31		−19·53
12	47·66		−48·12
13	20·38		−17·33
14	23·60		−13·84
	7	1	L
1	68·50		−70·61
2	76·27		−65·63
3	6·43	*	9·91
4	70·51		−63·30
5	109·06		109·80
6	36·54		−29·10
7	31·60		28·52
8	42·96		44·17
9	39·34		37·71
10	10·99	*	17·57
11	33·45		−31·50
12	24·28		24·43
13	20·10		−25·22
14	8·72	*	−28·62
	8	1	L
0	158·31		142·91
1	28·42		20·13
2	24·58		19·82
3	38·34		32·47
4	17·93		−16·62
5	7·94	*	6·17
6	8·62	*	−13·09
7	9·33	*	15·36
8	10·03	*	0·59
9	10·64	*	15·76
10	61·55		26·17
12	10·76	*	−17·10
13	9·88	*	10·46
14	8·49	*	5·92

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	9	1	L
1	25·12		−24·42
2	104·45		105·16
3	44·58		−39·06
4	180·42		176·20
5	16·47		−16·85
6	36·62		38·29
7	21·37		−18·82
8	25·02		25·89
9	10·77	*	2·59
10	49·67		−51·00
11	11·10		6·12
12	10·63	*	−22·58
13	19·35		−23·70
14	27·26		19·85
	10	1	L
0	28·99		−18·48
1	76·67		−70·47
2	78·93		72·60
3	19·87		19·03
4	113·22		−115·89
5	100·00		−98·22
6	20·48		−13·11
7	9·80	*	7·81
8	10·40	*	−9·85
9	15·40		11·61
10	40·17		−39·14
11	34·89		37·72
12	10·47	*	17·35
13	29·82		23·24
14	7·90	7	12·68
	11	1	L
1	60·54		51·79
2	7·56	*	0·91
3	24·97		−20·78
4	8·34	*	−14·63
5	58·82		−61·22
6	32·73		29·79
7	38·89		−41·48
8	28·01		−29·88
9	39·65		−36·70
10	29·51		23·84
11	18·94		10·56
12	25·16		23·50
13	9·14	*	13·37
14	7·52	*	−5·37

TABLE V
(*continued*)

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	12	1	L
0	20·39		15·62
1	30·11		−27·29
2	94·01		−84·66
3	8·29	*	−7·89
4	35·90		−41·28
5	9·20	*	−1·99
6	33·75		−30·34
7	34·11		19·58
8	10·76	*	7·83
9	24·79		−20·61
10	49·78		59·13
11	10·80		−11·96
12	10·03	*	−19·32
13	34·12		−35·39
14	15·82		18·27
	13	1	L
1	71·10		65·42
2	87·65		−72·70
3	8·69	*	11·49
4	27·24		28·50
5	39·33		33·21
6	38·87		−40·14
7	51·53		−49·30
8	80·25		84·51
9	11·14	*	−8·63
10	11·07	*	−3·98
11	21·23		−21·02
12	9·74	*	−1·76
13	11·91		13·23
	14	1	L
0	8·59	*	−1·30
1	56·04		50·78
2	8·82	*	−3·41
3	48·12		44·66
4	59·04		−59·74
5	84·38		90·75
6	41·28		−32·88
7	10·74	*	1·97
8	46·87		41·07
9	22·31		−9·42
10	10·95	*	13·74
11	32·82		−31·98
12	32·53		31·30
13	19·54		−16·42

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	15	1	L
1	69·76		−68·72
2	36·96		38·41
3	34·23		−31·28
4	63·65		70·01
5	53·00		49·85
6	10·59	*	−1·55
7	10·92	*	7·31
8	11·13	*	−8·07
9	47·14		47·45
10	50·57		−54·34
11	10·08	*	−10·47
12	8·99	*	−3·00
13	7·46	*	−12·28
	16	1	L
0	96·93		−100·62
1	38·03		38·58
2	74·14		−67·35
3	9·88	*	−0·95
4	10·17	*	2·08
5	10·50	*	7·15
6	45·89		48·63
7	24·73		−30·44
8	31·55		36·55
9	11·01	*	17·68
10	25·82		−26·20
11	9·72	*	−5·36
12	28·26		−36·47
13	16·75		19·24
	17	1	L
1	45·45		−45·03
2	51·22		−50·08
3	44·65		−41·70
4	10·49	*	−20·43
5	10·76	*	0·09
6	11·00	*	−17·93
7	11·14	*	20·85
8	19·24		23·69
9	41·93		45·37
10	10·23	*	−14·10
11	22·76		22·39
12	7·98	*	−4·85
	18	1	L
0	10·26	*	14·33
1	35·67		−30·29
2	10·41	*	5·82

L	$\lvert F_c\rvert$	sg	$\lvert F_c\rvert$
	18	1	L
3	46·08		−42·73
4	35·73		−36·07
5	66·73		−63·41
6	11·12	*	8·42
7	11·15	*	−15·39
8	10·98	*	15·56
9	10·57	*	−5·58
10	9·84	*	10·55
11	8·79	*	3·13
12	7·35	*	5·04
	19	1	L
1	69·71		77·52
2	64·30		−59·95
3	10·84	*	10·66
4	10·99	*	3·26
5	11·11	*	17·18
6	24·94		−24·78
7	27·10		−31·04
8	15·23		20·71
9	10·22	*	−5·56
10	9·38	*	−4·44
11	8·21	*	−4·90
12	14·71		12·64
	20	1	L
0	64·36		60·36
1	69·79		−72·73
2	112·31		120·65
3	11·04	*	21·64
4	29·42		−31·69
5	38·64		−38·34
6	24·80		−23·05
7	10·88	*	16·49
8	23·38		−23·42
9	9·78	*	−9·82
10	8·83	*	−5·56
11	13·04		−4·94
12	19·30		21·97
	21	1	L
1	19·19		20·05
2	41·58		−38·79
3	11·14	*	18·94
4	35·26		−32·63
5	11·09	*	17·36
6	10·92	*	−5·22
7	10·59	*	−6·51

