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7-{Diphenyl[(triphenylphosphine)aurio]phosphine(1+)-*P*}-8-methyl-7,8-dicarba-*nido*-undecaborate(1−) dichloromethane hemisolvate

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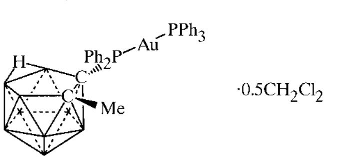
The title compound, 7-[(Ph_2P)Au(PPh_3)]-8-(CH_3)-7,8-*nido*-$C_2B_9H_{10}$]·0.5CH_2Cl_2 or [Au($C_{15}H_{23}B_9P$)($C_{18}H_{15}P$)]·0.5-CH_2Cl_2, is the first reported gold derivative of the ligand [7-(Ph_2P)-8-(CH_3)-7,8-*nido*-$C_2B_9H_{10}$]$^-$. It has a mononuclear structure with the gold centre in an essentially linear coordination [P—Au—P 174.041 (15)°]. The open C_2B_3 face contains one H atom that is strongly bonded to the central B atom and semi-bridging to a neighbouring B atom [B—H distances 1.070 (16) and 1.45 (3) Å].

Comment

We are interested in *closo*- and *nido*-carborane derivatives, and have reported several complexes in which a gold moiety is coordinated to the anionic *nido*-diphosphine ligand 7,8-bis(diphenylphosphino)-7,8-dicarba-*nido*-undecaborate(1−) (Crespo, Gimeno, Laguna & Jones, 1996; Crespo *et al.*, 1997, 1998). We report here the structure of the neutral title complex 7-{diphenyl[(triphenylphosphine)aurio]phosphine(1+)-*P*}-8-methyl-7,8-dicarba-*nido*-undecaborate(1−) dichloromethane hemisolvate, (I). The synthetic reaction (see *Experimental*) results in the coordination of an {Au(PPh_3)} fragment and partial degradation of the icosahedral carborane phosphine 1-(Ph_2P)-2-(CH_3)-1,2-*closo*-$C_2B_{10}H_{10}$. The product is the first reported gold derivative of the parent carbaborylphosphine and one of the few metallorganic derivatives described with the *nido*-phosphine [7-(Ph_2P)-8-(CH_3)-7,8-*nido*-$C_2B_9H_{10}$]$^-$ (*cf.* Viñas *et al.*, 1996).



(I)

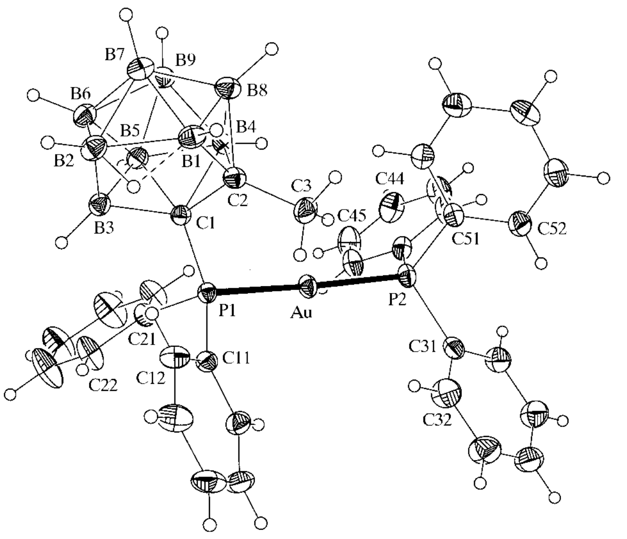


Figure 1
The molecular structure of the title compound (solvent omitted). Ellipsoids are drawn at the 50% probability level and H-atom radii are arbitrary. The semi-bridging H02*B*—B1 interaction is indicated by a dashed line (see text).

The structural study of (I) (Fig. 1) reveals for the C_2B_9 cage a geometry corresponding to an icosahedron from which one vertex has been removed. The C1—C2 distance of 1.588 (2) Å is shorter than that found in the corresponding *closo*-phosphine 1-(Ph_2P)-2-(CH_3)-1,2-*closo*-$C_2B_{10}H_{10}$ [1.702 (6) Å; Kivekäs *et al.*, 1994], but very similar to those observed in the various ruthenium complexes, which range from 1.538 (9) Å in [Ru{7-(PPh_2)-8-(CH_3)-7,8-$C_2B_9H_{10}$}$_2$] (Viñas *et al.*, 1995) to 1.561 (8) Å in [RuCl{7-(PPh_2)-8-(CH_3)-7,8-$C_2B_9H_{10}$}(PPh_3)$_2$] (Viñas *et al.*, 1996). The P1—Au—P2 fragment shows the linearity usually associated with two-coordinate gold(I) complexes, with Au—P distances of 2.3060 (5) and 2.3159 (5) Å, and a P—Au—P angle of 174.041 (15)°. A search of the Cambridge Structural Database (Allen & Kennard, 1993) for neutral molecules containing the P—Au—P group yielded 12 hits with a mean Au—P bond length of 2.317 Å.

