

The Structure of Sodium *scyllo*-Inositol Diborate Decahydrate

By C. T. GRAINGER

School of Physics, The University of New South Wales, Kensington 2033, Australia

(Received 12 June 1979; accepted 17 October 1980)

Abstract

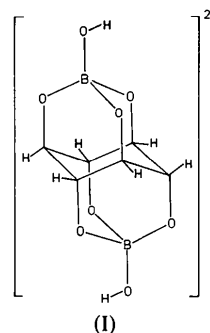
The crystal structure of $\text{Na}_2(\text{C}_6\text{H}_8\text{B}_2\text{O}_8) \cdot 10\text{H}_2\text{O}$ has been determined by three-dimensional X-ray analysis. The space group is *Fdd2* with $a = 24.203(8)$, $b = 23.115(8)$, $c = 6.831(2)$ Å at 295 K, and $Z = 8$. The structure was solved using direct methods. After stacking faults had been taken into account by employing separate scale factors for reflexions with even and odd indices, full-matrix least-squares refinement with slack constraints on the H atoms resulted in a final unweighted R of 0.051. The *scyllo*-inositol diborate ions themselves comprise rigid cage structures stacked in columns along the diad axes. The C atoms in the inositol ring are in a chair conformation with normal distances and angles. The two B atoms, situated on either side of the ring, are each bonded axially to alternate ring C atoms *via* three O atoms with B–O distances of 1.48 to 1.51 Å and O–C distances of 1.42 to 1.45 Å. The unbonded O...O distances, 2.40 to 2.44 Å, are slightly shorter than the corresponding distances, 2.47 to 2.51 Å, between alternate ring C atoms. Completing the tetrahedral coordination, each B atom is bonded also to an OH at 1.42 Å. The mean B–O bond length is 1.475(5) Å. The Na^+ ions are situated in parallel columns close to the screw axes. The $\text{Na} \cdots \text{Na}$ distance is 3.42 Å. The water molecules participate in an extensive hydrogen-bonding network along and between the columns of inositol diborate ions. Each Na^+ ion is octahedrally coordinated to the O atoms of six water molecules, with $\text{Na} \cdots \text{O}$ distances 2.36 to 2.44 Å.

Introduction

The preparation of the sodium salt of *scyllo*-inositol diborate (formerly known as *scyllitol* diborate) was first reported by Weissbach (1958). Available evidence supported the formula $\text{Na}_2(\text{C}_6\text{H}_8\text{B}_2\text{O}_8) \cdot 9\text{H}_2\text{O}$ for the crystalline salt. The weight loss on drying at 373 K for 18 h he found to be 37.27% which is also close to the value expected for this formula.

The present investigation was undertaken to confirm the postulated structure of the ion (I), and this it has done. For the crystal used in this study, however,

crystallographic evidence favours $10\text{H}_2\text{O}$ rather than $9\text{H}_2\text{O}$, and for this reason the former has been favoured in the title of this paper.



Experimental

Colourless needle-shaped crystals of rectangular cross-section and elongated along the c axis, prepared according to the method of Weissbach (1958) and crystallized from water, were kindly provided by Professor S. J. Angyal of the School of Chemistry, the University of New South Wales.

Weissenberg $hk0$, $hk1$ and $hk2$ and precession $h0l$ and $0kl$ photographs enabled the space group to be determined uniquely as *Fdd2* (No. 43), the conditions for reflexion being those listed in *International Tables for X-ray Crystallography* (1952).

The crystal density was measured by flotation in a mixture of cyclohexane and 1,2-dibromoethane; $D_{\text{meas}} = 1.550 \pm 0.050$, $D_{\text{calc}} = 1.584$ (for $10\text{H}_2\text{O}$) or 1.521 Mg m^{-3} (for $9\text{H}_2\text{O}$).

The intensities of 993 independent reflexions with $\theta < 70^\circ$ were measured on a Siemens automatic single-crystal diffractometer with Ni-filtered $\text{Cu K}\alpha$ radiation and a $\theta/2\theta$ scan. The stated cell dimensions, calculated from the diffraction angles, are based on $\text{Cu K}\alpha_1 = 1.5406$ Å. For each reflexion, Lorentz, polarization and absorption corrections were applied, and the equivalent path length for extinction corrections was calculated. The calculated absorption coefficient is 1.770 mm^{-1} .

