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The crystal structure of meyerhofferite, $CaB_3O_3(OH)_5 \cdot H_2O$ *

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With 2 figures

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Auszug

Meyerhofferit, $CaB_3O_3(OH)_5 \cdot H_2O$ ist triklin, in der Raumgruppe $P\bar{1}$ mit $a = 6{,}63$, $b = 8{,}35$, $c = 6{,}46$ Å (sämtlich $\pm$ 0,015 Å), $\alpha = 90°46'$, $\beta = 101°59'$, $\gamma = 86°55'$ (sämtlich $\pm$ 5'), $Z = 2$; Dichte: berechnet 2,125, gemessen 2,120. Die mit Hilfe der statistischen Methode von HAUPTMAN und KARLE aus 4193 Interferenzen berechneten Strukturfaktorvorzeichen ließen die Aufstellung einer Struktur zu, deren Parameter anschließend durch mehrfache Elektronendichte-Projektionen und mittels der Methode der kleinsten Quadrate verfeinert wurden. Für die endgültige Struktur ist der restliche Diskrepanzfaktor 0,14 aus 2678 Interferenzen mit $|F_{hkl}| > 0$.

Meyerhofferit enthält Polyionen $[B_3O_3(OH)_5]$ aus zwei B—O-Tetraedern und einem B—O-Dreieck, die durch gemeinsame Sauerstoffionen ringförmig verbunden sind. Die Polyion-Inseln sind miteinander durch Ca—O-Bindungen zu Zickzackstreifen parallel [001] verknüpft. Diese Streifen wechseln mit Wassermolekülen ab, mit denen sie über ein Netzwerk von Wasserstoffbindungen zusammenhängen. Die Struktur ist im Einklang mit der beobachteten Spaltbarkeit.

Abstract

Meyerhofferite, $CaB_3O_3(OH)_5 \cdot H_2O$, is triclinic $P\bar{1}$, $a = 6.63$, $b = 8.35$, $c = 6.46$ Å (all $\pm$ 0.015 Å), $\alpha = 90°46'$, $\beta = 101°59'$, $\gamma = 86°55'$ (all $\pm$ 05'), $Z = 2$, density (g. cm^{-3}): calc. 2.125, obs. 2.120. Signs for the observed reflections, calculated by the statistical method of HAUPTMAN and KARLE, using 4193 three-dimensional data, permitted the establishment of a trial structure which was refined by successive electron-density projections and least-squares analysis. For the final structure, the residual factor is 0.14 for 2678 terms with $|F_{hkl}| > 0$. Meyerhofferite contains the polyion $[B_3O_3(OH)_5]^{-2}$ consisting of two boron-oxygen tetrahedra and a boron-oxygen triangle linked at corners to form

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a ring. The insular polyions are linked together by calcium-oxygen bonds to form zigzag strings along [001]; the strings and the water molecule are in turn linked by a network of hydrogen bonds. The structure is in good accord with the observed cleavage.

Introduction

In the series $2CaO \cdot 3B_2O_3 \cdot xH_2O$, meyerhofferite has $x = 7$; other known members of the series are colemanite, with $x = 5$; a synthetic compound, with $x = 9$; and inyoite, with $x = 13$. As part of a systematic investigation of the crystal structures of borate minerals, the crystal structure of each of the members of this series has been determined. Colemanite was described by CHRIST, CLARK, and EVANS (1958), a preliminary note has appeared on meyerhofferite (CHRIST and CLARK, 1956), the synthetic compound was described by CLARK and CHRIST (1959), and inyoite was reported by CLARK (1959). The present paper presents the detailed results of the structure investigation of meyerhofferite; the phase determination which led to the solution of the crystal structure is described in detail in an accompanying paper (CLARK and CHRIST, 1960).

Experimental work

Crystal description, space group, and unit-cell dimensions

The crystals of meyerhofferite used in this study are synthetic and were grown by W. T. SCHALLER, U.S. Geological Survey, on ulexite fragments placed in water and held at 70–80 °C for approximately six months (CHRIST, 1953). X-ray powder data and a description of the morphology, together with the cell constants originally obtained by SWITZER (PALACHE, 1938) are given by CHRIST (1953). Subsequently, x-ray precession patterns were taken with Zr-filtered Mo radiation on a quartz-calibrated precession camera. Film measurements were corrected for both horizontal and vertical film shrinkage to obtain the crystallographic data given in CHRIST and CLARK (1956). These data are repeated here in Table 1, together with the reciprocal cell elements, the projection elements, and the direct and reciprocal Cartesian matrices.

Intensity measurements

For the intensity measurements multiple-film Weissenberg patterns were prepared using chiefly Zr-filtered Mo radiation; some small-angle reflections were obtained with Ni-filtered Cu radiation. For the Mo

Table 1. *Crystallographic data for meyerhofferite*

Triclinic, space group $P\bar{1} - C_i^1$, $Z = 2$ $[CaB_3O_3(OH)_5 \cdot H_2O]$

Direct-cell elements:

$a = 6.63_0$ Å	$\alpha = 90°46'$
$b = 8.35_2$	$\beta = 101°59'$
$c = 6.46_2$	$\gamma = 86°55'$
(all $\pm$ 0.015 Å)	(all $\pm$ 05′)

$$a:b:c = 0.794:1:0.774$$

$$(\lambda_{MoK\alpha} = 0.7107 \text{ Å}; \quad \lambda_{CuK\alpha} = 1.5418 \text{ Å})$$

Volume = 349.5 Å^3 Density (g.cm^{-3}), calc. 2.125, obs. [SCHALLER (1916) for the mineral] 2.120

Reciprocal-cell elements:

$a^* = 0.1544$ Å^{-1}	$\alpha^* = 89°52'$
$b^* = 0.1199$	$\beta^* = 78°02'$
$c^* = 0.1582$	$\gamma^* = 92°59'$

$$p_0:q_0:r_0 = 0.976:0.758:1$$

Projection elements:

$x_o' = 0.2124$	$p_o' = 0.9977$
$y_o' = 0.0024$	$q_o' = 0.7748$

$$\nu = 92°59'$$

Cartesian matrices*:

$$v_1 = 0.0521_4 \qquad v_2 = 0.9985_5$$

Direct matrix:

$$\begin{Vmatrix} 6.485 & 0.435 & 0 \\ 0 & 8.340 & 0 \\ -1.377 & -0.112 & 6.462 \end{Vmatrix} \quad \text{(in Å)}$$

Reciprocal matrix:

$$\begin{Vmatrix} 0.1542 & 0 & 0.0329 \\ -0.0080 & 0.1199 & 0.0004 \\ 0 & 0 & 0.1548 \end{Vmatrix} \quad \text{(in Å}^{-1}\text{)}$$

* Values calculated from direct- and reciprocal-cell elements using equations given by EVANS (1948).

patterns three films interleaved with 0.0005 in. Ni foil were used for each exposure. Patterns about [001] were taken for each level up to *hk*9, and the 0*kl*, *h*0*l* and *h*1*l* levels were also recorded. Exposure times were approximately 70 hours at 50 kV and 20 ma for each level, and, as necessary, short exposures of the order of 2–4 hours were also

made in order to bring the stronger reflections into the range of measurement. Intensities were estimated visually by comparison with a standard spot strip of intensities, prepared from the same meyerhofferite crystal used for the other patterns. Controlled exposures on the standard strip were made so that the intensity of the nth spot is given by $I_n = I_0(2)^{n/2}$, where I_0 corresponds to a barely perceptible blackening of the film. In the reciprocal sphere of radius $s = (\sin\theta)/\lambda = 0.9$ Å^{-1} there are 4342 independent reflections; 2678 of these had observable intensities, 1515 had intensities below the threshold of observation and were assigned the value zero, and 149 reflections, all with $s > 0.8$ Å^{-1}, were not recorded and were omitted from the compilation of data.

The equi-inclination Weissenberg instrument used for this study covered a 200° traverse at one setting, so that two settings were required for each upper level in order to record the entire 360° range. Weissenberg-instrument geometry for upper-level films is such that distortion occurs in the recording of reflections, some being contracted and some, extended. Systematic differences in intensity readings are therefore inevitable. In order to eliminate the systematic errors, comparison was made of different readings for the same reflection which had registered on a given level with each type of distortion. The comparison was carried out for as many reflections as possible on each level, and an average linear scale factor relating readings for contracted and extended reflections was found. This scale factor was then multiplied into readings for the extended reflections in order to place all readings on approximately the same scale.

The estimated intensities were corrected for Lorentz and polarization factors to obtain the F^2_{hkl}'s. For this correction the $1/Lp$ factor was calculated for each reciprocal lattice point, as a function of its cylindrical coordinates, by punched-card methods. Small and nearly equant crystals, about 0.2 mm on edge, were used to minimize absorption effects, but no absorption corrections were made. The F^2_{hkl}'s from various films were all put on the same relative scale by the use of appropriate film factors. Through the use of the triple-film technique, together with short and long exposures, a range of intensities from 1 to 8000 and of F^2_{hkl} from 1 to about 800 was obtained*.

* The ranges of 1 to 10,000 and 1 to 3,000 referred to as ranges of F^2_{hkl} in previous publications by the authors on colemanite, inyoite and $CaB_3O_3(OH)_5 \cdot 2H_2O$ are actually ranges of intensities.

