Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetraethylammonium 12-phenylethynyl-carba-*closo*-dodecaborate, $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$

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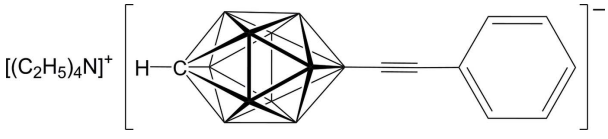
Received 23 March 2009; accepted 7 April 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.101; data-to-parameter ratio = 11.6.

The asymmetric unit of the title compound, $C_8H_{20}N^+\cdot C_9H_{16}B_{11}^-$ or $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$, consists of one cation and one anion. The $[12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]^-$ anion is close to possessing a non-crystallographic plane of mirror symmetry with a nearly linear B—C≡C—C group, with B—C≡C and C≡C—C angles of 177.15 (16) and 176.64 (17)°, respectively.

Related literature

Carba-*closo*-dodecaborate anions with functional groups are potential building blocks for a variety of applications, for example ionic liquids and liquid crystals, see: Körbe *et al.* (2006). Recently, we have shown that {*closo*-CB_{11}} clusters with one or two alkynyl groups bonded to boron are accessible by Pd-catalysed Kumada-type cross-coupling reactions starting from the corresponding mono- and diiodinated clusters, see: Finze (2008, 2009).



Experimental

Crystal data

$C_8H_{20}N^+\cdot C_9H_{16}B_{11}^-$
M_r = 373.38
Triclinic, $P\overline{1}$
a = 8.8201 (6) Å
b = 12.0929 (11) Å
c = 12.1858 (11) Å
α = 81.032 (7)°
β = 79.899 (7)°
γ = 71.553 (7)°
V = 1206.82 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.05 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

Data collection

Stoe Stadi CCD diffractometer
Absorption correction: none
17081 measured reflections
4219 independent reflections
3228 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.101
S = 1.00
4219 reflections
363 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *CrysAlis CCD* (Kuma, 2000); cell refinement: *CrysAlis RED* (Kuma, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2008); software used to prepare material for publication: *SHELXL97*.

Financial support from the Fonds der Chemischen Industrie (FCI) is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2103).

References

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Kuma (2000). *CrysAlis CCD* and *CrysAlis RED*. Kuma Diffraction, Wrocław, Poland.
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supplementary materials

Acta Cryst. (2009). E**65**, o1048 [**doi:10.1107/S1600536809013300**]

Tetraethylammonium 12-phenylethynylcarba-*closo*-dodecaborate, $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$

M. Finze and G. J. Reiss

Comment

Carba-*closo*-dodecaborate anions with functional groups are potential building blocks for a variety of applications, for example ionic liquids and liquid crystals (Körbe, 2006). Recently, we have shown that {*closo*-CB_{11}} clusters with one or two alkynyl groups bonded to boron are accessible by Pd-catalyzed Kumada-type cross-coupling reactions starting from the corresponding mono- and diiodinated clusters, respectively (Finze, 2008, 2009). The title compound $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$ crystallizes in the triclinic centrosymmetric space group $P\bar{1}$ with one formula unit in the asymmetric unit. The bond lengths and angles in the $[12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]^-$ anion in its $[Et_4N]^+$ salt are close to the values reported for the respective Cs^+ salt (Finze, 2008). However, the quality of the data for the $[Et_4N]^+$ salt described herein is significantly better than the quality of the data obtained for the Cs^+ salt. The thermal properties of $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$ were studied by differential scanning calorimetry (DSC). The salt melts at 433 K and is thermally stable up to 518 K.

Experimental

$[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$ was synthesized according to a published procedure (Finze, 2008). The spectroscopic data have been reported earlier (Finze, 2008). The salt was dissolved in acetonitrile and slow evaporation of the solvent resulted in colorless crystals.

Refinement

All hydrogen atom positions were obtained from difference fourier maps. The hydrogen atoms of the methyl groups were included in the latest stages of the refinement with a riding model and for each methyl group a common U_{iso} value was refined. The positional parameters and the isotropic displacement parameters of all other hydrogen atoms were refined freely.

Figures

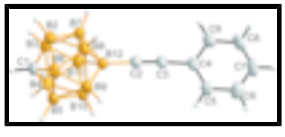

Fig. 1. : The 12-phenylethinylcarba-*closo*-dodecaborate cation in $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$. Hydrogen atoms are drawn with an arbitrary radius and the displacement ellipsoids are shown at the 40% probability level.