TABLE V
(*continued*)

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	21	1	L
8	10·04	*	−11·12
9	18·51		−13·18
10	8·18	*	−0·45
11	17·74		−13·05
	22	1	L
0	24·94		−29·08
1	11·15	*	1·32
2	11·15	*	19·46
3	67·71		66·74
5	36·18		32·63
6	23·77		19·60
7	10·18	*	16·25
8	13·47		−4·14
9	8·63	*	−9·64
10	7·42	*	−8·07
	23	1	L
1	11·10	*	−29·58
2	11·06	*	−5·26
3	10·98	*	−4·74
4	10·84	*	8·12
5	10·60	*	6·73
6	10·22	*	19·47
7	9·66	*	10·67
8	17·81		17·85
9	13·66		13·54
10	6·49	*	−14·75
	24	1	L
0	10·92	*	31·48
1	10·89	*	17·03
2	10·83	*	16·91
3	10·70	*	−18·61
4	10·48	*	3·60
5	10·15	*	−5·07
6	9·86	*	−2·48
7	9·03	*	−10·49
8	8·17	*	8·86
9	17·16		19·08
10	5·18	*	−29·95
	25	1	L
1	10·54	*	2·77
2	10·44	*	7·46
3	10·26	*	−9·44
4	24·46		28·51
5	9·58	*	2·24
6	20·18		16·93
7	16·56		13·72
8	25·25		−23·33
9	5·85	*	11·33
	26	1	L
0	20·16		−17·18
1	10·04		−13·11
2	9·91	*	−1·89
3	33·56		−37·11
4	9·35	*	21·07
5	8·88	*	−13·21
6	8·24	*	18·78
7	7·38	*	−2·03
8	18·54		18·59
	27	1	L
1	28·17		31·78
2	9·24	*	−9·37
3	8·98	*	4·05
4	8·58	*	7·54
5	22·74		−24·69
6	7·30	*	9·17
7	6·25	*	−20·93
	28	1	L
0	22·90		−29·47
1	8·60	*	−0·19
2	8·42	*	−4·36
3	24·35		25·78
4	7·66	*	−12·64
5	19·85		25·00
6	6·09	*	−12·89
7	10·82		5·32
	29	1	L
2	21·03		−22·77
3	7·07	*	−9·90
4	15·95		−15·16
	30	1	L
0	27·69		−28·88
1	6·44	*	−3·17
2	6·17	*	−11·18
3	24·03		32·51
	0	2	L
1	118·55		−171·39
2	30·21		37·14
3	189·60		200·97
4	117·94		118·55
5	41·20		−44·22
6	31·84		29·87
7	8·57	*	7·03
8	42·05		36·74
9	10·16	*	−10·14
10	69·66		−67·85
11	11·03	*	1·00
12	10·87	*	−12·00
13	10·17	*	26·42
14	33·46		−37·18
15	29·44		−33·43
	1	2	L
1	51·42		60·20
2	65·19		71·39
3	75·27		74·22
4	20·90		−14·65
5	52·92		−57·62
6	134·29		138·00
7	8·59	*	−10·83
8	57·28		51·67
9	33·73		−25·13
10	10·75	*	22·01
11	33·10		20·09
12	36·03		−28·73
13	10·16	*	6·33
14	29·62		−23·08
15	20·14		19·39
	2	2	L
0	85·59		89·05
1	72·38		−70·38
2	19·57		−16·75
3	42·12		36·53
4	53·34		47·31
5	38·11		−32·34
6	7·80	*	4·63
7	8·64	*	14·32
8	48·22		−43·83
9	10·20	*	−5·86
10	10·77	*	−3·16
11	11·03	*	−14·36
12	10·85	*	23·04
13	10·13	*	9·98

TABLE V
(*continued*)

