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(*S*)-2,2′-Bis(dicyclohexylphosphinoamino)-1,1′-binaphthyl

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.007 Å
R factor = 0.068
wR factor = 0.155
Data-to-parameter ratio = 19.3

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

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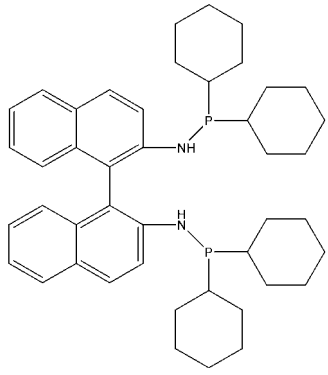
The the crystal structure of the title compound, $C_{44}H_{58}N_2P_2$, the molecule has a non-crystallographic twofold axis. The bond lengths and angles are within the expected ranges.

Comment

Aminophosphine ligands exhibit good to excellent enantioselectivity and high reactivity in the hydrogenation of prochiral olefins (Zhang *et al.*, 1998; Chan *et al.*, 1997). Recent research shows that the aminophosphine ligands derived from dialkylphosphine chloride exhibit significantly different enantioselectivity in the catalytic hydrogenation reaction due to the steric and electronic modulation of the substituent groups on the P atom (Trabesinger *et al.*, 1997; RajanBabu *et al.*, 1997). As part of our investigation of this phenomenon, we report the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) (Fig. 1) shows that the torsion angles C9—C10—C11—C12 and C1—C10—C11—C20 are 81.4 (4) and 79.9 (4)°, respectively.

Experimental

All reactions were carried out under N_2 using Schlenk techniques. (*S*)-2,2′-diamino-1,1′-binaphthyl (284 mg, 1.0 mmol) and 4-*N*,*N*-dimethylaminopyridine (15 mg) were mixed in a 50 ml round-bottomed Schlenk flask with a stirrer bar. The atmosphere was replaced with N_2 several times. 0.8 ml dry Et_3N and 30 ml dry CH_2Cl_2 were added. The mixture was cooled to 273 K with an ice bath, followed by dropwise addition of dicyclohexylphosphine chloride (0.6 ml, 2.5 mmol) over a period of 20 min. The solvent was removed under vacuum after stirring at room temperature for another 3 h. The residues were dissolved in 15 ml toluene and purified on a flash silica-gel column (30 ml toluene as eluent). Toluene was removed and 530 mg of a white solid was obtained (yield: 78%). A colorless crystal suitable for X-ray diffraction was obtained by recrystallization from a solution in EtOH and CH_2Cl_2. ^{31}P NMR ($CDCl_3$): δ 42.8 p.p.m.

Crystal data

$C_{44}H_{58}N_2P_2$	D_x = 1.113 Mg m^{-3}
M_r = 676.89	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 5501 reflections
a = 10.316 (3) Å	θ = 1–27.5°
b = 16.988 (5) Å	μ = 0.14 mm^{-1}
c = 11.635 (3) Å	T = 294 (2) K
β = 97.971 (7)°	Prism, colorless
V = 2019.4 (10) Å^3	0.30 × 0.28 × 0.26 mm
Z = 2	

Data collection

Bruker CCD area-detector diffractometer	4786 independent reflections
	2931 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.055
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 27.6°
	$h = -11 \rightarrow 13$
T_{min} = 0.960, T_{max} = 0.965	$k = -18 \rightarrow 22$
13673 measured reflections	$l = -15 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.068	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.155	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
S = 1.13	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
8354 reflections	Absolute structure: Flack (1983)
433 parameters	Flack parameter = 0.01 (10)
H-atom parameters constrained	

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

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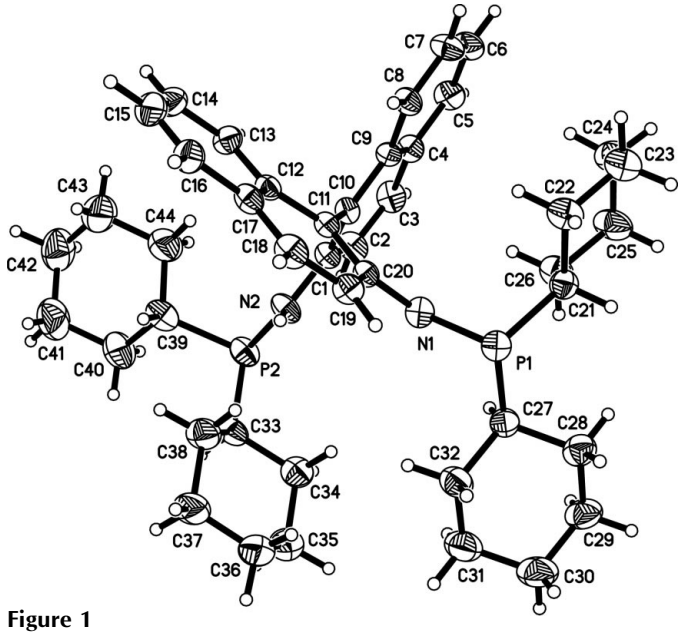

Figure 1
The molecular structure of (I), with displacement ellipsoids at the 30% probability level.

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