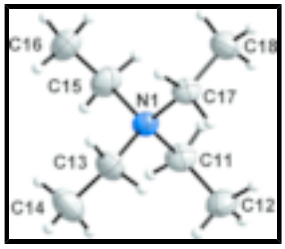

Fig. 2. : The tetraethylammonium cation in $[Et_4N][12\text{-PhCC-}closo\text{-}CB_{11}H_{11}]$. Hydrogen atoms are drawn with an arbitrary radius and the displacement ellipsoids are shown at the 40% probability level.

Tetraethylammonium 12-phenylethynylcarba-*closo*-dodecaborate

Crystal data

$C_8H_{20}N^+\cdot C_9H_{16}B_{11}^-$	$Z = 2$
$M_r = 373.38$	$F_{000} = 400$
Triclinic, $P\overline{1}$	$D_x = 1.031$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 433 K
$a = 8.8201$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 12.0929$ (11) Å	Cell parameters from 4623 reflections
$c = 12.1858$ (11) Å	$\theta = 6.8$–20.7º
$\alpha = 81.032$ (7)º	$\mu = 0.05$ mm^{-1}
$\beta = 79.899$ (7)º	$T = 293$ K
$\gamma = 71.553$ (7)º	Block, colourless
$V = 1206.82$ (18) Å^3	0.3 × 0.25 × 0.2 mm

Data collection

Stoe Stadi CCD diffractometer	3228 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.051$
Monochromator: graphite	$\theta_{max} = 25.0$º
$T = 293$ K	$\theta_{min} = 5.1$º
ω scans	$h = -10\rightarrow10$
Absorption correction: none	$k = -14\rightarrow14$
17081 measured reflections	$l = -14\rightarrow14$
4219 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 0.45P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.101$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
4219 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}

363 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.037 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.02767 (14)	0.15270 (10)	0.23820 (10)	0.0443 (3)
C11	0.1693 (2)	0.11482 (16)	0.30484 (17)	0.0572 (4)
H11A	0.145 (2)	0.1795 (17)	0.3523 (15)	0.075 (6)*
H11B	0.264 (2)	0.1153 (15)	0.2471 (15)	0.067 (5)*
C12	0.1927 (2)	−0.00116 (17)	0.37487 (17)	0.0756 (6)
H12A	0.0981	0.0009	0.4282	0.115 (5)*
H12B	0.2842	−0.0170	0.4139	0.115 (5)*
H12C	0.2110	−0.0617	0.3274	0.115 (5)*
C13	0.0403 (3)	0.06144 (18)	0.16256 (17)	0.0621 (5)
H13A	0.034 (2)	−0.0082 (16)	0.2173 (15)	0.071 (5)*
H13B	−0.055 (2)	0.0949 (16)	0.1246 (15)	0.074 (6)*
C14	0.1930 (3)	0.0312 (2)	0.08048 (18)	0.0937 (7)
H14A	0.1895	−0.0262	0.0355	0.149 (6)*
H14B	0.2844	0.0001	0.1208	0.149 (6)*
H14C	0.2024	0.1005	0.0329	0.149 (6)*
C15	0.0310 (3)	0.26924 (16)	0.17111 (18)	0.0655 (5)
H15A	0.021 (2)	0.3226 (17)	0.2329 (15)	0.074 (6)*
H15B	0.140 (2)	0.2534 (16)	0.1284 (16)	0.075 (6)*
C16	−0.0984 (3)	0.32222 (19)	0.09677 (17)	0.0891 (7)
H16A	−0.0897	0.2691	0.0434	0.126 (5)*
H16B	−0.0861	0.3948	0.0577	0.126 (5)*
H16C	−0.2023	0.3366	0.1416	0.126 (5)*
C17	−0.12997 (19)	0.16523 (15)	0.31589 (15)	0.0494 (4)
H17A	−0.119 (2)	0.0852 (16)	0.3536 (14)	0.062 (5)*
H17B	−0.207 (2)	0.1839 (14)	0.2658 (14)	0.060 (5)*
C18	−0.1717 (2)	0.25361 (16)	0.39823 (15)	0.0660 (5)
H18A	−0.1861	0.3305	0.3586	0.101 (4)*
H18B	−0.0860	0.2361	0.4430	0.101 (4)*

