−46·5 (2)° in (4). These three systems [(1), (3) and (4)] are in an almost perfect staggered conformation. This difference in conformation between phosphonamide (2) and [(1), (3) and (4)] is most probably due to electronic effects caused by the presence of an azido group.

The molecules are held together by van der Waals forces. The closest approach is observed between O and C(9) at 3·162 (5) Å.

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Structures of Two Carbon-to-Boron Bridged *o*-Carboranes

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Abstract. The structures of two substituted *o*-carboranes, icosahedral cages in which two adjacent positions are CH and the remaining vertices BH, are described. These carboranes [(1) and (2)] are the first molecules in which one C atom is attached to an adjacent framework B atom through a carbocyclic bridge. In (1) the C—B bridge is CH_2—CH_2—CO—CH_2, in (2) it is CH_2—CO—CH_2. The cage C—C bond in (1) can be assigned unambiguously because of the absence of rotational disorder. Compound (1): 1,4-[1,2-dicarbadodecaborane(12)-1,4-diyl]-2-butanone, $C_6H_{16}B_{10}O$, $M_r = 212·3$, monoclinic, $C2/c$, $a = 21·702$ (6), $b = 8·662$ (2), $c = 13·610$ (4) Å, $\beta = 102·11$ (2)°, $V = 2501$ (1) Å^3, $Z = 8$, $D_x = 1·13$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 4·1$ cm^{-1}, $F(000) = 880$, $T = 293$ K, $R = 0·056$ for 1283 reflections. Compound (2): 1,3-[1,2-dicarbadodecaborane(12)-1,4-diyl]-2-propanone, $C_5H_{14}B_{10}O$, $M_r = 198·3$, orthorhombic, $Pbca$, $a = 22·822$ (7), $b = 10·797$ (4), $c = 9·280$ (3) Å, $V = 2286$ (1) Å^3, $Z = 8$, $D_x = 1·15$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 4·22$ cm^{-1}, $F(000) = 816$, $T = 293$ K, $R = 0·067$ for 1096 reflections.

Introduction. Although myriad *o*-carboranes are known (Muetterties, 1975; Olah, Prakash, Williams, Field & Wade, 1987) and many substitution patterns have been examined by X-ray crystallography, simple compounds in which one of the framework carbons is linked to one of the borons through a simple bridge were unknown until recently. Until 1986, when we reported a synthesis of such compounds using an intramolecular insertion reaction of a ketocarbene (Wu & Jones, 1986), only relatively exotic metallocycles were known [C—P—Ir—B: Hoel & Hawthorne (1975), NMR analysis; C—P—Pt—B: Manojlovic-Muir, Muir & Solomun (1980), X-ray analysis; C—C—N—Pd—B: Kalinin, Usatov & Zakharkin (1985), NMR analysis]. In addition, X-ray structure determination of *o*-carboranes has been plagued with problems of rotational disorder resulting in a general inability to

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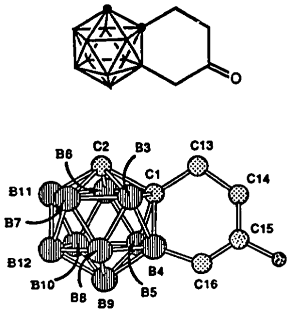
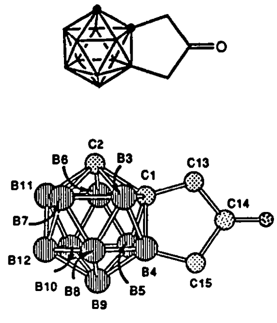
distinguish framework boron from carbon. Beall notes two such cases in his 1975 review of X-ray structure determination of *o*-carborane structures (Beall, 1975), and examples continue to appear. Manojlovic and her co-workers note such a possibility, for instance (Manojlovic-Muir, Muir & Solomun, 1980). In two very recent papers from the People's Republic of China on rhenium substituted *o*-carboranes, one reports a C—C bond distance of 1·65 Å (Chen, Lei, Xu, Zhang, Xu & Tang, 1986), but the other reports 1·70 Å, a distance likely to be probably too long for a cage C—C bond and probably the average of several C—C and C—B bond distances (Chen, Lei, Xu & Tang, 1987). Here we report for the first time the structures of two *o*-carboranes [(1) and (2)] in which one carbon is connected to a boron through a simple aliphatic bridge.