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	3	2	L
1	54·96		−50·48
2	34·85		31·64
3	13·35		8·75
4	112·80		119·63
5	7·07	*	−0·05
6	40·23		31·67
7	34·87		31·92
8	26·93		22·12
9	10·25	*	11·28
10	37·42		27·34
11	11·04	*	29·31
12	10·82	*	6·00
13	10·08	*	−9·07
	4	2	L
0	205·19		−252·27
1	4·43	*	9·13
2	43·51		32·30
3	26·69		−12·47
4	6·44	*	−12·97
5	45·66		39·84
6	35·86		28·38
7	8·83	*	22·55
8	9·61	*	10·49
9	30·95		31·89
10	30·66		15·94
11	58·41		−64·52
12	32·35		30·48
13	28·30		−22·76
	5	2	L
1	51·32		−47·50
2	68·99		59·15
3	19·80		−20·62
4	43·71		−41·57
5	20·95		19·36
6	43·27		−40·68
7	42·98		46·19
8	9·73	*	11·53
9	10·40	*	−6·51
10	37·70		35·28
11	11·03	*	3·21
12	42·92		48·51
13	9·91	*	−5·46
	6	2	L
0	22·68		−16·70
1	33·40		−30·80
2	63·09		59·90
3	6·28	*	−6·65
4	23·98		−23·86
5	7·63	*	−13·51
6	20·49		15·88
7	55·50		−49·73
8	54·89		−55·25
9	10·50	*	26·35
10	10·93	*	−15·17
11	11·02	*	−14·21
12	10·66	*	13·35
13	42·64		36·37
	7	2	L
1	30·66		−18·75
2	55·14		−46·37
3	49·50		45·03
4	60·74		−52·41
5	7·87	*	0·09
6	19·19		−16·03
7	81·15		68·15
8	43·62		37·78
9	29·98		−18·55
10	68·53		75·83
11	11·00	*	14·31
12	31·69		28·24
13	9·63	*	1·87
	8	2	L
0	122·58		112·47
1	49·86		−37·76
2	39·15		43·94
3	52·13		−46·25
4	7·52	*	6·40
5	36·42		−35·24
6	72·72		−78·27
7	23·30		−25·19
8	33·72		−34·61
9	10·70	*	5·91
10	11·01	*	−8·07
11	43·82		41·57
12	10·44	*	21·79
13	9·44	*	23·11
	9	2	L
1	53·34		53·85
2	110·65		108·42
3	25·40		13·16
4	82·26		88·18
5	8·43	*	−6·19
6	25·67		27·13
7	53·28		−50·17
8	29·23		−31·23
9	10·81	*	15·66
10	31·21		−24·19
11	10·89	*	−1·16
12	10·29	*	−22·47
13	9·22	*	11·70
	10	2	L
0	134·14		−132·49
1	90·68		−86·36
2	95·41		−87·22
3	48·15		48·82
4	35·68		36·32
5	28·99		−26·77
6	37·37		33·22
7	55·41		43·48
8	29·70		30·98
9	10·90	*	16·89
10	11·04	*	−17·50
11	26·44		−12·93
12	10·10	*	−12·53
13	8·95	*	−3·69
	11	2	L
1	56·91		53·70
2	99·00		100·91
3	8·10	*	18·39
4	28·33		22·08
5	54·36		−59·91
6	9·62	*	−13·91
7	63·56		−59·33
8	71·52		−70·99
9	10·98	*	−29·00
10	36·52		−36·43
11	10·66	*	−0·60
12	36·95		−45·66
13	21·17		15·96
	12	2	L
0	37·92		−41·69
1	7·97	*	0·16
2	27·11		−25·16
3	93·00		92·38
4	8·90	*	−11·46

TABLE V
(*continued*)

L	$\|F_o\|$	sg	$\|F_c\|$
	12	2	L
5	9·38	*	−0·61
6	32·84		30·31
7	10·40	*	4·41
8	35·84		9·93
9	11·03	*	−20·28
10	10·95	*	−1·09
11	10·49	*	−25·96
	13	2	L
1	92·51		−98·27
2	37·46		−31·45
3	8·89	*	−1·48
4	66·18		−66·20
5	32·20		36·89
6	76·13		−84·70
7	85·49		97·81
8	37·84		−37·79
9	11·04	*	−19·92
10	35·96		33·38
11	10·27	*	−4·50
	14	2	L
0	8·79	*	15·37
1	8·84	*	19·62
2	37·16		38·32
3	9·28	*	−0·29
4	9·63	*	−17·62
5	30·07		33·77
6	10·43	*	−14·65
7	10·78	*	−4·50
8	31·13		25·15
9	11·00	*	2·89
10	30·22		26·55
11	9·99	*	14·89
	15	2	L
1	9·27	*	5·30
2	9·42	*	−13·81
3	9·28	*	−0·29
4	49·87		−46·69
5	10·32	*	−4·01
6	26·11		−28·68
7	10·92	*	3·48
8	49·36		50·00
9	10·91	*	−11·21
10	10·47	*	20·16
11	9·66	*	−8·00

L	$\|F_o\|$	sg	$\|F_c\|$
	16	2	L
0	50·97		54·07
1	45·40		−45·69
2	9·82	*	−2·33
3	47·04		−46·07
4	23·02		−30·70
5	10·59	*	−31·90
6	10·85	*	−8·69
7	11·01	*	−15·01
8	11·01	*	−23·35
9	32·26		26·00
10	10·18	*	−1·89
11	9·27	*	20·36
	17	2	L
1	10·06	*	6·65
2	101·8	*	−20·61
3	10·36	*	6·26
4	10·58	*	21·04
5	55·09		−55·23
6	32·93		−31·62
7	36·61		−36·90
8	10·91	*	−13·41
9	10·52	*	−17·78
10	9·83	*	−1·88
11	8·81	*	15·42
	18	2	L
0	49·76		−56·99
1	10·41	*	16·91
2	10·50	*	−3·31
3	10·65	*	32·00
4	10·81	*	−4·09
5	10·96	*	16·16
6	11·04	*	−15·19
7	10·98	*	0·19
8	26·27		29·39
9	10·21	*	−5·74
10	9·40	*	−3·29
11	24·80	*	−22·98
	19	2	L
2	70·61		84·69
3	10·87	*	5·35
4	10·97	*	7·79
5	31·21		26·05
6	11·01	*	14·24
7	10·83	*	−4·72
8	25·60		−36·44
9	9·82	*	4·19

