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rac-2,2′-Bis(diphenylphosphinoyl)-1,1′-binaphthyl

Marie-Hélène Thibault and Frédéric-Georges Fontaine*

Département de Chimie, Université Laval, Québec, QC, Canada G1K 7P4

Correspondence e-mail:
frederic.fontaine@chm.ulaval.ca

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In the structure of the title compound, $C_{44}H_{32}O_2P_2$, the dihedral angle between the naphthyl ring systems is 88.74 (4)°.

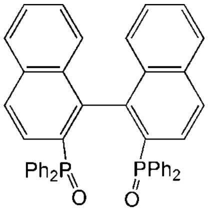
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.130
Data-to-parameter ratio = 14.9

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

Comment

In the course of our studies, the title compound, (I), was obtained from the oxidation of the corresponding bisphosphine with H_2O_2. The bisphosphine was being used as a ligand to coordinate palladium in a Buchwald–Hartwig coupling reaction.



(I)

The title compound crystallizes in the space group $P2_1/n$ (Fig. 1). The crystal packing is very similar to that of the previously reported structure of (*R*)-(+)-2,2′-bis(diphenylphosphinoyl)-1,1′-binaphthyl, (II) (Bunten *et al.*, 2000), which crystallizes in the space group $P2_1$.

The solid-state structure of (I) is maintained by short contacts between O atoms and one of the H atoms on one of the benzene groups of an adjacent molecule, giving a one-dimensional stacking of the molecules. The H43···O2 and C43···O2 distances are, respectively, 2.49 and 3.217 (2) Å, with a C43—H43···O2 angle of 136°, while the H30···O1 and C30···O1 distances are, respectively, 2.39 and 3.232 (2) Å, with a C30—H30···O1 angle of 150°.

The main difference between (I) and the enantiopure compound, (II), resides in the *b* axis being twice as long in the centrosymmetric crystal structure of (I) as in (II) [36.969 (4) *versus* 18.9903 (8) Å]. The structural parameters of both bis(diphenylphosphinoyl)-1,1′-binaphthyl structures are quite similar, with the exception of the torsion angle between the naphthyl rings, which is smaller in (II) [88.74 (4)° *versus* 94.17 (3)°].

Experimental

A Hartwig–Buchwald coupling was performed using rac-BINAP [*rac*-2,2′-bis(diphenylphosphino)-1,1′-binaphthalene] (6.1 mg, 0.0098 mmol) in toluene (3 ml). The reaction mixture was refluxed for 72 h and hydrogen peroxide (0.08 ml) was then added to oxidize the phosphine. After evaporation of the toluene *in vacuo*, the remaining brown mixture was dissolved in CH_2Cl_2 (3 ml) and extracted with water (3 × 2 ml). The aqueous phase was then washed with CH_2Cl_2 (3 × 3 ml). The organic phases were combined and dried with $MgSO_4$. Crystals of (I) were grown by slow evaporation of the solvent.

Crystal data

$C_{44}H_{32}O_2P_2$
$M_r = 654.64$
Monoclinic, $P2_1/n$
$a = 8.630$ (1) Å
$b = 36.969$ (4) Å
$c = 10.605$ (1) Å
$\beta = 104.111$ (1)°
$V = 3281.3$ (7) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.17$ mm^{-1}
$T = 273$ (2) K
0.37 × 0.27 × 0.17 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: integration (*XPREP*; Bruker, 2005)
$T_{\min} = 0.939$, $T_{\max} = 0.971$
34454 measured reflections
6449 independent reflections
5919 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.130$
$S = 1.12$
6449 reflections
433 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

H atoms were placed in idealized positions, with C—H = 0.93 Å, and refined using a riding model, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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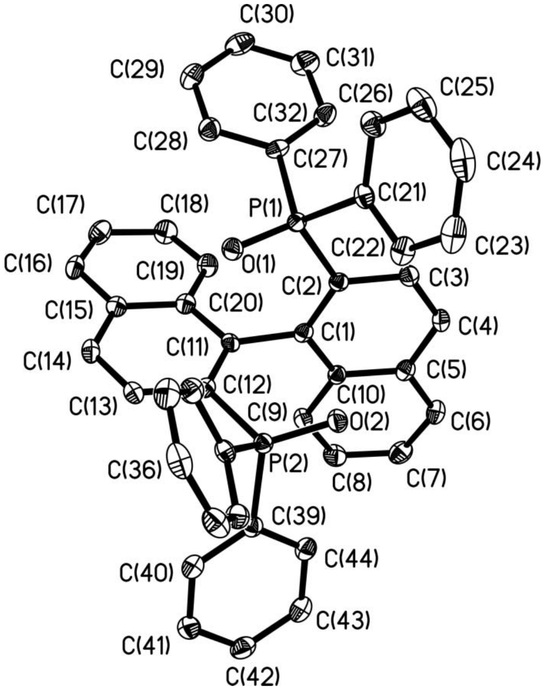

Figure 1
The molecular structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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