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Acta Cryst. (1994). **C50**, 2027–2030

1-Diphenylphosphino-2-methyl-1,2-dicarba-*closo*-dodecaborane(12)

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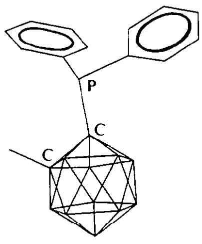
(*Received* 22 *November* 1993; *accepted* 28 *March* 1994)

Abstract

Two C atoms from two phenyl groups and one C atom of the 2-methyl-1,2-dicarba-*closo*-dodecaborane cage in the title compound, $C_{15}H_{23}B_{10}P$, are bonded to the P atom in a pyramidal arrangement. In the carbaborane moiety, the C—C bond length is 1.702 (6) Å; lengthening of this bond is discussed.

Comment

1-Diphenylphosphino-2-methyl-1,2-dicarba-*closo*-dodecaborane (I) is an exceptionally stable compound which has the potential for extensive derivatization, both of the carbaborane polyhedron and of the phosphine moiety. The latter may be of interest in the search for new compounds of phosphines.



(I)

Surprisingly, there is no report in the literature on the use of this phosphine nor of similar compounds containing the *nido* derivative $[7\text{-}(PPh_2)\text{-}8\text{-}Me\text{-}nido\text{-}C_2B_9H_{10}]^-$. We have studied several thioxaether macrocycles of different ring sizes (9, 12 and 13 atoms) containing as a ring component the *closo*-borane moiety with sulfur, silicon or carbon directly bonded to the cluster C atoms. As a general trend, in the compounds substituted with sulfur the cluster C(1)—C(2) distance is longer than in the unsubstituted compounds or in compounds substituted with silicon or carbon. In the compounds containing two S atoms (Table 1), the C(1)—C(2) distance is 1.858 (5) and 1.826 (5) Å (Teixidor, Viñas, Rius, Miravitlles & Casabó, 1990) and 1.816 (6) Å (Teixidor, Romerosa, Rius, Miravitlles, Casabó, Viñas & Sanchez, 1990), whereas in 9,12-isopropylidenedithio-1,2-dicarba-*closo*-dodecaborane(12), a compound with unsubstituted cluster C atoms, it is only 1.634 (3) Å (Šubrtová, Línek & Hašek, 1980). Similarly, when the cluster C atoms are connected directly to silicon the C(1)—C(2) distance is 1.688 (5) Å (Kivekäs, Romerosa & Viñas, 1994), and when connected to carbon, 1.684 (6) Å (Holbrey, Iveson, Lockhart, Tomkinson, Teixidor, Romerosa, Viñas & Rius, 1993).

Apart from the crowding effects noted by Lewis & Welch (1993), another reason for C(cage)—C(cage) bond lengthening could be the partial overlap of the tangentially oriented *p* atomic orbitals (AO's) of the cluster CH unit with appropriate-symmetry AO's of the exocluster atom connected to the cluster C atom. Such an explanation is indicated schematically for

Acta Crystallographica Section C
ISSN 0108-2701 © 1994

aryl and S-atom substitution in Fig. 1. In the first case, partial overlap of a tangentially oriented *p* AO with an aromatic p_π orbital is invoked; in the second case, partial overlap with a *d* orbital of appropriate symmetry and energy is invoked. These interactions weaken the C—C bond and increase the bond length. When such overlap is not possible, as for Si and sp^3 C atoms, the C—C distance should not be affected by electronic considerations. If the C—C distance is increased, intramolecular crowding effects probably are responsible instead.

In the title compound, two C atoms from two phenyl groups and the C atom C(1) of the 2-methyl-1,2-dicarba-*closo*-dodecaborane moiety are bonded to the P atom in a pyramidal arrangement. The P—C(cage) bond is significantly longer than the P—C(aryl) bonds, and the C—P—C angles vary from 102.9 (2) to 105.1 (2)°.

The angles around the phenyl C atoms C(14) and C(20) are not equivalent and vary from 118.5 (4) to 126.5 (3)° for C(14) and from 114.1 (3) to 128.2 (4)° for C(20). The opening of the P—C(14)—C(15) and P—C(20)—C(25) angles can be assumed to result from the mutual repulsion between the two phenyl rings.

In the borane polyhedron, the C(1)—B bond lengths vary from 1.700 (7) to 1.745 (7) Å and the P—C(1)—*X* angles from 112.0 (3) to 129.0 (3)°. The largest angle is P—C(1)—B(5). The orientation of the phenyl groups results in shortest C(14)···B and C(20)···B contacts to B(6) and B(4), respectively. Thus the observed movement of the P atom from its expected position and the considerable opening of the P—C(1)—B(5) angle can be assumed to result from the need to avoid repulsion between the phenyl groups and the borane polyhedron. There is no indication of a C(13)—H···P interaction.