H18C	−0.2697	0.2509	0.4459	0.101 (4)*
C1	0.8578 (2)	−0.42097 (13)	0.37589 (14)	0.0537 (4)
H1	0.905 (2)	−0.5044 (15)	0.4091 (13)	0.064 (5)*
B2	0.7262 (3)	−0.39328 (16)	0.28148 (17)	0.0561 (5)
H2	0.6959 (19)	−0.4675 (14)	0.2554 (13)	0.064 (5)*
B3	0.9243 (2)	−0.38352 (16)	0.23998 (17)	0.0555 (5)
H3	1.012 (2)	−0.4499 (15)	0.1888 (13)	0.065 (5)*
B4	0.9825 (2)	−0.33524 (16)	0.35199 (17)	0.0535 (5)
H4	1.108 (2)	−0.3739 (14)	0.3728 (13)	0.064 (5)*
B5	0.8195 (2)	−0.31531 (15)	0.46186 (16)	0.0516 (5)
H5	0.8448 (19)	−0.3415 (14)	0.5490 (14)	0.062 (5)*
B6	0.6607 (2)	−0.35139 (16)	0.41916 (17)	0.0560 (5)
H6	0.588 (2)	−0.3994 (14)	0.4814 (13)	0.065 (5)*
B7	0.7589 (2)	−0.27118 (15)	0.19136 (16)	0.0503 (4)
H7	0.7377 (18)	−0.2580 (13)	0.1044 (13)	0.057 (4)*
B8	0.5950 (2)	−0.25187 (16)	0.30200 (17)	0.0523 (5)
H8	0.465 (2)	−0.2256 (14)	0.2866 (13)	0.067 (5)*
B9	0.6529 (2)	−0.20266 (15)	0.41506 (16)	0.0496 (4)
H9	0.5641 (19)	−0.1430 (14)	0.4736 (13)	0.057 (4)*
B10	0.8521 (2)	−0.19299 (14)	0.37266 (15)	0.0463 (4)
H10	0.8952 (18)	−0.1288 (13)	0.4032 (12)	0.054 (4)*
B11	0.9180 (2)	−0.23517 (15)	0.23469 (16)	0.0486 (4)
H11	1.002 (2)	−0.1994 (14)	0.1743 (13)	0.063 (5)*
B12	0.7131 (2)	−0.15303 (14)	0.27349 (14)	0.0436 (4)
C2	0.64183 (18)	−0.02308 (13)	0.22826 (12)	0.0467 (4)
C3	0.58462 (17)	0.07904 (13)	0.19784 (12)	0.0446 (4)
C4	0.51356 (16)	0.20251 (12)	0.16824 (12)	0.0420 (3)
C5	0.46196 (19)	0.27790 (14)	0.25139 (15)	0.0503 (4)
H5A	0.4757 (19)	0.2488 (14)	0.3277 (14)	0.059 (5)*
C6	0.3917 (2)	0.39677 (15)	0.22449 (18)	0.0633 (5)
H6A	0.357 (2)	0.4489 (17)	0.2854 (16)	0.078 (6)*
C7	0.3720 (2)	0.44060 (16)	0.11525 (18)	0.0662 (5)
H7A	0.318 (2)	0.5237 (19)	0.0958 (16)	0.089 (6)*
C8	0.4210 (2)	0.36716 (16)	0.03291 (18)	0.0647 (5)
H8A	0.408 (2)	0.3951 (16)	−0.0451 (16)	0.074 (5)*
C9	0.4921 (2)	0.24830 (15)	0.05830 (15)	0.0536 (4)
H9A	0.530 (2)	0.1947 (15)	−0.0011 (14)	0.062 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0430 (7)	0.0412 (7)	0.0525 (7)	−0.0178 (6)	−0.0068 (6)	−0.0043 (5)
C11	0.0394 (9)	0.0633 (11)	0.0709 (12)	−0.0172 (8)	−0.0107 (9)	−0.0057 (9)
C12	0.0600 (12)	0.0723 (13)	0.0766 (13)	0.0020 (10)	−0.0135 (10)	0.0039 (10)
C13	0.0695 (13)	0.0592 (11)	0.0616 (11)	−0.0210 (10)	−0.0051 (10)	−0.0189 (9)
C14	0.1051 (18)	0.0901 (16)	0.0746 (14)	−0.0207 (13)	0.0179 (13)	−0.0270 (12)
C15	0.0741 (13)	0.0518 (10)	0.0705 (12)	−0.0272 (10)	−0.0061 (11)	0.0080 (9)
C16	0.1112 (18)	0.0705 (13)	0.0693 (13)	−0.0084 (12)	−0.0202 (13)	0.0107 (10)

