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(2*R*,5*S*)-2,3-Diphenyl-1,3,2-diazaphosphabicyclo[3.3.0]octane–Borane

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Abstract

The title complex, $C_{17}H_{22}BN_2P$, (3), is one of a series of new chiral diazaphospholidine–borane complexes used as catalysts in the enantioselective borane reduction of ketones. We describe herein the determination of the molecular structure of (3) and the absolute configuration about the P atom.

Comment

Recently, we reported a new method for the enantioselective reduction of ketones with BH_3:THF, catalyzed by a chiral tricoordinated phosphorus–borane complex (Brunel, Pardigon, Faure & Buono, 1992; Buono, Brunel, Faure & Pardigon, 1993). The study of the title compound, (3), was undertaken in order to probe the mechanism of the reduction and to establish the absolute configuration about the P atom. Complex (3) was synthesized by reaction of bis(dimethylamino)phenylphosphine, (1), with (*S*)-(+)-2-anilinomethylpyrrolidine, (2), in refluxing toluene and complexation by one equivalent of BH_3:THF.

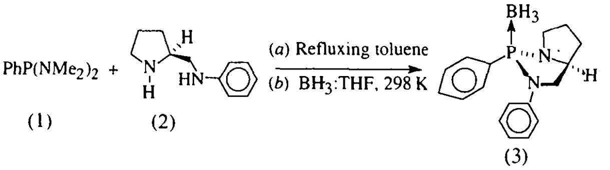

Fig. 1 is a view of the title molecule showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978).

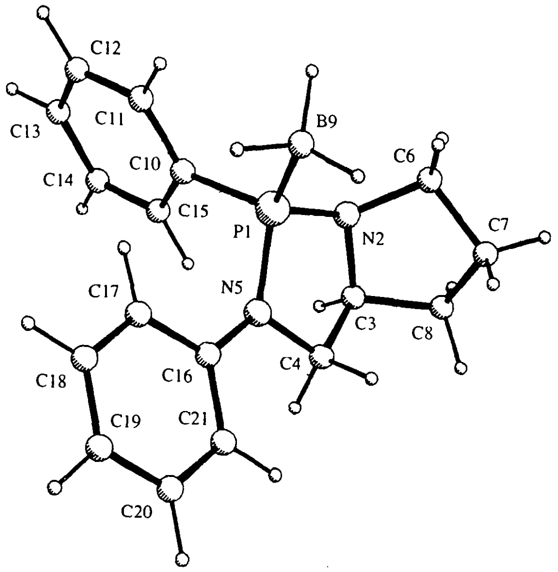

Fig. 1. View of the title compound (*PLUTO*; Motherwell & Clegg, 1978) showing the atom-labelling scheme.

Experimental

Crystal data

$C_{17}H_{22}BN_2P$
$M_r = 296.16$
Orthorhombic
$P2_12_12_1$
$a = 10.197$ (2) Å
$b = 9.086$ (2) Å
$c = 17.807$ (5) Å
$V = 1649.8$ (6) Å^3
$Z = 4$
$D_x = 1.19$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 30 reflections
$\theta = 15$–$35°$
$\mu = 1.402$ mm^{-1}
$T = 291$ K
Parallelepiped
0.60 × 0.60 × 0.50 mm
White

Data collection

Huber four-circle diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2969 measured reflections
2969 independent reflections
2793 observed reflections $[I \geq 2.5\sigma(I)]$

$\theta_{\max} = 67°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 21$
1 standard reflection monitored every 50 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.050$
$wR = 0.061$
$S = 0.47$
2793 reflections
155 parameters
H atoms isotropic with common $U = 0.094$ Å^2
$w = 1/[\sigma^2(F) + 0.0318F^2]$

$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.53$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
P1	0.7887 (1)	0.8765 (1)	0.1096 (1)	0.051 (1)
N2	0.7470 (2)	0.7076 (3)	0.1357 (1)	0.058 (1)
C3	0.8478 (3)	0.6421 (3)	0.1864 (2)	0.060 (1)
C4	0.9771 (3)	0.7180 (3)	0.1685 (2)	0.058 (1)
N5	0.9427 (2)	0.8670 (2)	0.1434 (1)	0.054 (1)
C6	0.6980 (4)	0.5936 (4)	0.0836 (2)	0.075 (1)
C7	0.8015 (4)	0.4750 (4)	0.0870 (2)	0.081 (1)
C8	0.8458 (4)	0.4771 (3)	0.1686 (2)	0.076 (1)
B9	0.7647 (5)	0.9174 (5)	0.0053 (2)	0.082 (1)
C10	0.7064 (3)	1.0031 (3)	0.1720 (1)	0.055 (1)
C11	0.6269 (3)	1.1126 (4)	0.1431 (2)	0.077 (1)
C12	0.5597 (4)	1.2077 (5)	0.1909 (3)	0.099 (2)
C13	0.5715 (4)	1.1913 (5)	0.2663 (4)	0.100 (2)
C14	0.6506 (4)	1.0832 (5)	0.2970 (3)	0.093 (1)
C15	0.7197 (4)	0.9901 (4)	0.2495 (2)	0.072 (1)
C16	1.0414 (3)	0.9689 (3)	0.1276 (1)	0.051 (1)
C17	1.0121 (3)	1.1139 (3)	0.1085 (2)	0.063 (1)
C18	1.1114 (4)	1.2135 (3)	0.0918 (2)	0.070 (1)
C19	1.2404 (3)	1.1738 (4)	0.0957 (2)	0.073 (1)
C20	1.2701 (3)	1.0312 (4)	0.1160 (2)	0.072 (1)
C21	1.1739 (3)	0.9285 (3)	0.1313 (2)	0.060 (1)