As a rule, C(cage)—C(cage) bond lengths in 7,8-dicarba-*nido*-undecaborate $[C_2B_9H_{12}]^-$ species differ considerably from those in 1,2-dicarba-*closo*-dodecaborane species. Short C(cage)—C(cage) bonds of 1.547 (8) (Kivekäs, Sillanpää, Teixidor, Viñas & Ayllon, 1994) and 1.555 (11) Å (Teixidor, Casabó, Viñas, Sanchez, Escriche & Kivekäs, 1991) have been reported recently for *nido*-carbaborane compounds with short and clearly strained C,C′-connected external S—CH$_2$—S′ and S—CH$_2$—CH$_2$—S′ strings, respectively. A short C(cage)—C(cage) bond of 1.542 (4) Å has also been reported for [7,8-*nido*-$C_2B_9H_{12}]^-$ (Buchanan, Hamilton, Reed & Welch, 1990). Not only are the C(cage)—C(cage) bonds in 1,2-*closo*-dicarbaboranes longer than those in the *nido* compounds, but the distances seem to be longer in C(aryl)- than in C(non-aryl)-substituted *closo* species (Lewis & Welch, 1993). Thus, a relatively long average C(cage)—C(cage) distance of 1.727 (6) Å has been reported recently for two crystallographically independent molecules of 1,2-Ph$_2$-1,2-*closo*-$C_2B_{10}H_{10}$, in which some degree of interphenyl intramolecular crowding exists, and in which an electronic contribution probably contributes to the lengthening of the C(1)—C(2) bond (Lewis & Welch, 1993). In the title compound, where non-aryl substituents are attached to the C(cage) atoms, the C(1)—C(2) distance is just significantly shorter than this.

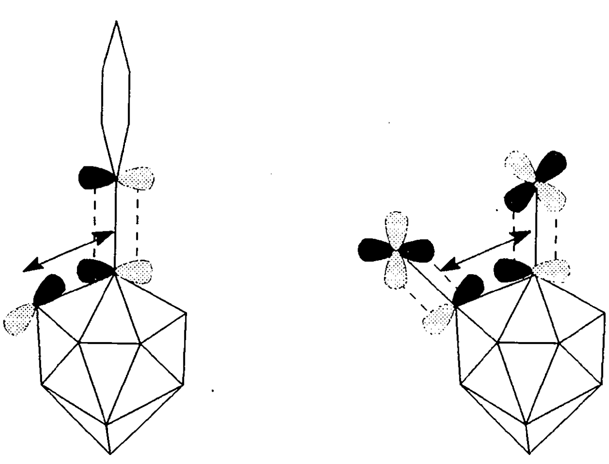


Fig. 1. C_c—C_c lengthening by partial overlap of appropriate atomic orbitals in the cluster and substituents.

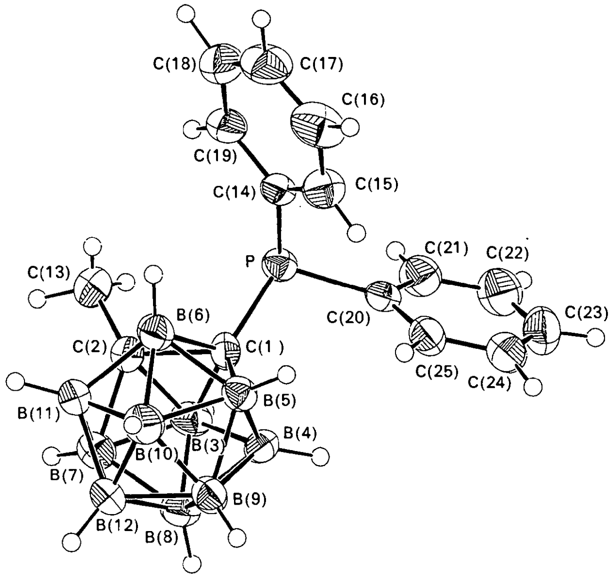


Fig. 2. *ORTEP* (Johnson, 1965) plot of the title compound. Displacement ellipsoids are shown at the 50% probability level, except for H atoms which are drawn as small circles of arbitrary radii.