Other considerations

In preparation for application of the Hauptman-Karle phase-determination procedures to the meyerhofferite data, a $K(s)$ curve (KARLE and HAUPTMAN, 1953) was prepared for meyerhofferite (CLARK and CHRIST, 1960). Initially the factor $[K(0)]^{1/2}$ was used to place the data on an approximate absolute scale. At all later stages of the study, observed and calculated structure factors were related by use of the scaling constant k, where $k\Sigma|F_o| = \Sigma|F_c|$.

Refinement of the projection data was carried out with an average isotropic temperature factor, determined by the method of WILSON (1942). Maxima on the electron-density projections were located by the method of BOOTH (1948). Refinement of the three-dimensional data was carried out using one-fifth of the 2678 data that have $|F_o| > 0$, selected by arranging the terms in order of increasing s and taking every fifth one. This portion of the data was given eleven cycles of least-squares refinement on a Burroughs 205 digital computer with a program developed by D. E. APPLEMAN and E. MONASTERSKI, U.S. Geological Survey. Non-diagonal terms were neglected in the solution of the normal equations, except for those terms involving both the temperature and scale factors. Atomic scattering curves were used as follows: for boron, the B^0 curve of IBERS (1957), for all oxygen atoms, the O^0 curve of BERGHUIS *et al.* (1955), and for calcium, a curve constructed by plotting the BERGHUIS *et al.* values for Ca^0 for $(\sin\theta)/\lambda \geq 0.3$ Å^{-1} and smoothing in to $f = 18$ at $(\sin\theta)/\lambda = 0$.

Determination and refinement of the structure

The structural problem consists in determining the coordinates for one calcium, three boron, and nine oxygen atoms (including OH^- and H_2O) in the positions $2(i)$ of space group $P\bar{1}$ (Internat. Tables, 1952). Direct determination of the signs of 2303 of the 2678 terms with $|F_o| > 0$ was made with Hauptman-Karle statistical procedures (CLARK and CHRIST, 1960). The projection data with known signs (F_{hk0}, F_{h0l}, F_{0kl}) were then used to calculate the three electron-density projections, each taken on a plane normal to a principal crystallographic axis. One of these, $\varrho_y(x, z)$, was shown in the preliminary note (Fig. 1, CHRIST and CLARK, 1956). The three maps considered together with a ball model were readily interpreted, and from positions of peak maxima, atomic coordinates were assigned to all thirteen atoms in the asymmetric unit (Table 2, column 1). From structure factors based on these coordinates signs were obtained for over 90% of the

Table 2. *Atomic positional parameters and temperature coefficients for meyerhofferite*

Atom	Parameter[2]	Stage of refinement[1] (1)	(2)	(3)	(4)
Ca	x	0.014	0.010	0.010	0.010
	y	0.377	0.377	0.377	0.377
	z	0.243	0.243	0.244	0.244
	B				0.75
O_1	x	0.410	0.407	0.406	0.406
	y	0.732	0.734	0.733	0.732
	z	0.313	0.327	0.331	0.331
	B				0.99
O_2(OH)	x	0.419	0.422	0.423	0.423
	y	0.890	0.889	0.887	0.888
	z	0.660	0.651	0.643	0.643
	B				1.36
O_3	x	0.118	0.115	0.116	0.116
	y	0.782	0.776	0.776	0.776
	z	0.500	0.502	0.495	0.493
	B				1.09
O_4(OH)	x	0.340	0.339	0.336	0.335
	y	0.461	0.461	0.461	0.461
	z	0.206	0.211	0.203	0.201
	B				1.15
O_5	x	0.058	0.058	0.062	0.063
	y	0.644	0.642	0.650	0.652
	z	0.148	0.145	0.150	0.151
	B				0.70
O_6(OH)	x	0.173	0.165	0.167	0.167
	y	0.370	0.374	0.378	0.378
	z	0.615	0.615	0.617	0.617
	B				1.02
$O_7(H_2O)$	x	0.157	0.132	0.148	0.151
	y	0.108	0.104	0.109	0.109
	z	0.202	0.211	0.216	0.217
	B				1.55
O_8(OH)	x	0.146	0.145	0.151	0.151
	y	0.117	0.126	0.123	0.123
	z	0.792	0.797	0.800	0.800
	B				1.19
O_9(OH)	x	0.329	0.334	0.330	0.330
	y	0.672	0.673	0.668	0.668
	z	0.955	0.955	0.956	0.957
	B				1.08

Table 2 (*continued*)

Atom	Parameter[2]	Stage of refinement[1] (1)	(2)	(3)	(4)
B_1	x	0.312	0.318	0.322	0.321
	y	0.798	0.792	0.798	0.798
	z	0.489	0.497	0.487	0.485
	B				0.86
B_2	x	0.308	0.308	0.290	0.285
	y	0.642	0.642	0.634	0.634
	z	0.183	0.183	0.173	0.167
	B				0.74
B_3	x	0.050	0.030	0.034	0.037
	y	0.233	0.278	0.269	0.267
	z	0.710	0.683	0.695	0.698
	B				0.78
Residual, R					
	$hk0$	0.19	0.18	—	—
	$h0l$	0.19	0.16	—	—
	$0kl$	0.21	0.16	—	—
	hkl	—	—	0.147[3]	0.145[4]
Scale factor, k					
	$hk0$	2.5	2.5	—	—
	$h0l$	2.4	2.4	—	—
	$0kl$	2.6	2.7	—	—
	hkl	—	—	2.9[3]	2.7_3[4]
Average isotropic B					
	$hk0$	0.66	0.66	—	
	$h0l$	1.00	1.00	—	
	$0kl$	0.94	0.94	—	
	hkl	—	—	0.87[3]	

[1] Stages of refinement: (1) from peak positions on electron-density projections calculated with signs determined by Hauptman-Karle procedures; (2) from peak positions of second set of electron-density projections; (3) at end of 5 cycles of least-squares refinement for one-fifth of three-dimensional data, average isotropic B; (4) final parameters at end of 11 cycles of least-squares refinement for one-fifth of three-dimensional data, individual isotropic B's.

[2] x, y, z in cycles; B in Å^2; standard errors in B: $\varepsilon_{Ca} = 0.03$, $\varepsilon_O = 0.17$ $\varepsilon_B = 0.17$ Å^2.

[3] For 535 terms with $|F_{hkl}| > 0$.

[4] For 2678 terms with $|F_{hkl}| > 0$.

Table 3. *Comparison of observed and calculated structure factors for hkl data of meyerhofferite.* Calculated F_{hkl} are based on the atomic parameters of Table 2, column 4

