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(2*R*,4*S*,5*R*)-3,4-Dimethyl-5-phenyl-2-[4-(trifluoromethyl)phenyl]-1,3,2-oxazaphospholidine(*P*—*B*)borane

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A P-chiral phosphinite borane complex, $C_{17}H_{20}BF_3NOP$, derived from (2*R*,4*S*,5*R*)-2-chloro-3,4-dimethyl-5-phenyl-1,3,2-oxazaphospholidine and [4-(trifluoromethyl)phenyl]-magnesium bromide is reported. The absolute configuration (*R* at P) is deduced from the known chirality of (1*R*,2*S*)-ephedrine and confirmed by the refinement.

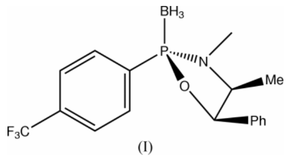
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.134
Data-to-parameter ratio = 16.7

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

Comment

Phosphorus-chiral ligands have proven useful for a great variety of asymmetric organic transformations. Ephedrine-derived oxazaphospholidines are very interesting precursors for the design and synthesis of novel P-chiral ligands (Juge *et al.*, 1990; Brunel *et al.*, 1994; Leung *et al.*, 2001). P-Chirality moves the stereogenic center close to both the metal and the substrate. To this date, only a few P-chiral monophosphines have been applied to transition metal catalysis, probably due to troublesome synthesis of P-chiral ligands. The chemoselective opening of the oxazaphospholane boranes with either lithium or Grignard reagents provides for the synthesis of a great variety of chiral phosphines. Having introduced a *p*-CF_3 substituent on the phenyl group a more electron-deficient oxazaphospholidine is obtained (Fig. 1). To our knowledge, no such structure has been reported. Bond lengths and angles within the phosphorus-containing five-membered ring are in accordance with earlier reported oxazaphospholidine borane complexes (Juge *et al.*, 1990; Brunel *et al.*, 1994; Leung *et al.*, 2001).



(I)

Experimental

(2*R*,4*S*,5*R*)-2-Chloro-3,4-dimethyl-5-phenyl-1,3,2-oxazaphospholidine was dissolved in THF, cooled to 195 K, and [4-(trifluoromethyl)phenyl]magnesium bromide (1 equivalent) was added dropwise to the solution. (Caution: the preparation of the Grignard

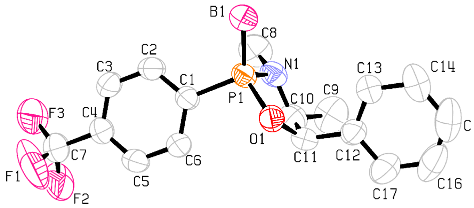


Figure 1
View of (I) (50% probability displacement ellipsoids). Only one conformation of the disordered trifluoromethyl group is shown.

reagent is a highly exothermic reaction; explosions have been reported.) The resulting mixture was stirred under an argon atmosphere for 18 h. BH_3*DMS (1 equivalent) was then added dropwise at 273 K and the resulting solution stirred for an additional 18 h, then concentrated under reduced pressure to yield a white solid. The product was crystallized from 2-propanol to give colourless rod-shaped crystals.

Crystal data

$C_{17}H_{20}BF_3NOP$
$M_r = 353.13$
Orthorhombic, $P2_12_12_1$
$a = 9.1911 (16)$ Å
$b = 12.8581 (17)$ Å
$c = 15.827 (2)$ Å
$V = 1870.4 (5)$ Å^3
$Z = 4$
$D_x = 1.254$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 25 reflections
$\theta = 37.7–49.6°$
$\mu = 0.18$ mm^{-1}
$T = 293 (2)$ K
Rod, colourless
1.00 × 0.75 × 0.40 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: none
6482 measured reflections
4085 independent reflections
2990 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$

$\theta_{\max} = 27.0°$
$h = -11 \rightarrow 4$
$k = -16 \rightarrow 16$
$l = -20 \rightarrow 0$
2 standard reflections every 123 reflections intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.134$
$S = 1.04$
4085 reflections
244 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0841P)^2 + 0.0091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}
Absolute structure: Flack (1983), 1755 Friedel pairs
Flack parameter = 0.04 (12)

Owing to poor quality, a large crystal was used, and no absorption correction was made. Attempts to reduce the size by cutting was not successful. The absolute configuration was established by the known stereochemistry of the ephedrine moiety. It was also confirmed by the refinement of the Flack (1983) parameter. H atoms were placed in calculated positions (C—H = 0.93–0.98 Å and B—H = 0.96 Å) and were allowed to ride on their parent atoms. The $U_{\rm iso}$(H) values were set to $1.5U_{\rm eq}$(parent) for the methyl and borane H atoms and $1.2U_{\rm eq}$(C) for other H atoms. The occupancies of the F atoms in the rotationally disordered trifluoromethyl group were initially refined [0.48 (2) and 0.52 (2) for the two components] and later fixed at 0.5.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Brunel, J. M., Buono, G., Baldy, A., Feneau-Dupont, J. & Declercq, J. P. (1994). *Acta Cryst.* C**50**, 954–955.

Enraf–Nonius (1994). *CAD*-4 *EXPRESS Software*. Enraf–Nonius, Delft, The Netherlands.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

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

Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.

Juge, S., Stephan, M., Laffitte, J. A. & Genet, J. P. (1990). *Tetrahedron Lett.* **31**, 6357–6360.

Leung, W., Cosway, S., Jones, R. H. V., McCann, H. & Wills, M. (2001). *J. Chem. Soc. Perkin Trans.* 1, pp. 2588–2594.

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

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