

Fig. 2. The crystal structure viewed along the c axis. The hydrogen bonds are drawn by broken lines. N and W indicate nitrogen and water O atoms, respectively. Other atom numbering is for O atom. Parentheses of the atom numbering are omitted for clarity.

oxygen Ow(1–3,5,8) at the Eu sites, and lattice water oxygen Ow(9,10) atoms indicate the hydrogen bonds with the neighboring anion, leading to a three-dimensional hydrogen bonding network.

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Diiodinated Derivatives of 1,2-Diarsenadecaborane: $\text{As}_2\text{B}_{10}\text{H}_8\text{I}_2$

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Abstract. $\text{As}_2\text{B}_{10}\text{H}_8\text{I}_2$, $M_r = 519.8$, monoclinic, $I2/a$, $a = 14.262$ (3), $b = 8.128$ (2), $c = 21.575$ (4) Å, $\beta = 92.65$ (2)°, $V = 2498$ (2) Å³, $Z = 8$, $D_x = 2.76$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 101.8$ cm⁻¹, $F(000) =$

1840, $T = 294$ K, $R = 0.036$ for 2066 observed reflections. The structure contains a disordered distorted dodecahedron of As_2B_{10} atoms with the As atoms adjacent. Two diiodinated (B–I) isomers (9,12- and 8,12-diiodo forms) are co-crystallized in the asymmetric unit, with the former predominating. The packing of the molecules in the crystal is

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determined by steric requirements of the adjacent *exo*-cage I atoms. Packing of the two isomers in the crystal lattice then results in apparent disorder of the two As atoms over four sites. Bond distances are As—As 2.435 (2) and 2.421 (2) Å respectively for the predominant forms of the 9,12- and 8,12-diiodo isomers. The B—I distances are 2.161 (7) and 2.170 (7) Å, and the mean B—B distance for boron positions which are not affected by arsenic disorder is 1.786 (11) Å.

Introduction. Our studies of heteroborane cluster compounds and their metal derivatives (Faridooon, Ni Dhubhghaill, Spalding, Ferguson, Kaitner, Fontaine & Kennedy, 1989) have led us to investigate the structural details of certain basic types of heteroboranes. The structures of 1-SeB₁₁H₁₁ (Ferguson, Parvez, MacCurtain, Ni Dhubhghaill, Spalding & Reed, 1987) and the [*nido*-7-SeB₁₀H₁₁]⁻ anion (Reed, Ferguson, Ruhl, Ni Dhubhghaill & Spalding, 1988) were both crystallographically disordered. To establish accurate Se—B bond distances, the monoiodo derivative of 1-SeB₁₁H₁₁ was prepared and the analysis of this non-disordered 12-I-1-SeB₁₁H₁₀ system has been reported recently (Ni Dhubhghaill, MacCurtain, Myers, Spalding, Brint, McCabe, Ferguson & Reed, 1989). We have now initiated studies of heteroboranes which contain the Main Group 5 atoms P, As and Sb (Brint, Sangchakr, McGrath, Spalding & Suffolk, 1990) and decided to analyse the 1,2-As₂B₁₀ structure. Bearing in mind our experiences with 1-SeB₁₁H₁₁, we prepared the diiodinated derivative by reacting 1,2-As₂B₁₀H₁₀ and excess iodine in refluxing dichloromethane in the presence of AlCl₃ catalyst. This paper reports the X-ray diffraction study of the diiodinated product.