h k l	F_c	$\lvert F_o\rvert$	h k l	F_c	$\lvert F_o\rvert$	h k l	F_c	$\lvert F_o\rvert$	h k l	F_c	$\lvert F_o\rvert$	h k l	F_c	$\lvert F_o\rvert$	h k l	F_c	$\lvert F_o\rvert$
11 6 0	-3		9 $\bar{8}$ 1	-4		8 8 3	3		7 $\bar{4}$ 2	13	12	6 9 1	-3		6 0 6	-15	15
5	11	15	9 9 2	12	13	7	-14	12	-5	-2		8	-3	5	-1	7	6
4	-10	10	8	1		6	5		-6	7	9	7	8	7	-2	5	5
3	3		7	-10	14	5	2		-7	5	6	6	-10	9	-3	-11	10
2	9	11	6	2		4	-5	5	-8	-12	13	5	-5	7	-4	5	
1	-7	6	5	-4		3	7	6	-9	6		4	5	3	-5	1	
0	-1		4	3		2	2		-10	2		3	-13	12	-6	2	
-1	-3		3	-2		1	4		7 11 3	8	6	2	21	19	-7	8	9
-2	-2		2	-5	7	0	2		10	-4		1	-1		-8	-13	11
-3	6		1	13	16	-1	-11	9	9	2		0	-0		6 7 7	-3	
-4	-4		0	0		-2	1		8	-1		-1	12	12	6	6	6
11 5 1	-0		-1	3		-3	0		7	-7	6	-2	-23	19	5	-3	
4	7	8	-2	-3		-4	-7	6	6	11	9	-3	6	3	4	-1	
3	-4		-3	-6	5	-5	9	9	5	-5	5	-4	9	8	3	5	
2	6	6	-4	0		-6	-2		4	-5	6	-5	-14	11	2	-5	6
1	-4	6	-5	-5		-7	4		3	15	12	-6	4		1	3	
0	-3	6	-6	2		-8	1		2	-8	5	-7	3		0	-2	
-1	10	12	-7	9	10	8 9 4	-6		1	-4		-8	-1		-1	-2	
-2	-9	10	-8	-8		8	10	9	0	7	6	-9	11	8	-2	1	
-3	-5	8	9 8 3	2		7	-3		-1	-12	10	-10	-12	9	-3	-3	
11 3 2	-6		7	-6		6	-1		-2	12	10	-11	1		-4	6	
2	-4		6	11	13	5	5		-3	-4		-12	4		-5	6	8
1	7	6	5	-4		4	-11	11	-4	-3		6 12 2	7	8	-6	-12	10
0	-2		4	-15	15	3	8	7	-5	8	8	11	-4	6	6 5 8	11	10
-1	-1		3	13	10	2	-2		-6	-7	6	10	-6	8	4	-5	
10 8 0	3		2	-2		1	-9	8	-7	1		9	5	6	3	5	7
7	-3		1	0		0	9	10	-8	4		8	-0		2	-3	6
6	-4		0	2		-1	-1		-9	-4		7	1	5	1	-8	11
5	10	11	-1	-5	5	-2	1		-10	1		6	2		0	6	6
4	-2		-2	7	6	-3	8	8	7 10 4	-2		5	-1		-1	-3	6
3	-0		-3	-0		-4	-12	14	9	-7		4	12	11	-2	1	
2	6	6	-4	-6	6	-5	8	8	8	7	8	3	-7	7	-3	7	8
1	-5		-5	5		-6	-3		7	-2		2	-8	9	-4	-5	
0	3	5	-6	-2		-7	-11	10	6	0		1	-1		5 14 0	-4	
-1	-4		-7	-2		8 7 5	11	10	5	3		0	-14	12	13	2	
-2	-10	12	9 6 4	-3		6	-4		4	-2		-1	15	15	12	-3	
-3	9	9	5	7	8	5	4		3	4	5	-2	-2		11	10	12
-4	-1		4	-4		4	5		2	-4	6	-3	3		10	4	
-5	5	7	3	9	9	3	-10	8	1	-9	10	-4	10	10	9	-9	8
-6	1		2	-4		2	-1		0	16	14	-5	-8	8	8	10	10
-7	-5		1	-11	14	1	-1		-1	-8	7	-6	3		7	-1	
10 8 1	-1		0	13	13	0	-4		-2	-4	5	-7	5	6	6	-19	18
7	8	10	-1	-6		-1	6	5	-3	11	12	-8	-20	18	5	-3	
6	-9	9	-2	-4		-2	-3		-4	-8	8	-9	3		4	-14	17
5	3		-3	5		-3	5		-5	4		-10	6		3	30	32
4	4	5	-4	-3		-4	4		-6	0		-11	-2		2	9	11
3	-5	6	-5	7	7	-5	-6		-7	-7	9	6 12 3	-2	6	1	-1	
2	5	7	9 4 5	5	8	-6	3		-8	4		11	8	6	0	13	16
1	-5	5	3	-10	6	8 5 6	-6		-9	1		10	-13	9	-1	-11	10
0	-6	8	2	6		4	9	11	7 9 5	1		9	2		-2	-6	7
-1	12	13	1	1		3	-4		8	-1		8	-0		-3	-8	8
-2	-6		0	-6		2	-2		7	5		7	-17	13	-4	-15	16
-3	5		-1	2		1	10	9	6	-6	6	6	21	17	-5	8	7
-4	-1		-2	-1		0	-8	9	5	2		5	7	7	-6	2	
-5	-9	11	-3	3		-1	-1		4	-1		4	-13	12	-7	5	4
-6	10	10	8 11 0	-3		-2	7	7	3	-11	12	3	19	16	-8	20	21
10 7 2	-1		10	2		-3	-10	9	2	10	10	2	-12	11	-9	-18	18
6	8	9	9	-7	7	-4	4		1	-1		1	2		-10	1	
5	-5		8	8	9	7 12 0	-10	11	0	0		0	-3	4	-11	5	6
4	1		7	4		11	-1		-1	7	7	-1	-18	16	-12	-9	10
3	1		6	-11	14	10	12	12	-2	-8	10	-2	11	9	-13	4	
2	-9	10	5	14	17	9	-14	15	-3	11	7	-3	2		5 13 1	4	
1	10	10	4	-5		8	13	16	-4	-9	10	-4	2		12	3	
0	-0		3	-2		7	7	11	-5	-9	9	-5	10	10	11	-10	8
-1	-4		2	3	5	6	-7	8	-6	11	9	-6	-9	8	10	11	10
-2	5		1	-11	11	5	0		-7	-5		-7	2		9	0	
-3	-5		0	2		4	-15	15	-8	1		-8	1		8	-8	7
-4	5		-1	5	6	3	-1		7 7 6	5	7	-9	-7	6	7	14	11
-5	-1		-2	-1		2	3		6	6	6	-10	8	7	6	-23	20
10 5 3	-10	11	-3	6	5	1	-11	13	5	-9	8	-11	-11		5	18	17
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3	9	9	-5	0		-1	10	12	3	1		10	-1		3	-41	43
2	-8	8	-6	1		-2	-6	5	2	-8	10	9	-2		2	19	18
1	4		-7	-7	10	-3	7	7	1	7		8	6	6	1	5	6
0	6		-8	7	7	-4	-18	19	0	-5		7	-2		0	-11	10
-1	-8	6	-9	-5	6	-5	9	10	-1	5		6	-5	5	-1	18	17
-2	11	8	-10	2		-6	-7	6	-2	3		5	1		-2	-14	14
-3	-5		8 11 1	-2		-7	-14	19	-3	-7	7	4	-11	9	-3	-1	
-4	-7	6	10	7	8	-8	18	20	-4	4		3	12	13	-4	23	23
10 3 4	-2		9	-3	4	-9	1		-5	2		2	2		-5	-20	18
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-7	1		9	-2		-3	-15	13	10	12	12	1	5	7	7	-13	12

Table 3 (*continued*)

h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$
5 6 5	14	11	5 0 9	2		4 7 4	- 6	6	3 8 0	- 2		3 1 4	-23	22	3 1 10	1	
5	- 2		- 1	9	7	6	- 4	5	7	-13	13	0	19	20	0	-11	11
4	- 3	4	- 2	-12	11	5	8	8	6	6	8	- 1	-11	10	- 1	7	9
3	2		- 3	3		4	-13	12	5	18	19	- 2	- 0		- 2	0	
2	-19	18	4 14 0	- 1		3	13	12	4	2		- 3	16	16	- 3	- 6	
1	6	7	13	5	6	2	13	13	3	16	20	- 4	- 5	6	- 4	12	
0	11	11	12	- 4	7	1	-18	17	2	1		- 5	1		2 15 0	3	
- 1	- 2		11	6	8	0	25	21	1	-27	30	- 6	4	5	14	5	
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- 3	- 9	9	9	-17	19	- 2	-19	19	- 1	-58	67	- 8	4	5	12	-10	11
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- 6	-22	19	6	16	16	- 5	6	6	- 4	- 7	5	-11	4		9	-13	11
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4	-15	12	- 9	-13	12	5	6	6	10	6	7	0	- 6	6	- 6	-14	12
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5 10 6	- 2		9	11	9	-10	- 3		1	31	32	8	7	5	2 14 2	0	
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5 9 7	1		-11	- 8		- 9	- 7		10	- 9	8	8	5		- 6	1	
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- 7	0		0	2		- 7	0		- 6	- 8	8	- 8	8	9	7	- 5	4
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6	0		- 3	-21	18	5	6	6	- 9	- 4		6	-12	9	4	- 1	
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0	1		- 9	2		- 1	9	10	12	-12	11	0	- 2		- 2	7	
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- 6	- 2		12	- 7	6	13	6	7	6	0		- 6	6	7	- 8	1	
5 4 9	0		11	11	8	12	- 4	6	5	25	21	- 7	- 4	6	- 9	- 6	6
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2	6	5	9	-17	15	10	- 0		3	7	6	1	- 8		-11	- 3	
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Table 3 (*continued*)

h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$
2-13 3	5		2 $\bar{8}$ 8	10	13	1 12 3	- 2		1 $\bar{3}$ 7	-16	15	0 4 2	39	37	0 $\bar{5}$ 6	- 7	5
-14	- 6		- 9	- 4		11	11	8	- 4	1		7	4	5	- 6	- 3	
2 13 4	4	8	2 8 9	- 1		10	- 5	6	- 5	2		2	1	4	- 7	5	5
12	- 6	6	7	6	7	9	2		- 6	- 1		1	7	4	- 8	-13	9
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9	-13	11	4	- 1		6	- 1		- 9	- 5	7	- 2	-40	36	-11	- 2	
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7	-15	14	2	2		4	18	10	-11	- 6	9	- 4	29	28	0 11 7	6	
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5	21	18	0	- 2		2	3	4	9	- 3		- 6	9	10	9	2	
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3	11	11	- 2	- 3		0	-11	14	7	- 8	6	- 8	-18	18	7	-11	8
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- 7	2		2	- 3		-10	6	6	- 3	9	9	11	11	9	- 3	5	6
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Table 3 (*continued*)

