Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-Benzylamino-5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphorinane 2-oxide

Shi-Guan Wan, Chang Liu, Yan Yu and Xing-Yu Yang*

Institute of Organic Synthesis, Central China Normal University, Wuhan, Hubei 430079, People's Republic of China

Correspondence e-mail: wsg_stone@tom.com

Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.121
Data-to-parameter ratio = 15.8

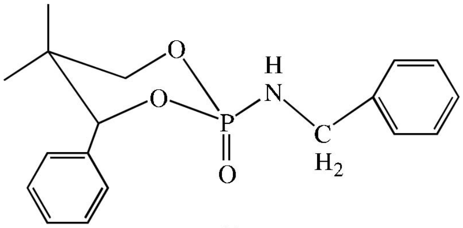
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 June 2005
Accepted 7 June 2005

In the title compound, $C_{18}H_{22}NO_3P$, the molecules are linked by two N—H···N hydrogen bonds to form sheets built from $R_2^2(8)$ rings.

Comment

2-Chloro-1,3,2-dioxaphosphorinane is an important heterocycle which shows good biological and pharmaceutical activity (Wu & Casida, 1995; Yang *et al.*, 1995; Matsumoto *et al.*, 1992; Jacobson & Nguyan, 1991), with some derivatives exhibiting good fungicidal or antitumour properties. As amino groups are often important substituents in structures which show good biological activity, we have embarked upon the preparation of a series of 2-chloro-1,3,2-dioxaphosphorinane derivatives containing an amine unit, including the title compound, (I).



(I)

The crystal structure of (I) (Fig. 1) reveals that the phosphorinane ring in the molecule is in a *trans* conformation. Bond distances and angles in (I) are as expected (Table 1).

N—H···O hydrogen bonds between centrosymmetrically related molecules are present (Fig. 2). The phosphoryl O atom participates in intermolecular N—H···O interactions with a neighbouring molecule to form $R_2^2(8)$ rings (Table 2).

Experimental

Compound (I) was prepared according to the procedure of Shao *et al.* (1995). Benzylamine (5 mmol), triethylamine (5 mmol) and dry dichloromethane (30 ml) were placed in a 100 ml three-necked flask and a solution of 2-chloro-1,3,2-dioxaphosphorinane (5 mmol) in dry dichloromethane (8 ml) was added dropwise over a period of 1 h at room temperature of 298 K. The mixture was then stirred at room temperature for 2–4 h. The solvent was removed under reduced pressure and the residual mixture was washed twice with a little water (2 × 5 ml) and then recrystallized from ethanol–water (2:3) to give compound (I). Suitable crystals were obtained by vapour diffusion of dioxane in dimethylformamide at room temperature (m.p. 453 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3190, 1583, 1453, 1233, 1038, 1018, 988; ^{1}H NMR (DMSO-d$_6$, δ, p.p.m.): 7.40–7.12 (*m*, 10H), 6.13–6.10 (*t*, 1H), 5.35 (*s*, 1H), 4.18–3.85 (*dd*, 4H), 0.85 (*s*, 3H), 0.70 (*s*,

doi:10.1107/S1600536805018003

3H); analysis, calculated for $C_{18}H_{22}NO_3P$: C 65.46, H 6.65, P 9.47%; found: C 65.21, H 6.58, P 9.25%.

Crystal data

$C_{18}H_{22}NO_3P$
$M_r = 331.34$
Triclinic, $P\bar{1}$
$a = 9.1916$ (10) Å
$b = 9.9676$ (11) Å
$c = 10.0055$ (11) Å
$\alpha = 86.049$ (2)°
$\beta = 72.405$ (2)°
$\gamma = 79.700$ (2)°
$V = 859.61$ (16) Å^3

$Z = 2$
$D_x = 1.280$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4140 reflections
$\theta = 2.4$–28.2°
$\mu = 0.17$ mm^{-1}
$T = 292$ (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.934$, $T_{\max} = 0.966$
5225 measured reflections

