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N-Benzylidene-3-(diphenylphosphino)-1-propanamine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.099
Data-to-parameter ratio = 14.8

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

The title compound, $C_{22}H_{22}NP$, a kind of Schiff base, which contains soft donor phosphines and hard donor nitrogen, has been synthesized. The C═N double bond has length 1.253 (2) Å. There is a zigzag chain with a diphenylphosphine and a benzene ring.

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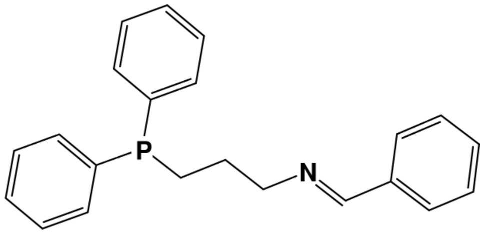
Comment

Phosphines are among the most common ancillary ligands for transition metal complexes with the result that there are many reported systems (Newkome, 1993*a*,*b*; Uriarte *et al.*, 1980). Ligands possessing both softer donor phosphines and harder donor nitrogen or oxygen sites have been studied extensively because they not only stabilize homobimetallic or heterobimetallic complexes but also provide catalyst systems with hemilabile ligands that readily dissociate to provide vacant coordination sites for incoming substrates (Elliott *et al.*, 1999). Issleib first synthesized the $[Ph_2P(CH_2)_3N{=}CH]C_6H_5$ compound and prepared the Ni (Issleib *et al.*, 1978), Pd and Pt (Issleib & Kipke, 1980) complexes with the ligand. Blinn *et al.* (1990) reported a new method to synthesize the ligand, which was obtained by reacting $Ph_2P(CH_2)_3NH_2$, from the reduction of $Ph_2P(CH_2)_3CN$, with benzaldehyde in the presence of molecular sieves. However, they did not report a single-crystal structure. In addition, Hussain *et al.* (1990) and Banbery *et al.* (1991) prepared the ligand $2\text{-}[Ph_2P(CH_2)_3N{=}CH]C_6H_4OH$ and its Re^V complex. As the distance for C16═N1 is 1.256 (3) Å in the title compound, (I), typical for a double bond, this is a novel kind of Schiff base. There is a zigzag chain with a diphenylphosphine and a benzene ring in the molecule.



(I)

Experimental

All commercially available reagents were used as supplied. ^1H NMR spectra were obtained from $CDCl_3$ solution using TMS as internal standard on a Bruker DRX 200 instrument. FT–IR spectra were measured by a Nicolet 1795X FT–IR spectrophotometer. Elemental analyses were performed on a Carlo–Erba 1106 elemental analyser. The compound $[Ph_2P(CH_2)_3N{=}CH]C_6H_5$ was prepared by the

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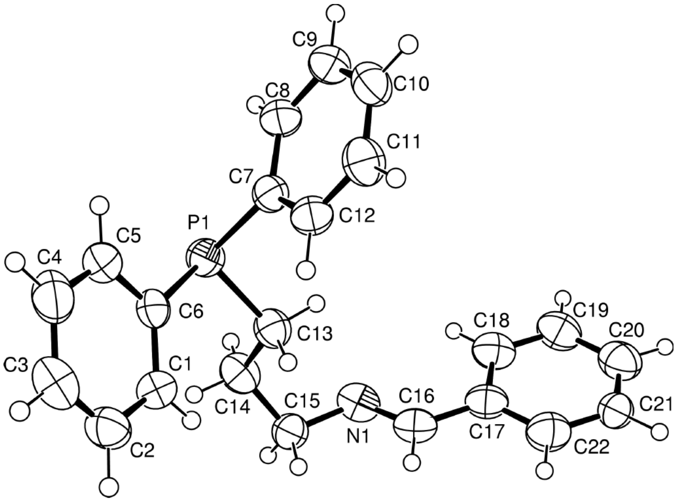

Figure 1
The molecule with labelling of the non-H atoms and 30% probability ellipsoids.

reaction between $Ph_2P(CH_2)_3NH_2$ and benzaldehyde in the presence of molecular sieves in benzene according to previously described procedures (Blinn *et al.*, 1990); m.p. 352–353 K. ^{1}H NMR ($CDCl_3$, 200 MHz, p.p.m.): 8.26 (*s*, 1H, –N═CH), 7.71–7.73 (*m*, 15H, Ph), 3.70 (*t*, 2H, –CH_2—N), 2.10 (*t*, 2H, –P—CH_2), 1.88 (*m*, 2H, –CH_2—CH_2—CH_2). IR (KBr, cm^{-1}): 1574, 1478 (C═C), 1697 (C═N), 1492 (C—P). Analysis calculated: C 79.74, H 6.69, N 4.23%; found: C 79.57, H 6.43, N 4.18%.

Crystal data

$C_{22}H_{22}NP$
$M_r = 331.38$
Orthorhombic, $P2_12_12_1$
$a = 5.768$ (2) Å
$b = 17.702$ (3) Å
$c = 17.823$ (4) Å
$V = 1819.8$ (8) Å^3
$Z = 4$
$D_x = 1.209$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 15.1$–15.9°
$\mu = 0.15$ mm^{-1}
$T = 293$ (2) K
Prismatic, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.946$, $T_{\max} = 0.970$
3202 measured reflections
3202 independent reflections
3014 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25.0°$
$h = -6 \rightarrow 6$
$k = -20 \rightarrow 21$
$l = -20 \rightarrow 21$
5 standard reflections every 300 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.099$
$S = 1.05$
3202 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.35P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.10$ e Å^{-3}
Absolute structure: Flack (1983); 1327 Friedel pairs
Flack parameter = −0.09 (10)

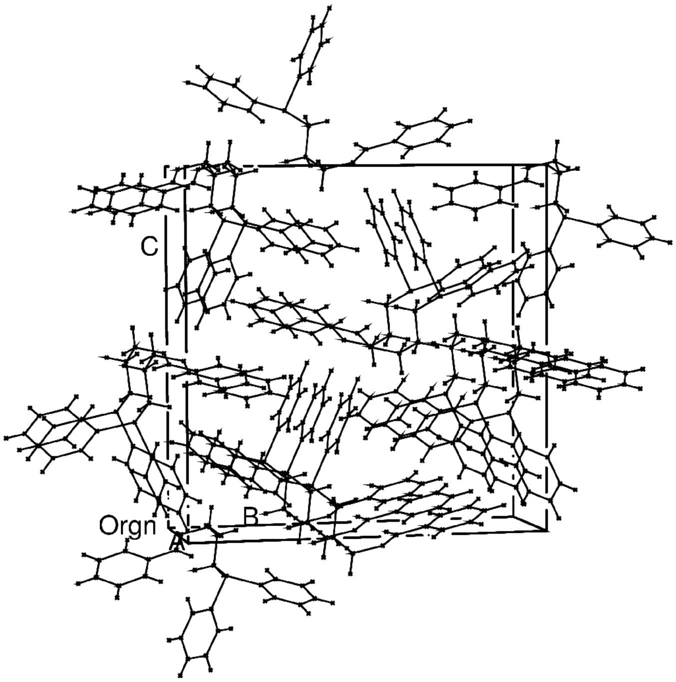

Figure 2
A view of the packing.

The positions of all H atoms were fixed geometrically.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4-*PC Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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