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[2-(1*H*-Inden-3-yl)ethyl]diphenylphosphine–borane

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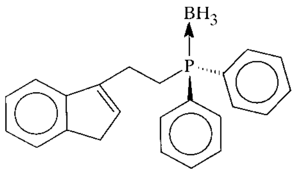
The title compound, $C_{23}H_{24}BP$, crystallizes in the centrosymmetric space group $P2_1/c$, with one molecule in the asymmetric unit. The indene moiety is essentially planar. The P—B bond length is 1.923 (3) Å, which is within the expected range.

Comment

The title compound, (I), is a protected bidentate phosphino-indenyl ligand synthesized during the course of our studies on transition metal complexes bearing chelating indenyl ligands. We studied this compound in order to compare its strucural parameters with those found in complexes wherein the indenyl moiety is coordinated to a transition metal. The borane protects the phosphine against oxidation.

N-[2-(1*H*-Inden-3-yl)ethyl]-*p*-toluenesulfonamide (Gainsford & Lensink, 1996) and *N*-[3-(inden-3-yl)propyl]benzyl-ammonium bromide (Groux *et al.*, 1999) present virtually the same structural parameters.



(I)

Experimental

IndH-$(CH_2)_2PPh_2$ was prepared following Kauffmann's procedure (Kauffmann *et al.*, 1985). BH_3·THF (6.1 ml of a 1 *M* solution in THF, 6.1 mmol) was syringed onto a 60 ml solution of the ligand (1.33 g, 4.05 mmol) in THF and stirred for 1 h at room temperature. The reaction mixture containing a white precipitate was evaporated to dryness. The crude material was purified by flash chromatography (silica gel, 1:3 ethyl acetate/hexane). White crystals were obtained after recrystallization from ethyl acetate/hexane (1:1). The compound was characterized by NMR spectroscopy and elemental analysis. ^{31}P {^1H} ($CDCl_3$): 16.6; ^1H ($CDCl_3$): 7.7, 7.5 and 7.2 (*m*, aromatic protons of indene and phenyls), 6.25 (*s*, H2), 3.29 (*s*, H3), 2.78 and 2.60 (*m*, CH_2–CH_2), 1.15 (*br*, BH_3); ^{13}C {^1H} ($CDCl_3$): 144.5 and 143.6 (C3*A* and C7*A*), 132.3, 131.4, 129.4, 129.0, 128.6, 124.5, 118.9 (C2), 24.6 (*d*, CH_2P), 21.5 (Ind-CH_2). Analysis calculated for $C_{23}H_{24}BP$: C 80.7, H 7.1%; found: C 80.6, H 7.3%.

Crystal data

$C_{23}H_{24}BP$
M_r = 342.20
Monoclinic, $P2_1/c$
a = 14.061 (5) Å
b = 6.644 (3) Å
c = 21.018 (10) Å
β = 96.82 (3)°
V = 1949.6 (15) $Å^3$
Z = 4
D_x = 1.166 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20–23°
μ = 1.232 mm^{-1}
T = 293 (2) K
Plate, white
0.88 × 0.12 × 0.02 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration (*ABSORP* in *NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.695, T_{max} = 0.978
30 965 measured reflections
3689 independent reflections
1679 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 69.83°
$h = -17 \rightarrow 17$
$k = -8 \rightarrow 8$
$l = -25 \rightarrow 25$
5 standard reflections
frequency: 60 min
intensity decay: no decay or variation

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.067
S = 0.853
3689 reflections
228 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0184P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.20 e $Å^{-3}$
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.00064 (6)

Table 1
Selected geometric parameters (Å, °).

P—C9	1.801 (2)	P—C11	1.810 (3)
P—C21	1.802 (2)	P—B	1.923 (3)
C9—P—C21	108.69 (12)	C8—C9—P	111.82 (18)
C9—P—C11	105.85 (12)	C16—C11—P	119.6 (2)
C21—P—C11	104.86 (12)	C12—C11—P	122.6 (2)
C9—P—B	112.41 (13)	C22—C21—P	119.9 (2)
C21—P—B	112.24 (13)	C26—C21—P	122.6 (2)
C11—P—B	112.32 (14)		
C7*A*—C1—C2—C3	−0.4 (3)	P—C11—C12—C13	−179.6 (2)
C1—C8—C9—P	165.44 (18)	P—C11—C16—C15	178.8 (2)
C21—P—C9—C8	−177.69 (18)	C9—P—C21—C22	−173.8 (2)
C11—P—C9—C8	−65.5 (2)	C11—P—C21—C22	73.4 (3)
B—P—C9—C8	57.4 (2)	B—P—C21—C22	−48.8 (3)
C9—P—C11—C16	146.3 (2)	C9—P—C21—C26	11.7 (3)
C21—P—C11—C16	−98.9 (2)	C11—P—C21—C26	−101.2 (2)
B—P—C11—C16	23.3 (3)	B—P—C21—C26	136.6 (2)
C9—P—C11—C12	−35.3 (2)	P—C21—C22—C23	−175.6 (2)
C21—P—C11—C12	79.5 (2)	P—C21—C26—C25	175.2 (2)
B—P—C11—C12	−158.3 (2)		

The space group was confirmed by *PLATON* (Spek, 1995). H atoms were constrained to the parent site using a riding model; *SHELXL*96 defaults, C—H = 0.93–0.97 and B—H = 1.10 Å. The isotropic displacement parameters, U_{iso}, were adjusted to a value 50% higher than those of the parent sites for B—H and 20% higher for the other H atoms. A final verification of possible voids was performed using the *VOID* routine of *PLATON*.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: locally modified *NRC*-2 and *NRC*-2*A* (Ahmed *et al.*, 1973); program(s) used to solve structure: *SHELXS*93 (Sheldrick, 1990); program(s) used to refine structure: *NRCVAX* (Gabe *et al.*, 1989) and *SHELXL*93 (Sheldrick, 1993); software used to prepare material for publication: *NRCVAX* and *SHELXL*93.

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