L	$\|F_o\|$	sg	$\|F_c\|$
	20	2	L
0	75·47		81·53
1	10·91	*	0·07
2	10·95	*	18·98
3	11·00	*	7·93
4	11·04	*	2·76
5	11·01	*	−4·21
6	10·88	*	33·20
7	31·76		13·93
8	10·68	*	4·78
	21	2	L
2	11·04	*	−20·04
3	11·04	*	−22·82
4	11·00	*	22·48
5	10·88	*	26·99
6	10·64	*	1·78
7	10·23	*	6·74
8	23·56		28·56
	22	2	L
0	24·66		25·62
1	11·02	*	−35·73
2	58·21		71·95
3	10·95	*	22·34
4	10·83	*	−42·78
5	10·63	*	−17·61
6	29·09		−33·93
7	9·77	*	−16·08
	23	2	L
2	10·83	*	18·32
3	10·72	*	−2·40
4	10·54	*	−7·79
5	10·25	*	−23·66
6	32·55		31·58
	24	2	L
0	10·63	*	−3·90
	1	3	L
1	3·08	*	6·27
2	75·27		−99·25
3	39·74		16·73
4	100·26		−112·53
5	6·90	*	17·92
6	7·73	*	−2·35

TABLE V
(*continued*)

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	1	3	L
7	8·53	*	32·21
8	35·93		−31·03
9	9·90	*	−1·87
10	10·31		−28·19
11	10·36	*	−23·49
12	53·69		−49·19
13	9·10	*	−27·79
	2	3	L
0	52·86		−62·11
1	33·69		−40·66
2	39·13		−42·61
3	95·71		115·13
4	53·19		56·19
5	6·97	*	8·70
6	82·37		85·00
7	8·57	*	20·98
8	30·88		22·49
9	9·93	*	13·99
10	10·32	*	−8·77
11	27·40		28·76
12	29·85		−22·30
13	9·06	*	32·91
	3	3	L
1	24·15		−24·37
2	44·44		−47·12
3	37·96		42·27
4	6·28	*	5·47
5	39·41		−39·44
6	48·79		−48·52
7	34·59		33·62
8	9·37	*	−29·62
9	9·96	*	−19·76
10	10·33	*	−30·37
11	10·34	*	12·71
12	9·91	*	7·05
13	9·00	*	7·01
	4	3	L
0	83·76		72·91
1	40·80		−43·16
2	58·10		60·94
3	99·03		−132·14
4	47·04		59·12
5	60·45		−51·56
6	7·99	*	−8·95
7	34·99		30·48
8	9·45	*	−0·50
9	10·02	*	33·81
10	10·35	*	−30·00
11	10·33	*	25·46
12	9·86	*	14·77
13	8·92	*	−18·82
	5	3	L
1	37·80		38·10
2	43·06		−38·87
3	34·45		−29·55
4	6·68	*	17·95
5	58·32		−66·81
6	8·15	*	16·19
7	45·24		−51·85
8	9·54	*	13·16
9	10·08	*	27·46
10	31·10		−28·63
11	10·30	*	39·04
12	32·46		34·00
13	8·81	*	28·31
	6	3	L
0	205·32		181·75
1	5·38	*	25·94
2	79·00		76·60
3	55·32		56·76
4	46·00		40·04
5	42·41		44·14
6	91·93		−104·53
7	9·02	*	−0·91
8	66·87		−73·43
9	10·14	*	−17·19
10	10·38	*	21·37
11	61·54		−63·40
12	25·64		25·52
13	8·67	*	−15·65
	7	3	L
1	22·54		15·66
2	39·86		33·38
3	40·89		39·36
4	97·58		105·40
5	7·85	*	−24·52
6	47·46		45·52
7	35·56		28·40
8	72·48		72·57
9	36·82		−41·64
10	10·39	*	−34·44
11	10·20	*	−11·43
12	25·32		−25·28
13	38·00		−16·61
	8	3	L
0	87·23		−81·02
1	6·26	*	20·66
2	38·71		32·85
3	6·98	*	10·06
4	41·83		37·01
5	8·11	*	16·28
6	59·29		59·39
7	9·36	*	−15·87
8	9·90	*	−6·10
9	10·28	*	3·98
10	55·91		−51·87
11	10·12	*	2·72
12	9·42	*	−1·96
	9	3	L
1	81·88		−79·63
2	6·94	*	18·50
3	7·33	*	13·58
4	7·83	*	16·93
5	8·39	*	−6·22
6	95·81		97·60
7	84·82		87·54
8	40·11		37·79
9	10·33	*	−4·43
10	41·43		46·58
11	26·48		24·88
12	9·24	*	13·99
	10	3	L
0	148·82		−150·45
1	7·11	*	−0·56
2	59·64		−51·12
3	23·10		−23·20
4	32·61		32·18
5	8·67	*	4·09
6	104·23		107·07
7	9·73	*	1·89
8	22·69		16·40
9	10·37	*	−3·88
10	30·93		27·17
11	9·87	*	13·36
12	9·03	*	−31·58

TABLE V
(*continued*)