(1)



(2)

Compound (2) has a pseudo twofold axis [through C(14), O and the centroids of the B(11)—B(12) and C(1)—B(4) bonds] as well as a pseudo mirror plane [B(7), B(10), B(3), B(5), C(14), O on mirror]. As expected, this structure is rotationally disordered resulting in hybrid $\frac{1}{2}$ C, $\frac{1}{2}$ B atoms at the 1 and 4 positions and $\frac{1}{4}$ C, $\frac{3}{4}$ B atoms at the 2, 6, 8 and 9 positions. Compound (1) has no such pseudo symmetry and consequently does not display rotational disorder. Thus we are able to assign the C—C bond in (1) unambiguously.

Experimental. All X-ray data were collected at room temperature on a Nicolet *R3m* diffractometer equipped with a graphite monochromater and Cu $K\alpha$ radiation. The structures were solved by direct methods and refined using a blocked cascade least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized (*SHELXTL* package of programs; Sheldrick, 1980). Atomic scattering factors were those incorporated in *SHELXTL*.

Compound (1). Compound (1) was prepared by the method of Wu & Jones (1986) and recrystallized from hexane to yield clear crystals. Diffraction data were collected from a clear crystal, 0·10 × 0·15 × 0·25 mm, using a 1·5° ω-scan mode for all data with $3 \leq 2\theta \leq 114°$, $-23 \leq h \leq 23$, $0 \leq k \leq 9$, $0 \leq l \leq 14$. The lattice parameters were obtained from a least-squares fit of 23 centered reflections with $40 \leq 2\theta \leq 45°$. Standard reflections $\bar{5}16$, 240 were monitored after every 50 reflections and showed a 5% linear decline in intensity. The 1895 measured intensities were reduced by applying Lorentz, polarization and decay corrections. No correction for absorption was applied. Systematically absent reflections were eliminated and equivalent reflections were averaged ($R_{\rm int}$ = 0·012) to give 1669 unique data of which 1283 were considered to be observed [$F_o > 3\sigma(F_o)$].

Table 1. *Atomic coordinates and isotropic thermal parameters* (Å^2) *for* (1) *with e.s.d.'s in parentheses*

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
C(1)	0·3789 (1)	0·3381 (3)	−0·1223 (2)	0·043 (1)
C(2)	0·4132 (1)	0·2431 (4)	−0·2009 (2)	0·059 (1)
B(3)	0·4383 (1)	0·2084 (4)	−0·0743 (2)	0·053 (1)
B(4)	0·3691 (1)	0·2233 (4)	−0·0246 (2)	0·043 (1)
B(5)	0·3047 (1)	0·2690 (4)	−0·1271 (2)	0·044 (1)
B(6)	0·3345 (2)	0·2848 (4)	−0·2375 (2)	0·054 (1)
B(7)	0·4281 (2)	0·0562 (5)	−0·1625 (3)	0·070 (2)
B(8)	0·3991 (2)	0·0393 (4)	−0·0512 (2)	0·054 (1)
B(9)	0·3162 (2)	0·0754 (4)	−0·0836 (2)	0·050 (1)
B(10)	0·2952 (2)	0·1166 (4)	−0·2148 (3)	0·060 (1)
B(11)	0·3649 (2)	0·1048 (5)	−0·2628 (3)	0·072 (2)
B(12)	0·3526 (2)	−0·0265 (4)	−0·1682 (3)	0·068 (1)
C(13)	0·3944 (1)	0·5077 (3)	−0·1046 (2)	0·057 (1)
C(14)	0·3754 (2)	0·5655 (3)	−0·0082 (3)	0·069 (1)
C(15)	0·4025 (1)	0·4650 (4)	0·0803 (2)	0·060 (1)
C(16)	0·3751 (1)	0·3063 (4)	0·0806 (2)	0·058 (1)
O	0·4464 (1)	0·5106 (3)	0·1454 (2)	0·086 (1)

Table 2. *Atomic coordinates and isotropic thermal parameters* (Å^2) *for* (2) *with e.s.d.'s in parentheses*

