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Diphenyl(3-phenylpropyl)phosphine

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The crystal structure of diphenyl(3-phenylpropyl)phosphine, $C_{21}H_{21}P$, has been determined at 180 (1) K. The molecules may be considered to be linked into chains running along screw diads in space group $P2_1/c$, with molecules adjacent in these chains associated *via* face-to-face and edge-to-face interactions between phenyl rings. Edge-to-face interactions also exist between chains giving rise to centrosymmetric fourfold motifs.

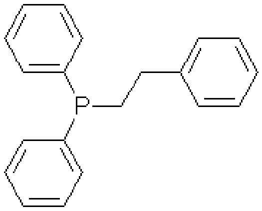
Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.153
Data-to-parameter ratio = 19.0

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

Comment

Diphenyl(3-phenylpropyl)phosphine, (I), is a potentially chelating ligand that has been prepared by several groups for the study of tethered arene–Rh^{I} and Ru^{II} complexes of the type [Rh{η^1-$Ph_2P(CH_2)_3C_6H_5$}{η^1:η^6-$Ph_2P(CH_2)_3C_6H_5$}] (Singewald *et al.*, 1994, 1996) and [$RuCl_2${η^1:η^6-Ph_2P-$(CH_2)_3C_6H_5$}] (Bennett *et al.*, 2001; Ghebreyessus & Nelson, 2000; Smith & Wright, 1998). (I) may coordinate to the metal centre through both the phosphorus donor atom and the alkyl arene. The chelate effect may stabilize oxidation states of the metal that are otherwise unstable by inhibiting decomposition, thought to proceed through loss of the η^6-arene (Bennett & Harper, 2001). We report here the crystal structure of the free phosphine (I), determined at 180 (1) K.



(I)

In the solid state, the molecular conformation of (I) is such that the 3-phenylpropyl chain is extended away from the P atom (Fig. 1). The major directional intermolecular interactions in (I) are between phenyl rings, which adopt both face-to-face and edge-to-face arrangements (Fig. 2). The molecules may be considered to be linked into chains running along the screw diads parallel to the *b* direction. Molecules adjacent within these chains associate *via* face-to-face and edge-to-face interactions between four phenyl rings, two of which are bound directly to the P atom and two of which belong to 3-phenylpropyl chains. This arrangement is reminiscent of the so-called 'phenyl embrace' (Dance & Scudder, 1996). Edge-to-

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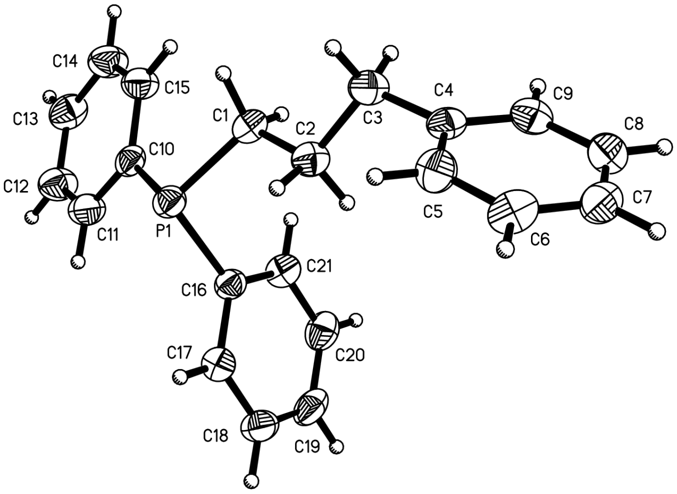

Figure 1
The molecular unit of (I) showing displacement ellipsoids at the 50% probability level (XP; Sheldrick, 1993).

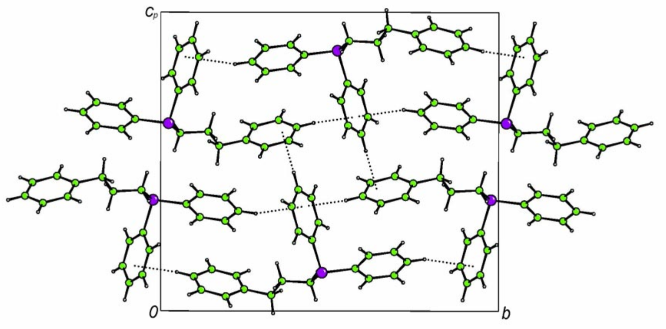

Figure 2
View along the *a* direction showing molecules linked into chains along screw diads by edge-to-face interactions between phenyl rings, denoted by dotted lines (*CAMERON*; Watkin *et al.*, 1996).

face interactions also exist between chains giving rise to centrosymmetric fourfold motifs (Fig. 2).

Experimental

1-Bromo-3-phenylpropane (15.3 ml, 0.10 mol) was added dropwise to a stirred suspension of magnesium (2.55 g, 0.11 mol) in dry THF (30 ml). Dry THF (20 ml) was added and the reaction mixture was heated at reflux for 30 min. The solution was allowed to cool, transferred to a separate flask with dry ether (30 ml), stirred and treated dropwise with chlorodiphenylphosphine (17 ml, 0.095 mol) in dry ether (50 ml) at 273 K. The mixture was heated at reflux for 30 min, cooled to 273 K and treated dropwise with degassed 10% aqueous NH_4Cl (30 ml). The mixture was allowed to come to room temperature, the organic phase removed and the aqueous phase extracted with dry ether (3 × 40 ml). The combined organic phases were dried (Na_2SO_4) and the solvents were removed *in vacuo*. The residue was recrystallized from dry ethanol to afford (I) as a white solid [melting point 329–331 K (23.17 g, 76%)]. Full spectroscopic and analytical data for (I) are included in Bennett *et al.* (2001). Single crystals suitable for X-ray diffraction were obtained from a CH_2Cl_2–toluene solution.

Crystal data

$C_{21}H_{21}P$
$M_r = 304.35$
Monoclinic, $P2_1/c$
$a = 5.7499$ (2) Å
$b = 18.0970$ (11) Å
$c = 16.0028$ (8) Å
$\beta = 91.421$ (3)°
$V = 1664.67$ (14) Å^3
$Z = 4$
$D_x = 1.214$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6675 reflections
$\theta = 1.0$–27.5°
$\mu = 0.16$ mm^{-1}
$T = 180$ (2) K
Block, orange
0.21 × 0.18 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Thin-slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.868$, $T_{\max} = 0.978$
13 665 measured reflections
3785 independent reflections
2390 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -23 \rightarrow 23$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.153$
$S = 0.99$
3785 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.083P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.015$
$\Delta\rho_{\max} = 0.67$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}

H atoms were placed geometrically and refined using a riding model with an isotropic displacement parameter fixed at $1.2U_{\rm eq}$ for the C atom to which they are attached.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; 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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