Experimental. A colourless block crystal was grown from tetrahydrofuran:*di-n*-butyl ether solution (5:1). Accurate cell dimensions and crystal orientation matrix determined on CAD-4 diffractometer by least-squares treatment of the setting angles of 25 reflections, 14 < θ < 19°. Crystal dimensions 0.20 × 0.25 × 0.32 mm; intensities of reflections with indices *h* 0 to 18, *k* 0 to 10, *l* -27 to 27, with 2 < 2θ < 54° measured; ω-2θ scans; ω-scan width (0.60 + 0.35tanθ)°; graphite-monochromated Mo Kα radiation; 3106 reflections measured, 2727 unique, 2066 with *I* > 3σ(*I*) labelled observed and used in structure solution and refinement, *R*_{int} = 0.016. Data corrected for Lorentz, polarization and absorption effects (max. and min. transmission factors = 0.73 and 1.30, respectively). Space group *I*2/*a* or *Ia* allowed by the systematic absences (*hkl*, *h* + *k* + *l* = 2*n* + 1, *h*0*l*, *h*, *l* = 2*n* + 1), *I*2/*a* chosen and confirmed by the successful refinement. Coordinates of the I

Table 1. *Positional and thermal parameters and their e.s.d.'s*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
I(1)	0.28234 (3)	0.14822 (6)	-0.10448 (3)	3.43 (1)
I(2)	-0.00078 (3)	0.14761 (5)	-0.07419 (2)	3.17 (1)
As/B(1)*	0.03075 (6)	0.7384 (1)	-0.16333 (4)	3.30 (2)
As/B(2)*	0.19890 (9)	0.7374 (1)	-0.17839 (6)	3.54 (2)
As/B(3)*	0.1442 (1)	0.7333 (2)	-0.07639 (7)	3.18 (3)
B(4)	0.0331 (5)	0.5548 (9)	-0.0911 (3)	2.1 (1)
B(5)	0.0127 (5)	0.4789 (9)	-0.1715 (4)	2.3 (1)
As/B(6)*	0.1006 (5)	0.5621 (8)	-0.2301 (4)	5.6 (2)
B(7)	0.2369 (5)	0.557 (1)	-0.1122 (4)	2.4 (1)
B(8)	0.1466 (5)	0.4767 (9)	-0.0643 (4)	2.3 (1)
B(9)	0.0694 (5)	0.3557 (8)	-0.1137 (4)	2.0 (1)
B(10)	0.1110 (5)	0.3583 (9)	-0.1899 (4)	2.3 (1)
B(11)	0.2148 (5)	0.482 (1)	-0.1901 (4)	2.4 (1)
B(12)	0.1919 (5)	0.3576 (9)	-0.1254 (4)	2.1 (1)

* These sites had disorder of the As and B atoms. Occupancies for sites 1, 2 and 3 (refined as As atoms) were 0.921 (2), 0.681 (2) and 0.492 (2) respectively. The occupancy factor for site 6 (refined as a B atom) was 1.655 (23).

atoms determined from analysis of three-dimensional Patterson function and remaining non-H atoms found in difference Fourier syntheses. It was obvious from the peak heights in the As₂B₁₀ moiety that the two As atoms were effectively disordered over adjacent sites (three main sites and a fourth minor one). Occupancy factor refinement with isotropic thermal parameters for the As and B atoms determined the various As/B ratios in the four sites. H atoms were visible in difference maps and were included in the final rounds of refinement but restrained to ride on the atoms to which they were bonded (with B—H 1.08 Å). Final refinement by full-matrix least-squares calculations on *F*, with anisotropic thermal parameters for non-H atoms. The final cycle of refinement included 132 variable parameters, *R* = 0.036, *wR* = 0.050, goodness-of-fit 2.36, *w* = 1/[σ²(*F*_o) + 0.030(*F*_o)²]. Max. shift/e.s.d. less than 0.02; density in final difference map ± 1.08 e Å⁻³ (adjacent to the I atoms), no significant chemical features remained. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations performed on a PDP11/73 computer using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1984). Atomic coordinates and some details of molecular geometry are given in Tables 1* and 2 respectively. Fig. 1, prepared using *ORTEPII* (Johnson, 1976), shows a general view of the disordered structure.