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
C(1)*	0·1356 (2)	0·0194 (3)	0·2115 (4)	0·058 (1)
C(2)*	0·1753 (2)	−0·1065 (4)	0·2582 (4)	0·070 (2)
B(3)	0·1010 (2)	−0·1109 (3)	0·2677 (4)	0·053 (1)
B(4)*	0·0783 (2)	0·0357 (3)	0·3203 (4)	0·061 (1)
B(5)	0·1391 (2)	0·1289 (4)	0·3440 (4)	0·062 (1)
B(6)*	0·2001 (2)	0·0378 (4)	0·3045 (5)	0·081 (2)
B(7)	0·1410 (2)	−0·1764 (4)	0·4055 (4)	0·068 (2)
B(8)*	0·0803 (2)	−0·0841 (4)	0·4436 (4)	0·070 (2)
B(9)*	0·1033 (2)	0·0654 (4)	0·4944 (4)	0·071 (2)
B(10)	0·1809 (2)	0·0653 (4)	0·4833 (5)	0·076 (2)
B(11)	0·2033 (2)	−0·0826 (4)	0·4287 (6)	0·084 (2)
B(12)	0·1434 (2)	−0·0656 (4)	0·5459 (4)	0·078 (2)
C(13)	0·1167 (2)	0·0645 (3)	0·0616 (4)	0·078 (2)
C(14)	0·0536 (2)	0·1034 (3)	0·0746 (4)	0·071 (1)
O	0·0262 (1)	0·1399 (3)	−0·0271 (3)	0·108 (1)
C(15)	0·0280 (2)	0·0917 (4)	0·2249 (4)	0·079 (2)

* C(1), C(2), B(4), B(6), B(8), B(9) represent hybrid B, C atoms.

All atoms of the carborane group were initially refined as C atoms with isotropic temperature factors. The carbon and boron assignements were then made based on the following observations. The temperature factor (U) for C(1) was 0·030 *vs* 0·048 Å^2

for B(4) and that for C(2) was 0·041 *vs* 0·052–0·062 Å^2 for B(3), B(5), B(6), B(8) and B(9). In addition, the C(1)—C(2) distance was 1·64 (1) Å, the C(1) and C(2) to B distances were 1·70 (1)–1·74 (1) Å while the B—B distances were all in the range 1·79 (1)–1·84 (1) Å. With no exceptions the bond distances fall in the ranges found for C—C, C—B and B—B bonds in other structurally characterized carboranes (Beall, 1975). Following refinement of all non-H atoms with anisotropic temperature factors, a difference Fourier map displayed peaks at plausible positions for all H atoms. The H atoms were entered at the observed positions and fixed with $U = 0·07$ Å^2. Refinement converged at $R = 0·056$, $wR = 0·060$ $\{w = [\sigma^2(F_o) + 0·00066F_o^2]^{-1}\}$, $S = 1·66$ for 154 parameters. In the final cycle of refinement $(\Delta/\sigma)_{\max} = 0·026$, $(\Delta/\sigma)_{\text{mean}} = 0·005$ and the largest variation from zero on the final difference Fourier synthesis was 0·17 e Å^{-3}.

Compound (2). Compound (2) was also prepared by the method of Wu & Jones (1986) and recrystallized from hexane to give clear crystals. Diffraction data were collected from a clear crystal 0·20 × 0·25 × 0·25 mm, using a 1·0° ω-scan mode for all data with $3 \leq 2\theta \leq 114°$, $0 \leq h \leq 24$, $0 \leq k \leq 11$, $0 \leq l \leq 10$. The lattice parameters were obtained from a least-squares fit of 15 centered reflections with $40 \leq 2\theta \leq 45°$. Standard reflections 250, 922 were monitored after every 50 reflections and showed a 22% linear decline in intensity. A linear-decay correction was applied to the data. The 1813 reflections were reduced by applying Lorentz and polarization corrections. No correction for absorption was applied. Systematically absent reflections were eliminated to give 1541 unique data of which 1096 were considered to be observed $[F_o > 3\sigma(F_o)]$.

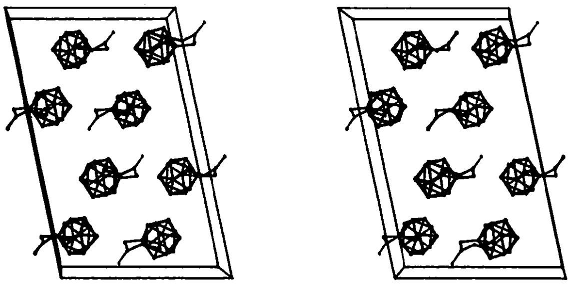


Fig. 1. Packing of (1) in the unit cell.

