Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Chang Liu, Yan Yu and Xingyu Yang*

Department of Chemistry, Central China Normal University, Wuhan, Hubei 430079, People's Republic of China

Correspondence e-mail: yangxy524@126.com

In the crystal structure of the title compound, $C_{18}H_{21}N_2O_3P$, molecules form chains of rings *via* C—H···O and N—H···O hydrogen bonds.

Received 2 November 2005
Accepted 14 December 2005
Online 11 January 2006

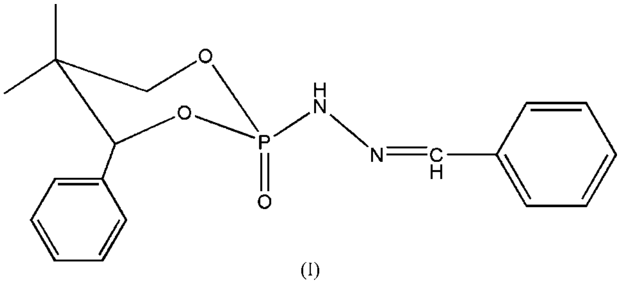
Key indicators

Single-crystal X-ray study
T = 292 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.140
Data-to-parameter ratio = 19.3

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

Comment

2-Chloro-1,3,2-dioxaphosphinane is an important heterocycle as its derivatives can be good intumescent flame retardants (Yang & Lee, 1986; Wang & Shau, 1998; Li & Shi, 2002). These derivatives were found to decompose rapidly in the temperature range 493–753 K. Preliminary tests showed these compounds have high flame retardance activity for alkyd resins and epoxy E-44 varnish. In view of this, we have prepared a series of 2-chloro-1,3,2-dioxaphosphinane derivatives containing the benzoylhydrazone system. We report the crystal structure of the title compound, (I).



(I)

In the molecular structure of (I) (Fig. 1), the dioxaphosphorinane ring adopts a chair conformation. Molecules form $R_2^2(8)$ rings (Bernstein *et al.*, 1995) *via* N—H···O hydrogen bonds. These rings are, in turn, linked into chains *via* weak C—H···O hydrogen bonds (Table 2 and Fig. 2).

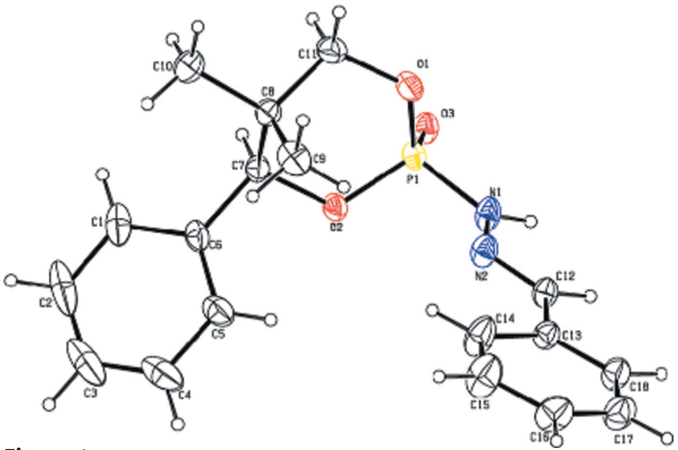


Figure 1
View of (I), shown with 50% probability displacement ellipsoids.

Experimental

The title compound was prepared according to the procedure of Yang (2004). Suitable crystals were obtained from an acetonitrile solution at room temperature (m.p. 453 K). IR (KBr, ν cm^{-1}): 3123, 1606, 1481, 1240, 1096, 1047, 928; ^{1}H NMR (DMSO-d$_6$): δ 9.98–9.90 (*d*, 1H), 7.98 (*s*, 1H), 7.64–7.33 (*m*, 10H), 5.55 (*s*, 1H), 4.43–3.99 (*d*, 2H), 1.12–0.78 (*d*, 6H); analysis calculated for $C_{18}H_{21}N_2O_3P$: C 62.78, H 6.15, P 8.99%; found: C 62.56, H 6.08, P 8.87%.

