Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methyl-1-propenyl-1,2-dicarba-*closo*-dodecaborane(12)

Gennadii V. Grintselev-Knyazev,[a]* Mikhail Yu. Antipin,[a] Sergey P. Knyazev,[b] Valerii N. Kirin[b] and Eugenii A. Chernyshev[b]

[a]X-Ray Structural Centre, Institute of Organoelement Compounds (INEOS), Russian Academy of Sciences, 28 Vavilov St., B-334, Moscow 119991, Russian Federation, and [b]Moscow State Academy of Fine Chemical Technology, Moscow 117571, Russian Federation

Correspondence e-mail: gena@xrlab.ineos.ac.ru

Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.057
wR factor = 0.128
Data-to-parameter ratio = 16.4

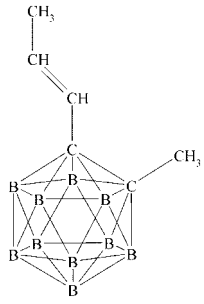
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 November 2001
Accepted 22 November 2001
Online 8 December 2001

The title compound, $C_6H_{18}B_{10}$, is the first X-ray investigated carborane with a propenyl substituent. The C—C bond length in the carborane cage is 1.688 (2) Å.

Comment

The propenyl substituent is situated in the plane bisecting the C1—B3—B4 triangular face, with a B4—C1—C13—C14 torsion angle of 36.8 (2)°.



(I)

The C1—C2 distance of 1.688 (2) Å in (I) is significantly longer than those in B-substituent carboranes (Grintselev-Knyazev *et al.*, 2001); this agrees with the general tendency of C—C bond elongation in aryl C-substituent carboranes (Lewis & Welch, 1993).

The crystal packing is shown in Fig. 2.

Experimental

A solution of 1-Me-1,2-$C_2B_{10}H_{12}$ was treated with *n*-butyllithium in ether to afford the intermediate lithiated species. The mixture obtained was added to allyl bromide to give 1-allyl-2-methyl-1,2–$C_2B_{10}H_{12}$ (Heying *et al.*, 1963). 1-Propenyl-2-methyl-1,2-$C_2B_{10}H_{12}$ was obtained by isomerization of 1-allyl-2-methyl-1,2-$C_2B_{10}H_{12}$ with an excess of alkali. The NMR spectra were obtained in d_6-acetone (5% solution). ^{1}H (360 MHz, δ, p.p.m.): 6.31 (*dq*, ═CHCH$_3$, 1H), 5.88 (*dd*, B—CH═, 1H), 2.00 (*s*, B—CH$_3$, 3H), 1.81 (*dd*, CH$_3$—CH═, 3H). ^{13}C (90.6 MHz, δ, p.p.m.): 139.55 and 123.50 (HC═), 78.75 and 76.02 (C_{carb}), 22.68 and 17.24 (CH_3). ^{11}B (115.54 MHz, δ, p.p.m.): −3.80, −4.91, −8.52, −9.16, −10.01 (relative intensity 1:1:2:4:2).

Crystal data

$C_6H_{18}B_{10}$
M_r = 198.30
Monoclinic, $P2_1/n$
a = 7.3615 (16) Å
b = 21.815 (5) Å
c = 7.6346 (16) Å
β = 91.541 (5)°
V = 1225.6 (5) Å^3
Z = 4
D_x = 1.075 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 821 reflections
θ = 3–30°
μ = 0.05 mm^{-1}
T = 110 (2) K
Prism, colorless
0.50 × 0.45 × 0.40 mm

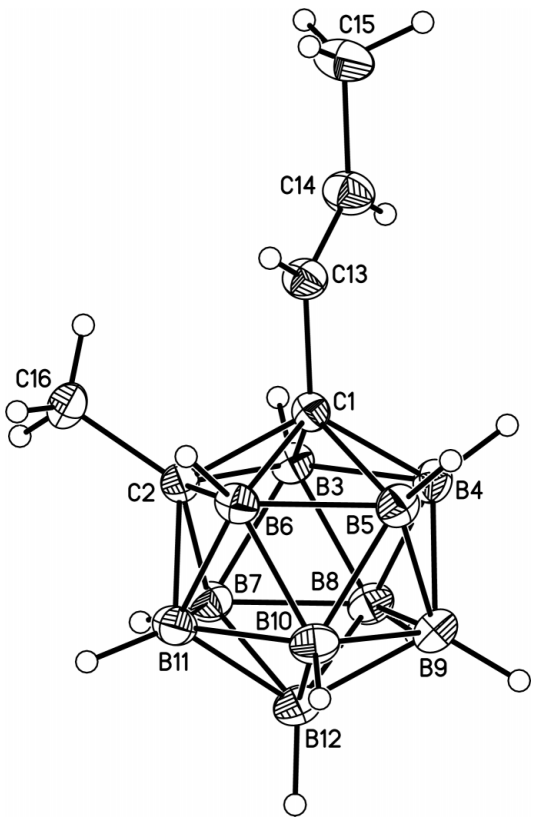


Figure 1
The molecular structure of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.981
14366 measured reflections
3557 independent reflections
2306 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 30.1°
$h = -10 \rightarrow 10$
$k = -30 \rightarrow 30$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.128
S = 1.03
3557 reflections
217 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.028P)^2 + 0.700P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

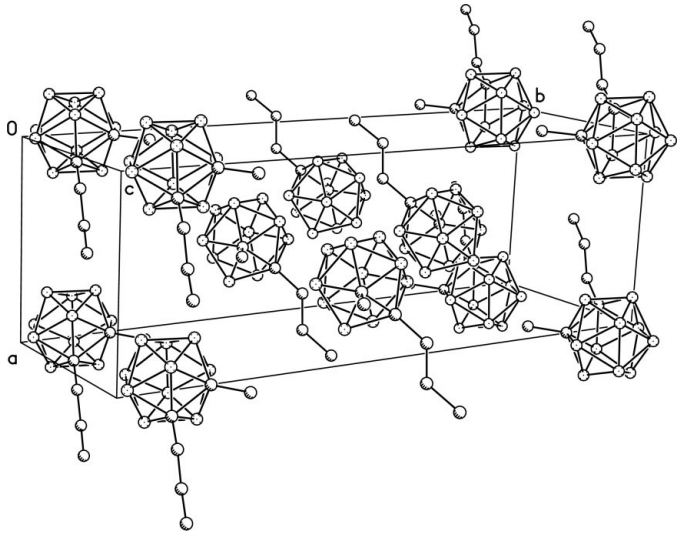


Figure 2
View of the crystal packing of (I).

Table 1
Selected geometric parameters (Å, °).

C1—C13	1.4843 (18)	C13—C14	1.312 (2)
C1—C2	1.6880 (19)	C14—C15	1.489 (2)
C2—C16	1.5062 (19)		
C13—C1—C2	117.63 (11)	C14—C13—C1	125.42 (13)
C16—C2—C1	117.51 (11)	C13—C14—C15	124.34 (14)

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART* and *SAINT* (Bruker, 1999); data reduction: *SMART* and *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SMART* (Version 5.053) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Grintselev-Knyazev, G. V., Antipin, M. Yu., Knyazev, S. P., Kirin, V. N. & Chernyshev, E. A. (2001). *Acta Cryst.* C**57**, 827–829.
Heying, T. L., Ager, J. W., Clark, S. L., Alexander, R. P., Papetti, S., Reid, J. A. & Trotz, S. I. (1963). *Inorg. Chem.* **2**, 1097–1105.
Lewis, Z. G. & Welch, A. J. (1993). *Acta Cryst.* C**49**, 705–710.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.