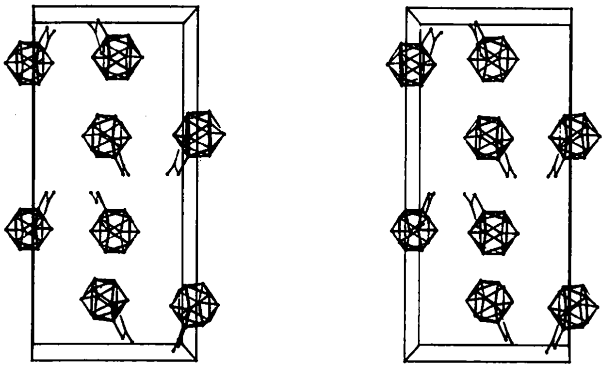


Fig. 2. Packing of (2) in the unit cell.

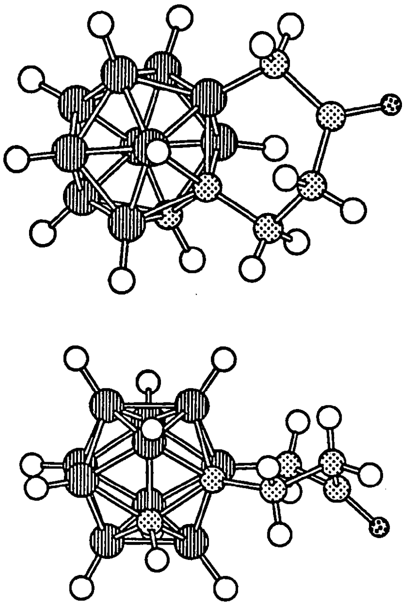


Fig. 3. Two views of compound (1).

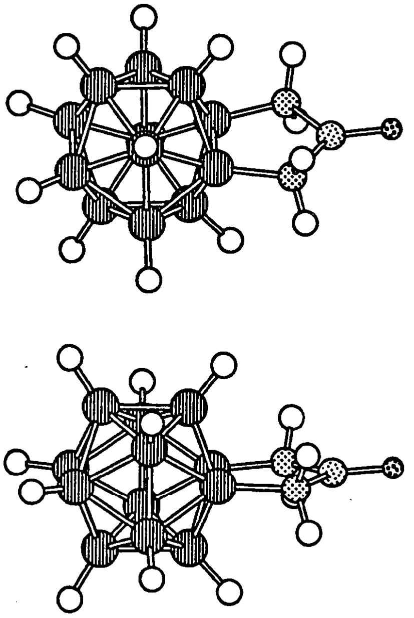


Fig. 4. Two views of compound (2).

Table 3. *Bond lengths* (Å)

(1)		(2)*	
C(1)—C(2)	1·646 (4)	C(1)—C(2)	1·688 (5)
C(1)—B(3)	1·732 (4)	C(1)—B(3)	1·695 (5)
C(1)—B(4)	1·710 (4)	C(1)—B(4)	1·662 (5)
C(1)—B(5)	1·706 (4)	C(1)—B(5)	1·706 (5)
C(1)—B(6)	1·721 (4)	C(1)—B(6)	1·717 (6)
C(1)—C(13)	1·515 (4)	C(1)—C(13)	1·535 (5)
C(2)—B(3)	1·721 (4)	C(2)—B(3)	1·698 (5)
C(2)—B(6)	1·714 (4)	C(2)—B(6)	1·710 (6)
C(2)—B(7)	1·711 (5)	C(2)—B(7)	1·745 (6)
C(2)—B(11)	1·695 (5)	C(2)—B(11)	1·726 (7)
B(3)—B(4)	1·778 (5)	B(3)—B(4)	1·733 (5)
B(3)—B(7)	1·765 (5)	B(3)—B(7)	1·722 (6)
B(3)—B(8)	1·755 (5)	B(3)—B(8)	1·723 (5)
B(4)—B(5)	1·799 (4)	B(4)—B(5)	1·727 (5)
B(4)—B(8)	1·787 (5)	B(4)—B(8)	1·727 (5)
B(4)—B(9)	1·793 (4)	B(4)—B(9)	1·743 (5)
B(4)—C(16)	1·582 (4)	B(4)—C(15)	1·569 (5)
B(5)—B(6)	1·761 (5)	B(5)—B(6)	1·742 (6)
B(5)—B(9)	1·778 (4)	B(5)—B(9)	1·756 (6)
B(5)—B(10)	1·763 (5)	B(5)—B(10)	1·747 (6)
B(6)—B(10)	1·748 (5)	B(6)—B(10)	1·741 (7)
B(6)—B(11)	1·755 (6)	B(6)—B(11)	1·737 (7)
B(7)—B(8)	1·765 (5)	B(7)—B(8)	1·741 (6)
B(7)—B(11)	1·771 (5)	B(7)—B(11)	1·758 (6)
B(7)—B(12)	1·774 (6)	B(7)—B(12)	1·768 (6)
B(8)—B(9)	1·788 (4)	B(8)—B(9)	1·759 (6)
B(8)—B(12)	1·790 (5)	B(8)—B(12)	1·735 (6)
B(9)—B(10)	1·784 (5)	B(9)—B(10)	1·773 (6)
B(9)—B(12)	1·765 (5)	B(9)—B(12)	1·750 (6)
B(10)—B(11)	1·772 (6)	B(10)—B(11)	1·749 (7)
B(10)—B(12)	1·777 (5)	B(10)—B(12)	1·749 (7)
B(11)—B(12)	1·780 (6)	B(11)—B(12)	1·756 (7)
C(13)—C(14)	1·540 (5)	C(13)—C(14)	1·505 (6)
C(14)—C(15)	1·502 (4)	C(14)—O	1·199 (5)
C(15)—C(16)	1·498 (4)	C(14)—C(15)	1·517 (6)
C(15)—O	1·223 (3)		