h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$
$\bar{1}$ 12 1	- 4		$\bar{1}$ 9 6	5	5	$\bar{2}$ 6 1	-16	18	$\bar{2}$ 0 5	- 0		$\bar{2}$ 4 11	4		$\bar{3}$ 11 4	3	
-13	- 6	6	8	-11	10	5	27	26	- 1	- 2		3	2			-12	-13	13
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-15	- 5		6	-10	10	3	-29	30	- 3	4	4	1	9	11		-14	- 0	
$\bar{1}$ 14 2	- 0		5	- 7	6	2	29	32	- 4	- 6	4	0	- 8	8	$\bar{3}$ 13 5	9	10	
13	0		4	18	18	1	- 1		- 5	9	8	- 1	2			12	- 3	
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0	-17	15	- 9	11	9	-12	5		8	- 8	7	6	- 9	8		- 1	2	3
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$\bar{1}$ 14 3	12	13	2	-11	8	3	11	12	- 7	3	5	- 9	8	7		10	2	
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$\bar{1}$ 14 4	- 1		- 4	-19	16	3	-12	14	-11	- 5		- 9	22	20		6	10	9
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$\bar{1}$ 13 5	4		5	-10		2	5	3	4	- 3	4	- 9	- 2			0	4	6
12	- 7	6	4	8		1	-16	15	3	- 3		-10	4	6		- 1	2	
11	- 3		3	- 4		0	18	18	2	10	6	-11	-10	8		- 2	10	8
10	16	14	2	- 1		- 1	-29	28	1	-10	9	-12	11	8		- 3	- 1	3
9	- 8	9	1	8	10	- 2	18	17	0	14	13	-13	1			- 4	-13	11
8	4	5	0	- 4		- 3	10	14	- 1	- 0		-14	- 6			- 5	5	5
7	8	8	- 1	9	8	- 4	-32	29	- 2	-13	9	$\bar{3}$ 13 4	5			- 6	1	
6	-20	20	- 2	- 2		- 5	27	24	- 3	12	9	12	- 6	7		- 7	- 4	
5	21	21	- 3	- 8		- 6	0		- 4	-10	8	11	11	10		- 8	1	
4	-19	21	- 4	5		- 7	- 9	9	- 5	- 4	3	10	- 4	6		- 9	- 5	
3	-23	20	- 5	- 7		- 8	9	9	- 6	9	9	9	- 7	6		-10	9	
2	25	16	- 6	0		- 9	- 9	9	- 7	- 6	7	8	12	11		-11	- 1	
1	7	7	- 7	4		-10	3		- 8	8	7	7	-15	14		$\bar{3}$ 9 9	- 9	8
0	2		$\bar{1}$ 4 11	6		-11	4	5	- 9	3	5	6	- 8	6		8	- 1	
- 1	10	9	3	- 4		-12	-15	17	$\bar{2}$ 7 10	2		5	18	16		7	5	5
- 2	-13	10	2	- 7		-13	12	10	6	0		4	-13	13		6	- 8	7
- 3	9	7	1	11	15	-14	1		5	- 5		3	26	24		5	10	10
- 4	- 6	5	0	- 7		$\bar{2}$ 13 5	5		4	6		2	- 1			4	- 5	5
- 5	-15	12	- 1	- 5		12	- 2		3	- 3		1	-13	14		3	3	
- 6	11	9	- 2	9		11	- 2		2	3		0	20	21		2	11	8
- 7	- 7	6	- 3	- 6		10	16	13	1	5		- 1	-34	34		1	-14	12
- 8	6	7	- 4	8		9	- 8	9	0	- 8	10	- 2	- 0			0	4	
- 9	3	5	$\bar{2}$ 14 1	- 7	5	8	- 5	6	- 1	3		- 3	- 3	7		- 1	- 1	
-10	- 1		13	- 7	7	7	- 5	6	- 2	- 4		- 4	-12	11		- 2	-12	8
-11	3		12	1		6	- 9	9	- 3	- 2		- 5	15	17		- 3	7	7
-12	- 4		11	-16	13	5	2	4	- 4	3		- 6	10	8		- 4	3	3
-13	- 2		10	21	18	4	4	4	- 5	- 1		- 7	0			- 5	3	3
$\bar{1}$ 12 6	5		9	- 3	5	3	8	7	- 6	3		- 8	6	6		- 6	6	6
11	- 4		8	- 8	8	2	20	12	- 7	3		- 9	-22	19		- 7	-11	9
10	1		7	7	6	1	- 5	6	- 8	- 6		-10	8	7		- 8	10	7

Table 3 (*continued*)

h k l	F_c	$\lvert F_o \rvert$
$\bar{3}$ $\bar{9}$ 9	- 2	
$\bar{3}$ 7 10	5	
6	- 9	
5	- 3	
4	14	
3	- 7	
2	5	
1	4	
0	- 7	10
- 1	4	
- 2	- 4	
- 3	- 6	
- 4	8	
- 5	- 7	
- 6	5	
- 7	7	
- 8	- 6	
$\bar{3}$ 4 11	3	
3	- 7	
2	- 1	
1	8	11
0	- 7	8
- 1	0	
- 2	8	
- 3	- 5	
- 4	3	
- 5	0	
$\bar{4}$ 13 1	7	6
12	- 9	9
11	0	
10	5	
9	- 7	7
8	5	6
7	- 1	
6	- 7	6
5	30	30
4	-28	28
3	- 9	9
2	24	24
1	-3[illegible]	38
0	30	30
- 1	11	13
- 2	-20	21
- 3	11	11
- 4	- 3	4
- 5	- 6	5
- 6	10	10
- 7	-26	21
- 8	13	11
- 9	12	10
-10	- 7	6
-11	5	
-12	- 8	8
-13	2	
-14	4	
$\bar{4}$ 13 2	- 9	7
12	8	8
11	- 2	
10	- 6	5
9	8	8
8	- 8	8
7	13	12
6	0	
5	-25	26
4	21	22
3	-11	13
2	9	12
1	4	5
0	-15	17
- 1	18	20
- 2	3	4
- 3	-20	21
- 4	14	17
- 5	-19	20
- 6	2	5
- 7	18	18
- 8	- 9	9
- 9	6	7
-10	- 8	9
-11	3	5
-12	not calc.	7
-13	-10	12
-14	1	
$\bar{4}$ 13 3	- 8	9
12	5	
11	3	
10	- 3	
9	13	13
8	- 0	
7	-14	14
6	5	5
5	-17	15
4	10	10
3	- 7	6
2	- 9	9
1	42	41
0	3	
- 1	-14	18
- 2	3	
- 3	-23	22
- 4	- 2	
- 5	14	13
- 6	-18	16
- 7	17	14
- 8	1	
- 9	- 2	
-10	9	7
-11	- 9	6
-12	- 6	
-13	4	
-14	0	
$\bar{4}$ 13 4	4	
12	- 7	
11	3	
10	1	
9	- 6	
8	11	9
7	- 4	6
6	6	7
5	- 0	
4	-14	16

h k l	F_c	$\lvert F_o \rvert$
$\bar{4}$ 3 4	15	16
2	-10	10
1	- 0	
0	-10	11
- 1	- 5	6
- 2	30	30
- 3	1	
- 4	-18	19
- 5	21	19
- 6	-16	13
- 7	1	
- 8	7	7
- 9	-20	18
-10	11	11
-11	4	6
-12	- 3	
-13	9	10
$\bar{4}$ 12 5	- 3	
11	- 6	6
10	11	7
9	-17	12
8	- 5	5
7	13	12
6	-17	14
5	26	18
4	11	8
3	-14	14
2	6	5
1	-15	15
0	-11	11
- 1	11	12
- 2	-21	20
- 3	11	13
- 4	20	18
- 5	1	
- 6	6	6
- 7	-17	17
- 8	2	
- 9	- 0	
-10	- 7	7
-11	6	6
-12	- 1	
-13	1	
$\bar{4}$ 12 6	2	
11	- 2	
10	3	
9	- 3	
8	- 4	5
7	12	13
6	-15	15
5	1	
4	7	11
3	-12	13
2	18	16
1	1	
0	-13	13
- 1	11	9
- 2	- 2	
- 3	- 5	6
- 4	5	6
- 5	-19	14
- 6	12	11
- 7	7	8
- 8	- 3	
- 9	5	4
-10	- 5	7
-11	2	
-12	2	
$\bar{4}$ 11 7	5	
10	- 7	7
9	11	11
8	1	
7	- 6	8
6	11	10
5	-11	11
4	1	
3	- 5	7
2	-11	11
1	15	17
0	5	5
- 1	- 1	
- 2	12	11
- 3	-18	18
- 4	4	4
- 5	3	5
- 6	-13	12
- 7	7	9
- 8	- 4	
- 9	7	7
-10	7	9
-11	- 8	11
$\bar{4}$ 10 8	- 2	
9	- 4	
8	9	9
7	- 7	8
6	5	6
5	- 4	
4	- 8	9
3	13	14
2	- 1	
1	- 4	4
0	7	6
- 1	-12	11
- 2	6	6
- 3	7	6
- 4	-15	14
- 5	11	10
- 6	- 3	
- 7	1	
- 8	8	7
- 9	- 9	8
-10	- 2	
$\bar{4}$ 8 9	1	
7	- 2	
6	- 4	3
5	7	5
4	- 2	7
3	1	4
2	- 1	
1	- 2	4
0	7	8