Crystal data

$C_{18}H_{21}N_2O_3P$
$M_r = 344.34$
Monoclinic, $P2_1/c$
$a = 7.5618$ (9) Å
$b = 17.683$ (2) Å
$c = 14.9324$ (17) Å
$\beta = 100.568$ (2)°
$V = 1962.8$ (4) Å^3
$Z = 4$

$D_x = 1.165$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6100 reflections
$\theta = 2.3$–26.0°
$\mu = 0.16$ mm^{-1}
$T = 292$ (2) K
Block, colorless
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
16102 measured reflections
4284 independent reflections

3365 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\rm max} = 27.0°$
$h = -9 \rightarrow 9$
$k = -22 \rightarrow 22$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.141$
$S = 1.02$
4284 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.073P)^2 + 0.3167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C7—O2	1.4639 (19)	C12—N2	1.268 (2)
C7—C8	1.541 (3)	N1—N2	1.390 (2)
C8—C11	1.521 (3)	N1—P1	1.6230 (16)
C8—C9	1.521 (3)	O1—P1	1.5701 (13)
C8—C10	1.534 (3)	O2—P1	1.5721 (12)
C11—O1	1.453 (2)	O3—P1	1.4641 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1$^{\rm i}$	0.93	2.55	3.429 (3)	158
N1—H1A···O3$^{\rm ii}$	0.85 (1)	2.01 (1)	2.856 (2)	174 (2)

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+1, -z+2$.

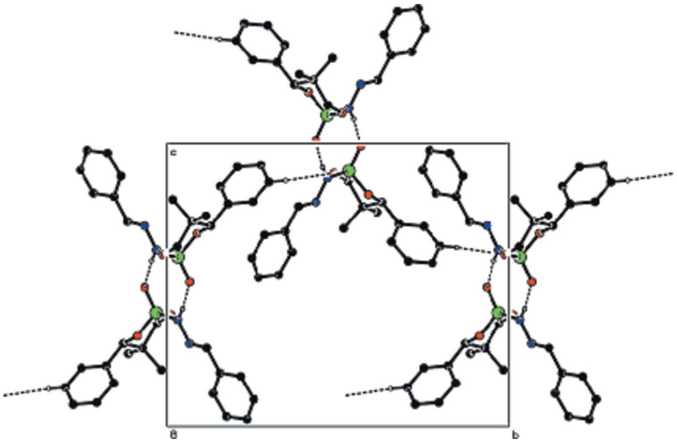


Figure 2
A view of the crystal packing of (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

All H atoms, except for H1A, were included in calculated positions and constrained to ride on their parent atoms, with methyl C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C), methylene C—H = 0.97 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C), methine C—H = 0.98 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C), and aromatic/C12 C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). Atom H1A was located in a difference map and then refined with the restraint N—H = 0.86 (1) Å and the $U_{\rm iso}$ value was set at 1.2$U_{\rm eq}$(N1). In the crystal structure, there are solvent-accesible voids of 98.0 Å^3. These voids may initially have contained solvent, but this has been lost without degradation of the structure. There is no significant residual electron density to suggest the presence of solvent of crystallization.

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* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, X. & Shi, Y. S. (2002). *Polym. Degr. Stab. J.* **77**, 383.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wang, T. S. & Shau, M. D. (1998). *Appl. Sci. J.* **70**, 877.
Yang, C. P. & Lee T. W. (1986). *Appl. Polym. Sci. J.* **32**, 3005–3007.
Yang, X. Y. (2004). *Org. Chem. Lett.* **8**, 793–796.

Acta Cryst. (2006). E**62**, o536–o537 Liu *et al.* · $C_{18}H_{21}N_2O_3P$ **o537**