The $0kl$ Weissenberg photograph of one crystal contained a streak linking diffuse 'forbidden' reflexions

031, 011, 011 and 031. The crystal used for intensity data collection was not examined photographically.

Structure determination and refinement

The structure was solved using the direct-methods program system *MULTAN 74* (Germain, Main & Woolfson, 1970; Woolfson, 1975). One feature of note was the weakness of the odd-index reflexions compared with those of even index. In determining the $|E|$'s this difference was nullified by specifying separate scale factors. The 200 reflexions with the largest $|E|$'s were used to generate 1200 triple phase relations. As the starting set for phase extension by tangent refinement, *MULTAN* chose the following:

<i>h</i>	<i>k</i>	<i>l</i>	Phase
0	28	0	0° from Σ_1 formula
17	13	1	0° origin definition
6	18	6	45/135/225/315°
3	1	5	45/135/225/315°
11	15	3	45/135° enantiomorph definition.

The best 12 refinements as judged by the combined figure of merit (CFOM), using equal weighting for the three components, were very similar. That with the highest CFOM failed to yield a solution, but that with the highest absolute figure of merit did; its CFOM was the fifth highest.

With 16 general equivalent positions in the cell and the calculated number of formula units 8, the ions had to be situated on the diad axes, and this part of the structure was found in the *E* map. Subsequent structure factor calculations and Fourier syntheses revealed peaks equal in number to the remaining heavy atoms. However, in no way could the value of *R* be reduced below 0.12 even after using the least-squares refinement program *RAELS* (Rae, 1975, 1977) to confirm a chemically sensible structure by objectively identifying the O and Na atoms and locating the H atoms along the hydrogen bonds.

A careful study of a difference Fourier map revealed the cause of the trouble. Several small peaks (about 0.1 times normal height) were found, corresponding to a partial superimposed structure displaced from the one determined by a translation $\frac{1}{2}c$. This was interpreted as a stacking disorder. It is noteworthy that accidental interatomic vectors of approximately $\frac{1}{2}c$ are common in this structure; this made it more difficult to detect the anomalous peak pattern, and may be of significance in the occurrence of this particular disorder.

If a fraction *f* of the volume is displaced by $\frac{1}{2}c$ and scatters coherently with the remainder, it may be shown (see Appendix) that the sole effect is to reduce the measured structure amplitudes for odd-*l*-index

reflexions by a factor $(1 - 2f)$, leaving the even-index reflexions unchanged. Accordingly, the stacking disorder was taken into account simply by refining two separate scale factors, one for the even-index and the other for the odd-index reflexions, without having to include additional 'atoms'. This was spectacularly effective, the final *R* value for all data falling to 0.051. The final values obtained for the scale factors were 0.617 (5) and 0.427 (3), giving $f = 0.154$ (4). This would seem to indicate that approximately one-sixth of the crystal was displaced in this way. (Complete disorder would correspond to $f = 0.5$.)

The parameters refined were the two scale factors, a parameter for extinction correction, all positional parameters, anisotropic temperature parameters for non-hydrogen atoms, and isotropic temperature parameters for H atoms. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Full correction was made for anomalous dispersion. Sodium was treated as Na⁺, but all other atoms were treated as neutral.

For the H atoms slack constraints were applied to the positional parameters as follows. The C—H distances in the inositol ring were constrained to values near 1.08 Å, but the angles were not constrained. In the water molecules the H—O bond length and the H—O—H angle were constrained to values near 0.95 Å and 109.5°. The isotropic temperature parameters of all the H atoms were refined in association with those of their respective bonded atoms in such a way that they remained about 10% above the isotropic components (Rae, 1975) of the latter.

The weight for each reflexion was taken as $(\sigma^2 + 0.0009F_{obs}^2)^{-1}$ where σ is the e.s.d. based on counting statistics.

The final unweighted residual *R* was 0.0514, taking into account all 993 reflexions. For the 534 even-index reflexions, *R* was 0.0508; for the 459 odd-index reflexions it was 0.0531.

