Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	$U_{\rm eq}$
O1	0.3655 (5)	0.23117 (11)	−0.0704 (5)	0.0534 (10)
O4	−0.1304 (5)	0.16407 (12)	−0.0474 (6)	0.0634 (11)
O7	0.0520 (5)	0.36750 (11)	−0.0305 (6)	0.0576 (10)
C2	0.3586 (8)	0.1870 (2)	−0.0723 (7)	0.0479 (12)
C3	0.2020 (7)	0.1624 (2)	−0.0675 (7)	0.0477 (12)
C4	0.0184 (8)	0.1842 (2)	−0.0567 (7)	0.0474 (12)
C4a	0.0269 (7)	0.2319 (2)	−0.0577 (7)	0.0446 (12)
C5	−0.1351 (7)	0.2577 (2)	−0.0514 (8)	0.0504 (13)
C6	−0.1240 (8)	0.3020 (2)	−0.0470 (8)	0.0517 (13)
C7	0.0550 (8)	0.3232 (2)	−0.0452 (7)	0.0480 (12)
C8	0.2163 (7)	0.2988 (2)	−0.0576 (7)	0.0496 (13)
C8a	0.1989 (7)	0.2536 (2)	−0.0612 (7)	0.0436 (12)
C9	0.2165 (7)	0.1143 (2)	−0.0643 (8)	0.0504 (13)
C10	0.1701 (8)	0.0900 (2)	0.0787 (9)	0.061 (2)
C11	0.1867 (10)	0.0455 (2)	0.0865 (11)	0.074 (2)
C12	0.2501 (10)	0.0234 (2)	−0.0489 (12)	0.082 (2)
C13	0.2970 (10)	0.0468 (2)	−0.1941 (11)	0.077 (2)
C14	0.2811 (9)	0.0917 (2)	−0.2017 (9)	0.064 (2)
C15	0.2343 (8)	0.3925 (2)	−0.0038 (9)	0.0551 (14)
C16	0.2521 (10)	0.4022 (2)	−0.2030 (10)	0.074 (2)
C17	0.2118 (10)	0.4337 (2)	0.1032 (10)	0.078 (2)

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

O1—C2	1.351 (6)	C2—C3	1.341 (6)
O1—C8a	1.373 (5)	C3—C4	1.473 (7)
O4—C4	1.232 (6)	C3—C9	1.476 (7)
O7—C7	1.361 (6)	C4—C4a	1.461 (7)
O7—C15	1.443 (6)		
C2—O1—C8a	118.1 (4)	C3—C2—O1	126.1 (5)
C7—O7—C15	120.2 (4)		
C2—C3—C9—C10	128.5 (5)	C7—O7—C15—C16	86.1 (6)
C2—C3—C9—C14	−50.2 (7)	C7—O7—C15—C17	−153.1 (5)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Software used to prepare material for publication: *FINISH* in *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1158). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Acharya, K. R., Puranik, V. G., Tavale S. S. & Guru Row, T. N. (1986). *Acta Cryst.* C**42**, 597–599.

Breton, M. B., Precigoux, G., Courseille, C. & Hospital, M. (1975). *Acta Cryst.* B**31**, 921–924.

Caballero, P. & Smith, C. M. (1986). *J. Nat. Prod.* **49**, 1126–1129.

Feuer, L., Nógrádi, M., Gottsegen, Á., Vermes, B., Streliszky, J., Wolfner, A., Farkas, L., Antus, S. & Kovács, M. (1971). Ger. Patent 2 125 245; *Chem. Abstr.* **76**, 72407.

Kaneda, M., Iitaka, Y. & Shibita, S. (1973). *Acta Cryst.* B**29**, 2827–2832.

Lányi, G., Nógrádi, M., Ecsery-Puskás M. & Hermecz, I. (1995). *Acta Pharm. Hung.* In the press

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany

Shoja, M. (1992*a*). *Acta Cryst.* C**48**, 2033–2035.

Shoja, M. (1992*b*). *Z. Crystallogr.* **199**, 161–166.

Varga, M., Bátori, S. & Hermecz, I. (1995). *Acta Pharm. Hung.* In the press

---

Acta Cryst. (1996). C**52**, 1024–1026

A New Crystalline Form of 1-Phenyl-1,2-dicarba-*closo*-dodecaborane(12)

RHODRI LL. THOMAS, GEORGINA M. ROSAIR AND ALAN J. WELCH

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

(*Received 5 June 1995; accepted 29 August 1995*)

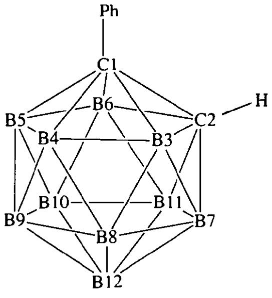
Abstract

A new polymorph of the title compound, $C_8H_{16}B_{10}$, is reported. The carborane icosahedron is relatively undistorted, with the phenyl substituent twisted out of the C2—C1—$C_{\rm ring}$ plane by 18.3 (2)°, which is in good agreement with the conformation predicted by molecular-orbital calculations.