h k l	F_c	$\lvert F_o \rvert$
$\bar{4}$ $\bar{1}$ 9	- 4	5
- 2	- 8	7
- 3	8	8
- 4	- 7	6
- 5	9	7
- 6	3	4
- 7	-12	11
- 8	7	7
- 9	- 1	
$\bar{4}$ 7 10	13	
6	- 2	
5	- 4	
4	10	
3	- 6	
2	1	
1	3	7
0	-16	16
- 1	7	
- 2	2	
- 3	0	
- 4	8	
- 5	- 8	
- 6	4	
- 7	- 0	
$\bar{4}$ 4 11	7	
3	- 2	
2	- 6	
1	8	9
0	- 1	
- 1	- 2	
- 2	4	
- 3	- 9	
- 4	5	
- 5	2	
$\bar{5}$ 13 1	9	6
12	- 4	4
11	-10	9
10	6	7
9	- 8	8
8	8	9
7	- 1	
6	-12	11
5	28	27
4	- 2	4
3	- 9	9
2	0	
1	-14	14
0	- 0	
- 1	9	8
- 2	-15	15
- 3	19	18
- 4	-11	9
- 5	4	4
- 6	22	17
- 7	-18	15
- 8	- 3	4
- 9	4	4
-10	- 4	4
-11	4	
-12	- 3	
-13	- 4	
-14	10	8
$\bar{5}$ 13 2	- 5	
12	7	7
11	- 2	
10	8	9
9	4	
8	-20	20
7	15	17
6	- 5	10
5	-15	16
4	1	
3	2	
2	25	33
1	16	16
0	-20	23
- 1	8	9
- 2	-16	19
- 3	-13	13
- 4	6	9
- 5	-16	17
- 6	20	24
- 7	- 0	
- 8	4	
- 9	15	14
-10	-19	19
-11	- 5	6
-12	10	13
-13	-11	12
-14	6	
$\bar{5}$ 13 3	- 6	
12	1	
11	7	7
10	-11	11
9	14	12
8	0	
7	- 8	8
6	4	4
5	-18	18
4	13	14
3	17	15
2	-33	29
1	12	12
0	25	25
- 1	-21	22
- 2	15	14
- 3	-17	17
- 4	- 1	
- 5	13	12
- 6	-10	10
- 7	2	
- 8	3	
- 9	- 7	5
-10	5	7
-11	2	
-12	- 1	
-13	- 1	
$\bar{5}$ 12 4	- 1	
11	5	
10	- 6	7
9	- 0	

h k l	F_c	$\lvert F_o \rvert$
$\bar{5}$ 8 4	1	
7	-11	12
6	18	18
5	- 2	
4	- 4	6
3	7	10
2	-11	12
1	9	9
0	2	
- 1	-39	40
- 2	30	31
- 3	14	17
- 4	-16	16
- 5	16	14
- 6	-12	12
- 7	0	
- 8	5	5
- 9	-13	11
-10	6	
-11	4	
-12	- 7	
-13	10	11
$\bar{5}$ 12 5	- 3	
11	-13	10
10	14	13
9	- 7	6
8	- 0	
7	9	8
6	- 7	5
5	5	5
4	2	
3	- 8	7
2	6	4
1	-18	15
0	3	5
- 1	21	21
- 2	- 8	10
- 3	6	7
- 4	-12	13
- 5	7	7
- 6	6	7
- 7	-17	15
- 8	3	4
- 9	8	6
-10	- 5	5
-11	7	6
-12	- 3	
-13	- 1	
$\bar{5}$ 11 6	- 5	
10	4	
9	- 5	
8	- 7	
7	18	17
6	- 2	
5	- 5	6
4	7	9
3	-11	12
2	- 1	
1	7	9
0	-11	12
- 1	12	13
- 2	-10	9
- 3	5	5
- 4	16	15
- 5	-17	17
- 6	- 2	
- 7	6	7
- 8	- 4	4
- 9	5	5
-10	- 5	4
-11	- 3	6
-12	10	12
$\bar{5}$ 10 7	- 7	9
9	12	14
8	- 3	6
7	- 8	9
6	- 3	4
5	- 4	4
4	9	12
3	4	5
2	- 6	7
1	14	13
0	- 6	7
- 1	- 2	5
- 2	0	
- 3	-14	16
- 4	6	8
- 5	- 5	5
- 6	9	9
- 7	12	11
- 8	-12	12
- 9	2	
-10	6	8
-11	-12	13
$\bar{5}$ 9 8	- 1	
8	4	
7	-10	11
6	8	8
5	2	
4	-22	19
3	13	14
2	7	10
1	- 7	7
0	15	11
- 1	-14	15
- 2	3	
- 3	5	6
- 4	-13	12
- 5	6	6
- 6	1	
- 7	- 7	5
- 8	9	9
- 9	- 0	
-10	- 1	
$\bar{5}$ 8 9	6	8
7	- 1	
6	- 1	
5	3	5
4	- 0	
3	6	6
2	- 4	5

h k l	F_c	$\lvert F_o \rvert$
$\bar{5}$ 1 9	-19	18
0	14	14
- 1	0	4
- 2	- 5	6
- 3	12	11
- 4	- 4	5
- 5	3	
- 6	2	
- 7	-11	9
- 8	2	
- 9	- 0	
$\bar{5}$ 6 10	2	
5	- 0	
4	7	
3	-10	
2	3	
1	5	
0	- 8	7
- 1	1	
- 2	- 5	
- 3	8	
- 4	4	
- 5	- 8	
- 6	4	
- 7	1	
$\bar{5}$ 3 11	3	
2	- 7	
1	7	8
0	- 5	
- 1	4	
- 2	8	
- 3	-11	
- 4	- 0	
$\bar{6}$ 12 1	- 1	
11	- 5	
10	6	5
9	- 3	
8	8	8
7	5	
6	- 9	8
5	- 4	4
4	- 4	
3	- 2	
2	16	15
1	-20	19
0	11	12
- 1	18	18
- 2	-17	18
- 3	6	5
- 4	- 7	6
- 5	- 8	8
- 6	11	12
- 7	- 2	
- 8	- 2	4
- 9	11	11
-10	- 9	8
-11	1	
-12	0	
-13	- 2	
$\bar{6}$ 12 2	8	10
11	- 7	8
10	6	7
9	1	
8	-10	12
7	5	
6	- 1	6
5	4	5
4	10	10
3	-14	16
2	9	11
1	0	
0	-12	13
- 1	12	14
- 2	-28	29
- 3	16	20
- 4	17	19
- 5	-16	16
- 6	13	11
- 7	1	
- 8	-15	15
- 9	12	12
-10	-11	11
-11	- 2	
-12	12	14
-13	- 9	9
$\bar{6}$ 12 3	9	11
11	5	
10	-13	11
9	8	6
8	- 3	6
7	-13	12
6	9	9
5	- 8	5
4	3	4
3	28	26
2	-13	13
1	6	4
0	-11	12
- 1	-12	12
- 2	7	8
- 3	- 8	9
- 4	1	
- 5	8	9
- 6	2	
- 7	8	9
- 8	- 7	8
- 9	- 5	6
-10	6	6
-11	-11	14
-12	6	
-13	1	
$\bar{6}$ 12 4	- 6	
11	7	10
10	- 2	
9	3	
8	2	
7	- 9	8
6	8	9
5	4	
4	-18	20
3	9	12

h k l	F_c	$\lvert F_o \rvert$
$\bar{6}$ 2 4	- 9	8
1	1	
0	22	21
- 1	-11	13
- 2	7	5
- 3	1	
- 4	- 9	10
- 5	2	
- 6	- 8	6
- 7	- 7	7
- 8	13	14
- 9	- 4	
-10	7	8
-11	3	
-12	- 6	
$\bar{6}$ 11 5	- 4	
10	0	
9	- 7	6
8	11	10
7	8	7
6	- 7	7
5	9	6
4	- 9	8
3	- 6	5
2	7	7
1	-23	21
0	14	16
- 1	6	7
- 2	1	
- 3	14	13
- 4	-10	10
- 5	- 4	4
- 6	4	4
- 7	-12	10
- 8	6	5
- 9	- 1	
-10	- 2	
-11	12	12
-12	- 9	8
$\bar{6}$ 11 6	- 4	
10	5	9
9	4	
8	- 9	10
7	3	
6	0	
5	- 3	
4	12	12
3	-15	15
2	9	10
1	8	9
0	-12	14
- 1	9	9
- 2	- 9	9
- 3	- 9	8
- 4	15	15
- 5	- 6	6
- 6	0	
- 7	11	9
- 8	-10	10
- 9	6	5
-10	- 3	
-11	- 6	6
$\bar{6}$ 10 7	- 4	
9	3	
8	0	
7	- 1	
6	0	
5	-10	11
4	7	10
3	2	5
2	0	
1	9	8
0	-14	16
- 1	8	9
- 2	- 1	
- 3	-12	14
- 4	10	10
- 5	- 3	
- 6	- 5	6
- 7	16	14
- 8	-10	10
- 9	0	
-10	6	8
-11	-10	13
$\bar{6}$ 9 8	- 4	
8	6	
7	-13	10
6	3	
5	12	10
4	-10	9
3	9	9
2	- 4	
1	- 3	
0	5	11
- 1	- 9	9
- 2	0	
- 3	4	6
- 4	- 2	
- 5	7	7
- 6	- 3	6
- 7	- 1	
- 8	2	
- 9	- 8	12
$\bar{6}$ 7 9	3	
6	- 7	8
5	7	7
4	- 8	8
3	2	4
2	9	10
1	-12	13
0	5	5
- 1	5	6
- 2	- 5	7
- 3	6	6
- 4	- 4	5
- 5	- 5	7
- 6	7	8
- 7	- 9	7
- 8	5	5
$\bar{6}$ 5 10	2	
4	3	

