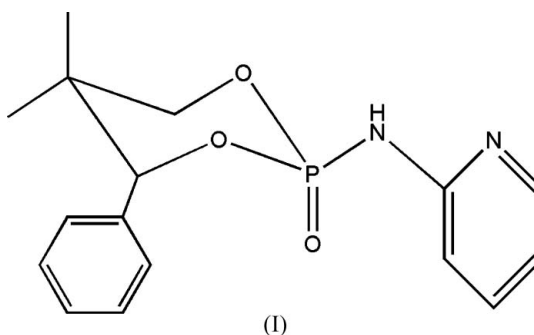


Yan Yu, Chang Liu and
Xingyu Yang*Department of Chemistry, Central China Normal
University, Wuhan, Hubei 430079, People's
Republic of ChinaCorrespondence e-mail:
yuyan801206@yahoo.com.cn

Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.047
 wR factor = 0.120
Data-to-parameter ratio = 15.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.5,5-Dimethyl-2-oxo-4-phenyl-2-(2-pyridyl-
amino)-1,3,2-dioxaphosphorinaneIn the crystal structure of the title compound, $\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_3\text{P}$, molecules are linked by two $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming rings with an $R_2^2(8)$ motif; $\pi-\pi$ stacking also contributes to the packing.Received 27 October 2005
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Comment

2-Chloro-1,3,2-dioxaphosphorinane is an important hetero-
cycle, and its derivatives show good biological and pharma-
ceutical activity (Wolter & Hans, 1985; Jacobson & Nguyan,
1991; Rui *et al.*, 1997; Yang *et al.*, 1991); they also exhibit good
fungicidal or antitumor activities. We report here the crystal
structure of the title compound, (I).

The structure of (I) (Fig. 1) shows that the P atom of the oxodioxaphosphorinane ring carries a pyridylamine substituent, with a phenyl substituent at C7 and two methyl groups on C8. Overall, the oxaphosphorinane ring adopts a chair conformation. In the crystal structure, $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link neighboring molecules, forming inversion-related rings in an $R_2^2(8)$ motif (Fig. 2) (Bernstein *et al.*, 1995). The distance between the inversion-related C1–C6 ring centroids ($\text{Cg}\cdots\text{Cg}^i$) is 3.5921 (13) Å [symmetry code: (i) $2 - x, -y, -z$],

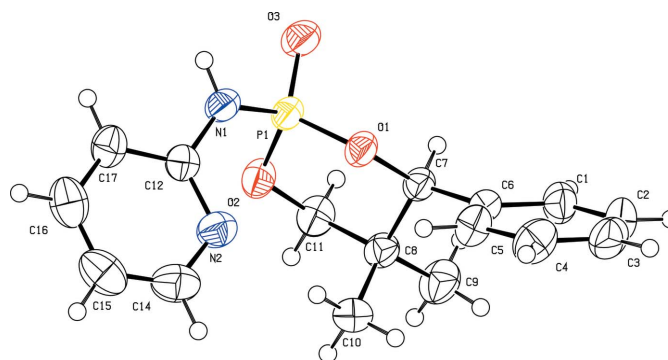


Figure 1
The structure of (I), showing the numbering scheme, with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

suggesting that significant π - π interactions further stabilize the structure.

Experimental

The title compound, (I), was prepared according to the procedure of Maier (1976); suitable crystals were obtained by vapor diffusion of dioxane into a dimethylformamide solution at room temperature (m.p. 515 K). IR (KBr, ν cm^{-1}): 3147, 1597, 1469, 1221, 1043, 1007, 971; ^1H NMR (DMSO- d_6): δ 7.33 (s, 5H), 8.30–6.90 (m, 4H), 9.09 (s, 1H), 4.45–3.95 (dd, 3H), 1.16 (s, 3H), 0.77 (s, 3H); analysis calculated for $\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_3\text{P}$: C 60.37 H 6.02, P 9.73%; found: C 60.21, H 6.12, P 9.60%.

Crystal data

$\text{C}_{16}\text{H}_{19}\text{N}_2\text{O}_3\text{P}$	$D_x = 1.335 \text{ Mg m}^{-3}$
$M_r = 318.30$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3123 reflections
$a = 10.3775$ (9) Å	$\theta = 2.6$ – 27.8°
$b = 7.4406$ (7) Å	$\mu = 0.19 \text{ mm}^{-1}$
$c = 20.6390$ (18) Å	$T = 292$ (2) K
$\beta = 96.231$ (2)°	Block, colorless
$V = 1584.2$ (2) Å ³	$0.30 \times 0.20 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3107 independent reflections
φ and ω scans	2913 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$R_{\text{int}} = 0.023$
$T_{\text{min}} = 0.946$, $T_{\text{max}} = 0.982$	$\theta_{\text{max}} = 26.0^\circ$
23120 measured reflections	$h = -12 \rightarrow 11$
	$k = -7 \rightarrow 9$
	$l = -25 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2 + 0.8267P]$
$R[F^2 > 2\sigma(F^2)] = 0.047$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.12$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
3107 reflections	$\Delta\rho_{\text{min}} = -0.39 \text{ e \AA}^{-3}$
204 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O3}^i$	0.85 (1)	1.98 (1)	2.823 (2)	171 (2)
$\text{C10}-\text{H10B}\cdots\text{N2}$	0.96	2.57	3.522 (3)	174

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

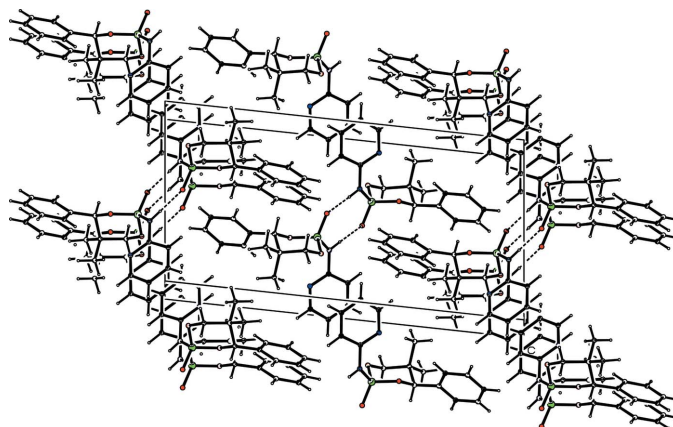


Figure 2

A view of the crystal packing. Hydrogen bonds are shown as dashed lines.

The coordinates of amino atom H1A were refined freely with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic or methylene and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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