Comment

1-Ph-1,2-*closo*-$C_2B_{10}H_{11}$ has been known since 1963 (Heyling *et al.*, 1963) and many derivatives of it have been structurally characterized (Clegg *et al.*, 1993; McGrath & Welch, 1995*a*,*b*,*c*,*d*). Surprisingly, it is only recently that a structural determination of the parent molecule has been reported (Brain *et al.*, 1996). This previous crystal form, which will be denoted as the α form, contained two crystallographically independent molecules, only one of which was sufficiently ordered for all cage atoms to be identified. In the other molecule, the position of atom C2 could not be determined unambiguously. In the new crystalline modification described herein, which will be referred to as the β form, there is only one molecule in the asymmetric unit and the cage C atom bearing the H atom is easily identified. All reference to the molecular parameters of the α form are to those of the ordered molecule. The determination of the β form, (I), proved to be more precise than that of the α form, with typical e.s.d.'s being one third of the values reported previously, presumably due in part to the greater order found in the present determination.

Acta Crystallographica Section C
ISSN 0108-2701 ©1996



(I)

The numbering scheme adopted for this molecule is shown in Fig. 1. This is similar to that used for many phenyl-substituted carboranes (McGrath & Welch, 1995*a*,*b*,*c*,*d*; Brain *et al.*, 1996), with the cage C atom bearing the phenyl substituent being labelled C1. The compound crystallizes with no close intermolecular contacts.

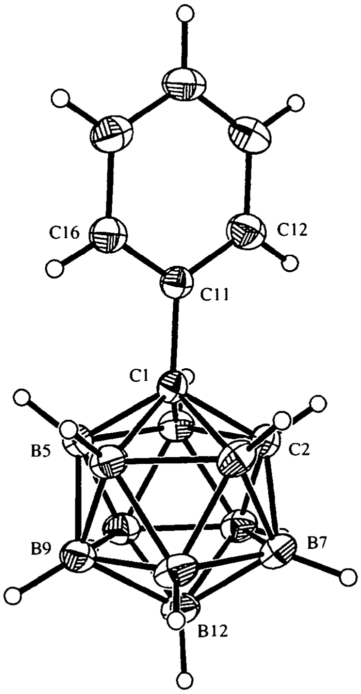


Fig. 1. Perspective view of one molecule of the β form of 1-Ph-1,2-*closo*-$C_2B_{10}H_{11}$. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of artificial radii for clarity.

In considering the structures of C-phenyl carboranes, it is convenient to discuss the conformation of the ring relative to the cage in terms of θ (Cowie, Reid, Watmough & Welch, 1994), the modulus of the average of the two C2—C1—C11—$C_{\rm ring}$ torsion angles. *Ab initio* calculations have predicted that the optimum θ angle for this molecule would be 65° (Brain *et al.*, 1996). The value of θ calculated from this structure is 71.7 (2)°, which is in excellent agreement with the *ab initio* value and only slightly greater than that found in the α form [68.6 (4)°].

Comparison of the other molecular parameters derived for the structure of the monophenylcarborane in its two crystalline forms shows no other significant differences in their values, except for a slight lengthening of the C1—B6 and C2—B3 distances of the β form relative to those of the α form.

In conclusion, this new crystalline form of 1-Ph-1,2-*closo*-$C_2B_{10}H_{11}$ shows excellent agreement with the theoretical conformation. This precise determination makes a good reference for comparison of carboranes in the series 1-Ph-2-*R*-$C_2B_{10}H_{10}$, with various different θ angles, where *R* is small, *e.g.* *R* = Me (McGrath & Welch, 1995*a*) and Br (McGrath & Welch, 1995*b*).

Experimental

The title compound was synthesized in good yield according to the method of Brain *et al.* (1996). Analytically pure crystals suitable for X-ray diffraction were grown by slow evaporation of a methanol solution at room temperature.

