

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

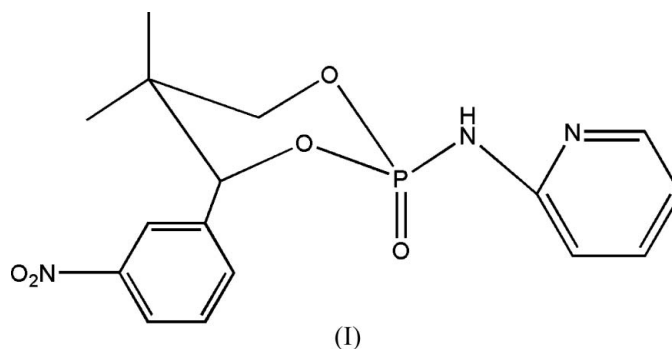
Single-crystal X-ray study  
 $T = 292$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.048  
 $wR$  factor = 0.161  
Data-to-parameter ratio = 14.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.5,5-Dimethyl-4-(3-nitrophenyl)-2-oxo-2-(2-pyridyl-  
amino)-1,3,2-dioxaphosphorinaneIn the crystal structure of the title compound,  $\text{C}_{16}\text{H}_{18}\text{N}_3\text{O}_5\text{P}$ ,  
molecules are linked by two  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds,  
forming rings with an  $R_2^2(8)$  motif.

Received 2 November 2005

Accepted 8 November 2005

Online 26 November 2005

## Comment

The development of intumescent flame-retardant systems  
(IFR) has become an active research field due to their low  
smoke and low toxicity characteristics (Leman & Robertson,  
1978; Halpen & Mott, 1984; Wolter & Hans, 1985). A number  
of phosphorus and nitrogen heterocyclic compounds have  
been shown to have good IFR properties (Jacobson *et al.*,  
1991; Rui *et al.*, 1997; Yang *et al.*, 1991) and we report here  
the molecular structure of one such compound, 5,5-dimethyl-4-(3-  
nitrophenyl)-2-oxo-2-(2-pyridylamino)-1,3,2-dioxaphosphorinane,  
(I).The structure of (I) is shown in Fig. 1. The P atom of the  
oxodioxaphosphorinane ring carries a pyridylamino substituent,  
with a 3-nitrophenyl substituent at C7 and two methyl  
groups on C8. The oxaphosphorinane ring adopts a chair  
conformation. In the crystal structure, molecules form  
hydrogen bonded dimers linked by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen-  
bonded dimers about an inversion centre in an  $R_2^2(8)$  motif  
(Fig. 2).

## 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. 521 K). IR (KBr,  $\nu$   $\text{cm}^{-1}$ ): 3134, 1596, 1469, 1220, 1045, 1006,  
976.  $^1\text{H}$  NMR (DMSO- $d_6$ ):  $\delta$  8.35–6.90 (*m*, 8H), 9.21 (*s*, 1H), 5.25–3.95  
(*dd*, 3H), 1.15 (*s*, 3H), 0.82 (*s*, 3H). Analysis calculated for  
 $\text{C}_{16}\text{H}_{18}\text{N}_3\text{O}_5\text{P}$ : C 52.90, H 4.99, P 8.53%; found: C 52.81, H 5.11, P  
8.40%.

## Crystal data

$C_{16}H_{18}N_3O_5P$   
 $M_r = 363.30$   
 Triclinic,  $P\bar{1}$   
 $a = 8.5644$  (14) Å  
 $b = 9.4816$  (16) Å  
 $c = 11.7155$  (19) Å  
 $\alpha = 79.093$  (3)°  
 $\beta = 73.925$  (3)°  
 $\gamma = 75.318$  (3)°  
 $V = 877.0$  (3) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.376$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 2765  
 reflections  
 $\theta = 2.2$ – $28.1$ °  
 $\mu = 0.19$  mm<sup>-1</sup>  
 $T = 292$  (2) K  
 Block, colorless  
 $0.30 \times 0.20 \times 0.10$  mm

## Data collection

Bruker SMART CCD area-detector  
 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2000)  
 $T_{\min} = 0.946$ ,  $T_{\max} = 0.981$   
 6266 measured reflections

3401 independent reflections  
 2822 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$   
 $\theta_{\text{max}} = 26.0$ °  
 $h = -9 \rightarrow 10$   
 $k = -11 \rightarrow 11$   
 $l = -12 \rightarrow 14$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.161$   
 $S = 1.09$   
 3401 reflections  
 232 parameters

H atoms treated by a mixture of  
 independent and constrained  
 refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.1005P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.24$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.48$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H2\cdots O5^i$	0.86 (1)	1.91 (1)	2.766 (2)	179 (2)

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

The methyl H atoms were constrained to an ideal geometry, with  $C-H = 0.96$  Å and  $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$ , but each group was allowed to rotate freely about its  $C-C$  bond. All aromatic 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  $N-H = 0.86$  Å,  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C,N)$ .