Experimental

The title compound was first reported about 20 years ago (Zakharkin, Zhubekova & Kazantsev, 1972). The synthetic procedure which we followed included substantial modifications to the original synthesis, which increased the yield. Before use, 1-methyl-*o*-carborane (Dexsil Chemical Corporation) was sublimed under a high vacuum. A 1.6 *M* solution of *n*-butyllithium (Fluka) in hexane was used as purchased. To a three-necked round-bottom flask (250 ml) fitted with a dinitrogen inlet/outlet and containing deoxygenated dry ether (50 ml) was added 1-methyl-*o*-carborane (1.5 g, 9.5 mmol). The mixture was cooled (ice–water) during the addition (10 min) of *n*-butyllithium (6 ml, 9.5 mmol). After stirring for 30 min at the ice–water temperature, the mixture was stirred at room temperature for 30 min, and again cooled to 273 K before the addition of chlorodiphenylphosphine (2.1 g, 9.5 mmol) over a period of 30 min. The ice bath was removed and the mixture was stirred for an extra 30 min at room temperature and under reflux for 2 h. Once cooled, the solvent was evaporated under vacuum. Water (20 ml) was added. Stirring was continued for 10 min, before the two layers were separated. The diethyl ether extract was dried and evaporated under vacuum. The evaporation of the solvent yielded an off-white powder (2.96 g, 91%). Analytically pure crystals of the title compound suitable for X-ray analysis were grown from acetone. Analysis: calculated for $C_{15}H_{23}B_{10}P$, C 52.61, H 6.77; found, C 52.61, H 6.73%. FTIR (KBr): $\nu = 2583\ \text{cm}^{-1}$ (B—H). ^{1}H FT-NMR (400 MHz, $CDCl_3$, 293 K, TMS): $\delta = 2.31$ (*s*, 3, CH_3), 7.18–7.21 (*m*, 5, Ar—H), 7.55–7.60 p.p.m. (*m*, 5, Ar—H). ^{11}B FTNMR (128 MHz, $CHCl_3$, 293 K, $BF_3.Et_2O$): $\delta = -1.49$ [*d*, ^1J(B,H) = 160.9 Hz, 1B], −5.74 [*d*, ^1J(B,H) = 146.7 Hz, 1B], −9.70 p.p.m. [*d*, ^1J(B,H) = 151.9 Hz, 8B]. ^{31}P FTNMR (161 MHz, $CDCl_3$, 293 K, H_3PO_4): $\delta = 11.18$ p.p.m. (*s*, PPh_2). Elemental analyses were performed using a Perkin-Elmer 240-B microanalyser, The ^{1}H NMR, ^{11}B NMR and ^{31}P NMR spectra were obtained using a Bruker AM 400WB or AC 400 instrument, and IR spectra of KBr pellets were recorded using a Nicolet 710-FT spectrophotometer.

Crystal data

$C_{15}H_{23}B_{10}P$
$M_r = 342.4$
Orthorhombic
$Pna2_1$
$a = 11.057$ (3) Å
$b = 12.937$ (2) Å
$c = 13.639$ (4) Å
$V = 1951.0$ (8) Å^3
$Z = 4$
$D_x = 1.166$ Mg m^{-3}

Mo *Kα* radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.2$–16.6°
$\mu = 0.13$ mm^{-1}
$T = 296$ K
Prism
0.40 × 0.38 × 0.38 mm
Colourless

Data collection

Rigaku AFC-5*S* diffractometer
ω–2θ scans
Absorption correction: empirical
$T_{\min} = 0.948$, $T_{\max} = 1.000$
2565 measured reflections
2565 independent reflections
1752 observed reflections
[$|F| > 3\sigma(F)$]

$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 17$
3 standard reflections
monitored every 150 reflections
intensity variation: 0.1%

Refinement

Refinement on *F*
$R = 0.048$
$wR = 0.047$
$S = 1.390$
1752 reflections
326 parameters
All H-atom parameters refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.043$
$\Delta\rho_{\max} = 0.2$ e Å^{-3}
$\Delta\rho_{\min} = -0.2$ e Å^{-3}
Atomic scattering factors from *Xtal*3.0 (Hall & Stewart, 1990)

Table 1. C(1)—C(2) *distance* (Å) *and size of exocyclic C,C′-connected ring for 1,2-dicarba-closo-dodecaborane compounds*

