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(+)-(*RR*)-{*μ*-*m*-Phenylenedimethylenebis[*tert*-butyl(phenyl)phosphine]}bis(borane)

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The title compound, $C_{28}H_{42}B_2P_2$, (II), was synthesized with >95% enantiomeric excess, and the relative and absolute configuration was determined. Both P atoms have distorted tetrahedral geometry, and the average B—P—C angle and P—B distance are 112.7 (6)° and 1.910 (6) Å, respectively.

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.088
Data-to-parameter ratio = 17.9

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

Comment

The importance of phosphine ligands in transition metal chemistry is remarkable. Among them optically active C_2-symmetric phosphine ligands proved to be instrumental in asymmetric catalysis (Yamanoi & Imamoto, 1999). Recently, P-chiral phosphines have emerged as a good chiral controller in transition metal-catalyzed reactions (Kolodiazhnyi, 1998). Transition metal complexes with phosphorus–carbon–phosphorus (PCP) tridentate or pincer ligands were studied extensively (Laurenti & Santelli, 1999). However, to our knowledge, no structure of a pincer ligand with P-chiral phosphines has been reported so far. Therefore, we wish to report the first crystallographic structure of a P-chiral PCP tridentate ligand as a bis-borone complex.

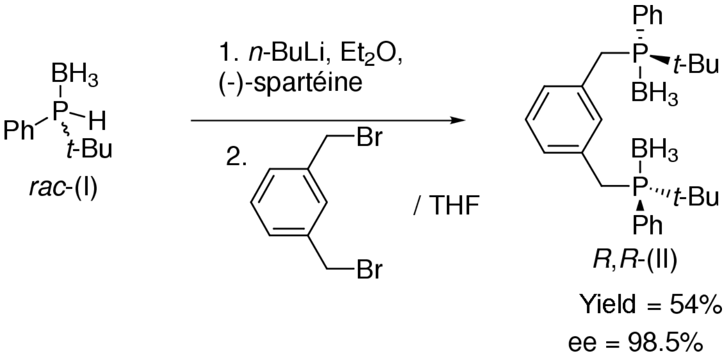

Fig. 1 clearly indicates the C_2-symmetry of (II). The two phosphorus substituents are on opposite sides of the aromatic ring. Both P atoms have adopted a distorted tetrahedral geometry. The average B—P—C angle and P—B distance are 112.7 (6)° and 1.910 (6) Å, respectively (Table 1).

Experimental

According to the literature procedure of Wolfe & Livinghouse (1998), the enantioselective alkylation of the phosphine–borane (I) with the lithium–sparteine complex afforded the desired C_2-

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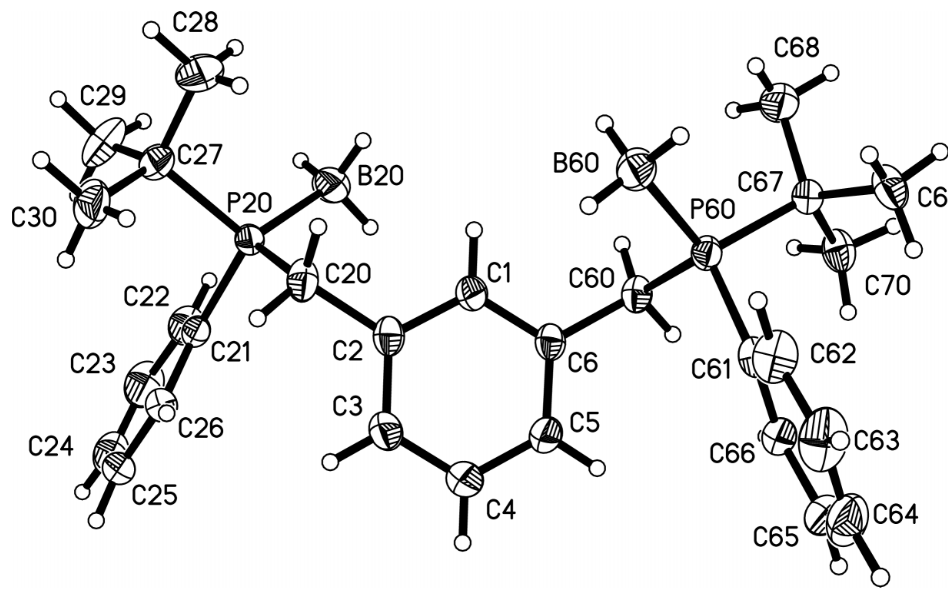