Table 3 (*continued*)

h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$	h k l	F_c	$\lvert F_o \rvert$
$\bar{6}$ 3 10	-12		$\bar{7}$ 1 5	- 7	7	$\bar{8}$ 10 2	0		$\bar{8}$ $\bar{5}$ 7	- 4	4	$\bar{9}$ 4 5	- 0		$\bar{10}$ $\bar{1}$ 4	- 8	10
2	3		0	10	9	9	2		- 6	- 1		3	8	9	- 2	5	
1	- 2		- 1	6	4	8	- 4		- 7	6	5	2	8	7	- 3	0	
0	3		- 2	- 9	8	7	8	9	- 8	- 6	6	1	- 9	12	- 4	- 9	9
- 1	7	9	- 3	- 1		6	- 2		- 9	- 3		0	5		- 5	13	20
- 2	- 4		- 4	-10	9	5	3		$\bar{8}$ 6 8	9		- 1	- 8	10	- 6	- 0	
- 3	3		- 5	5	6	4	1		5	3		- 2	- 7	7	- 7	0	
- 4	4		- 6	4	5	3	-10	11	4	- 8		- 3	6	6	- 8	1	
- 5	- 9		- 7	-12	9	2	- 2		3	5		- 4	- 1		$\bar{10}$ 6 5	- 2	
$\bar{7}$ 6	2		- 8	17	13	1	1		2	- 6	7	- 5	1		5	8	
$\bar{6}$ 1 11	8	9	- 9	- 2		0	- 5		1	- 3		- 6	5		4	- 8	
0	- 7		-10	- 5	6	- 1	8	7	0	7	8	- 7	- 0		3	2	
- 1	- 4		$\bar{11}$	7	7	- 2	0		- 1	- 9	9	- 8	7		2	6	
- 2	9		7 10 6	3		- 3	5	6	- 2	8	10	$\bar{9}$	- 6		1	- 6	
$\bar{7}$ 11 1	- 1		9	4		- 4	9	8	- 3	1		$\bar{9}$ 7 6	3		0	3	
10	5		8	- 4		- 5	-11	12	- 4	- 1		6	- 8	10	- 1	- 2	
9	- 5		7	-12	11	- 6	1		- 5	5		5	1		- 2	- 6	
8	3		6	-13	13	- 7	- 8	8	- 6	- 6		4	11	15	- 3	9	
7	6	6	5	- 2		- 8	- 2		- 7	- 3		3	-16	20	- 4	- 5	
6	- 8	8	4	14	15	- 9	9	12	$\bar{8}$	2		2	6	7	- 5	2	
5	14	11	3	-17	17	-10	- 3		$\bar{8}$ 5 9	3		1	3		- 6	3	
4	-13	12	2	1		-11	0		4	- 5	7	0	- 3		- 7	-11	
3	- 9	7	1	8	11	$\bar{8}$ 10 3	-11	15	3	5	6	- 1	9	10	$\bar{8}$	10	
2	22	20	0	- 7	6	9	8	9	2	8	10	- 2	- 8	11	$\bar{10}$ 5 6	- 0	
1	-16	17	- 1	8	8	8	- 8	7	1	-13	16	- 3	- 1		4	9	11
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- 4	0		- 6	2		3	5		- 4	- 8	9	$\bar{8}$	- 7	8	- 1	5	
- 5	- 5	7	- 7	4	5	2	-17	16	- 5	0		$\bar{9}$ 6 7	9	9	- 2	- 0	
- 6	13	10	- 8	- 3		1	4		$\bar{8}$ 6	8	10	5	- 6	7	- 3	- 1	
- 7	-13	13	- 9	2		0	- 7	6	8 1 10	4		4	1		- 4	3	
- 8	- 2		-10	- 4		- 1	- 5		0	2		3	- 4		- 5	- 4	
- 9	4		$\bar{11}$	4	6	- 2	18	16	- 1	3		2	- 3		- 6	3	
-10	- 2		7 9 7	2		- 3	-10	11	$\bar{8}$ 2	- 9		1	7	6	$\bar{8}$ 7	- 3	
-11	3	4	8	- 6	7	- 4	8	8	$\bar{9}$ 9 1	-10	11	0	-14	12	$\bar{10}$ 4 7	6	6
-12	- 4		7	- 2		- 5	2		8	2		- 1	7	8	3	- 8	8
$\bar{7}$ 11 2	- 1		6	7	9	- 6	- 4		7	5		- 2	12	9	2	- 3	
10	3		5	- 2		- 7	5		6	- 5	5	- 3	- 6	4	1	17	20
9	- 1		4	9	10	- 8	- 9	12	5	5	5	- 4	6	4	0	- 8	8
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6	- 5		1	5	5	-11	- 2		2	18	19	$\bar{8}$ 7	9	8	- 3	-11	10
5	0		0	-15	15	$\bar{8}$ 10 4	- 2		1	-18	21	$\bar{9}$ 5 8	4		- 4	5	5
4	15	15	- 1	- 6	7	9	1		0	1		4	- 3		- 5	- 1	
3	- 6	7	- 2	13	15	8	5		- 1	8	9	3	5		$\bar{10}$ 2 8	- 2	
2	10	11	- 3	- 8	7	7	-10	9	- 2	- 6	6	2	- 5		1	- 1	
1	-12	14	- 4	7	6	6	5	10	- 3	11	- 9	1	- 2		0	1	
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- 4	21	23	- 9	- 2		1	4		- 8	2		- 4	3		4	- 5	
- 5	- 5		$\bar{10}$	6	7	0	17	15	- 9	4		- 5	5		3	- 0	
- 6	3		7 8 8	4		- 1	-21	25	$\bar{10}$	- 7	5	$\bar{9}$ 6	- 4		2	- 0	
- 7	3		7	- 9		- 2	4		$\bar{9}$ 9 2	2		$\bar{9}$ 2 9	1	19	1	- 4	
- 8	- 7	9	6	7		- 3	2		8	- 1		1	- 7	10	0	6	7
- 9	5		5	- 3		- 4	-14	17	7	6	6	0	7	9	- 1	2	
-10	-12	14	4	- 3	11	- 5	12	17	6	- 3		- 1	- 3	7	- 2	- 4	7
-11	1		3	12	7	- 6	- 6	8	5	1		- 2	- 6	9	- 3	3	
$\bar{12}$	8	10	2	- 5		- 7	0		4	9	9	$\bar{8}$ 3	10	16	- 4	- 5	6
$\bar{7}$ 11 3	5		1	- 1		- 8	12	16	3	- 9	11	$\bar{10}$ 7 1	1		- 5	5	
10	-13	17	0	4		- 9	- 4		2	- 2		6	- 4		$\bar{11}$ 6	- 2	
9	5		- 1	- 8	8	-10	- 1		1	- 3		5	12	13	$\bar{11}$ 5 2	- 2	
8	0		- 2	6	7	-11	- 1		0	- 0		4	- 6	8	4	8	9
7	- 5		- 3	- 4	7	$\bar{8}$ 9 5	- 3		- 1	16	18	3	- 3	5	3	- 7	7
6	11	12	- 4	- 5	6	8	2		- 2	-16	20	2	11	12	2	4	
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4	12	13	- 6	-11	11	6	- 4		- 4	14	16	0	0		0	- 7	9
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2	-15	15	- 8	6	7	4	- 4		- 6	1		- 2	- 3		- 2	- 4	7
1	16	17	$\bar{9}$	- 5	6	3	1		- 7	- 0		- 3	6	6	- 3	- 1	
0	- 9	9	7 6 9	- 6	6	2	5		- 8	- 9	12	- 4	0		- 4	6	
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- 2	12	12	4	- 4	4	0	6	5	$\bar{10}$	- 3		- 6	2		6	6	9
- 3	- 8	9	3	- 4		- 1	1		$\bar{9}$ 9 3	8	9	- 7	- 4		$\bar{11}$ 5 3	- 4	
- 4	8	8	2	2		- 2	- 2		8	1		$\bar{8}$	6	6	4	3	
- 5	0		1	- 4	5	- 3	5	5	7	- 3		$\bar{10}$ 7 2	8	8	5	- 2	
- 6	-14	14	0	7	7	- 4	-16	14	6	6		2	- 9	10	- 2	- 1	
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-11	- 5		- 5	- 5	6	- 9	- 0		1	10	9	1	- 0		- 3	-10	12
-12	2		- 6	4	5	$\bar{10}$	- 4	6	0	- 5	6	0	- 5	8	- 4	3	
$\bar{7}$ 11 4	- 2		$\bar{9}$ 7	- 6	6	$\bar{8}$ 9 6	- 1		- 1	1		- 1	20	24	- 5	0	
10	1		7 4 10	2		8	-10	9	- 2	6		- 2	- 5		- 6	- 0	
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2	- 3		- 4	- 1		0	-11	11	$\bar{10}$	4		$\bar{10}$ 7 3	1		- 3	- 1	
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0	6		8 10 1	13	13	- 2	-13	15	7	-12	12	5	- 4		- 5	7	
- 1	- 7	6	9	-12	13	- 3	3		6	10	10	4	1		- 6	- 6	
- 2	9	9	8	4		- 4	13	13	5	5		3	- 8	13	$\bar{11}$ 4 5	- 0	
- 3	4	5	7	4		- 5	- 8	10	4	-15	15	2	- 0		3	- 1	
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-12	- 5		- 2	-17	17	5	- 3		- 5	16	21	- 7	9	9	$\bar{11}$ 2 6	3	
$\bar{7}$ 10 5	11	10	- 3	10	10	4	3		- 6	- 2		- 8	- 6		1	2	
9	-11	8	- 4	- 9	11	3	2		- 7	- 9	14	$\bar{10}$ 7 4	-12	15	0	- 4	
8	4	5	- 5	- 2		2	- 8	6	- 8	7	9	6	2		- 1	3	
7	8	7	- 6	15	13	1	5	4	- 9	- 5		5	4		- 2	- 3	
6	-12	12	- 7	- 9	8	0	- 5	3	$\bar{10}$	5		4	- 5		- 3	3	
5	4		- 8	4		- 1	- 1		$\bar{9}$ 8 5	- 1		3	13	14	- 4	- 4	
4	-12	11	- 9	- 1		- 2	9	7	7	0		2	- 5				
3	5	5	-10	- 6	7	- 3	- 4	4	6	- 4		1	- 2				
2	10	8	-11	9	8	- 4	3		5	- 0		0	3				

projection terms with $|F_o| > 0$. Using these terms a second set of electron-density projections was prepared. Coordinates (Table 2, column 2) found from the peak maxima of this second set of maps were taken as the beginning ones for digital-computer least-squares refinement.