* C(1), C(2), B(4), B(6), B(8) and B(9) represent hybrid B, C atoms.

Table 4. *A comparison of bond lengths in* (1)

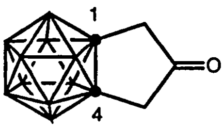
Experimental bond length (Å) with e.s.d.'s in parentheses	Experimental mean (Å)	Calculated mean* (Å)
C(1)—C(2) [1·646 (4)]	1·646	1·634
B(9)—B(12) [1·765 (5)]	1·765	1·779
B(3)—B(8) [1·755 (5)]	1·752	1·755
B(6)—B(10) [1·748 (5)]		
C(1)—B(4) [1·710 (4)]	1·705	1·700
C(2)—B(7) [1·711 (5)]		
C(1)—B(5) [1·706 (4)]		
C(2)—B(11) [1·695 (5)]		
B(8)—B(9) [1·788 (4)]	1·785	1·791
B(10)--B(9) [1·784 (5)]		
B(8)—B(12) [1·790 (5)]		
B(10)—B(12) [1·777 (5)]		

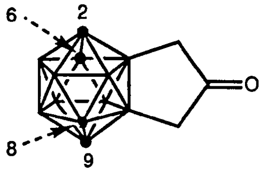
The following entries compare related bonds, one of which is attached to an atom shared between the cage and ring and one of which is not

B(4)—B(5) [1·799 (4)]	1·785	1·789
B(7)—B(11) [1·771 (5)]		
C(1)—B(3) [1·732 (4)]	1·722	1·729
C(2)—B(3) [1·721 (4)]		
C(1)—B(6) [1·721 (4)]		
C(2)—B(6) [1·714 (4)]		
B(3)—B(4) [1·778 (5)]	1·765	1·782
B(3)—B(7) [1·765 (5)]		
B(6)—B(5) [1·761 (5)]		
B(6)—B(11) [1·755 (6)]		
B(4)—B(8) [1·787 (5)]	1·772	1·777
B(7)—B(8) [1·765 (5)]		
B(5)—B(10) [1·763 (5)]		
B(11)—B(10) [1·772 (6)]		
B(4)—B(9) [1·793 (4)]	1·781	1·772
B(7)—B(12) [1·774 (6)]		
B(5)—B(9) [1·778 (4)]		
B(11)—B(12) [1·780 (6)]		

* Calculations for the parent *o*-carborane (Ott & Gimarc, 1986).

Because of the packing disorder of the molecule, the C atoms of the carborane are disordered throughout several possible positions as follows: one is disordered between the B(1) and B(4) positions, the second between B(2), B(6), B(8) and B(9).