The extinction parameter used in the above calculation was $r^* = 0.3526$ defined by the expression (Larson, 1970)

$$F_c^* = kF_c \left(1 + 158.8 r^* \bar{T} \frac{\lambda^3}{V^2} \gamma |F_c|^2 \right)^{-1/4},$$

where *k* = the scale factor, \bar{T} = effective path length in mm, F_c^* , F_c = corrected and uncorrected values, λ = the wavelength in Å, *V* = cell volume in Å³, and $\gamma = (1 + \cos^4 2\theta) / |\sin 2\theta(1 + \cos^2 2\theta)|$.

The final positional parameters from the constrained refinement are listed in Table 1.†

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35794 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$, for H $\times 10^3$) and isotropic temperature parameters (\AA^2)
$$B_{eq} = \frac{1}{3} \sum_i a_i^2 B_{ii}$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Na(1)	2525 (1)	-34 (1)	4757 (0)	2.3
C(1)	470 (1)	389 (1)	2324 (15)	2.0
C(3)	-115 (2)	-308 (2)	4147 (13)	2.0
C(5)	-118 (3)	-308 (3)	492 (12)	2.1
O(1)	903 (1)	-37 (1)	2323 (11)	2.0
O(3)	332 (2)	-715 (2)	4084 (11)	2.1
O(5)	330 (2)	-717 (2)	530 (11)	2.2
B(1)	685 (2)	-638 (2)	2314 (17)	1.8
O(7)	1143 (1)	-1022 (2)	2303 (12)	2.6
O(W1)	1068 (2)	-929 (2)	7290 (13)	3.1
O(W2)	936 (2)	-2135 (2)	6802 (11)	2.9
O(W3)	886 (2)	-2173 (2)	2727 (10)	2.4
O(W4)	2272 (2)	-705 (2)	7304 (12)	2.7
O(W5)	2232 (2)	-674 (2)	2219 (12)	2.4
H(C1)	69 (3)	79 (3)	213 (16)	2.3
H(C3)	-37 (4)	-35 (4)	544 (14)	2.3
H(C5)	-36 (4)	-37 (4)	-83 (13)	2.2
H(O7)	111 (3)	-148 (3)	208 (19)	3.2
H(1W1)	91 (5)	-77 (5)	616 (18)	3.3
H(2W1)	87 (5)	-82 (4)	840 (18)	3.3
H(1W2)	99 (3)	-173 (4)	701 (16)	3.3
H(2W2)	114 (4)	-234 (4)	771 (16)	3.3
H(1W3)	103 (4)	-238 (4)	378 (13)	2.7
H(2W3)	99 (4)	-234 (4)	154 (16)	2.7
H(1W4)	190 (4)	-75 (3)	772 (14)	2.9
H(2W4)	248 (4)	-101 (4)	778 (16)	2.9
H(1W5)	187 (3)	-77 (3)	255 (15)	2.6
H(2W5)	243 (3)	-101 (4)	181 (15)	2.6

When constraints are applied during least-squares refinement the calculation of the errors in the refined parameters may be affected. In order to avoid optimistic error estimates the following procedure was carried out after the final *RAELS* constrained refinement. A full-matrix refinement was performed in which all parameters were allowed to vary without the application of constraints. The (pessimistic) error estimates from this refinement are those listed in Table 1; these are also the error estimates input to *ORFFE* (Busing, Martin & Levy, 1964) *via* the matrix when calculating the errors in the distances and angles.

Description of the structure

The *scyllo*-inositol diborate ion

The ion (hereafter usually referred to as the 'molecule') is shown in Figs. 1, 2 and 3 which were drawn using *ORTEP* (Johnson, 1976). The relevant bond lengths and angles are in Table 2. The molecule lies on a twofold axis which passes through the mid-points of the bonds C(2)–C(3) and C(5)–C(6). The boron bonding is tetrahedral, the bond to O(7), 1.419 (6) Å, being noticeably shorter than those to

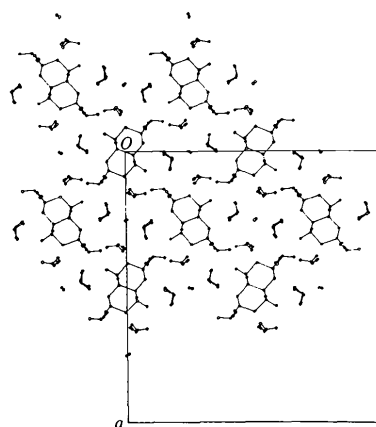


Fig. 1. General projection on to the *ab* plane showing the positions of the water molecules in relation to the columns of sodium and *scyllo*-inositol diborate ions. The chosen asymmetric unit includes half of the molecule at the origin in this projection and some of the nearby water molecules.