Table 2. *Selected geometric parameters (Å, °)*

N2—P1	1.659 (2)	N5—P1	1.684 (2)
B9—P1	1.910 (3)	C10—P1	1.806 (3)
C3—N2	1.492 (4)	C6—N2	1.477 (4)
C4—C3	1.522 (4)	C8—C3	1.531 (4)
N5—C4	1.468 (3)	C16—N5	1.396 (3)
C7—C6	1.510 (5)	C8—C7	1.523 (5)
C11—C10	1.383 (4)	C15—C10	1.391 (4)
C12—C11	1.392 (6)	C13—C12	1.357 (8)
C14—C13	1.384 (8)	C15—C14	1.389 (5)
C17—C16	1.393 (4)	C21—C16	1.402 (4)
C18—C17	1.389 (4)	C19—C18	1.366 (5)
C20—C19	1.379 (5)	C21—C20	1.381 (4)
N5—P1—N2	95.3 (1)	C8—C7—C6	103.7 (3)
B9—P1—N5	118.4 (2)	C11—C10—P1	120.1 (2)
C10—P1—N5	104.3 (1)	C15—C10—P1	120.7 (2)
C3—N2—P1	111.2 (2)	C12—C11—C10	120.5 (4)
C6—N2—C3	109.5 (2)	C14—C13—C12	121.4 (3)
C8—C3—N2	104.9 (2)	C14—C15—C10	120.2 (4)
N5—C4—C3	106.0 (2)	C21—C16—N5	120.8 (2)
C16—N5—P1	124.5 (2)	C18—C17—C16	120.8 (3)
C7—C6—N2	103.8 (3)	C20—C19—C18	118.3 (3)
C7—C8—C3	102.3 (3)	C20—C21—C16	119.9 (3)
B9—P1—N2	114.8 (2)	C15—C10—C11	119.2 (3)
C10—P1—N2	107.4 (1)	C13—C12—C11	119.5 (4)
C10—P1—B9	114.5 (2)	C15—C14—C13	119.2 (4)
C6—N2—P1	124.0 (2)	C17—C16—N5	121.5 (2)
C4—C3—N2	106.8 (2)	C21—C16—C17	117.8 (2)
C8—C3—C4	114.3 (3)	C19—C18—C17	121.2 (3)
C4—N5—P1	112.2 (2)	C21—C20—C19	122.0 (3)
C16—N5—C4	120.1 (2)		
N5—P1—N2—C3	10.7 (2)	N2—C3—C8—C7	28.2 (3)
N5—P1—N2—C6	−123.2 (2)	C4—C3—C8—C7	−88.4 (3)
B9—P1—N2—C3	135.4 (2)	C3—C4—N5—P1	−23.7 (3)
B9—P1—N2—C6	1.5 (3)	C3—C4—N5—C16	175.5 (2)
C10—P1—N2—C3	−96.0 (2)	P1—N5—C16—C17	26.0 (3)
C10—P1—N2—C6	130.0 (2)	P1—N5—C16—C21	−154.6 (2)
N2—P1—N5—C4	8.3 (2)	C4—N5—C16—C17	−175.6 (3)
N2—P1—N5—C16	168.0 (2)	C4—N5—C16—C21	3.8 (4)
B9—P1—N5—C4	−113.6 (2)	N2—C6—C7—C8	35.8 (3)
B9—P1—N5—C16	46.1 (3)	C6—C7—C8—C3	−39.6 (3)
C10—P1—N5—C4	117.7 (2)	P1—C10—C11—C12	177.9 (3)
C10—P1—N5—C16	−82.5 (2)	C15—C10—C11—C12	−0.8 (5)
N2—P1—C10—C11	−124.7 (2)	P1—C10—C15—C14	−176.7 (3)
N2—P1—C10—C15	53.9 (3)	C11—C10—C15—C14	2.0 (5)
N5—P1—C10—C11	135.0 (2)	C10—C11—C12—C13	−0.7 (6)
N5—P1—C10—C15	−46.3 (3)	C11—C12—C13—C14	1.0 (7)
B9—P1—C10—C11	4.0 (3)	C12—C13—C14—C15	0.2 (7)
B9—P1—C10—C15	−177.4 (3)	C13—C14—C15—C10	−1.7 (6)
P1—N2—C3—C4	−25.6 (3)	N5—C16—C17—C18	−179.0 (3)
P1—N2—C3—C8	−147.3 (2)	C21—C16—C17—C18	1.6 (4)
C6—N2—C3—C4	115.1 (3)	N5—C16—C21—C20	−179.5 (3)
C6—N2—C3—C8	−6.6 (3)	C17—C16—C21—C20	−0.1 (4)
P1—N2—C6—C7	116.5 (3)	C16—C17—C18—C19	−1.9 (5)
C3—N2—C6—C7	−18.1 (3)	C17—C18—C19—C20	0.5 (5)
N2—C3—C4—N5	30.1 (3)	C18—C19—C20—C21	1.0 (5)
C8—C3—C4—N5	145.6 (3)	C19—C20—C21—C16	−1.2 (5)

At the end of the refinement, the 1175 Friedel pairs of reflections present in the data set were sorted in decreasing magnitude according to $w^{1/2} = ||F_c(h,k,l)|-|F_c(-h,-k,-l)||$. Among the first 200 pairs of reflections, the signs of 172 observed differences were consistent with the signs of the corresponding calculated differences, establishing that the molecule is described with the correct absolute configuration, *R*.

The structure of the title complex was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms were placed in computed positions except for those attached to the B atom which were obtained from a difference Fourier synthesis. Anisotropic least-squares refinement was carried out using *SHELX*76 (Sheldrick, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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