Nardelli, M. (1983). *Comput. Chem.* 7, 95–98.
Rodier, P. N., Robert, J.-M. & Le Baut, G. (1992). *Acta Cryst.* C**48**, 572–574.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Acta Cryst. (1994). C**50**, 954–955

A (2*R*,5*S*)-2-*o*-Anisyl-3-oxa-1-aza-2-phosphabicyclo[3.3.0]octane–Borane Complex

J. M. Brunel and G. Buono*

Ecole Nationale Supérieure de Synthèses, de Procédés et d'Ingénierie Chimiques d'Aix-Marseille (ENSSPICAM), URA 1410, Faculté des Sciences et Techniques de St Jérome, Université Aix-Marseille III, Avenue Normandie Niemen, 13397 Marseille CEDEX 20, France

A. Baldy

CIMM, URA CNRS 1411, Faculté des Sciences et Techniques de St Jérome, Université Aix-Marseille III, Avenue Normandie Niemen, 13397 Marseille CEDEX 20, France

J. Feneau-Dupont and J.-P. Declercq

Laboratoire de Chimie Physique et de Cristallographie, Université Catholique de Louvain, 1 Place Pasteur, 1348 Louvain la Neuve, Belgium

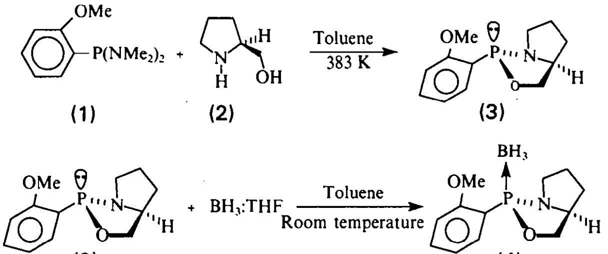
(*Received* 7 *June* 1993; *accepted* 21 *October* 1993)

Abstract

(2*R*,5*S*)-2-(2-Methoxyphenyl)-3-oxa-1-aza-2-phosphabicyclo[3.3.0]octane(*P*—*B*)borane, $C_{12}H_{19}BNO_2P$, is one of a series of new chiral oxazaphospholidine–borane complexes used as catalysts in the enantioselective borane reduction of ketones. The structure and the absolute configuration about the P atom were determined.

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 (4) (Brunel, Pardigan, Faure & Buono, 1992). Complex (4) was synthesized by the reaction of bis(dimethylamino)-*o*-anisylphosphine (1) with (*S*)-(+)-prolinol (2) in refluxing toluene and complexation by one equivalent of BH_3:THF.



The study of the title compound (4) was undertaken in order to probe the mechanism of the reduction and to establish the absolute configuration about the P atom. The structure is shown in Fig. 1. It is noteworthy that the sum of the bond angles around the N atom is 341.6°, showing that the configuration is non-planar. Nevertheless, the short P—N bond [1.661 (3) Å] involves a $p\pi$–$d\pi$ contribution but does not exclude complexation to another molecule of borane, as observed by ^{31}P and ^{11}B NMR spectroscopy (Buono, Brunel, Faure & Pardigon, 1993).

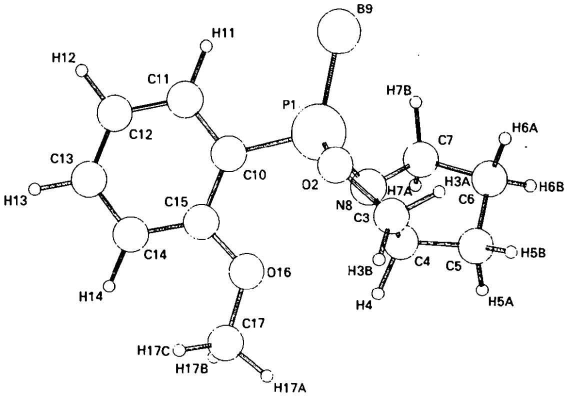


Fig. 1. A view of the molecule (*PLUTO*; Motherwell & Clegg, 1978), showing the atom-numbering scheme. The H atoms of the borane group are not shown.

Experimental

The compound was obtained as described previously (Buono, Brunel, Faure & Pardigon, 1993) and was recrystallized twice from hexane.

Crystal data

$C_{12}H_{19}BNO_2P$
$M_r = 251.1$
Orthorhombic
$P2_12_12_1$
$a = 7.197\ (1)$ Å
$b = 8.270\ (1)$ Å
$c = 22.684\ (1)$ Å
$V = 1350.1\ (2)$ Å^3
$Z = 4$
$D_x = 1.24$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 30 reflections
$\theta = 15$–$35°$
$\mu = 1.706$ mm^{-1}
$T = 291$ K
Parallelepiped
0.37 × 0.20 × 0.18 mm
White