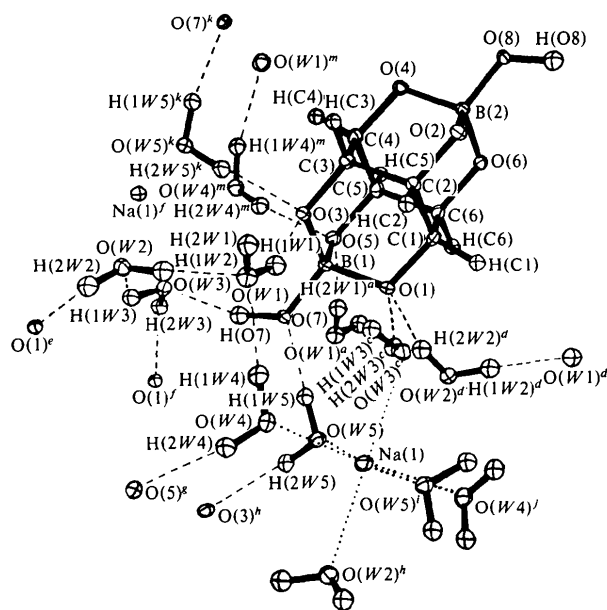


Fig. 2. Perspective diagram showing details of the *scyllo*-inositol diborate ion, the sodium coordination and the hydrogen bonding. This view is obtained by rotating Fig. 1 through an angle $+10^\circ$ about the *a* axis then -11° about the *b* axis.

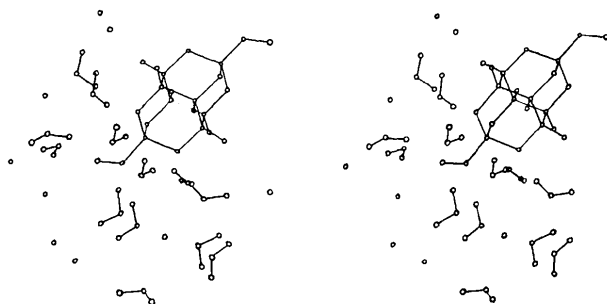


Fig. 3. Stereoscopic pair corresponding to Fig. 2

Table 2. Bond lengths (Å) and angles (°) for the molecule

The e.s.d.'s in the last digit(s) are in parentheses.

B(1)—O(1)	1.486 (5)	O(1)—B(1)—O(3)	108.2 (6)
B(1)—O(3)	1.492 (11)	O(1)—B(1)—O(5)	108.7 (6)
B(1)—O(5)	1.503 (11)	O(3)—B(1)—O(5)	108.3 (3)
B(1)—O(7)	1.419 (6)	O(7)—B(1)—O(1)	107.9 (3)
C(1)—C(6)	1.525 (11)	O(7)—B(1)—O(3)	112.2 (7)
C(1)—C(2)	1.525 (10)	O(7)—B(1)—O(5)	111.5 (7)
C(2)—C(3)	1.527 (14)	C(1)—O(1)—B(1)	112.4 (3)
C(5)—C(6)	1.533 (13)	C(3)—O(3)—B(1)	112.2 (5)
O(1)—O(3)	2.412 (7)	C(5)—O(5)—B(1)	111.4 (5)
O(3)—O(5)	2.428 (6)	C(6)—C(1)—C(2)	109.9 (4)
O(5)—O(1)	2.429 (7)	C(2)—C(3)—C(4)	108.6 (5)
C(1)—C(3)	2.479 (9)	C(4)—C(5)—C(6)	108.8 (5)
C(3)—C(5)	2.497 (7)	O(1)—C(1)—C(6)	108.8 (6)
C(5)—C(1)	2.487 (9)	O(1)—C(1)—C(2)	109.1 (6)
C(1)—O(1)	1.438 (5)	O(3)—C(3)—C(2)	109.7 (7)
C(3)—O(3)	1.434 (8)	O(3)—C(3)—C(4)	108.6 (5)
C(5)—O(5)	1.439 (9)	O(5)—C(5)—C(4)	109.0 (5)
		O(5)—C(5)—C(6)	109.3 (7)

O(1), O(3) and O(5), whose mean is 1.481 (6) Å. The mean length of all four B—O bonds is 1.475 (5) Å, a value of some significance (see below). The tetrahedral angles around the B atom also vary: four are close to 108° while the other two are about 112°. The mean of the B...O...C angles is 112.0 (3)°.