* Full details of molecular dimensions, calculated hydrogen coordinates, anisotropic thermal parameters, and a list of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53517 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and selected bond angles (°)

I(1)	B(12)	2.170 (7)	B(5)	As/B(6)	1.942 (11)		
I(2)	B(9)	2.161 (7)	B(5)	B(9)	1.766 (10)		
As/B(1)	As/B(2)	2.435 (2)	B(5)	B(10)	1.770 (11)		
As/B(1)	As/B(3)	2.421 (2)	As/B(6)	B(10)	1.872 (10)		
As/B(1)	B(4)	2.157 (7)	As/B(6)	B(11)	1.921 (10)		
As/B(1)	B(5)	2.131 (8)	B(7)	B(8)	1.811 (11)		
As/B(1)	As/B(6)	2.292 (8)	B(7)	B(11)	1.802 (11)		
As/B(2)	As/B(3)	2.369 (2)	B(7)	B(12)	1.763 (10)		
As/B(2)	As/B(6)	2.258 (7)	B(8)	B(9)	1.791 (10)		
As/B(2)	B(7)	2.098 (8)	B(8)	B(12)	1.781 (11)		
As/B(2)	B(11)	2.104 (8)	B(9)	B(10)	1.773 (11)		
As/B(3)	B(4)	2.161 (8)	B(9)	B(12)	1.777 (10)		
As/B(3)	B(7)	2.117 (8)	B(10)	B(11)	1.791 (11)		
As/B(3)	B(8)	2.102 (8)	B(10)	B(12)	1.767 (11)		
B(4)	B(5)	1.852 (11)	B(11)	B(12)	1.766 (11)		
B(4)	B(8)	1.808 (11)	I(1)...	I(2)	4.120 (1)		
B(4)	B(9)	1.774 (10)					
As/B(2)	As/B(1)	As/B(3)	58.39 (5)	As/B(3)	B(8)	B(4)	66.6 (3)
As/B(2)	As/B(1)	As/B(6)	57.0 (2)	As/B(3)	B(8)	B(7)	65.0 (4)
As/B(3)	As/B(1)	B(4)	56.0 (2)	B(4)	B(8)	B(9)	59.1 (4)
B(4)	As/B(1)	B(5)	51.2 (3)	B(7)	B(8)	B(12)	58.8 (4)
B(5)	As/B(1)	As/B(6)	51.9 (3)	B(9)	B(8)	B(12)	59.7 (4)
As/B(1)	As/B(2)	As/B(3)	60.50 (5)	I(2)	B(9)	B(4)	117.3 (5)
As/B(1)	As/B(2)	As/B(6)	58.3 (2)	I(2)	B(9)	B(5)	121.1 (4)
As/B(3)	As/B(2)	B(7)	56.2 (2)	I(2)	B(9)	B(8)	118.6 (5)
As/B(6)	As/B(2)	B(11)	52.1 (3)	I(2)	B(9)	B(10)	123.8 (4)
B(7)	As/B(2)	B(11)	50.8 (3)	I(2)	B(9)	B(12)	122.7 (4)
As/B(1)	As/B(3)	As/B(2)	61.11 (5)	B(4)	B(9)	B(5)	63.1 (4)
As/B(1)	As/B(3)	B(4)	55.8 (2)	B(4)	B(9)	B(8)	61.0 (4)
As/B(2)	As/B(3)	B(7)	55.4 (2)	B(5)	B(9)	B(10)	60.0 (4)
B(4)	As/B(3)	B(8)	50.2 (3)	B(8)	B(9)	B(12)	59.9 (4)
B(7)	As/B(3)	B(8)	50.8 (3)	B(10)	B(9)	B(12)	59.7 (4)
As/B(1)	B(4)	As/B(3)	68.2 (2)	B(5)	B(10)	As/B(6)	64.4 (4)
As/B(1)	B(4)	B(5)	63.7 (3)	B(5)	B(10)	B(9)	59.8 (4)
As/B(3)	B(4)	B(8)	63.2 (3)	As/B(6)	B(10)	B(11)	63.2 (4)
B(5)	B(4)	B(9)	58.2 (4)	B(9)	B(10)	B(12)	60.3 (4)
B(8)	B(4)	B(9)	60.0 (4)	B(11)	B(10)	B(12)	59.5 (4)
As/B(1)	B(5)	B(4)	65.1 (3)	As/B(2)	B(11)	As/B(6)	68.1 (3)
As/B(1)	B(5)	As/B(6)	68.3 (3)	As/B(2)	B(11)	B(7)	64.4 (4)
B(4)	B(5)	B(9)	58.7 (4)	As/B(6)	B(11)	B(10)	60.5 (4)
As/B(6)	B(5)	B(10)	60.4 (4)	B(7)	B(11)	B(12)	59.2 (4)
B(9)	B(5)	B(10)	60.2 (4)	B(10)	B(11)	B(12)	59.5 (4)
As/B(1)	As/B(6)	As/B(2)	64.7 (2)	I(1)	B(12)	B(7)	118.7 (4)
As/B(1)	As/B(6)	B(5)	59.8 (3)	I(1)	B(12)	B(8)	120.3 (5)
As/B(2)	As/B(6)	B(11)	59.8 (3)	I(1)	B(12)	B(9)	122.8 (4)
B(5)	As/B(6)	B(10)	55.2 (4)	I(1)	B(12)	B(10)	121.8 (4)
B(10)	As/B(6)	B(11)	56.3 (4)	I(1)	B(12)	B(11)	119.0 (5)
As/B(2)	B(7)	As/B(3)	68.4 (2)	B(7)	B(12)	B(8)	61.4 (4)
As/B(2)	B(7)	B(11)	64.8 (4)	B(7)	B(12)	B(11)	61.4 (4)
As/B(3)	B(7)	B(8)	64.1 (4)	B(8)	B(12)	B(9)	60.4 (4)
B(8)	B(7)	B(12)	59.8 (4)	B(9)	B(12)	B(10)	60.0 (4)
B(11)	B(7)	B(12)	59.4 (4)	B(10)	B(12)	B(11)	60.9 (4)

