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N,*N*′-Bis[(diphenylphosphinoyl)methyl]-1,4-phenylenediamine

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.007 Å
R factor = 0.078
wR factor = 0.251
Data-to-parameter ratio = 19.2

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

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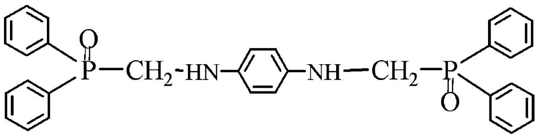
The molecule of the title compound, $C_{32}H_{30}N_2O_2P_2$, an oxidative derivative of *N*,*N*′-bis[(diphenylphosphino)methyl]-1,4-phenylenediamine, occupies a special position on an inversion center. The crystal structure contains N—H···O═P hydrogen bonds, connecting the molecules to form an infinite chain along [100].

Comment

In our studies of the coordination chemistry of diphosphines (Zhang *et al.*, 2002; Song *et al.*, 2002), we have focused our attention on the preparation of functionalized phosphines. In this paper, we present an X-ray crystallographic analysis of the title compound, (I).



(I)

Compound (I) is obtained when *N*,*N*′-bis[(diphenylphosphino)methyl]-1,4-phenylenediamine (Durran *et al.*, 2000) is mixed with $NiCl_2\cdot2H_2O$ in air, the oxidation state of the P atom being converted from III to V. The structure of (I) as determined by X-ray crystallography shows that the asymmetric unit consists of one half-molecule. The molecule (Fig. 1) occupies a special position of $\overline{1}$ site symmetry. In (I), the tertiary phosphine oxide has a distorted tetrahedral geometry with a P═O bond length of 1.483 (3) Å, which is consistent with appreciable double-bond character (Coles *et al.*, 2001).

The primary structural feature in the solid state is that supramolecular aggregation of (I) involves only intermolecular N—H···O═P hydrogen bonds (Table 2; Song *et al.*, 2000). Atom N acts as a hydrogen bond donor to O═P of an adjacent molecule (Fig. 2). The molecules are centrosymmetrically associated *via* a pair of N—H···O═P hydrogen bonds and propagation of these hydrogen bonds generates a chain running parallel to the [100] direction. This hydrogen-bonded structure may be regarded as a molecular ladder. There are neither hydrogen bonds nor aromatic π–π stacking interactions between adjacent chains, so the supramolecular structure is defined by the direction-specific interactions.

Experimental

A solution of $NiCl_2\cdot2H_2O$ (0.082 g, 0.50 mmol) in 15 ml of CH_3OH was added to CH_2Cl_2 (10 ml) containing *N*,*N*′-bis[(diphenylphosphino)methyl]-1,4-phenylenediamine (0.252 g, 0.5 mmol) and

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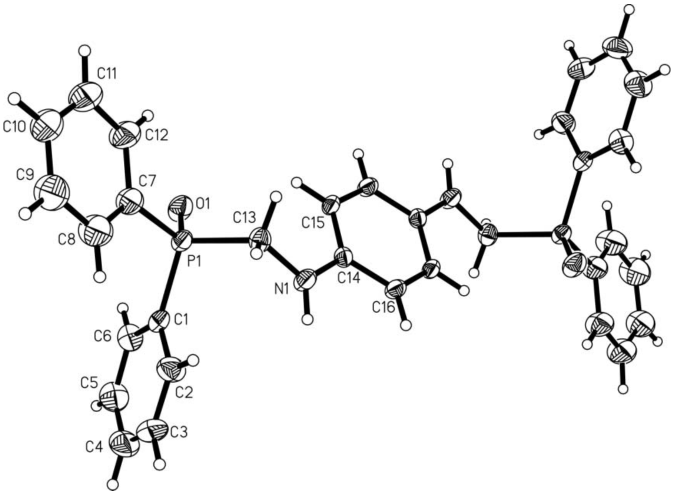

Figure 1

The molecular structure of (I), showing the atomic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radii. Unlabeled atoms are related to labeled atoms by ($1 - x$, $2 - y$, $-z$).

the solution was stirred at room temperature for 4 h. The filtrate was layered with acetone (15 ml). Brown crystals of the title compound suitable for X-ray diffraction analysis were obtained within a week.

Crystal data

$C_{32}H_{30}N_2O_2P_2$
$M_r = 536.52$
Monoclinic, $P2_1/c$
$a = 6.0688$ (7) Å
$b = 11.0033$ (13) Å
$c = 20.759$ (2) Å
$\beta = 91.284$ (2)°
$V = 1385.9$ (3) Å^3
$Z = 2$
$D_x = 1.286$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.19$ mm^{-1}
$T = 292$ (2) K
Prism, brown
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.956$, $T_{\max} = 0.981$
9412 measured reflections
3301 independent reflections
2043 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.072$
$\theta_{\max} = 28.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.251$
$S = 0.99$
3301 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1492P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.62$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

P1—O1	1.483 (3)	P1—C13	1.817 (4)
P1—C1	1.791 (4)	N1—C14	1.401 (5)
P1—C7	1.804 (4)	N1—C13	1.446 (5)
O1—P1—C1	112.67 (19)	C1—P1—C13	106.49 (19)
O1—P1—C7	110.9 (2)	C7—P1—C13	103.22 (19)
C1—P1—C7	109.06 (19)	C14—N1—C13	122.1 (3)
O1—P1—C13	113.96 (18)		

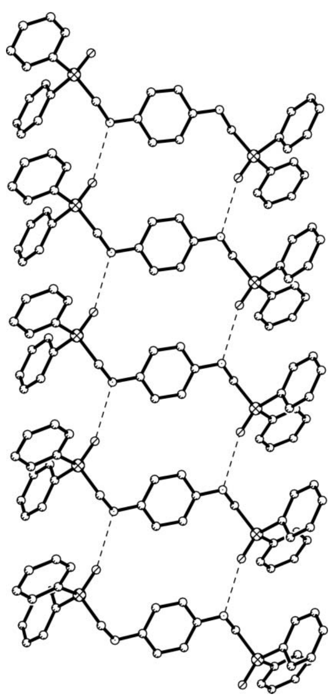

Figure 2

One-dimensional chain propagated by N—H···OP hydrogen bonding (dashed lines). H atoms have been omitted.

Table 2

Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.86	2.15	2.922 (4)	151

Symmetry code: (i) $x + 1, y, z$.

H atoms were observed in difference Fourier maps, but were placed in idealized positions and refined as riding on their parent atoms [C—H = 0.93–0.97 Å, N—H = 0.86 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C,N)].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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