Crystal data

$C_8H_{16}B_{10}$
$M_r = 220.32$
Monoclinic
$P2_1/c$
$a = 7.0250\,(14)$ Å
$b = 8.408\,(2)$ Å
$c = 21.890\,(4)$ Å
$\beta = 97.89\,(3)°$
$V = 1280.7\,(4)$ Å^3
$Z = 4$
$D_x = 1.143$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 250 reflections
$\theta = 2$–$20°$
$\mu = 0.052$ mm^{-1}
$T = 150\,(2)$ K
Plate
0.36 × 0.20 × 0.18 mm
Colourless

Data collection

Enraf–Nonius FAST diffractometer
MADNES (Pflugrath & Messerschmidt, 1991)
Absorption correction: none
5323 measured reflections
1964 independent reflections

1677 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0521$
$\theta_{\rm max} = 24.89°$
$h = -7 \rightarrow 8$
$k = -7 \rightarrow 9$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R(F) = 0.0544$
$wR(F^2) = 0.1431$
$S = 1.031$
1964 reflections
227 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1092P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.348$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.304$ e Å^{-3}
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)*

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

	x	y	z	$U_{\rm eq}$
C1	0.1711 (2)	0.3010 (2)	0.12266 (6)	0.0211 (4)
C2	0.3552 (2)	0.2692 (2)	0.17657 (7)	0.0252 (4)
B3	0.3993 (2)	0.2763 (2)	0.10132 (8)	0.0278 (5)
B4	0.2406 (2)	0.4270 (2)	0.06767 (8)	0.0268 (5)
B5	0.1017 (2)	0.4964 (2)	0.12460 (8)	0.0256 (4)
B6	0.1730 (2)	0.3905 (2)	0.19423 (8)	0.0255 (4)
B7	0.5585 (2)	0.3640 (2)	0.16229 (8)	0.0290 (5)
B8	0.4873 (2)	0.4708 (2)	0.09312 (8)	0.0306 (5)
B9	0.3039 (2)	0.6079 (2)	0.10756 (8)	0.0308 (5)
B10	0.2615 (2)	0.5844 (2)	0.18573 (8)	0.0288 (5)
B11	0.4200 (3)	0.4335 (2)	0.21946 (8)	0.0278 (5)
B12	0.5005 (3)	0.5686 (2)	0.16614 (8)	0.0285 (5)
C11	0.0288 (2)	0.1680 (2)	0.10596 (6)	0.0223 (4)
C12	0.0168 (2)	0.0389 (2)	0.14551 (8)	0.0336 (4)
C13	−0.1132 (2)	−0.0831 (2)	0.12821 (8)	0.0366 (5)
C14	−0.2325 (2)	−0.0777 (2)	0.07242 (8)	0.0328 (4)
C15	−0.2224 (2)	0.0511 (2)	0.03389 (8)	0.0327 (4)
C16	−0.0930 (2)	0.1739 (2)	0.05060 (7)	0.0280 (4)

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

C1—C11	1.511 (2)	B6—B10	1.764 (2)
C1—C2	1.649 (2)	B6—B11	1.784 (2)
C1—B5	1.716 (2)	B7—B8	1.772 (3)
C1—B4	1.724 (2)	B7—B12	1.773 (3)
C1—B6	1.736 (2)	B7—B11	1.786 (3)
C1—B3	1.742 (2)	B8—B9	1.789 (3)
C2—B11	1.698 (2)	B8—B12	1.789 (2)
C2—B7	1.701 (2)	B9—B12	1.782 (3)
C2—B3	1.718 (2)	B9—B10	1.788 (3)
C2—B6	1.722 (2)	B10—B11	1.780 (3)
B3—B8	1.766 (3)	B10—B12	1.794 (3)
B3—B4	1.779 (2)	B11—B12	1.776 (2)
B3—B7	1.780 (2)	C11—C16	1.385 (2)
B4—B9	1.780 (2)	C11—C12	1.398 (2)
B4—B5	1.783 (3)	C12—C13	1.391 (2)
B4—B8	1.785 (2)	C13—C14	1.384 (2)
B5—B6	1.777 (2)	C14—C15	1.380 (2)
B5—B9	1.784 (2)	C15—C16	1.391 (2)
B5—B10	1.785 (2)		
C11—C1—C2	118.76 (12)	B10—B6—B11	60.22 (9)
C11—C1—B5	122.26 (12)	B8—B7—B12	60.60 (10)
C11—C1—B4	121.82 (12)	C2—B7—B3	59.11 (9)
B5—C1—B4	62.45 (9)	B8—B7—B3	59.63 (10)
C11—C1—B6	117.52 (11)	C2—B7—B11	58.20 (9)
C2—C1—B6	61.07 (9)	B12—B7—B11	59.87 (10)
B5—C1—B6	61.95 (9)	B3—B8—B7	60.39 (10)
C11—C1—B3	116.70 (11)	B3—B8—B4	60.14 (9)
C2—C1—B3	60.81 (9)	B4—B8—B9	59.75 (10)
B4—C1—B3	61.77 (10)	B7—B8—B12	59.72 (10)
B11—C2—B7	63.39 (10)	B9—B8—B12	59.74 (10)
C1—C2—B3	62.29 (9)	B4—B9—B5	60.06 (10)
B7—C2—B3	62.72 (10)	B12—B9—B10	60.35 (10)
C1—C2—B6	61.98 (9)	B5—B9—B10	59.98 (10)
B11—C2—B6	62.90 (10)	B4—B9—B8	60.01 (10)
C2—B3—C1	56.90 (9)	B12—B9—B8	60.13 (10)
C1—B3—B4	58.60 (9)	B6—B10—B11	60.44 (9)
B8—B3—B4	60.44 (10)	B6—B10—B5	60.08 (9)
C2—B3—B7	58.17 (9)	B5—B10—B9	59.90 (10)
B8—B3—B7	59.98 (10)	B11—B10—B12	59.58 (9)
C1—B4—B3	59.63 (9)	B9—B10—B12	59.66 (10)
C1—B4—B5	58.57 (9)	B12—B11—B10	60.60 (10)
B9—B4—B5	60.08 (10)	C2—B11—B6	59.20 (9)
B3—B4—B8	59.42 (10)	B10—B11—B6	59.34 (10)
B9—B4—B8	60.25 (10)	C2—B11—B7	58.41 (9)
C1—B5—B6	59.58 (9)	B12—B11—B7	59.71 (10)
C1—B5—B4	58.98 (9)	B7—B12—B11	60.42 (10)
B4—B5—B9	59.86 (10)	B7—B12—B8	59.68 (10)
B6—B5—B10	59.37 (10)	B9—B12—B8	60.13 (11)
B9—B5—B10	60.12 (10)	B11—B12—B10	59.82 (10)
C2—B6—C1	56.95 (9)	B9—B12—B10	59.99 (10)
C1—B6—B5	58.47 (9)	C16—C11—C1	119.60 (13)
B10—B6—B5	60.54 (10)	C12—C11—C1	121.26 (14)
C2—B6—B11	57.89 (9)		