L	$\|F_o\|$	sg	$\|F_c\|$
	11	3	L
1	35·34		32·74
2	7·74	*	2·22
3	8·07	*	0·31
4	43·27		35·00
5	62·08		−51·99
6	9·45	*	14·61
7	38·38		−40·95
8	43·49		40·63
9	10·39	*	−11·38
10	50·10		54·07
11	9·70	*	3·19
12	8·77	*	25·57
	12	2	L
0	72·28		−72·19
1	48·37		−43·45
2	8·14	*	0·59
3	8·44	*	2·58
4	8·82	*	−39·96
5	41·36		−39·63
6	9·69	*	−20·38
7	10·07	*	−10·73
8	27·34		−19·33
9	10·37	*	−18·58
10	10·11	*	1·88
11	9·48	*	−19·16
12	18·95		27·05
	13	3	L
1	8·36	*	−18·58
2	8·53	*	−0·41
3	37·35		37·44
4	9·15	*	−6·26
5	9·53	*	−12·52
6	9·91	*	0·85
7	33·88		27·42
8	31·14		−18·27
9	34·21		−30·56
10	9·94	*	7·19
11	9·22	*	−8·72
12	8·12	*	20·63
	14	3	L
0	8·71	*	2·48
1	35·06		25·48
2	48·02		−45·14
3	9·16	*	1·50
4	9·46	*	28·24
5	9·79	*	25·22
6	72·10		−70·66
7	10·32	*	25·90
8	44·06		46·53
9	30·62		19·20
10	9·73	*	−1·16
11	8·91	*	−5·12
12	7·72	*	22·56
	15	3	L
1	42·90		−30·24
2	9·28	*	−11·45
3	9·49	*	23·54
4	9·75	*	−15·75
5	10·02	*	6·74
6	45·84		−44·22
7	55·90		68·86
8	10·34	*	17·88
9	10·04	*	−4·93
10	9·45	*	−32·99
11	8·54	*	13·43
12	26·12		−30·63
	16	3	L
0	9·46	*	14·74
1	28·51		36·35
2	34·67		−30·36
3	29·37		−32·30
4	33·16		18·40
5	10·20	*	35·03
6	27·39		−20·61
7	10·38	*	10·41
8	10·22	*	−1·41
9	9·82	*	7·91
10	9·12	*	−17·31
11	8·11	*	16·41
12	38·40		45·20
	17	3	L
1	9·82	*	−8·34
2	9·91	*	14·73
3	10·05	*	−9·15
4	10·20	*	−1·92
5	30·99		−26·34
6	46·46		−53·81
7	10·31	*	1·05
8	47·09		−45·77
9	9·52	*	−7·17
10	31·45		−31·97
11	20·12		15·56
	18	3	L
0	73·22		−70·59
1	30·24		−30·29
2	10·15	*	−31·21
3	57·03		−58·22
4	10·34	*	−1·59
5	46·45		−45·15
6	40·08		40·15
7	10·16	*	−31·84
8	9·78	*	15·91
9	9·15	*	1·95
10	8·25	*	4·12
	19	2	L
1	10·27	*	14·61
2	10·31	*	−23·15
3	10·36	*	29·47
4	37·46		−25·43
5	57·67		−55·37
6	53·11		−52·62
7	9·93	*	−5·69
8	31·29		−31·58
9	38·94		−32·67
	20	3	L
0	53·89		−39·50
1	46·40		41·89
2	40·23		38·75
3	10·39	*	10·82
4	10·35	*	−10·64
5	10·23	*	30·91
6	10·00	*	17·44
7	9·60	*	0·40
8	9·01	*	−15·66
9	27·13		−38·10
	21	3	L
1	34·41		38·10
2	10·35	*	4·07
3	39·90		40·74
4	10·19	*	−6·83
5	10·00	*	25·80
6	9·67	*	−24·20
7	9·18	*	−12·73
8	8·49	*	1·62
	22	3	L
0	41·07		42·34
1	27·13		27·41

TABLE V
(*continued*)

L	$\lvert F_o\rvert$	sg	$\lvert F_o\rvert$
	22	3	L
2	56·80		67·77
3	10·10	*	17·85
4	9·93	*	9·54
5	9·65	*	22·40
6	9·24	*	−2·73
7	8·66	*	19·21
8	28·39	*	−21·90
	23	3	L
1	49·01		−47·54
2	53·45		48·58
3	9·78	*	−27·34
4	31·65		24·92
5	9·19	*	18·87
	24	3	L
0	9·66	*	6·41
1	9·63	*	2·43
2	9·52	*	−22·69
3	9·33	*	−5·21
4	9·04	*	21·33
5	8·63	*	5·99
	25	3	L
0	9·16	*	0·00
1	9·12	*	−15·29
2	8·99	*	10·17
3	8·76	*	−4·72
4	8·42	*	13·92
5	30·75		−31·81
	26	3	L
0	25·61		20·05
1	8·49	*	8·35
2	8·33	*	−5·73
3	34·23		−32·37
	0	4	L
1	18·25		−24·81
2	82·92		90·51
3	60·92		−63·49
4	77·28		−65·01
5	94·00		−80·05
6	35·79		−35·80
7	8·34	*	1·38
8	40·00		−38·23

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	0	4	L
9	9·36	*	4·30
10	9·51	*	−7·39
11	76·61		81·15
	1	4	L
1	29·11		34·41
2	4·23	*	−0·78
3	35·22		−34·31
4	6·06	*	24·22
5	38·90		−30·71
6	7·65	*	22·99
7	64·16		−57·74
8	8·95	*	−0·86
9	9·37	*	10·76
10	9·51	*	13·60
11	9·29	*	8·31
	2	4	L
0	41·33		−56·14
1	3·52	*	0·48
2	44·71		37·00
3	21·90		20·14
4	54·97		−56·12
5	6·94	*	9·07
6	7·70	*	−19·37
7	8·39	*	23·47
8	8·98	*	21·86
9	9·38	*	5·49
10	9·51	*	−5·09
11	9·27	*	3·75
	3	4	L
1	87·52		108·70
2	4·71	*	2·52
3	25·78		21·81
4	79·17		−72·93
5	62·20		−63·54
6	7·78	*	20·78
7	71·22		−68·99
8	9·02	*	−9·21
9	9·40	*	−14·53
10	9·50	*	−10·56
11	9·24	*	20·61
	4	4	L
0	36·21		−38·89
1	68·47		80·82
2	75·94		−72·04