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.

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

## References

- Bruker (1997). SHELXTL. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Bruker (2000). SMART, SAINT and SADABS (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Halpen, Y. & Mott, D. M. (1984). *Ind. Eng. Chem.* **23**, 233–238.

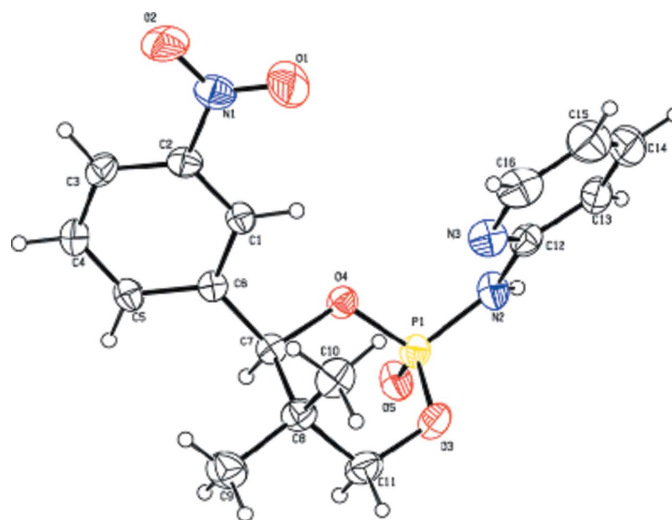


Figure 1

View of (I), showing the labelling of the non-H atoms and 50% probability ellipsoids.

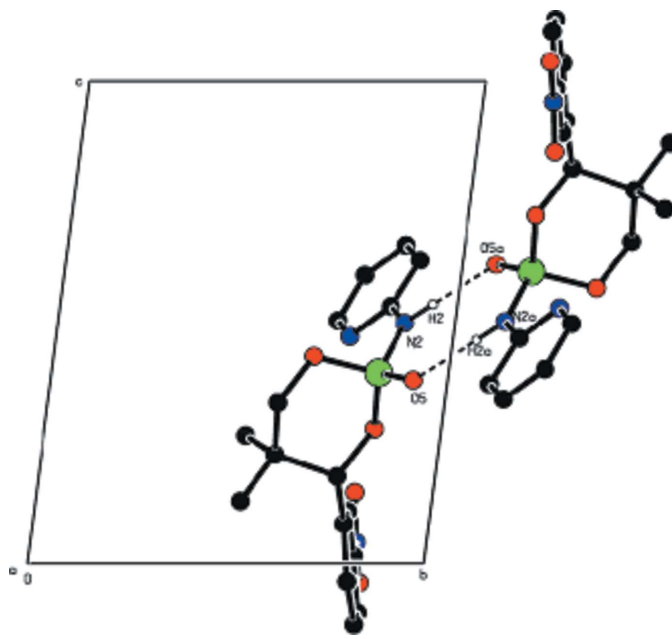


Figure 2

A view of the crystal packing down the  $a$  axis. Hydrogen bonds are shown as dashed lines. [Symmetry code: (a)  $1 - x, 2 - y, 1 - z$ .]

- Jacobson, R. M., Nguyen, L. T. & Ramsay, J. R. (1991). Patent Appl. EP 437 335.  
 Leman, J. D. & Robertson, A. J. (1978). US Patent No. 4 080 501.  
 Maier, L. (1976). *Metal Org. Chem.* **6**, 133–155.  
 Rui, L. S., Guang, F. Y. & Wei, S. M. (1997). *Chin. Chem. Lett.* **8**, 855–858.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
 Wolter, T. H. & Hans, W. (1985). *J. Org. Chem. Lett.* **50**, 4508–4514.  
 Yang, H. Z., Wu, Y. & Zhang, Y. F. (1991). *Chem. J. Chin. Univ.* **12**, 44–46.