C17	0.0387 (9)	0.0518 (10)	0.0590 (10)	−0.0155 (7)	−0.0070 (8)	−0.0048 (8)
C18	0.0640 (12)	0.0628 (11)	0.0675 (11)	−0.0110 (9)	−0.0052 (9)	−0.0162 (9)
C1	0.0603 (10)	0.0321 (8)	0.0662 (10)	−0.0067 (7)	−0.0200 (8)	−0.0001 (7)
B2	0.0654 (13)	0.0385 (9)	0.0701 (12)	−0.0170 (9)	−0.0233 (10)	−0.0037 (9)
B3	0.0587 (12)	0.0423 (10)	0.0620 (12)	−0.0061 (9)	−0.0080 (10)	−0.0137 (9)
B4	0.0475 (11)	0.0417 (9)	0.0684 (12)	−0.0036 (8)	−0.0184 (9)	−0.0056 (8)
B5	0.0625 (12)	0.0394 (9)	0.0506 (10)	−0.0089 (8)	−0.0179 (9)	0.0007 (8)
B6	0.0575 (12)	0.0431 (10)	0.0645 (12)	−0.0158 (9)	−0.0070 (10)	0.0033 (9)
B7	0.0608 (12)	0.0420 (9)	0.0505 (10)	−0.0138 (8)	−0.0177 (9)	−0.0041 (8)
B8	0.0461 (11)	0.0437 (10)	0.0675 (12)	−0.0125 (8)	−0.0165 (9)	0.0008 (8)
B9	0.0509 (11)	0.0395 (9)	0.0523 (10)	−0.0063 (8)	−0.0056 (9)	−0.0030 (8)
B10	0.0515 (10)	0.0353 (8)	0.0540 (10)	−0.0106 (8)	−0.0171 (8)	−0.0035 (7)
B11	0.0454 (10)	0.0439 (9)	0.0554 (11)	−0.0124 (8)	−0.0055 (8)	−0.0056 (8)
B12	0.0453 (10)	0.0358 (8)	0.0496 (10)	−0.0092 (7)	−0.0126 (8)	−0.0035 (7)
C2	0.0472 (9)	0.0422 (9)	0.0512 (9)	−0.0130 (7)	−0.0127 (7)	−0.0004 (7)
C3	0.0393 (8)	0.0413 (8)	0.0530 (9)	−0.0125 (7)	−0.0115 (7)	0.0025 (7)
C4	0.0329 (7)	0.0374 (7)	0.0552 (9)	−0.0127 (6)	−0.0097 (6)	0.0057 (7)
C5	0.0447 (9)	0.0457 (9)	0.0557 (10)	−0.0120 (7)	−0.0045 (7)	0.0034 (7)
C6	0.0558 (11)	0.0453 (9)	0.0826 (13)	−0.0113 (8)	−0.0001 (10)	−0.0065 (9)
C7	0.0557 (11)	0.0400 (9)	0.0959 (15)	−0.0129 (8)	−0.0150 (10)	0.0160 (10)
C8	0.0676 (12)	0.0563 (11)	0.0693 (12)	−0.0228 (9)	−0.0242 (10)	0.0227 (10)
C9	0.0554 (10)	0.0491 (9)	0.0581 (10)	−0.0189 (8)	−0.0156 (8)	0.0055 (8)

Geometric parameters (Å, °)

N1—C13	1.511 (2)	B3—H3	1.106 (17)
N1—C17	1.5173 (19)	B4—B5	1.767 (3)
N1—C11	1.519 (2)	B4—B10	1.768 (2)
N1—C15	1.521 (2)	B4—B11	1.772 (3)
C11—C12	1.498 (2)	B4—H4	1.112 (17)
C11—H11A	0.991 (19)	B5—B10	1.764 (2)
C11—H11B	0.996 (19)	B5—B9	1.768 (3)
C12—H12A	0.9600	B5—B6	1.771 (3)
C12—H12B	0.9600	B5—H5	1.101 (16)
C12—H12C	0.9600	B6—B8	1.771 (3)
C13—C14	1.509 (3)	B6—B9	1.771 (3)
C13—H13A	1.000 (19)	B6—H6	1.117 (17)
C13—H13B	0.973 (19)	B7—B12	1.773 (2)
C14—H14A	0.9600	B7—B11	1.777 (3)
C14—H14B	0.9600	B7—B8	1.778 (3)
C14—H14C	0.9600	B7—H7	1.087 (15)
C15—C16	1.501 (3)	B8—B12	1.779 (2)
C15—H15A	1.041 (19)	B8—B9	1.791 (3)
C15—H15B	0.98 (2)	B8—H8	1.127 (17)
C16—H16A	0.9600	B9—B10	1.778 (3)
C16—H16B	0.9600	B9—B12	1.782 (3)
C16—H16C	0.9600	B9—H9	1.118 (16)
C17—C18	1.497 (2)	B10—B12	1.777 (2)
C17—H17A	0.987 (17)	B10—B11	1.778 (3)

