Acta Cryst. (1996). C**52**, 2221–2222

Redetermination of 1,7-Diphenyl-1,7-dicarba-*closo*-dodecaborane(12)

GEORGINA M. ROSAIR, ALAN J. WELCH AND ANDREW S. WELLER

Department of Chemistry, Heriot-Watt University, Riccarton, Edinburgh EH14 4AS, Scotland. E-mail: chegmr@bonaly.hw.ac.uk

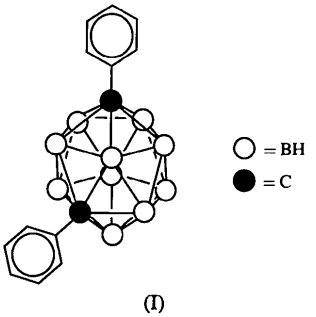
(*Received 13 December 1995; accepted 12 February 1996*)

Abstract

The previous determination of the structure of the title molecule, $C_{14}H_{20}B_{10}$ [Astakhin, Romanov, Gusev, Kalinin, Zakharkin & Los (1977). *Zh. Strukt. Khim.* **18**, 406–408] with R = 0.15 has been superseded by the present determination which has R = 0.06.

Comment

We are currently investigating the structural and chemical consequences of introducing deliberate overcrowding into heteroborane polyhedra. A substantial number of interesting results have recently been obtained using the {7,8-Ph_2-7,8-$C_2B_9H_9$} ligand, in which the cage-bound phenyl groups are adjacent not only to each other but also to a ligated metal fragment (*e.g.* Grädler, Reed, Welch & Weller, 1996). In contrast, relatively few studies have so far been reported involving the {7,9-Ph_2-7,9-$C_2B_9H_9$} ligand. The structure of the parent *closo* carborane, 1,7-Ph_2-1,7-$C_2B_{10}H_{10}$, has already been reported (Astakhin, Romanov, Gusev, Kalinin, Zakharkin & Los, 1977) but is of limited precision (R = 0.15). To establish an accurate structure of this molecule against which to discuss the structures of future derivatives, we herein report an accurate redetermination of the title compound, (I).



(I)

The resulting structure of 1,7-Ph_2-1,7-$C_2B_{10}H_{10}$ is accurately defined (e.s.d. on C—C distance typically 0.003 Å). The carborane cage has the expected near-icosahedral geometry with C—B distances in the range 1.701 (3)–1.732 (3) Å and B—B distances in the range 1.760 (4)–1.787 (4) Å. C_{cage}—C_{phenyl} distances are 1.514 (3) and 1.520 (3) Å. The phenyl substituents are planar within experimental error and stand orthogonal to the B_5 rings to which the cage C atoms are attached [dihedral angles 88.6 (2) and 92.1 (2)°]. The dihedral angle between the two phenyl rings is 113.1 (2)°.

An accurate crystallographic study of 1,2-Ph_2-1,2-$C_2B_{10}H_{10}$ (Lewis & Welch, 1993) has been reported recently. A further example of a related molecule is 1-Ph-1,2-$C_2B_{10}H_{11}$ in two crystalline modifications which has recently been structurally characterized (Thomas, Rosair & Welch, 1996; Brain, Cowie, Donohoe, Hnyk, Rankin, Reed, Reid, Robertson, Welch, Hofmann & Schleyer, 1996).

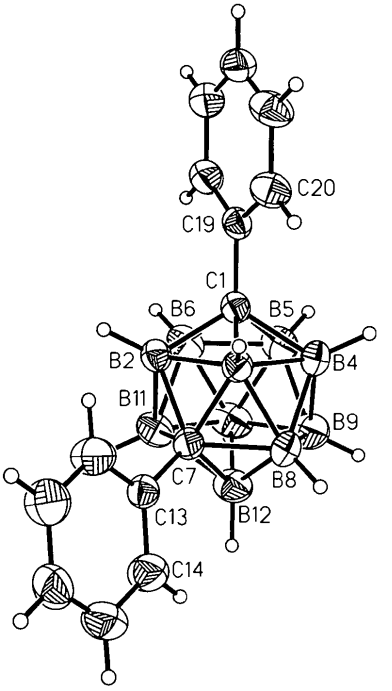


Fig. 1. Perspective view of 1,7-Ph_2-1,7-$C_2B_{10}H_{10}$ with displacement ellipsoids drawn at the 40% probability level, except for H atoms which have artificial radii of 0.1 Å for clarity.