Intensity data were collected assuming a triclinic unit cell. With a detector-to-crystal (DET) setting of 50.28 mm and a swing angle (θ_D) of −19.9°, reflections were found in two 5° ω-rotation regions separated by 90°. The orientation matrix and unit-cell dimensions were determined by *MADNES* software (Pflugrath & Messerschmidt, 1991) using 50 reflections taken from these regions. Accurate values of DET (49.410 mm) and θ_D (−19.973°) were determined, along with improved cell dimensions, using 250 high-angle reflections from the two regions. The refined mosaic spread was 0.754°. Unit-cell refinement was subsequently performed at ω-rotation intervals of 15° during data collection. Intensity data corresponding to slightly more than one hemisphere of reciprocal space were recorded using two ω-scan regions of 99.8°, with a φ shift of 90° at χ = 90°, to record the missing cusp data. Throughout data collection, the ω increment for each frame was 0.20° and the measuring time was 8 s, with a total data collection time of 6.5 h. No significant decay of intensity levels was observed throughout the experiment.

Data collection: *MADNES*. Cell refinement: *MADNES*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1994).

We thank Heriot-Watt University for provision of a studentship (RLT), the Callery Chemical Company for a generous gift of $B_{10}H_{14}$, and the EPSRC X-ray diffraction service at the University of Wales, Cardiff, for collection of the data.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1287). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

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. & von R. Schleyer, P. (1996). *Inorg. Chem.* In the press.

Clegg, W., Coult, R., Fox, M. A., Gill, W. R., MacBride, J. A. H. & Wade, K. (1993). *Polyhedron*, **12**, 2711–2717.

Cowie, J., Reid, B. D., Watmough, J. M. S. & Welch, A. J. (1994). *J. Organomet. Chem.* **481**, 283–293.

Heying, T. L., Ager, J. W., Clark, S. L., Mangold, D. J., Goldstien, H. L., Hillman, M., Polak, R. J. & Szymanski, J. W. (1963). *Inorg. Chem.* **2**, 1089–1092.

McGrath, T. D. & Welch, A. J. (1995*a*). *Acta Cryst.* **C51**, 646–649.

McGrath, T. D. & Welch, A. J. (1995*b*). *Acta Cryst.* **C51**, 649–651.

McGrath, T. D. & Welch, A. J. (1995*c*). *Acta Cryst.* **C51**, 651–654.

McGrath, T. D. & Welch, A. J. (1995*d*). *Acta Cryst.* **C51**, 654–657.

Pflugrath, J. W. & Messerschmidt, A. (1991). *MADNES. Munich Area Detector (New EEC) System.* Small Molecule Version EEC 11/09/89. Enraf-Nonius, Delft, The Netherlands.

Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

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