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$
	4	4	L
3	5·73	*	7·61
4	50·42		−53·79
5	65·72		56·43
6	64·06		55·38
7	8·54	*	−20·17
8	51·34		54·86
9	36·51		−33·09
10	9·50	*	13·96
11	37·94		−38·36
	5	4	L
1	65·02		−74·72
2	60·14		−60·83
3	6·00	*	10·41
4	6·67	*	−10·91
5	69·73	X	63·74
6	45·37		−48·80
7	91·41		92·17
8	75·39		−73·91
9	9·45	*	25·49
10	9·48	*	−48·61
11	9·14	*	21·33
	6	4	L
0	5·25	*	−15·79
1	71·96		81·49
2	46·91		39·57
3	46·31		−44·95
4	6·91	*	−2·09
5	33·74		31·03
6	8·18	*	−4·29
7	8·75	*	−39·36
8	47·89		46·72
9	9·48	*	−15·92
10	9·46	*	−10·46
11	9·07	*	11·96
	7	4	L
1	37·79		−20·18
2	6·15	*	−14·50
3	42·39		−37·91
4	33·63		−28·98
5	43·90		29·20
6	8·35	*	7·66
7	8·88	*	8·61
8	43·59		−35·69
9	9·50	*	40·35
10	9·42	*	−34·59
11	8·97	*	−15·98

TABLE V
(*continued*)

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	8	4	L
0	71·26		69·29
1	6·26	*	−33·47
2	40·18		35·39
3	6·95	*	−6·27
4	7·45	*	10·27
5	46·59		−45·58
6	35·17		−29·36
7	9·01	*	−14·01
8	9·37	*	−15·43
9	9·51	*	7·52
10	9·36	*	−3·63
11	30·67		31·87
	9	4	L
1	84·37		87·28
2	43·18		−41·08
3	7·28	*	23·72
4	7·74	*	−18·70
5	53·33		−45·83
6	8·72	*	12·88
7	44·79		−53·31
8	53·36		48·71
9	9·50	*	−8·83
10	9·28	*	11·82
	10	4	L
0	7·00	*	2·50
1	7·07	*	6·37
2	37·88		40·46
3	7·62	*	18·00
4	37·65		−33·29
5	8·47	*	−0·59
6	8·90	*	0·95
7	9·27	*	26·83
8	9·48	*	−0·42
9	9·57	*	−11·75
10	9·17	*	−1·41
	11	4	L
1	40·22		−23·26
2	7·66	*	−12·19
3	7·95	*	−1·51
4	8·31	*	−2·60
5	56·43		66·79
6	9·08	*	37·42
7	9·37	*	17·22
8	9·51	*	9·21
9	9·41	*	8·53
10	9·02	*	12·52

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	12	4	L
0	45·42		−40·46
1	7·85	*	2·88
2	46·74		43·07
3	8·27	*	−11·15
4	8·59	*	−3·31
5	9·93	*	30·55
6	49·76		35·78
7	46·31		44·11
8	9·50	*	−1·09
9	9·32	*	23·00
10	8·84	*	−28·59
	13	4	L
1	8·21	*	−12·14
2	9·36	*	−3·78
3	35·38		−36·96
4	8·85	*	−4·16
5	9·13	*	−18·60
6	63·55		76·85
7	9·50	*	−25·29
8	9·46	*	13·49
9	9·18	*	4·32
10	8·61	*	18·07
	14	4	L
0	6·50	*	13·14
1	8·55	*	−1·87
2	8·67	*	0·76
3	8·86	*	−13·58
4	9·08	*	5·06
5	9·30	*	−30·10
6	9·46	*	7·30
7	9·51	*	−32·42
8	9·37	*	−19·78
9	8·99	*	−2·38
10	8·34	*	−2·00
	15	4	L
1	8·85	*	−19·42
2	9·96	*	−0·15
3	9·11	*	−14·99
4	71·26		74·13
5	46·19		−47·74
6	51·20		54·78
7	9·46	*	19·64
8	9·22	*	26·49
9	8·75	*	−4·33
10	8·01	*	8·21

L	$\lvert F_o \rvert$	sg	$\lvert F_c \rvert$
	16	4	L
0	47·22		−38·80
1	9·12	*	16·53
2	9·19	*	16·74
3	9·30	*	−15·73
4	9·42	*	10·20
5	7·50	*	14·51
6	9·49	*	−49·46
7	9·35	*	−3·18
8	9·02	*	5·14
9	8·45	*	−1·60
10	7·63	*	5·22
	17	4	L
1	52·73		51·98
2	9·37	*	32·43
3	9·44	*	29·47
4	9·50	*	−23·43
5	9·50	*	−11·29
6	9·41	*	19·26
7	9·17	*	−27·80
8	8·74	*	1·63
9	8·09	*	−11·45
10	7·17	*	−22·00
	18	4	L
0	9·45	*	−0·42
1	9·46	*	−7·20
2	9·48	*	−51·24
3	9·51	*	−24·71
4	9·50	*	3·88
5	9·43	*	−3·70
6	9·25	*	14·43
7	8·92	*	31·11
8	8·40	*	−5·33
9	7·66	*	6·25
10	6·64	*	−4·21
	19	4	L
2	51·20		−52·83
3	9·49	*	−25·48
4	9·42	*	−17·73
5	9·27	*	−8·31
6	9·01	*	−34·49
7	8·59	*	−28·04
8	7·99	*	−17·76
9	7·16	*	−6·85
10	5·99	*	−21·10