Experimental

The title compound was synthesized by the thermolysis of 1,2-Ph_2-1,2-$C_2B_{10}H_{10}$ in a sealed tube at 670 K for 48 h. The crude product was extracted into pentane and purified by column chromatography using pentane as eluent. Purity was confirmed by microanalysis and ^{11}B NMR spectroscopy. Crystals were grown by the slow evaporation of a pentane solution at 290 K.

Crystal data

$C_{14}H_{20}B_{10}$ — Mo $K\alpha$ radiation
M_r = 296.40 — λ = 0.71073 Å

Monoclinic
$P2_1/n$
a = 10.8980 (13) Å
b = 7.6993 (6) Å
c = 20.713 (2) Å
β = 103.971 (8)°
V = 1686.6 (3) Å^3
Z = 4
D_x = 1.167 Mg m^{-3}
D_m not measured

Cell parameters from 29 reflections
θ = 4.72–12.44°
μ = 0.057 mm^{-1}
T = 293 (2) K
Block
0.6 × 0.5 × 0.4 mm
Colourless

Data collection

Siemens P4 diffractometer
ω scans
Absorption correction:
empirical *via* ψ scans (*SHELXTL/PC*; Sheldrick, 1994)
T_{min} = 0.748, T_{max} = 0.803
3949 measured reflections
2929 independent reflections
2012 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0371
θ_{max} = 25.00°
$h = -1 \rightarrow 12$
$k = -1 \rightarrow 9$
$l = -24 \rightarrow 24$
3 standard reflections
monitored every 97 reflections
intensity decay: 5.9%; corrected for

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0601
$wR(F^2)$ = 0.2465
S = 1.020
2927 reflections
227 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0856P)^2 + 0.4235P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.190 e Å^{-3}
$\Delta\rho_{min}$ = −0.170 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	−0.0264 (2)	0.4434 (3)	0.15469 (10)	0.0402 (5)
B2	−0.0955 (2)	0.2469 (3)	0.13132 (11)	0.0428 (6)
B3	−0.0890 (2)	0.4099 (3)	0.07179 (12)	0.0409 (6)
B4	−0.1033 (3)	0.6141 (4)	0.10786 (14)	0.0524 (7)
B5	−0.1195 (3)	0.5758 (4)	0.1905 (2)	0.0573 (8)
B6	−0.1150 (3)	0.3486 (4)	0.20431 (13)	0.0523 (7)
C7	−0.2257 (2)	0.3010 (3)	0.06970 (10)	0.0419 (5)
B8	−0.2348 (3)	0.5203 (4)	0.0517 (2)	0.0541 (7)
B9	−0.2542 (3)	0.6222 (4)	0.1256 (2)	0.0702 (10)
B10	−0.2617 (3)	0.4583 (5)	0.1847 (2)	0.0656 (9)
B11	−0.2469 (3)	0.2540 (4)	0.14776 (13)	0.0530 (7)
B12	−0.3325 (3)	0.4246 (4)	0.0991 (2)	0.0598 (8)
C13	−0.2699 (2)	0.1747 (3)	0.01281 (10)	0.0439 (5)
C14	−0.3924 (3)	0.1737 (4)	−0.02450 (13)	0.0675 (8)
C15	−0.4312 (3)	0.0584 (4)	−0.0767 (2)	0.0802 (9)
C16	−0.3487 (3)	−0.0575 (4)	−0.09244 (14)	0.0703 (8)
C17	−0.2270 (3)	−0.0580 (4)	−0.05596 (15)	0.0777 (9)
C18	−0.1879 (3)	0.0565 (4)	−0.00398 (14)	0.0677 (8)
C19	0.1167 (2)	0.4520 (3)	0.17884 (11)	0.0425 (5)
C20	0.1897 (2)	0.5056 (4)	0.13637 (12)	0.0550 (6)
C21	0.3201 (2)	0.5111 (4)	0.15818 (15)	0.0654 (8)
C22	0.3780 (3)	0.4632 (3)	0.2217 (2)	0.0660 (8)
C23	0.3066 (3)	0.4118 (4)	0.2643 (2)	0.0691 (8)
C24	0.1763 (2)	0.4062 (3)	0.24295 (13)	0.0574 (7)