In the inositol ring the distances between adjacent C atoms are equal within experimental error, the mean value being 1.527 (4) Å. The distances from the O atoms to the respective ring C atoms are also equal within experimental error, the mean being 1.437 (4) Å. The angles at each of the ring C atoms involving bonded C and O atoms are all within the range 108.0–110.4°. The distances and angles involving H atoms are of little significance owing to constraints during refinement, and are not listed. The unbonded O—O distances, mean 2.423 (4) Å, are slightly shorter than the corresponding distances, mean 2.488 (5) Å, between alternate ring C atoms.

After preliminary trials only the Na atoms were treated as having a charge, and this was justified by the final difference Fourier synthesis which shows insignificant electron density at all atomic sites. However, it does leave unexplained the question as to where the negative charge of the ion is located.

The only significant discrepancies in the difference map are three peaks lying in the plane of O(1), O(3) and O(5) and forming with them an approximate regular hexagon. Their peak electron densities, 0.79, 0.79 and 0.58 e Å⁻³, exceed the average discrepancy, 0.06 (5) e Å⁻³, in the map by 14, 14 and 10 e.s.d.'s, respectively. Their electron contents are estimated as approximately 0.4, 0.4 and 0.3 e respectively. Perhaps these three peaks comprise a distributed negative charge on the *scyllo*-inositol ion. No other discrepancies exceed 0.32 e Å⁻³.

Table 3. Symmetry code

(a)	$x, y, z - 1$	(g)	$\frac{1}{2} + x, -\frac{1}{2} - y, z + \frac{3}{2}$
(b)	$x, y, z + 1$	(h)	$\frac{1}{2} + x, -\frac{1}{2} - y, z - \frac{1}{2}$
(c)	$\frac{1}{2} - x, \frac{1}{2} + y, z + \frac{1}{2}$	(i)	$\frac{1}{2} - x, -y, z + \frac{1}{2}$
(d)	$\frac{1}{2} - x, \frac{1}{2} + y, z - \frac{3}{2}$	(j)	$\frac{1}{2} - x, -y, z - \frac{1}{2}$
(e)	$\frac{1}{2} - x, -\frac{1}{2} + y, z + \frac{3}{2}$	(k)	$-\frac{1}{2} + x, -\frac{1}{2} - y, z + \frac{1}{2}$
(f)	$\frac{1}{2} - x, -\frac{1}{2} + y, z - \frac{1}{2}$	(m)	$-\frac{1}{2} + x, -\frac{1}{2} - y, z - \frac{1}{2}$

Table 4. Hydrogen bonding

(a) Distances (Å) between oxygen atoms involved in hydrogen bonding			
O(W2) ^d —H(2W2) ^d ...O(1)	2.771 (8)	O(7)—H(O7)...O(W3)	2.749 (6)
O(W3) ^e —H(2W3) ^e ...O(1)	2.758 (8)	O(W5)—H(W5)...O(7)	2.758 (6)
O(W1)—H(W1)...O(3)	2.867 (11)	O(W2)—H(W2)...O(W1)	2.827 (7)
O(W5) ^h —H(2W5) ^h ...O(3)	2.981 (7)	O(W4)—H(W4)...O(W1)	2.960 (7)
O(W1) ^g —H(2W1) ^g ...O(5)	2.887 (11)	O(W3)—H(W3)...O(W2)	2.788 (6)
O(W4) ^m —H(2W4) ^m ...O(5)	2.876 (8)		
(b) Angles (°) between oxygen atoms involved in hydrogen bonding			
O(1)...O(W2) ^d ...O(W1) ^g	100.3 (3)	O(5)...O(W1) ^g ...O(3) ^e	127.6 (2)
O(1)...O(W3) ^e ...O(W2) ^d	134.5 (3)	O(5)...O(W4) ^m ...O(W1) ^g	108.1 (2)
O(3)...O(W1) ^g ...O(5) ^h	99.9 (1)	B(1)...O(7)...O(W3)	115.4 (3)
O(3)...O(W5) ^h ...O(7) ^f	102.5 (2)		
(c) Distances (Å) between non-bonded oxygen atoms			
O(7)...O(W1)	3.418 (11)	O(W4)...O(W5)	3.475 (9)
O(7)...O(W1) ^g	3.436 (11)	O(W4)...O(W5) ^h	3.360 (8)
O(W3)...O(W2) ^d	4.050 (6)		