Figure 1
ORTEP drawing (*SHELXTL*; Bruker, 1997) of the title molecule. Ellipsoids correspond to 40% probability.

symmetric diphosphine in 54% yield and 98.5% ee, contaminated with 5% of the *meso* diastereoisomer. Recrystallization from dichloromethane/ether afforded the enantio- and diastereomerically pure (>99%) bis(phosphine–borane) (II). Optical rotation of (II): $[a]_D$ = +127.6 (c 2.78, $CHCl_3$).

Crystal data

$C_{28}H_{42}B_2P_2$
$M_r = 462.18$
Orthorhombic, $P2_12_12_1$
$a = 7.380$ (6) Å
$b = 13.515$ (6) Å
$c = 28.189$ (14) Å
$V = 2812$ (3) Å^3
$Z = 4$
$D_x = 1.092$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 20.0$–$21.0°$
$\mu = 1.48$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.83 × 0.22 × 0.14 mm

Data collection

Nonius CAD-4 diffractometer
ω scans
Absorption correction: by integration (*ABSORP* in *NRCVAX*; Gabe *et al.*, 1989)
$T_{\min} = 0.539$, $T_{\max} = 0.830$
30 802 measured reflections
5320 independent reflections
4737 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.062$
$\theta_{\max} = 69.8°$
$h = -8 \rightarrow 8$
$k = -16 \rightarrow 16$
$l = -34 \rightarrow 34$
5 standard reflections
frequency: 60 min
intensity decay: 21.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.088$
$S = 1.03$
5320 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*96
Extinction coefficient: 0.0028 (2)
Absolute structure: Flack (1983), 2274 Friedel pairs
Flack parameter = −0.004 (18)

Table 1
Selected geometric parameters (Å, °).

C20—P20	1.824 (2)	C60—P60	1.829 (2)
P20—C21	1.802 (2)	P60—C61	1.806 (2)
P20—C27	1.858 (3)	P60—C67	1.862 (2)
P20—B20	1.904 (3)	P60—B60	1.916 (3)
C21—P20—C20	107.36 (11)	C61—P60—C60	106.62 (11)
C21—P20—C27	105.59 (11)	C61—P60—C67	105.07 (10)
C20—P20—C27	105.53 (11)	C60—P60—C67	105.96 (10)
C21—P20—B20	113.39 (13)	C61—P60—B60	113.24 (13)
C20—P20—B20	111.91 (13)	C60—P60—B60	112.13 (12)
C27—P20—B20	112.51 (13)	C67—P60—B60	113.20 (12)

The title compound, (II), is quite stable and can be stored for months on the bench, without any significant decomposition. However, it seems to be X-ray sensitive, giving a linear intensity decay during exposure; this was corrected. H atoms were constrained to the parent site using a riding model; *SHELXL*96 defaults, C—H = 0.93–0.97 Å and B—H = 0.96 Å. The isotropic factors, $U_{\rm iso}$, were adjusted to 150% of the value of the parent site for methyl and B—H H atoms and 120% for other H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *NRC*-2 and *NRC*-2*A* (Ahmed *et al.*, 1973); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL*96.

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References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1973). *NRC Crystallographic Computer Programs for the IBM*/360. Accession Nos. 133–147. *J. Appl. Cryst.* **6**, 309–346.

Bruker (1997). *SHELXTL*. Release 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Kolodiazhnyi, O. I. (1998). *Tetrahedron Asymmetry*, **9**, 1279–1332.

Laurenti, D. & Santelli, M. (1999). *Org. Prep. Proced. Int.* **31**, 245–294.

Sheldrick, G. M. (1996). *SHELXL*96. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS*97. University of Göttingen, Germany.

Wolfe, B. & Livinghouse, T. (1998). *J. Am. Chem. Soc.* **120**, 5116–5117.

Yamanoi, Y. & Imamoto, T. (1999). *Rev. Heterocycl. Chem.* **20**, 227–248.