	Ring size	C(1)—C(2)
9,12-μ-[$(CH_3)_2CS_2$]-1,2-$C_2B_{10}H_{10}$[a]	0	1.634 (3)
1,2-μ-($CH_2CH_2SCH_2CH_2OCH_2CH_2SCH_2CH_2$)-$C_2B_{10}H_{10}$[b]	13	1.684 (6)
1,1′-$Si(CH_3)_2$-2,2′-$Si(CH_3)_2$-1,2-$C_2B_{10}H_{10}$[c]	6	1.688 (5)
1-$P(C_6H_5)_2$-2-CH_3-1,2-$C_2B_{10}H_{10}$[d]	0	1.702 (6)
1,2-$(C_6H_5)_2$-1,2-$C_2B_{10}H_{10}$[e]	0	1.733 (4), 1.720 (4)
1,2-μ-S($CH_2CH_2OCH_2CH_2$)S-$C_2B_{10}H_{10}$[f]	9	1.816 (6)
1,2-μ-S($CH_2CH_2OCH_2CH_2OCH_2CH_2$)S-$C_2B_{10}H_{10}$[g]	12	1.858 (5), 1.826 (5)

References: (*a*) Šubrtová, Línek & Hašek (1980); (*b*) Holbrey, Iveson, Lockhart, Tomkinson, Teixidor, Romerosa, Viñas & Rius (1993); (*c*) Kivekäs, Romerosa & Viñas (1994); (*d*) this work; (*e*) Lewis & Welch (1993); (*f*) Teixidor, Romerosa, Rius, Miravitlles, Casabó, Viñas & Sanchez (1990); (*g*) Teixidor, Viñas, Rius, Miravitlles & Casabó (1990).

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

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

	x	*y*	*z*	U_{eq}
P	0.16126 (9)	0.38418 (7)	0.3000	0.0352 (3)
C(1)	0.0879 (3)	0.5047 (3)	0.3498 (3)	0.034 (1)
C(2)	−0.0656 (4)	0.5029 (3)	0.3414 (4)	0.042 (1)
B(3)	0.0147 (5)	0.5752 (4)	0.2582 (4)	0.043 (2)
B(4)	0.1387 (4)	0.6267 (4)	0.3248 (4)	0.038 (2)
B(5)	0.1306 (4)	0.5793 (4)	0.4465 (4)	0.037 (1)
B(6)	0.0023 (5)	0.4968 (4)	0.4542 (5)	0.045 (2)
B(7)	−0.1204 (5)	0.6221 (4)	0.3104 (6)	0.053 (2)
B(8)	0.0079 (4)	0.7030 (4)	0.3022 (6)	0.046 (2)
B(9)	0.0781 (5)	0.7065 (4)	0.4183 (5)	0.043 (2)
B(10)	−0.0059 (5)	0.6251 (4)	0.4994 (5)	0.051 (2)
B(11)	−0.1276 (5)	0.5742 (4)	0.4327 (5)	0.050 (2)
B(12)	−0.0807 (5)	0.7028 (4)	0.4112 (5)	0.049 (2)
C(13)	−0.1281 (5)	0.4066 (4)	0.3039 (8)	0.062 (2)
C(14)	0.1831 (4)	0.3052 (3)	0.4098 (3)	0.039 (1)
C(15)	0.2693 (5)	0.3197 (4)	0.4812 (4)	0.050 (2)
C(16)	0.2779 (6)	0.2515 (5)	0.5606 (5)	0.068 (2)
C(17)	0.1994 (6)	0.1695 (4)	0.5673 (4)	0.066 (2)
C(18)	0.1142 (5)	0.1537 (4)	0.4961 (4)	0.060 (2)
C(19)	0.1048 (4)	0.2209 (3)	0.4184 (4)	0.049 (2)
C(20)	0.3141 (4)	0.4238 (3)	0.2643 (3)	0.039 (1)
C(21)	0.3532 (5)	0.3821 (4)	0.1765 (4)	0.052 (2)
C(22)	0.4692 (5)	0.3999 (5)	0.1418 (5)	0.065 (2)
C(23)	0.5452 (5)	0.4621 (5)	0.1930 (5)	0.062 (2)
C(24)	0.5100 (5)	0.5057 (4)	0.2803 (5)	0.058 (2)
C(25)	0.3942 (4)	0.4873 (3)	0.3153 (4)	0.046 (2)

Table 3. *Selected geometric parameters* (Å, °)

P—C(1)	1.884 (4)	B(4)—B(8)	1.779 (7)
P—C(14)	1.829 (4)	B(4)—B(9)	1.772 (8)
P—C(20)	1.832 (4)	B(5)—B(6)	1.778 (8)
C(1)—C(2)	1.702 (6)	B(5)—B(9)	1.787 (7)
C(1)—B(3)	1.745 (7)	B(5)—B(10)	1.775 (8)
C(1)—B(4)	1.709 (6)	B(6)—B(10)	1.772 (8)
C(1)—B(5)	1.700 (7)	B(6)—B(11)	1.775 (8)
C(1)—B(6)	1.714 (8)	B(7)—B(8)	1.766 (7)
C(2)—B(3)	1.718 (7)	B(7)—B(11)	1.78 (1)