The water of hydration

The five water molecules in the asymmetric unit are designated *W*(1), *W*(2), ..., *W*(5) (Fig. 2). Superscripts *a*, *b*, *c*, ... (Table 3) designate symmetry-related water molecules. The water molecules play an essential part in binding together the whole structure. Each inositol molecule is stabilized longitudinally, laterally and vertically by a network of hydrogen bonds linking it *via* water molecules to inositol molecules in other columns and to those in the same column. Fig. 1 gives an overall view of the hydrogen-bonding network; Fig. 2 shows individual bonds. Atoms O(1), O(3), O(5) and O(7) are each involved in two such hydrogen bonds.

Table 4 lists the hydrogen-bond distances and angles between the bonded O atoms; they vary respectively from 2.74 to 2.99 Å and from 100 to 135°. Due to the constraints during refinement, there is no point in listing distances and angles directly involving the H atoms.

As a matter of interest, Table 4 also lists the distances between neighbouring O atoms which are *not* hydrogen bonded. They are all greater than 3.41 Å.

The sodium ions

The Na⁺ ions are only 0.098 Å from the twofold screw axes, and the Na...Na distance is 3.42 Å. Each Na⁺ is octahedrally coordinated to six O atoms belonging to waters of hydration. The Na(1) atom is coordinated to O(W4), O(W5), O(W4)ⁱ, O(W5)ⁱ, O(W3)^c and O(W2)^h (Fig. 2). The coordination

Table 5. Sodium coordination: distances (Å) between pairs of atoms involved

	O(W2) ^a	O(W3) ^c	O(W5) ^f	O(W4) ^f	O(W5)	O(W4)
Na(1)	2.355 (6)	2.377 (7)	2.419 (8)	2.442 (8)	2.387 (8)	2.410 (8)
O(W4)	3.572 (9)	3.199 (8)	3.406 (4)	4.848 (6)	3.475 (8)	
O(W5)	3.320 (8)	3.436 (9)	4.801 (6)	3.406 (4)		
O(W4) ^f	3.303 (8)	3.466 (9)	3.360 (8)			
O(W5) ^f	3.514 (9)	3.208 (7)				
O(W3) ^c	4.732 (5)					

distances Na...O lie within the range 2.40 ± 0.05 Å. Table 5 lists these together with the distances between all pairs of atoms involved in the coordination. Angles are not listed as they can be calculated from the distances.

Tetrahedral bonding of boron to oxygen

When Zachariasen (1963a) modified Pauling's (1929) electrostatic valence principle to take account of varying bond lengths, he verified the idea in the case of several structures involving triangular and/or tetrahedral coordination of B to O with the aid of empirical tables numerically relating electrostatic bond strength to bond length for B—O and O—H...O bonding. Since then, many structures involving B—O bonding have been completed. As the present structure has tetrahedral B—O bonding it is of interest to examine the present situation for such configurations. Earlier relevant

surveys have been made by Merlino & Sartori (1972) and by Frohnecke, Hartl & Heller (1977).

For tetrahedral B—O bonding one can calculate the sum of the bond strengths ($\sum S$) for B either by consulting Zachariasen's table or equivalently (if no bond is shorter than 1.365 Å — a condition always met so far in tetrahedral cases) by means of the expression $\sum S = 16.409 - 9.091 \bar{L}$, which is valid for the linear part of Zachariasen's table, where \bar{L} (Å) is the mean bond length. According to the modified principle this sum should be close to 3.000, the valence of B, and hence \bar{L} close to 1.475 Å if the modified principle and the table are both correct. This is a simple test to apply.

Table 6 lists 14 structures containing tetrahedral B—O bonding. In all cases it has been possible to calculate both the mean bond length \bar{L} and its e.s.d. from information given by the author(s). The last column lists the sum of the bond strengths calculated from the above relation, and its e.s.d. It is seen that the Pauling—Zachariasen principle is well supported as $\sum S$ is within one e.s.d. of 3.000 in almost all cases.