See the coordinate table for an explanation of the As/B disorder which affects atoms 1, 2, 3 and 6.

Discussion. Our analysis shows that the reaction of As₂B₁₀H₁₀ with I₂ has produced two distinct isomers which could not be easily separated during post reaction work-up. The four sites fractionally occupied by either As or B atoms correspond to either the 9,12- or 8,12-diiodo isomers; because the 12-atom cage is essentially spherical, the two isomers pack in such a way that the coordinates of the adjacent I atoms remain identical, but the As atoms are disordered over four sites. The ORTEP plot shown in Fig. 1 has the correct numbering scheme for the 9,12 isomer [site occupancies for atoms at sites 1, 2, 3 and

6 (in the 9,12 numbering scheme) are given in Table 1]; the disorder caused by the co-crystallization of the two isomers only appears to affect these four sites. A somewhat similar problem (with iodination occurring at two sites) has also been encountered in an electron-diffraction study of the product obtained by diiodinating 1,7-C₂B₁₀H₁₂ (Vilkov, Khaikin, Zhigach & Siryatskaya, 1968), where a 1:1 mixture of 9,10- and 5,12-diiodo isomers was assumed in the analysis of the structure.

Because we started our synthesis with the two As atoms immediately adjacent, only models which have the two As atoms directly bonded are physically reasonable in interpreting the disorder. The main isomer present is the 9,12 one, with As—As 2.435 (2) Å. A choice of any other two adjacent As atoms (and appropriate renumbering) leads to the 8,12 isomer (or its enantiomorph); the As—As distance in the predominant form of this isomer [corresponding to As(1)—As(3) in Fig. 1] is 2.421 (2) Å. A 'normal' range of 2.43–2.46 Å for two-electron As—As single bonds has been suggested (Rheingold & Sullivan, 1983). The distances between As(1) and the 100% boron sites B(4) and B(5) are 2.157 (7) and 2.131 (8) Å respectively. Comparable data in the literature refer to Me₃AsBX₃ adducts only. For X = H, the As—B distance was estimated as 2.035 Å from a combined microwave, infrared and Raman spectroscopic study (Durig, Hughes & Odom, 1974). For X = Cl, Br and I the distances were determined to be 2.065 (6), 2.04 (1) and 2.03 (1) Å respectively in an X-ray diffraction study (Chadha, Chehayber & Drake, 1985).