As stated above, in order to facilitate refinement only one-fifth of the 2678 data with $|F_o| > 0$ were used. Five cycles of refinement with a general isotropic temperature factor were completed and the resulting coordinates and factors are given in Table 2, column 3, together with the residual (0.14_7) for the 536 *hkl* terms used. Finally six cycles of least-squares refinement (536 terms) using an individual isotropic temperature factor for each atom were completed; this refinement was stopped when the shifts indicated in coordinates became less than the calculated errors. The final set of coordinates, the individual temperature factors, the scale factor and residual (0.14_5) for all 2678 *hkl* terms with $|F_o| > 0$ are given in Table 2, column 4. Calculated structure factors based on these parameters are compared with the observed structure factors in Table 3.

The standard errors associated with the atomic parameters calculated from the least-squares analysis were obtained by the same procedure as that outlined in Table 3 of our paper on the structure of $CaB_3O_3(OH)_5 \cdot 2H_2O$ (CLARK and CHRIST, 1959). Because of the use of three-dimensional data in the present case the errors are smaller than those found for $CaB_3O_3(OH)_5 \cdot 2H_2O$. Taking into account the uncertainty in the values of the cell dimensions, we assign the following standard errors for the interatomic distances in meyerhofferite:

$$\text{Ca–O} \pm 0.02 \text{ Å}, \quad \text{O–O} \pm 0.025 \text{ Å}, \quad \text{B–O} \pm 0.03 \text{ Å}, \quad \text{B–B} \pm 0.03 \text{ Å},$$
$$\text{Ca–Ca} \pm 0.01 \text{ Å}.$$

Description and discussion of the structure

The principal structural features of meyerhofferite are illustrated in Figs. 1 and 2. The crystal contains insular polyion rings of composition $[B_3O_3(OH)_5]^{-2}$ consisting of two boron-oxygen tetrahedra and a boron-oxygen triangle linked at corners. This is the same polyion found in the synthetic compound $CaB_3O_3(OH)_5 \cdot 2H_2O$ (CLARK and CHRIST, 1959), and in inyoite $CaB_3O_3(OH)_5 \cdot 4H_2O$ (CLARK, 1959); the infinite chains found in colemanite, $CaB_3O_4(OH)_3 \cdot H_2O$ (CHRIST, CLARK, and EVANS, 1958), consist of the polymerization product of these insular polyions described by the schematic reaction:

$$n[B_3O_3(OH)_5]^{-2} = [B_3O_4(OH)_3]_n^{-2n} + nH_2O.$$

As in the three previous studies mentioned above, assignment of the hydrogen atoms was carried out by assuming that those oxygens not shared between two borons in the polyion represent hydroxyl groups, and that the single unattached oxygen represents a water molecule. Thus the formula $2CaO \cdot 3B_2O_3 \cdot 7H_2O$ representing the

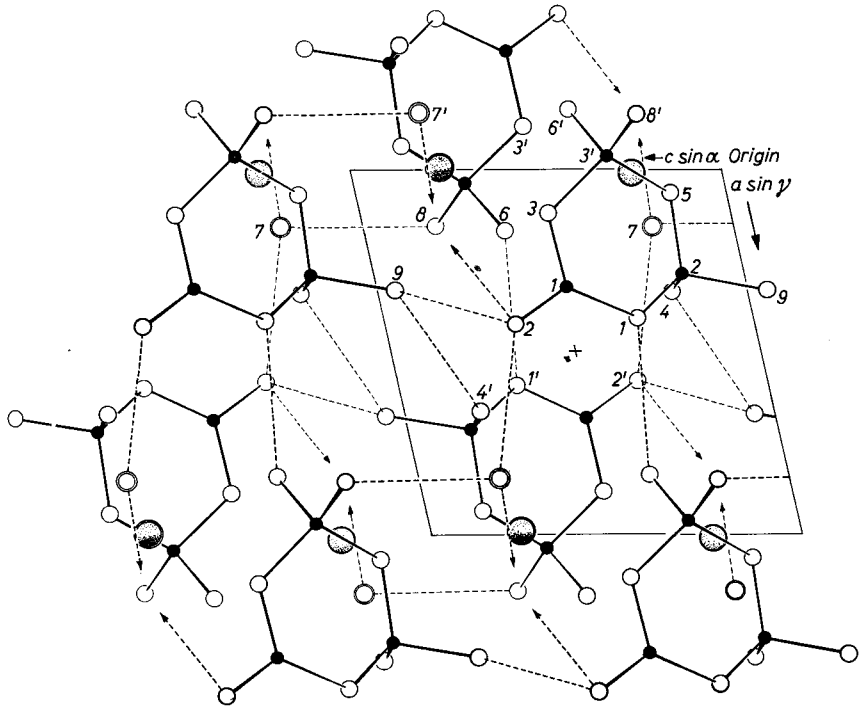

Fig. 1. Projection of the structure of meyerhofferite, $CaB_3O_3(OH)_5 \cdot H_2O$, on a plane normal to [010]. The small black circles represent boron atoms, the small open circles oxygen atoms, the double circles water molecules, and the larger stippled circles calcium atoms. The number designations of the atoms correspond to those given in Tables 4, 6, and 8. The dashed lines represent hydrogen bonds. Those dashed lines ending in an arrowhead indicate that the bond is associated with the atom translated one unit in the *b* direction.

chemical analysis becomes $CaB_3O_3(OH)_5 \cdot H_2O$. The arrangement of oxygens bonded to two borons, and hydroxyls bound to one boron (together with the single water molecule) accounts satisfactorily for the hydrogen content of the crystal. In fact, this way of inferring the positions of the hydrogen atoms is of general validity in hydrated borates and constitutes one of four rules that govern the structure and

composition of the polyions (or their polymerization products) occurring in these compounds*.

In meyerhofferite, successive polyions are linked through calcium-oxygen bonds to form endless zigzag strings along [001]; these strings are viewed along [001] in Fig. 2. Each calcium is surrounded by an irregular octahedron of four hydroxyls, one oxygen, and one water molecule as nearest neighbors, with an additional oxygen and hydroxyl

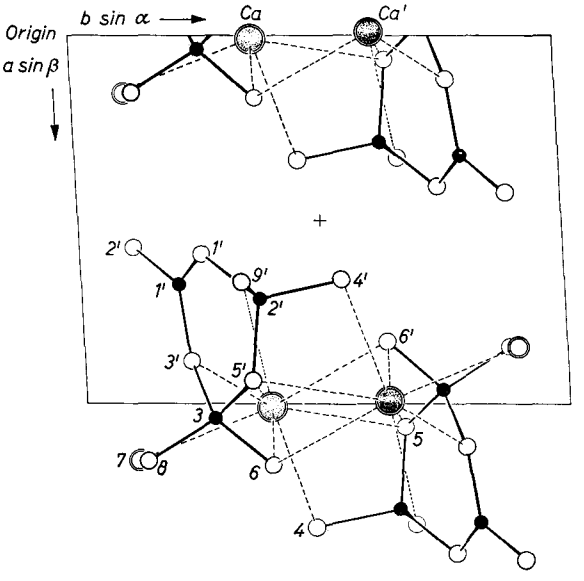

Fig. 2. Projection of the structure of meyerhofferite, $CaB_3O_3(OH)_5 \cdot H_2O$, on a plane normal to [001]. The dashed lines show the coordination of the calcium. The Ca—O_5' and Ca—O_9' bonds (and their centrosymmetric equivalents) shown are between the calcium and the oxygens in the adjoining cell below the plane of the drawing, so that endless zigzag strings are formed along [001]. The number designations of the atoms correspond to those given in Table 7.

* It has been shown elsewhere (CHRIST, 1959) that four rules seemingly govern the nature of the polyions occurring in hydrated borates: (1) boron will link either three oxygens to form a triangle, or four oxygens to form a tetrahedron; (2) polynuclear anions are formed by corner sharing only of boron-oxygen triangles or tetrahedra in such a manner that a compact group of low to medium negative charge results; (3) in the polyions of hydrated borates, those oxygens not shared by two borons always attach a proton and exist as hydroxyl groups; (4) the insular groups may polymerize in various ways by splitting out water.