Table 2. *Selected geometric parameters (Å, °)*

C1—C19	1.520 (3)	B5—B9	1.771 (5)
C1—B3	1.707 (3)	B5—B6	1.772 (4)
C1—B2	1.707 (3)	B5—B10	1.773 (4)
C1—B4	1.725 (3)	B6—B10	1.767 (4)
C1—B5	1.727 (3)	B6—B11	1.777 (4)
C1—B6	1.732 (3)	C7—C13	1.514 (3)
B2—C7	1.715 (3)	C7—B12	1.725 (3)
B2—B6	1.760 (4)	C7—B11	1.726 (3)
B2—B11	1.764 (4)	C7—B8	1.727 (3)
B2—B3	1.773 (3)	B8—B12	1.774 (4)
B3—C7	1.701 (3)	B8—B9	1.777 (4)
B3—B8	1.761 (4)	B9—B12	1.765 (4)
B3—B4	1.763 (4)	B9—B10	1.774 (5)
B4—B8	1.768 (4)	B10—B11	1.773 (4)
B4—B9	1.771 (4)	B10—B12	1.773 (4)
B4—B5	1.787 (4)	B11—B12	1.777 (5)
C19—C1—B3	117.8 (2)	C13—C7—B3	116.7 (2)
C19—C1—B2	118.7 (2)	C13—C7—B2	118.3 (2)
C19—C1—B4	118.2 (2)	C13—C7—B12	120.9 (2)
C19—C1—B5	119.7 (2)	C13—C7—B11	120.3 (2)
C19—C1—B6	119.9 (2)	C13—C7—B8	118.0 (2)

Atomic coordinates for B and C atoms taken from the Cambridge Structural Database (refcode DPMCBO; Allen & Kennard, 1993) were used as a starting point for refinement, after transformation by the matrix (−1,1,0; 0,0,−1; 0,1,0). The phenyl H atoms were constrained to idealized positions (C—H 0.93 Å) as were the cage H atoms (B—H 1.10 Å). The isotropic displacement parameters of both sets of H atoms were defined as 1.2 × U_{iso} of the bound atom.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

We thank Heriot-Watt University (GMR) and the EPSRC (ASW) for provision of postdoctoral fellowships and the Callery Chemical Company for a generous gift of $B_{10}H_{14}$.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1346). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8** 31–37.

Astakhin, A. V., Romanov, V. V., Gusev, A. I., Kalinin, V. N., Zakharkin, L. I. & Los, M. G. (1977). *Zh. Strukt. Khim.* **18**, 406–408.

Brain, P. T., Cowie, J., Donohoe, D. J., Hnyk, D., Rankin, D. W. H., Reed, D., Reid, B. D., Robertson, H. E., Welch, A. J., Hofmann, M. & Schleyer, P. von R. (1996). *Inorg. Chem.* In the press.

Grädler, U., Reed, D. R., Welch, A. J. & Weller, A. S. (1996). *J. Chem. Soc. Dalton Trans.* 335–342.

Lewis, Z. G. & Welch, A. J. (1993). *Acta Cryst.* C**49**, 705–708.

Thomas, R. Ll., Rosair, G. M. & Welch, A. J. (1996). *Acta Cryst.*C**52**. 1024–1026.

Sheldrick, G. M. (1994). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.