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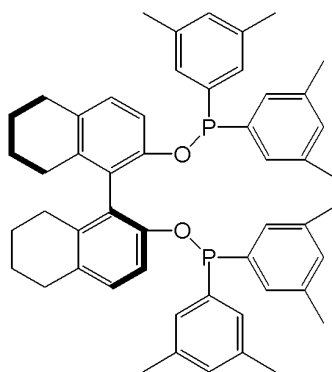
Key indicators

Single-crystal X-ray study
 $T = 294\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.049
 wR factor = 0.128
Data-to-parameter ratio = 20.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(S)-2,2'-Bis[bis(3,5-dimethylphenyl)phosphinoyl]-
5,5',6,6',7,7',8,8'-octahydro-1,1'-binaphthyl**

The title compound, $\text{C}_{52}\text{H}_{56}\text{O}_2\text{P}_2$, was synthesized conveniently from (*S*)-2,2'-dihydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-binaphthyl and bis(3,5-dimethylphenyl)phosphine chloride in high yield. The molecule has crystallographic twofold rotation symmetry.

Comment

Phosphinite ligands exhibit good-to-excellent enantioselectivity and high reactivity in the hydrogenation of prochiral olefins (Zhang *et al.*, 1998; Chan *et al.*, 1997). The phosphinite (I) can be conveniently obtained by reacting (*S*)-2,2'-dihydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-binaphthyl (H8-BINOL) with bis(3,5-dimethylphenyl)phosphine chloride. Recent research shows that the phosphinite ligands derived from bis(3,5-dimethylphenyl)phosphine chloride exhibit higher enantioselectivity in the catalytic hydrogenation reaction than those derived from chlorodiphenylphosphine, because of 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 effort to investigate this phenomenon, we present the crystal structure of (I).



(I)

As shown in Fig. 1, the title molecule has crystallographic twofold rotation symmetry. The $\text{Csp}^3-\text{Csp}^3$ bonds C7—C8, C8—C9 and C9—C10 [1.476 (3), 1.441 (3) and 1.463 (3) Å, respectively] are shorter than the normal $\text{Csp}^3-\text{Csp}^3$ bond (1.51–1.55 Å) and differ significantly in length from the $\text{Csp}^3-\text{Csp}^2$ bonds C3—C7 and C4—C10 [1.513 (4) and 1.510 (4) Å, respectively] in the H8-naphthyl unit. Disorder, suggested to be due to the puckering of the ring, which is suggested by the high U_{eq} values of atoms C8 and C9.

Experimental

All reactions were carried out under N₂ using Schlenk techniques. (*S*)-2,2'-Dihydroxy-5,5',6,6',7,7',8,8'-octahydro-1,1'-binaphthyl (0.30 g, 1.0 mmol) and 4-*N,N*-dimethylaminopyridine (15 mg) were mixed in a 50 ml round-bottomed Schlenk flask with a stir bar. The atmosphere was replaced with N₂ several times, and 0.8 ml dry Et₃N and 30 ml dry Et₂O were added. The mixture was cooled to 273 K with an ice-bath, followed by dropwise addition of bis(3,5-dimethylphenyl)phosphine chloride (0.6 ml, 2.5 mmol) over 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 with a flash silica-gel column (30 ml toluene as eluent). Toluene was removed and 670 mg white solid obtained (yield: 87%). Colorless crystals suitable for X-ray diffraction were obtained by recrystallization from Et₂O/CH₂Cl₂ (5:1). ³¹P NMR (CDCl₃): δ 109.7. ¹³C NMR (CDCl₃): δ 21.39, 21.48, 23.22, 23.30, 27.80, 29.76, 115.28, 115.41, 127.36, 127.54, 127.65, 127.84, 129.09, 130.85, 131.01, 131.33, 137.03, 137.59, 142.11, 142.26. ¹H NMR (500 MHz, CDCl₃): δ 1.52–1.68 (*m*, 8H), 2.21 (*s*, 12H), 2.19 (*s*, 12H), 2.14–2.23 (*m*, 2H), 2.38–2.42 (*m*, 2H), 2.60–2.64 (*m*, 2H), 2.69–2.75 (*m*, 2H).

Crystal data

C ₅₂ H ₅₆ O ₂ P ₂	Mo K α radiation
<i>M_r</i> = 774.91	Cell parameters from 5442 reflections
Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2	θ = 1–27.5°
<i>a</i> = 13.1983 (18) Å	μ = 0.14 mm ⁻¹
<i>b</i> = 19.015 (3) Å	<i>T</i> = 294 (2) K
<i>c</i> = 8.8486 (12) Å	Block, colorless
<i>V</i> = 2220.7 (5) Å ³	0.34 × 0.28 × 0.26 mm
<i>Z</i> = 2	
<i>D_x</i> = 1.159 Mg m ⁻³	

Data collection

Bruker CCD area-detector diffractometer	5134 independent reflections
φ and ω scans	3079 reflections with <i>I</i> > 2 σ (<i>I</i>)
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	<i>R</i> _{int} = 0.045
<i>T</i> _{min} = 0.955, <i>T</i> _{max} = 0.965	θ _{max} = 27.6°
15328 measured reflections	<i>h</i> = -17 → 11
	<i>k</i> = -24 → 24
	<i>l</i> = -11 → 11

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.128$	(Δ/σ) _{max} < 0.001
<i>S</i> = 0.93	$\Delta\rho$ _{max} = 0.27 e Å ⁻³
5134 reflections	$\Delta\rho$ _{min} = -0.28 e Å ⁻³
253 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	2216 Friedel pairs
	Flack parameter = -0.03 (12)

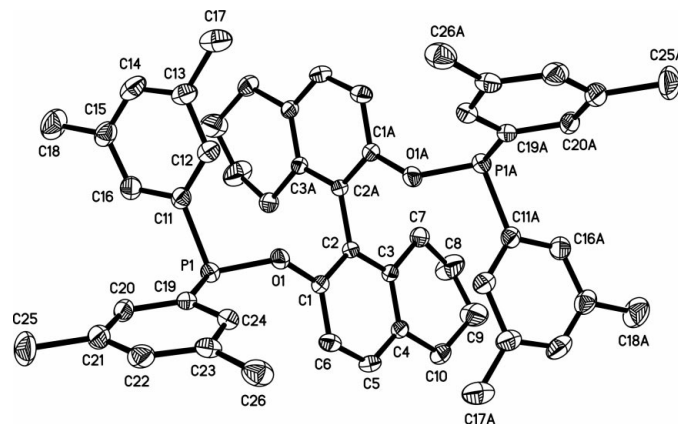


Figure 1

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

Data collection: *SMART* (Bruker, 1995); cell refinement: *SMART*; data reduction: *SHELXTL-NT* (Bruker, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-NT*; software used to prepare material for publication: *SHELXTL-NT*.

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