Data collection

Huber four-circle diffractometer with a Rigaku rotating anode
$\theta/2\theta$ scans
Absorption correction: none
2927 measured reflections
2927 independent reflections
2217 observed reflections [$I \geq 2.5\sigma(I)$]
$\theta_{\max} = 67°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 26$
1 standard reflection monitored every 50 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.054$
$wR = 0.057$
$S = 0.6$
2217 reflections
155 parameters
H-atom positions calculated
$w = 1/[\sigma^2(F) + 0.01823F^2]$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.65$ e Å^{-3}
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	0.8725 (1)	0.5396 (1)	0.3829 (1)	0.047 (1)
O2	1.0582 (3)	0.4427 (3)	0.3664 (1)	0.062 (1)
C3	1.0205 (6)	0.2971 (7)	0.3339 (2)	0.087 (2)
C4	0.8188 (6)	0.2494 (5)	0.3430 (2)	0.066 (1)
C5	0.7154 (8)	0.1980 (6)	0.2868 (2)	0.088 (2)
C6	0.6202 (9)	0.3453 (7)	0.2651 (2)	0.100 (2)
C7	0.5659 (5)	0.4376 (5)	0.3209 (2)	0.070 (1)
N8	0.7214 (4)	0.3991 (3)	0.3619 (1)	0.050 (1)
B9	0.8501 (8)	0.7455 (5)	0.3447 (2)	0.073 (1)
C10	0.8756 (4)	0.5518 (3)	0.4622 (1)	0.046 (1)
C11	0.8355 (5)	0.7001 (4)	0.4880 (1)	0.058 (1)
C12	0.8291 (6)	0.7164 (6)	0.5491 (2)	0.071 (1)
C13	0.8630 (6)	0.5864 (5)	0.5834 (1)	0.071 (1)
C14	0.8994 (5)	0.4343 (5)	0.5593 (1)	0.064 (1)
C15	0.9034 (4)	0.4169 (4)	0.4986 (1)	0.050 (1)
O16	0.9337 (4)	0.2752 (3)	0.4710 (1)	0.065 (1)
C17	0.9288 (6)	0.1297 (5)	0.5047 (2)	0.072 (1)

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

O2—P1	1.602 (2)	N8—P1	1.661 (3)
B9—P1	1.917 (4)	C10—P1	1.802 (3)
C3—O2	1.438 (6)	C4—C3	1.518 (7)
C5—C4	1.536 (5)	N8—C4	1.486 (4)
C6—C5	1.482 (8)	C7—C6	1.529 (6)
N8—C7	1.489 (4)	C11—C10	1.389 (4)
C15—C10	1.403 (4)	C12—C11	1.392 (5)
C13—C12	1.350 (6)	C14—C13	1.397 (5)
C15—C14	1.384 (4)	O16—C15	1.346 (4)
C17—O16	1.427 (4)		
N8—P1—O2	97.5 (1)	B9—P1—O2	114.1 (2)
B9—P1—N8	115.9 (2)	C10—P1—O2	104.5 (1)
C10—P1—N8	109.4 (1)	C10—P1—B9	113.7 (2)
C3—O2—P1	112.4 (2)	C4—C3—O2	109.1 (3)
C5—C4—C3	114.9 (4)	N8—C4—C3	105.9 (3)
N8—C4—C5	104.0 (3)	C6—C5—C4	105.8 (4)
C7—C6—C5	104.7 (4)	N8—C7—C6	102.6 (3)
C4—N8—P1	110.9 (2)	C7—N8—P1	121.4 (2)
C7—N8—C4	110.6 (3)	C11—C10—P1	117.9 (2)
C15—C10—P1	123.0 (2)	C15—C10—C11	118.9 (3)
C12—C11—C10	120.8 (3)	C13—C12—C11	119.4 (4)
C14—C13—C12	121.6 (3)	C15—C14—C13	119.2 (3)
C14—C15—C10	120.0 (3)	O16—C15—C10	116.2 (2)
O16—C15—C14	123.9 (3)	C17—O16—C15	118.7 (3)

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H-atom positions were calculated; the values given in the deposit material were computed using *SHELX*76 (Sheldrick, 1976). At the end of the refinement the 901 Friedel pairs of reflections present in the data set were sorted according to $w^{1/2}||F_c(h,k,l)| - |F_c(-h,-k,-l)||$ in order of decreasing magnitude. 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.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71778 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1059]

References

Brunel, J. M., Pardigon, O., Faure, B. & Buono, G. (1992). *J. Chem. Soc. Chem. Commun.* pp. 287–288.
Buono, G., Brunel, J. M., Faure, B. & Pardigon, O. (1993). *Phosphorus Sulfur Silicon*, pp. 43–46.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* Univ. of Cambridge, England.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Acta Cryst. (1994). C**50**, 955–957

4-(4,6-Diméthylpyrid-2-yl)-5-éthyl-3-phényl-4*H*-1,2,4-triazole

N. RODIER

Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques, 5 Rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

S. ROBERT-PIESSARD, J.-M. ROBERT ET G. LE BAUT

Laboratoire de Chimie thérapeutique,
Faculté de Pharmacie, 1 Rue Gaston-Veil,
44035 Nantes CEDEX, France

(*Reçu le* 1 *juin* 1993, *accepté le* 27 *septembre* 1993)

Abstract

The title compound, $C_{17}H_{18}N_4$, is a structural analogue of *N*-(4,6-dimethylpyrid-2-yl)benzamide, with a triazolic heterocycle instead of the amide

Acta Crystallographica Section C
ISSN 0108-2701 ©1994