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O,O′-(*R*)-(1,1′-Dinaphthyl-2,2′-diyl) *N*-benzyl-*N*-(2-pyridyl)phosphoramidite

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The title compound, $C_{32}H_{23}N_2O_2$, is a moisture- and oxygen-sensitive phosphite ligand. It has been shown to be effective in asymmetric conjugate addition of diorganozinc reagents to α,β-unsatuated cyclic enones.

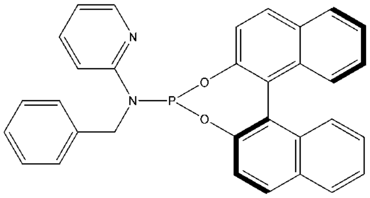
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.058
Data-to-parameter ratio = 17.4

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

Comment

Recently, some striking results have been obtained in the Cu-catalysed enantioselective conjugate addition of organometallic reagents; in particular, the addition of diorganozinc reagents to α,β-unsaturated esters, acyclic and cyclic enones, using chiral phosphoramidite ligands (Feringa *et al.*, 1997). Our research shows that the phosphoramidite ligand derived from 2-aminopyridine and binaphthol is an efficient ligand in the 1,4-conjugate addition of diphenylzinc to cyclic enones. Here, as part of our investigation, we report the crystal structure of the title compound, (I). Bond lengths and angles are within normal ranges (Table 1).



(I)

Experimental

All reactions were carried out under N_2, using Schlenk techniques. To a cooled solution (213 K) of PCl_3 (270 ml, 3.0 mmol), EtN_3(860 ml, 6.0 mmol), and toluene (5 ml) was added a warm solution (333 K) of (*R*)-2,2′-binaphthol (860 mg, 3.0 mmol) in toluene (25 ml) over a period of 25 min. After stirring for 2 h, the reaction mixture was warmed to room temperature and filtered under a nitrogen atmosphere. The filtrate was a solution of the chlorophosphite. The title compound was prepared by the reaction of the chlorophosphite and 2.9 mmol of the benzyl-2-pyridine compound at 233 K in the presence of Et_3N (410 ml, 2.9 mmol) and 4-dimethylaminopyridine. The crude products were purified by flash silica-gel chromatography. 1185 mg of a white solid was obtained (yield: 82%). A colorless crystal suitable for X-ray diffraction was obtained by recrystallization from a solution in CH_2Cl_2 and ether. ^{31}P NMR (CD_2Cl_2): δ 144.47 p.p.m.

Crystal data

$C_{32}H_{23}N_2O_2P$
M_r = 498.49
Orthorhombic, $P2_12_12_1$
a = 9.5168 (11) Å
b = 10.3952 (11) Å
c = 25.398 (3) Å
V = 2512.6 (5) Å^3
Z = 4
D_x = 1.318 Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3922 reflections
θ = 1–27.5°
μ = 0.14 mm^{-1}
T = 294 (2) K
Block, colorless
0.36 × 0.32 × 0.30 mm

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Data collection

Siemens CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.950$, $T_{\max} = 0.958$
17396 measured reflections
5805 independent reflections
2424 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.075$
$\theta_{\max} = 27.6°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -22 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.058$
$S = 0.90$
5805 reflections
334 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.002P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.15 (9)

Table 1
Selected geometric parameters (Å, °).

P1—N1	1.733 (2)	C1—C2	1.398 (3)
O1—C1	1.392 (3)	C2—C3	1.350 (4)
N1—C28	1.387 (3)	C4—C5	1.416 (4)
N1—C21	1.460 (3)	C4—C9	1.418 (4)
N2—C28	1.338 (3)	C5—C6	1.337 (4)
O2—P1—O1	100.09 (9)	C3—C2—C1	119.4 (3)
O2—P1—N1	103.69 (10)	C2—C3—C4	121.2 (3)
O1—P1—N1	93.54 (11)	C6—C5—C4	121.4 (4)
C1—O1—P1	118.97 (16)	C7—C8—C9	120.8 (3)
C10—C1—C2	123.2 (3)	C8—C9—C10	121.6 (3)
N1—P1—O2—C20	46.8 (2)	P1—O1—C1—C10	74.9 (3)
O2—P1—N1—C28	76.7 (2)	C1—C10—C11—C20	−52.8 (4)
O2—P1—N1—C21	−92.7 (2)	C9—C10—C11—C12	−59.6 (4)

H atoms were included in the riding-model approximation, with $U_{\rm iso}$ values equal to the $U_{\rm eq}$ of the atom to which they were bound.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SHELXTL* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

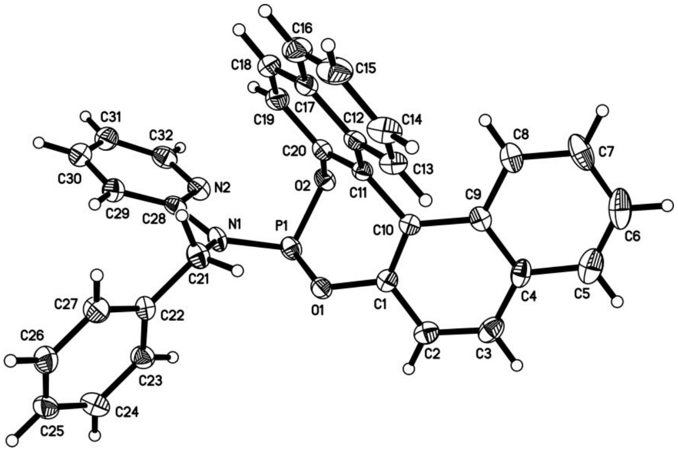

Figure 1
The molecular structure of (I), showing ellipsoids at the 50% probability level (Siemens, 1995).

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