For those sites which are 100% boron, the range of B—B distances in the present As₂B₁₀H₈I₂ arsenaborane is notably narrow, 1.763 (10)–1.852 (11) Å, with a mean value 1.786 (11) Å; a much broader range of B—B distances was found in 12-I-1-

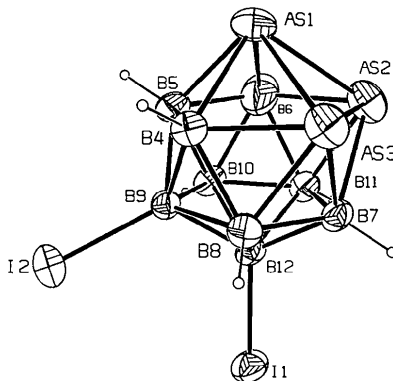


Fig. 1. View of the As₂B₁₀H₈I₂ molecule showing the numbering scheme for the 9,12-diiodo isomer. Ellipsoids are at the 50% level.

SeB₁₁H₁₀ 1.715 (12)–1.934 (13), mean 1.762 (12) Å (Ni Dhubhghaill *et al.*, 1989). The B—I distances in As₂B₁₀H₈I₂ [2.161 (7) and 2.170 (7) Å] span the B—I distance found in 12-I-1-SeB₁₁H₁₀ of 2.167 (7) Å and are also comparable with the value found in 1-I-B₁₀H₁₃ [2.17 (1) Å] (Sequeira & Hamilton, 1967).

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Structure of Sodium β'' -Gallate

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Abstract. Na_{1+x}Ga_{11-x/3}O₁₇ ($x = 0.98$), $M_r = 1061.72$, rhombohedral, $R\bar{3}m$, with hexagonal cell; $a = 5.855$ (1), $c = 34.570$ (10) Å, $V = 1026.3$ (5) Å³, $Z = 3$, $D_x = 5.15$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 219$ cm⁻¹, $F(000) = 1466$, $T = 296$ K, final $R = 0.033$ for 707 independent reflections. Sodium β'' -gallate is isostructural with sodium β'' -alumina. Sodium substitutes for 16.3% of Ga atoms at tetrahedral sites in the spinel block. The size of the tetrahedron is enlarged compared with that of the usual GaO₄ moiety because of this substitution. In the conduction plane, an Na⁺ ion is located at the 6(c) and 18(h) sites with occupancies 35 and 16%, respectively, and an O atom is randomly distributed at the 18(h) sites, or at six positions around each 3(b) site. This is the first case where a random distribution of O atoms in the conduction plane is observed for a β'' -gallate, although it has been known in some β'' -aluminas. The deviation of the O atom from the 3(b) site is 0.54 Å.

Introduction. β - and β'' -gallates, which are gallium analogs of β - and β'' -aluminas, which are well known as solid electrolytes, also exhibit high ionic

conductivities. The synthesis, structure and conductivities of these gallates have been widely studied. Sodium β'' -gallate was first synthesized by Boilot, Thery & Collongues (1973), and its single crystals were grown by the evaporation method (Foster & Scardefield, 1977). The ionic conductivities of sodium β'' -gallate were examined by Chandrashekhar & Foster (1977), who discussed charge compensation mechanisms of sodium β'' -gallate on the basis of the results of structure analysis (Anderson, Foster & La Placa, 1981). However, no atomic coordinates were given in their paper and structural details of sodium β'' -gallate have never been discussed. The reason is that the material is unstable due to its hygroscopic properties (Foster & Arbach, 1977), which gives rise to difficulty in the precise refinement of structural parameters. The present study has been undertaken in order to obtain stable single crystals of sodium β'' -gallate and to determine the structural parameters with an accuracy comparable to the results for potassium and ammonium β'' -gallate (Tsurumi, Ikawa, Nishimura, Urabe & Udagawa, 1987). Although they are isostructural with each other, these compounds show some inter-