3313 independent reflections
3056 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 26.0°$
$h = -11 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.121$
$S = 1.07$
3313 reflections
210 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.2466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C7—N1	1.465 (2)	O1—P1	1.5814 (11)
C8—O3	1.448 (2)	O2—P1	1.4650 (13)
C12—O1	1.4592 (19)	O3—P1	1.5888 (12)
N1—P1	1.6013 (15)		
N1—C7—C1	115.78 (15)	C12—O1—P1	118.75 (10)
O3—C8—C9	113.17 (13)	C8—O3—P1	115.72 (10)
O1—C12—C13	106.99 (13)	O2—P1—O1	113.64 (7)
O1—C12—C9	108.87 (12)	O2—P1—O3	114.16 (7)
C7—N1—P1	125.52 (12)	O1—P1—O3	102.17 (6)
C12—O1—P1—N1	−162.65 (11)	C7—N1—P1—O1	55.08 (16)
C8—O3—P1—N1	161.07 (12)	C7—N1—P1—O3	−53.02 (16)
C7—N1—P1—O2	−178.65 (14)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2[i]	0.86	2.04	2.8690 (19)	161

Symmetry code: (i) $-x+1, -y+1, -z+2$.

The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å for phenyl H, 0.97 Å for methylene H and 0.98 Å for H on C12, N—H distances of 0.86 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C,N})$.

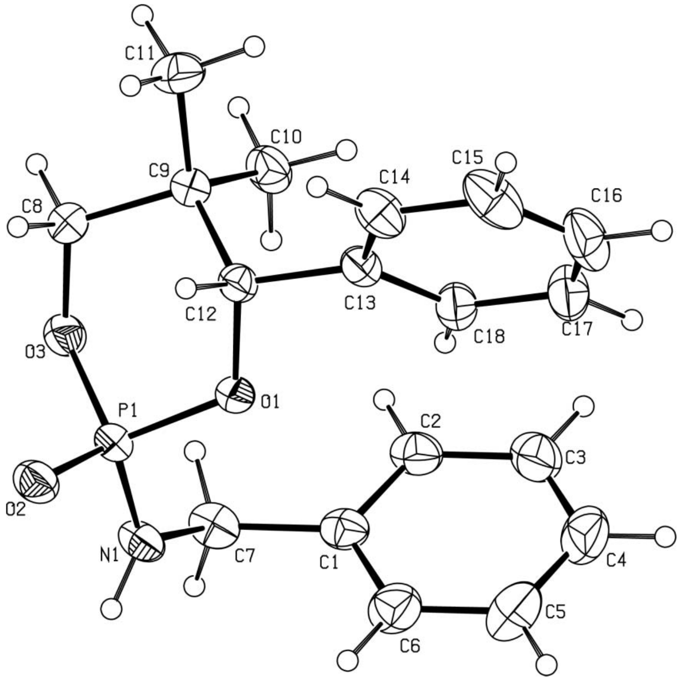


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

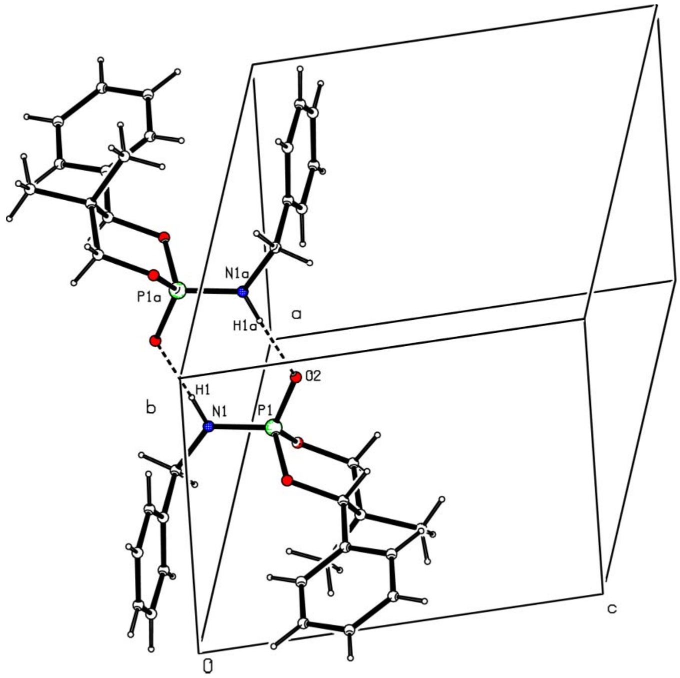


Figure 2
The molecular packing of (I), viewed along the c axis. The hydrogen-bonding interactions are indicated by dashed lines. Atoms labelled with the suffix a are at the symmetry position $-x+1, -y+1, -z+2$.

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

The authors acknowledge financial support from the Science Foundation of Wuhan City (grant No. 20021002052).

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