C(2)—B(6)	1.715 (8)	B(7)—B(12)	1.78 (1)
C(2)—B(7)	1.711 (7)	B(8)—B(9)	1.76 (1)
C(2)—B(11)	1.696 (8)	B(8)—B(12)	1.78 (1)
C(2)—C(13)	1.513 (8)	B(9)—B(10)	1.788 (8)
B(3)—B(4)	1.775 (7)	B(9)—B(12)	1.759 (7)
B(3)—B(7)	1.762 (8)	B(10)—B(11)	1.752 (8)
B(3)—B(8)	1.760 (7)	B(10)—B(12)	1.772 (9)
B(4)—B(5)	1.771 (7)	B(11)—B(12)	1.767 (7)

C(1)—P—C(14)	102.9 (2)	B(6)—C(2)—C(13)	117.7 (5)
C(1)—P—C(20)	105.1 (2)	B(7)—C(2)—C(13)	119.8 (4)
C(14)—P—C(20)	104.6 (2)	B(11)—C(2)—C(13)	120.8 (4)
P—C(1)—C(2)	113.1 (3)	P—C(14)—C(15)	126.5 (3)
P—C(1)—B(3)	112.0 (3)	P—C(14)—C(19)	115.0 (4)
P—C(1)—B(4)	123.4 (3)	C(15)—C(14)—C(19)	118.5 (4)
P—C(1)—B(5)	129.0 (3)	P—C(20)—C(21)	114.1 (3)
P—C(1)—B(6)	119.2 (3)	P—C(20)—C(25)	128.2 (4)
C(1)—C(2)—C(13)	119.3 (4)	C(21)—C(20)—C(25)	117.6 (4)
B(3)—C(2)—C(13)	117.5 (5)		

P—C(1)—C(2)—C(13)	−4.4 (7)	C(20)—P—C(1)—B(4)	−19.7 (4)
C(14)—P—C(1)—C(2)	95.8 (3)	C(20)—P—C(1)—B(5)	60.2 (4)
C(14)—P—C(1)—B(5)	−49.1 (4)	C(20)—P—C(14)—C(15)	−34.7 (5)
C(14)—P—C(1)—B(6)	28.1 (4)	C(14)—P—C(20)—C(25)	65.2 (4)
C(20)—P—C(1)—C(2)	−154.9 (3)		

Cell refinement, data collection and data reduction: Rigaku AFC-5*S* software. Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *Xtal*3.0 (Hall & Stewart, 1990). Refinement of the enantiomeric model did not change the *R* factors. Molecular graphics: *ORTEP* (Johnson, 1965). Programs used to prepare material for publication: *BONDLA* and *ATABLE* from *Xtal*3.0.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 2030–2032

3-Methyl-5-(2,6,6-trimethyl-1-cyclohexenyl)-2(5*H*)-furanone

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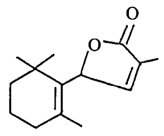
(*Received* 2 *September* 1993; *accepted* 30 *March* 1994)

Abstract

The title compound, $C_{14}H_{20}O_2$, can be regarded as a synthetic derivative resembling tobacco isoprenoids and is structurally related to primary precursor α- and β-ionones. The double bond distance C(3)═C(4) in the furanone ring is 1.329 (8) Å and the double bond distance C(1′)═C(2′) in the cyclohexene ring is 1.331 (6) Å. The bond between the rings, C(5)—C(2′), is 1.513 (7) Å and the bond torsion angle O(1)—C(5)—C(2′)—C(1′), which describes the relative twist of the rings, is ±51.3 (7)°.

Comment

The title compound (I) was isolated as a side product in phase-transfer dihalocarbene addition to β-ionone and was a result of chemoselective adduct formation from a dichloro ether intermediate hydrolysed to the γ lactone (Díaz, Alvarez, Toscano, Shoolery & Jankowski, 1990). A structure determination was undertaken in order to confirm the identity of the compound and establish the relative positions of the rings.



(I)

The cyclohexene ring adopts a half-chair conformation [Cremer & Pople (1975) parameters: $Q = 0.408$ (7) Å, $\theta = 51.7$ (8)° and $\varphi = -151$ (1)°] while the methyl benzofuranone ring is almost planar. The

Acta Crystallographica Section C
ISSN 0108-2701 ©1994