Among all the cases listed in Table 6 *scyllo*-inositol diborate is unique in its symmetry, having one short B—O bond and three longer ones, yet the mean is exactly 1.475 Å.

The author wishes to express thanks to Professor S. J. Angyal for suggesting the problem, for providing the crystals and for helpful discussions, Dr A. D. Rae for the use of his program *RAELS* and for assistance in using it, Mr D. C. Craig for collecting the reflexion data and for assistance in computing, and the University of New South Wales for the provision of computing time.

Table 6. Tetrahedral boron—oxygen coordination

Source	Structure	Mean B—O* distance \bar{L} (Å)	Sum of bond† strengths $\sum S$
Zachariasen (1963a)	Monoclinic metaborate acid	1.472 (4)	3.027 (30)
Zachariasen (1963b)	Cubic metaborate acid	1.472 (4)	3.027 (30)
Zachariasen & Plettinger (1963)	Potassium pentaborate tetrahydrate	1.477 (4)	2.982 (30)
Dunitz <i>et al.</i> (1971)	Boromycin	1.471 (8)‡	3.036 (70)
Fedder, von Schnering & Umland (1971)	Bis(dihydroxoboroxalene-diamide dioximate)nickel(II)	1.510 (30)	2.68 (54)
Karipides & Graf (1972)	Bis(tropolonato)boron(III) bromide	1.474 (8)	3.027 (70)
Dal Negro, Ungaretti & Perotti (1972)	Tetraacetyl diborate	1.475 (2)	3.000 (18)
Krogh-Moe (1972)	Potassium pentaborate	1.473 (3)	3.018 (27)
Merlino & Sartori (1972)	Sborgite	1.468 (3)	3.063 (27)
Mariezcurrera & Rasmussen (1973)	Potassium boromalate	1.478 (2)	2.973 (18)
Krogh-Moe (1974)	Caesium triborate	1.473 (5)	3.018 (45)
Dal Negro, Rossi & Perotti (1975)	Potassium tetraacetoborate	1.472 (5)	3.027 (45)
Frohnecke <i>et al.</i> (1977)	Caesium tetrahydroxohexa-oxo-pentaborate	1.470 (10)	3.045 (90)
Present work	<i>scyllo</i> -Inositol diborate	1.475 (5)	3.000 (45)

* The standard deviation given in parentheses has been calculated as $\frac{1}{2}(\sum_{i=1}^4 \sigma_i^2)^{1/2}$.

† The sum of the bond strengths has been calculated using $\sum S = 16.409 - 9.091 \bar{L}$.

‡ Calculated using corrected value 0.3557 for the x coordinate of atom O(23) of boromycin (Dunitz, 1979).

APPENDIX

Stacking disorder formula for *scyllo*-inositol diborate

For a perfect crystal the structure factor is

$$F_{\text{perf}} = \sum_j f_j \exp[2\pi i(hx_j + ky_j + lz_j)]$$

where f_j is the scattering factor of the j th atom after correcting for thermal vibration.

If a fraction f is displaced by a distance $z = \frac{1}{2}$ due to a stacking disorder and scatters coherently with the remaining fraction $(1 - f)$, the structure factor will become:

$$\begin{aligned} F_{\text{dis}} &= f \sum_j f_j \exp\{2\pi i(hx_j + ky_j + l(z_j + \frac{1}{2}))\} \\ &\quad + (1 - f) \sum_j f_j \exp[2\pi i(hx_j + ky_j + lz_j)] \\ &= [1 - f + f \exp(il\pi)] F_{\text{perf}} \\ &= \begin{cases} (1 - 2f) F_{\text{perf}} & \text{for } l \text{ odd,} \\ F_{\text{perf}} & \text{for } l \text{ even.} \end{cases} \end{aligned}$$

Thus the odd- l reflexions are reduced by a factor $(1 - 2f)$ leaving the even- l structure factors unchanged. Consequently one can deal with such disorder by using separate scale factors for odd and even l indices. One then expects

$$\frac{\text{scale factor for odd } l}{\text{scale factor for even } l} = \frac{1 - 2f}{1},$$

$$\text{whence } f = \frac{1}{2} \left(1 - \frac{\text{scale factor for odd } l}{\text{scale factor for even } l} \right).$$

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