TABLE V
(*continued*)

L	$\lvert F_o\rvert$	sg	$\lvert F_c\rvert$	L	$\lvert F_o^9\rvert$	sg	$\lvert F_c^c\rvert$	L	$\lvert F_9^9\rvert$	sg	$\lvert F_c^c\rvert$
	20	4	L		21	4	L		23	4	L
0	9·48	*	−62·75	3	9·15	*	−12·45	3	51·12		20·45
1	9·47	*	37·59								
2	9·44	*	2·97		22	4	L		24	4	L
3	53·01		59·30								
4	9·24	*	−6·49	0	35·21		−36·04	0	38·74		−39·60
5	9·03	*	40·90	1	9·06	*	10·06				
6	8·68	*	7·58	2	8·98	*	16·78				
7	36·61		−23·53	3	8·83	*	−39·57				
8	7·50	*	10·45								
9	29·31		−40·97								
10	5·15	*	−4·38								

Valence sums, $\sum v$, of bonds reaching each oxygen anion were calculated from the empirical equation[17]

$$\sum v = \sum v_i \left(\frac{\bar{L}}{L}\right)^p , \tag{3}$$

where v_i is the ideal bond valence, *i.e.* formal cationic charge divided by coordination number; L is the observed bond length and $\bar{L}$ is the average bond length for each coordination polyhedron in the structure. Summation is taken over all bonds reaching the oxygen atom.

Exponent p is given by[17]

$$p = \frac{\bar{L}}{L_{max} - \bar{L}} , \tag{4}$$

where L_{max} is the upper limit of an atomic approach that will still be considered as an interatomic bond, so that coordination number is the number of anions at distances smaller than L_{max} from the cation. The values of L_{max} are available in [17]. L_{max} for B—O is 1·87 Å, for Mg—O is 2·85 Å. The values v_i, $\bar{L}$ and p for the BO_4, BO_3 and MgO_6 polyhedra are given in Table VII.

The valence sum, $\sum v$, reaching each oxygen anion (Table VIII) gives the answer to the question of the hydrogen distribution in the aksait structure. O(1), O(4), O(10), O(11), O(12) and O(13) belong to hydroxyl groups (the valence sum is approximately equal to 1). O(14) and O(15) belong to water molecules (the valence sum is approximately equal to 0). The remaining anions are oxygen ions (the sum is approximately

TABLE VI

Bond Distances and Angles in the Crystal Structure of $MgO.3\ B_2O_3.5\ H_2O$, with Standard Deviations

Distances, Å		Angles	
		VO_3-triangles	
B(1)—0(1)	1·37(2)	0(1)—B(1)—0(2,	124(2)°
B(1)—0(2)	1·32(3)	0(1)—B(1)—0(7)	113(2)°
B(1)—0(7)	1·42(3)	0(2)—B(1)—0(7)	123(2)°
B(3)—0(3)	1·37(3)	0(3)—B(3)—0(4)	121(2)°
B(3)—0(4)	1·38(2)	0(3)—B(3)—0(5)	122(1)°
B(3)—0(5)	1·39(3)	0(4)—B(3)—0(5)	117(2)°
B(6)—0(8)	1·35(3)	0(8)—B(6)—0(9)	125(3)°
B(6)—0(9)	1·37(3)	0(8)—B(6)—0(10)	119(2)°
B(6)—0(10)	1·42(4)	0(9)—B(6)—0(10)	115(2)°
Average distance 1·38		*Average bond angle 120°*	
		BO_4-tetrahedra	
B(2)—0(2)	1·48(2)	0(2)—5(2)—0(3)	107(1)°
B(2)—0(3)	1·51(2)	0(2)—B(2)—0(6)	109(2)°
B(2)—0(6)	1·49(3)	0(2)—B(2)—0(11)	113(2)°
B(2)—0(11)	1·46(3)	0(3)—B(2)—0(6)	108(2)°
		0(3)—B(2)—0(11)	112(2)°
		0(6)—B(2)—0(11)	109(1)°
B(4)—0(6)	1·53(2)	0(6)—B(4)—0(7)	109(1)°
B(4)—0(7)	1·45(2)	0(6)—B(4)—0(8)	106(2)°
B(4)—0(8)	1·47(3)	0(6)—B(4)—0(12)	107(2)°
B(4)—0(12)	1·46(3)	0(7)—B(4)—0(8)	109(2)°
		0(7)—B(4)—0(12)	113(2)°
		0(8)—B(4)—0(12)	112(1)°
B(5)—0(5)	1·45(2)	0(5)—B(5)—0(6)	106(2)°
B(5)—0(6)	1·57(3)	0(5)—B(5)—0(9)	112(2)°
B(5)—0(9)	1·40(3)	0(5)—B(5)—0(13)	109(2)°
B(5)—0(13)	1·52(3)	0(6)—B(5)—0(9)	109(2)°
		0(6)—B(5)—0(13)	105(2)°
		0(9)—B(5)—0(13)	116(2)°
Average distance 1·48		*Average bond angle 109·5°*	