C17—H17B	0.942 (17)	B10—H10	1.101 (15)
C18—H18A	0.9600	B11—B12	1.785 (3)
C18—H18B	0.9600	B11—H11	1.092 (16)
C18—H18C	0.9600	B12—C2	1.548 (2)
C1—B5	1.692 (2)	C2—C3	1.202 (2)
C1—B4	1.698 (3)	C3—C4	1.4393 (19)
C1—B2	1.698 (2)	C4—C5	1.389 (2)
C1—B3	1.699 (3)	C4—C9	1.389 (2)
C1—B6	1.703 (3)	C5—C6	1.385 (2)
C1—H1	1.010 (17)	C5—H5A	0.956 (16)
B2—B8	1.762 (3)	C6—C7	1.373 (3)
B2—B7	1.767 (3)	C6—H6A	0.993 (19)
B2—B3	1.767 (3)	C7—C8	1.366 (3)
B2—B6	1.773 (3)	C7—H7A	0.98 (2)
B2—H2	1.118 (16)	C8—C9	1.383 (2)
B3—B7	1.767 (3)	C8—H8A	0.973 (18)
B3—B11	1.769 (3)	C9—H9A	0.990 (17)
B3—B4	1.775 (3)		
C13—N1—C17	106.34 (12)	C3—C2—B12	177.15 (16)
C13—N1—C11	110.83 (14)	C2—C3—C4	176.64 (17)
C17—N1—C11	110.72 (13)	C5—C4—C9	118.84 (14)
C13—N1—C15	111.48 (14)	C5—C4—C3	119.59 (14)
C17—N1—C15	111.00 (13)	C9—C4—C3	121.55 (14)
C11—N1—C15	106.54 (13)	C6—C5—C4	120.32 (16)
C12—C11—N1	115.71 (14)	C7—C6—C5	120.04 (19)
C14—C13—N1	115.43 (16)	C8—C7—C6	120.15 (17)
C16—C15—N1	115.55 (16)	C7—C8—C9	120.53 (18)
C18—C17—N1	116.43 (13)	C8—C9—C4	120.11 (18)
C2—C3—C4—C5	8(3)	C5—C6—C7—C8	0.2 (3)
C2—C3—C4—C9	−171 (3)	C6—C7—C8—C9	−0.5 (3)
C9—C4—C5—C6	−0.6 (2)	C7—C8—C9—C4	0.2 (3)
C3—C4—C5—C6	−179.29 (14)	C5—C4—C9—C8	0.3 (2)
C4—C5—C6—C7	0.4 (3)	C3—C4—C9—C8	178.98 (15)

Fig. 1

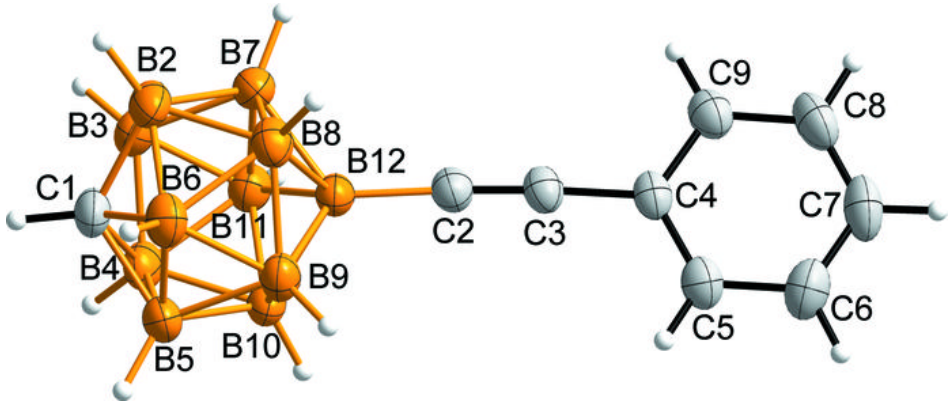

Fig. 2