The atoms at positions 1 and 4 were refined as $\frac{1}{2}$ B, $\frac{1}{2}$ C, and those at the 2, 6, 8 and 9 positions as $\frac{1}{4}$ C, $\frac{3}{4}$ B. Following refinement of all non-H atoms with anisotropic temperature factors, a difference Fourier map displayed peaks at plausible positions for all H atoms (0·31–0·48 e Å^{-3}). The H atoms were fixed at the observed positions with U = 0·08 Å^2. Refinement converged at $R = 0{\cdot}067$, $wR = 0{\cdot}077$ $\{w = [\sigma^2(F_o) + 0{\cdot}0015F_o^2]^{-1}\}$, and $S = 1{\cdot}54$ for 146 parameters. In the final cycle of refinement $(\Delta/\sigma)_{\max} = 0{\cdot}011$, $(\Delta/\sigma)_{\text{mean}} = 0{\cdot}001$ and the largest variation from zero on the final difference Fourier synthesis was 0·19 e Å^{-3}. Analysis of $\sum w(F_o^2 - F_c^2)^2$ as a function of F_o^2, setting angles and Miller indices showed no unusual features indicating an appropriate decay correction had been applied.

Atomic coordinates for (1) and (2) are given in Tables 1 and 2, and the packing of (1) and (2) in the unit cell is shown in Figs. 1 and 2.

Discussion. Figs. 3 and 4 show a number of views of the bridged molecules (1) and (2). The bond distances for (1) and (2) are given in Table 3.*

In compound (1), the asymmetry introduced by the exocage ring eliminates rotational disorder in the crystal and the C(1)—C(2) bond distance can be identified with certainty. At 1·646 (4) Å, it falls squarely in the normal range (Beall, 1975). The icosahedron is not severely distorted by the presence of the ring, and the B—C bonds are also within

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* Lists of structure factors, anisotropic thermal parameters for (1) and (2) and bond angles for (1) and (2) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52421 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. A copy of the Chem3D™ files used to create Figs. 1 and 2 from the crystal coordinates can be obtained from the authors if a 3·5" disk is supplied. 'Chem3D' is a trade mark of Cambridge Scientific Computing Inc., PO Box 2123, Cambridge, MA 02238, USA.

normal ranges. Similarly, the 'cyclohexenone' ring has normal bond lengths and angles, with the C—B bond shared with the cage having a normal carborane bond length [1·710 (4) Å].

The exocage ring in (1) does exert an influence on the detailed structure of the carborane in the vicinity of the ring. For instance, the bonds to the C and B atoms shared with the exocage ring are slightly longer than the related bonds on the other side of the molecule. Table 4 summarizes the situation and provides a comparison of all bond lengths with the results of calculations by Ott & Gimarc (1986) on *o*-carborane itself.

As noted earlier, compound (2) is rotationally disordered, and therefore some distances reported in Table 3 represent averaged B—C, C—C or B—C B—B distances. Nevertheless, it is possible to say that the bond distances and angles in the exocyclic ring are normal, with a single exception. Because of our earlier structural assignment which showed that the ring connects carbon and boron as shown in (2), (Wu & Jones, 1986), we can identify with certainty the framework bond making up part of the five-membered ring. Its length is 1·662 (5) Å, shorter than the corresponding bond in (1) [1·710 (4) Å] and other carboranes (Beall, 1975). Presumably, this reflects the inability of the three-carbon bridge to span a 'normal' C—B distance of about 1·70 Å.

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5-(2-Bromo-4,5-dimethoxyphenyl)-4-(2-hydroxy-3,4-dimethoxyphenyl)pyrimidine

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Abstract. 6-[5-(2-Bromo-4,5-dimethoxyphenyl)-4-pyrimidinyl]-2,3-dimethoxyphenol, $C_{20}H_{19}BrN_2O_5$, $M_r = 447·3$, triclinic, $P\bar{1}$, $a = 9·360\ (2)$, $b = 11·771\ (3)$, $c = 10·236\ (3)$ Å, $\alpha = 78·23\ (2)$, $\beta = 65·28\ (2)$, $\gamma = 72·19\ (2)°$, $V = 971·5\ (5)$ Å^3, $Z = 2$, $D_x = 1·52$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 2·13$ mm^{-1}, $F(000) = 456$, $T = 293$ K, $R = 0·050$ for 2123 observed reflections. The crystal structure is in agreement with that predicted from IR and NMR spectral studies in solution. The bromobenzene ring at C(7) is nearly perpendicular [105·8 (2)°] to the heterocyclic ring, while the other aryl substituent at C(12) is rotated out of the pyrimidine ring plane by 20·8 (2)°. An intramolecular OH···N bond is observed with the distance O···N = 2·55 (1) Å and the angle O—H···N = 150 (7)°.

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