With these four rules it has been found possible to correlate the hydrated borates of known crystal structures and to postulate the structural formulas for a number of hydrated borates whose crystal structures have not yet been worked out (CHRIST, 1960).

at larger distances. Adjacent polyion-calcium strings are linked only through hydrogen bonds (discussed below). The perfect cleavage parallel to (010) and the secondary cleavages parallel to (100) and ($\bar{1}10$), first noted by SCHALLER (1916), are satisfactorily explained by the structure since these cleavages involve only the breaking of hydrogen bonds.

Table 4. *Boron-oxygen bond lengths and bond angles for meyerhofferite* (See Fig. 1)

B—O bonds		Bond angles	
	Triangle around B_1		
B_1—O_1	1.34 Å	O_1—B_1—O_2*	124.0°
B_1—O_2*	1.35	O_1—B_1—O_3	121.3
B_1—O_3	1.39	O_3—B_1—O_2*	114.7
Average	1.36 Å		$\Sigma = 360.0°$
	Tetrahedron around B_2		
B_2—O_1	1.46 Å	O_1—B_2—O_4*	112.5°
B_2—O_4*	1.47	O_1—B_2—O_5	114.6
B_2—O_5	1.45	O_1—B_2—O_9*	110.8
B_2—O_9*	1.48	O_5—B_2—O_4*	104.6
Average	1.47 Å	O_5—B_2—O_9*	108.4
		O_4*—B_2—O_9*	105.2
		Average	109.4°
	Tetrahedron around B_3'		
		O_3—B_3'—O_5	110.7°
B_3'—O_3	1.48 Å	O_3—B_3'—O_6'*	103.4
B_3'—O_5	1.43	O_3—B_3'—O_8'*	111.0
B_3'—O_6'*	1.47	O_5—B_3'—O_6'*	109.7
B_3'—O_8'*	1.47	O_5—B_3'—O_8'*	108.1
Average	1.46 Å	O_6'*—B_3'—O_8'*	114.0
		Average	109.5°
		B_1—O_1—B_2	120.5°
		B_1—O_3—B_3'	121.5
		B_2—O_5—B_3'	125.2
(B—O bonds all ± 0.03 Å)		(All angles	± 1.5°)

* Hydroxyl oxygen.

The principal interatomic distances and angles are listed in Tables 4 to 8. For meyerhofferite the average B—O distance in the triangle is 1.36 Å, and in the two tetrahedra, 1.46 Å and 1.47 Å, respectively (Table 4). These results are in excellent agreement with the results previously found in other borates, as is shown in Table 5. The difference in distances between triangular B—O and tetrahedral B—O is by

Table 5. *Comparison of average boron-oxygen bond lengths for meyerhofferite and other borates*

Compound	Average B—O length		Reference
	Triangle	Tetrahedron	
Colemanite	1.37 Å	1.48 Å	CHRIST, CLARK and EVANS, 1958
$CaB_3O_3(OH)_5 \cdot 2\,H_2O$	1.36	1.48	CLARK and CHRIST, 1959
Inyoite	1.38	1.47	CLARK, 1959
Borax	1.36	1.48	MORIMOTO, 1956
Metaboric acid	1.36	1.46	ZACHARIASEN, 1952
Meyerhofferite	1.36	1.47	Present study

now well established. On the basis of the present study no significance can be attached to the variation of individual B—O bond lengths within the tetrahedra or triangle. The O—B—O bond angles appear reasonable, the average for the two tetrahedra being 109.4° and 109.5°, respectively. The sum of the three O—B—O angles in the triangle is exactly 360.0°, indicating that within limits of error the boron lies strictly in the plane of the triangle. The same finding has been made for the other members of this series. The variation in bond angles within the triangle and within the tetrahedra is believed to be real, the distortion probably representing accommodation of the polyion to its surroundings in the crystal.

For oxygens bonded to the same boron, the average O—O separation is 2.35 Å in the triangle and 2.39 Å in each of the two tetrahedra (Table 6). It had been suggested previously (CLARK and CHRIST. 1959) that in the borates of this series the O—O separation is a little larger, on the average, in the tetrahedra than in th etriangle. This difference is to be expected because the B—O distances in the tetrahedra are appreciably larger than those in the triangle. For an equilateral triangle with an average B—O length of 1.36 Å (Table 4), and a regular tetrahedron with an average B—O length of 1.46_5 Å (Table 4), the calculated O—O separations are 2.36 Å and 2.39 Å, respectively, in excellent agreement with those found.

Table 6. *Oxygen-oxygen distances in meyerhofferite for oxygens bonded to the same boron*
(See Fig. 1)

Triangle around B_1

O_1—O_2*	2.37 Å
O_1—O_3	2.38
O_2*—O_3	2.31
Average =	2.35 Å

Tetrahedron around B_2

O_1—O_4*	2.44 Å
O_1—O_5	2.45
O_1—O_9*	2.42
O_5—O_4*	2.32
O_5—O_9*	2.38
O_4*—O_9*	2.35
Average =	2.39 Å

Tetrahedron around B_3'

O_3—O_5	2.40 Å
O_3—O_6'*	2.32
O_3—O_8'*	2.43
O_5—O_6'*	2.37
O_5—O_8'*	2.35
O_6'*—O_8'*	2.47
Average =	2.39 Å

(All O—O bonds ± 0.025 Å)

* Hydroxyl oxygen.

From Fig. 1 it is seen that the polyion is based on a six-membered boron-oxygen ring, containing the successively linked atoms B_1, O_1, B_2, O_5, B_3', O_3. The plane defined by the three boron atoms obeys the equation (in perpendicular form):

$$0.0267x - 0.8253y + 0.5640z - 0.0660 = 0.$$

Oxygen O_5 lies very nearly in this plane, O_1 is 0.16 Å above the plane, and the largest deviation occurs for O_3 which lies 0.30 Å below the plane. The ring is therefore nearly planar. As might be expected from the presence of both triangularly and tetrahedrally coordinated boron in the same ring, there is considerable departure from the shape of a regular hexagon.

The average distance between borons in the same ring is 2.50 Å (Table 7). The Ca—O distances are listed in Table 7. There are six oxygens at an average distance of 2.41 Å making an approximately octahedral array about the Ca, and two oxygens making longer bonds of 2.51 Å and 2.55 Å; the average for all eight Ca—O bonds is 2.44 Å. The two distances of closest approach for the calciums are 3.76 Å and 3.89 Å (Table 7).

Table 7. *Calcium-oxygen bond lengths, calcium-calcium and boron-boron interatomic distances in meyerhofferite*
(See Fig. 2)

Ca—O bonds

Ca—O_4*	2.37 Å	Ca—O_6'*	2.55
Ca—O_7**	2.40	Ca—O_5'	2.51
Ca—O_9'*	2.40	Average of two =	2.53
Ca—O_6*	2.42		
Ca—O_5	2.44		
Ca—O_3'	2.45		
Average of six =	2.41 Å		

Average of eight = 2.44 Å

(All Ca—O bonds ± 0.02 Å)

Ca—Ca distances

Ca—Ca′ (z = 0.756 cycles) 3.89 Å
Ca—Ca′ (z = − 0.244 cycles) 3.76 Å
(Ca—Ca distances ± 0.01 Å)

B—B distances

B_1'—B_2'	2.43 Å
B_1'—B_3	2.51
B_2'—B_3	2.56
Average =	2.50 Å

(B—B distances ± 0.03 Å)

* Hydroxyl oxygen. ** Water oxygen.

Distances of less than 3.00 Å between interpolyion oxygens, and between these oxygens and the water molecules, are considered indicative of possible hydrogen bonding. There are eight such distances, three between 2.70 Å and 2.80 Å, two between 2.80 Å and 2.90 Å, and three between 2.90 Å and 3.00 Å (Table 8). Those interatomic distances considered to represent hydrogen bonds are shown by dashed lines in Fig. 1. The assignment of the proton donor atoms and the proton acceptor atoms was carried out by first assuming that the two protons of the water molecule O_7 are directed toward the centrosymmetrically-related atoms O_8 and O_8', making bonds of length 2.70 Å and 2.84 Å respectively. Since O_8 and O_8' are centrosymmetric equivalents this means that each of these atoms receives one normal and one weak hydrogen bond from a water molecule. Having made

this assignment, the remainder of the assignments immediately follow: O_7 receives a hydrogen bond from O_2' of length 2.78 Å, O_2' receives two very weak bonds of lengths 2.91 Å and 2.94 Å from O_8' and O_9', respectively, and O_9' receives a bond from O_4 of length 2.79 Å. The only ring oxygen involved in hydrogen bonding, O_1', receives a bond from O_6 of length 2.88 Å. The remaining distance less than 3.00 Å, that of 2.95 Å between O_7 and O_3', is considered to be a van der Waals contact.

Table 8. *Oxygen-oxygen distances in meyerhofferite for oxygens not bonded to the same boron*
(See Fig. 1)

(Only distances < 3.0 Å listed)

O_7**—O_8*	2,70 Å
O_7**—O_2'*	2.78
O_4'*—O_9*	2.79
O_7**—O_8'*	2.84
O_1'—O_6*	2.88
O_2*—O_8*	2.91
O_2*—O_9*	2.94
O_7**—O_3'	2.95

(All O—O bonds ± 0.025 Å)

* Hydroxyl oxygen.
** Water oxygen.

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