TABLE VI
(continued)

Distances, Å		Angles	
		MgO_6-octahedron	
Mg—0(10)	2·13(2)	0(10)—Mg—0(11)	96(1)°
Mg—0(11)	2·12(1)	0(10)—Mg—0(12)	91(1)°
Mg—0(12)	2·11(2)	0(10)—Mg—0(14)	88(1)°
Mg—0(13)	2·03(2)	0(10)—Mg—0(15)	83(1)°
Mg—0(14)	2·08(2)	0(11)—Mg—0(12)	87(1)°
Mg—0(15)	2·13(1)	0(11)—Mg—0(13)	93(1)°
		0(11)—Mg—0(14)	89(1)°
		0(12)—Mg—0(13)	87(1)°
		0(12)—Mg—0(15)	91(1)°
		0(13)—Mg—0(14)	94(1)°
		0(13)—Mg—0(15)	88(1)°
		0(14)—Mg—0(15)	93(1)°
		Bond angles of the bridging oxygen atoms	
B(1)—0(2)—B(2)	123(2)°	B(4)—0(8)—B(6)	124(2)°
B(2)—0(3)—B(3)	117(1)°	B(5)—0(9)—B(6)	118(2)°
B(3)0(5)—B(5)	127(2)°		
B(2)—0(6)—B(4)	116(2)°	B(6)—0(10)—Mg	132(1)°
B(2)—0(6)—B(5)	120(1)°	B(2)—0(11)—Mg	118(1)°
B(4)—0(6)—B(5)	116(1)°	B(4)—0(12)—Mg	118(1)°
B(1)—0(7)—B(4)	120(1)°	B(5)—0(13)—Mg	119(1)°

equal to 2). The deviations from the integral values 0, 1 or 2 indicate the presence of hydrogen bonding. The values of hydrogen-bond valence *vs* oxygen–oxygen separation have been calculated[18] for a linear O—H···O arrangement. If one of the two oxygen ions in a pair does not belong to either a hydroxyl group or a water molecule, then it can be only an acceptor ion. Thus, its valence sum, $\sum v$, must be increased by the amount due to the hydrogen bonding as it is case of 0(3), 0(5), 0(7), 0(8), 0(9), and the valence sum of the donor oxygen ion must be decreased by the same amount (oxygens 0(10), 0(11), 0(13), 0(14), 0(15)). Table IX gives short oxygen–oxygen distances and estimated hydrogen-bond valences in the aksait structure for asymmetric and linear O—H···O bonds. Hydrogen bonds of the type O—H···O take important part in the intermolecular cohesive forces. Six hydroxyl groups and two water molecules create eight O—H···O contacts with neighbouring molecules at the

TABLE VII

Bond Valences v_i, Average Bond Lengths L and Values of p for Coordination Polyhedra Used to Construct Curves of Bond Valences *vs* Bond Lengths for Aksait $Mg\,B_6O_7(OH)_6.2\,H_2O$

Group	v_i	L	p
BO_4	3/4	1·483	3·83
BO_3	1	1·377	2·79
MgO_6	1/3	2·102	2·81

TABLE VIII

Estimated Valence Sums $\sum v$ Reaching each Oxygen Anion

Atom		Oxygen bonding	Atom		Oxygen bonding
0(1)	1·02	OH donor–acceptor	0(9)	1·95	O^{2-} acceptor
0(2)	1·88	O^{2-}	0(10)	1·25	OH donor
0(3)	1·73	O^{2-} acceptor	0(11)	1·13	OH donor
0(4)	0·99	OH donor–acceptor	0(12)	1·14	OH
0(5)	1·80	O^{2-} acceptor	0(13)	1·12	OH donor
0(6)	2·04	O^{2-}	0(14)	0·34	H_2O donor
0(7)	1·76	O^{2-} acceptor	0(15)	0·32	H_2O donor
0(8)	1·85	O^{2-} acceptor			

TABLE IX

Short Oxygen–Oxygen Distances (Å) Representing Hydrogen Bonds of the Type O—H...O and Estimated Hydrogen–Bond Valences in Aksait According to[18]

Atoms	Distances, Å	Hydrogen-Bond valences
0(1)....0(8)	2·81(2)	0·161
0(1)....0(15)	2·82(2)	0·158
0(3)....0(13)	2·76(2)	0·182
0(4)....0(11)	2·79(2)	0·165
0(4)....0(14)	2·82(2)	0·158
0(5)....0(14)	2·79(1)	0·165
0(7)....0(10)	2·62(2)	0·246
0(9)....0(14)	2·65(2)	0·231

oxygen–oxygen distances less than 3·00 Å. Table IX summarizes the short hydrogen bond lengths.

It is not surprising that the hydroxyl groups are preserved at all terminal oxygen atoms of the polyanion $(B_6O_7(OH)_6)^{2-}$. According to the Pauling's electronegativity scale[16], the electronegativity values for the boron and hydrogen atoms are comparable: 2·0 and 2·1. Boric acid and its condensation products act, not as proton donors, but as a Lewis acid, accepting the electron pairs of the OH groups to form boron–oxygen tetrahedra. The presence of the hydroxyl groups in the structure of $Mg(B_6O_7(OH)_6).2\,H_2O$ also influences dehydration process. Dehydration of the compound[5] is completed at 500°C.

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