The 'extra' H atom (H02*B*) in the pentagonal C_2B_3 open face (atoms C1, C2, B1, B2 and B3; B2 bears two H atoms) is strongly bonded to the central B atom [B2—H02*B* 1.070 (16) Å], but can be considered as semi-bridging to B1 [B1—H02*B* 1.45 (3) Å]. This distance is similar to those in related derivatives such as [AuCl$_2${(PPh_2)$_2$$C_2B_9H_{10}$}] (1.37 Å; Jones *et al.*, 1997), [Au{(PPh_2)$_2$$C_2B_9H_{10}$}{($PPh_2$)$_2$$C_2B_{10}H_{10}$}] (1.35 Å; Crespo *et al.*, 1997) or [Au_2($C_{26}H_{30}B_9C_2$)(C_6F_5)]·CH_2Cl_2 [1.50 (8) Å; Crespo *et al.*, 1998]. We attributed the imprecision of these values to the presence of heavy atoms; the current structure is appreciably more precise despite the presence of gold. We have observed a similar arrangement of H atoms in silver complexes of the open-face carborane ligand (Crespo, Gimeno, Jones & Laguna, 1996) and in the light-atom derivative [$C_2B_9H_{10}$(SC_5H_4NH)$_2$)](CF_3SO_3), in which the H atoms were freely refined, clearly showing the semi-bridging nature of this H atom [B—H distances 1.12 (3) and 1.39 (3) Å; Jones *et al.*, 1997].

Experimental

To a suspension of 1-(Ph_2P)-2-(CH_3)-1,2-*nido*-$C_2B_{10}H_{10}$ (0.034 g, 0.1 mmol; Kivekäs *et al.*, 1994) in ethanol (30 ml), [Au(PPh_3)(SC_4H_8)]ClO_4 (0.096 g, 0.15 mmol) was added. The mixture was refluxed for 30 min and the complex (a white solid) filtered off and washed with ethanol (10 ml) (yield: 72%). Analysis calculated for $C_{33}H_{38}AuB_9P_2$: C 50.05, H 4.08%; found: C 49.65, H 4.6%. NMR data: $^{31}P\{^1H\}$ (*AB* system) 17.21 p.p.m., $J = 327.5$ Hz; ^1H −2.25 (*m*, *br*, 1H), 1.41 (*s*, 3H, CH_3), 6.7–7.8 (*m*, *br*, 15H, C_6H_5) p.p.m. Single crystals were obtained from an *n*-heptane–dichloromethane solution.

Crystal data

[Au($C_{15}H_{23}B_9P$)($C_{18}H_{15}P$)]·0.5CH_2Cl_2
$M_r = 833.29$
Triclinic, $P\bar{1}$
$a = 11.3770$ (8) Å
$b = 12.5180$ (10) Å
$c = 15.4837$ (12) Å
$\alpha = 69.925$ (3)°
$\beta = 72.338$ (3)°
$\gamma = 66.953$ (3)°
$V = 1869.1$ (2) Å^3
$Z = 2$
$D_x = 1.481$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 8011 reflections
$\theta = 2$–28°
$\mu = 4.116$ mm^{-1}
$T = 143$ (2) K
Irregular wedge, colourless
0.29 × 0.22 × 0.16 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.400$, $T_{max} = 0.518$
22 230 measured reflections
10 779 independent reflections
9788 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 30°$
$h = -15 \rightarrow 16$
$k = -17 \rightarrow 17$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.048$
$S = 0.990$
10 779 reflections
445 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.027$
$\Delta\rho_{max} = 1.07$ e Å^{-3}
$\Delta\rho_{min} = -0.81$ e Å^{-3}

The dichloromethane solvate molecule is disordered over an inversion centre; C—Cl bond lengths were restrained to be equal. The high displacement parameters of this molecule may indicate partial occupation. All non-H atoms, except solvent C atoms, were refined anisotropically. H atoms of the carbaborane open face were located in difference syntheses and refined freely, but with B—H distances restrained to be equal. The methyl H atoms were identified from difference syntheses, idealized and refined as a rigid group allowed to rotate but not tip. Other H atoms were included using a riding model. A total of 433 restraints were applied, involving local phenyl-ring symmetry and light-atom displacement parameters, in addition to the B—H restraints. The largest feature of residual electron density (1.07 e Å^{-3}) is 0.79 Å from the Au atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

Table 1
Selected geometric parameters (Å, °).

Au—P2	2.3060 (5)	C1—C2	1.588 (2)
Au—P1	2.3159 (5)	C2—C3	1.510 (2)
P1—C11	1.8072 (18)	B1—H01	1.056 (15)
P1—C21	1.8158 (18)	B2—H02*A*	1.070 (15)
P1—C1	1.8197 (17)	B2—H02*B*	1.070 (16)
P2—C41	1.808 (2)	B3—H03	1.074 (15)
P2—C31	1.8130 (19)	B1—H02*B*	1.45 (3)
P2—C51	1.8153 (18)		
P2—Au—P1	174.041 (15)	C41—P2—C51	105.28 (9)
C11—P1—C21	104.95 (8)	C31—P2—C51	106.42 (8)
C11—P1—C1	106.93 (8)	C41—P2—Au	113.87 (6)
C21—P1—C1	108.63 (8)	C31—P2—Au	113.12 (6)
C11—P1—Au	110.30 (6)	C51—P2—Au	108.33 (6)
C21—P1—Au	111.18 (6)	H02*A*—B2—H02*B*	110.2 (18)
C1—P1—Au	114.35 (6)	B2—H02*B*—B1	93.7 (17)
C41—P2—C31	109.24 (9)		

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1352). Services for accessing these data are described at the back of the journal.

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