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5,5-Dimethyl-2-(naphthalen-1-yloxy)-1,3,2-dioxaphosphinane 2-oxide

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
R factor = 0.048
wR factor = 0.116
Data-to-parameter ratio = 17.8

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

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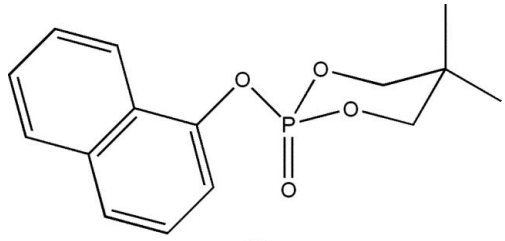
In the crystal structure of the title compound, $C_{15}H_{17}O_4P$, molecules are linked into chains by C—H···O hydrogen bonds.

Comment

2-Chloro-1,3,2-dioxaphosphinane and its derivatives exhibit high flame-retardance (Wang & Shau, 1998; Li *et al.*, 2002) as well as biological and pharmaceutical activity (Jacobson & Nguyan, 1991; Hoeve & Wynberg, 1985). The title compound, (I), is a 2-chloro-1,3,2-dioxaphosphinane derivative containing the naphthyloxy group.



(I)

Bond distances and angles in (I) are as expected, and the dioxaphosphorinane ring adopts a chair conformation (Fig. 1). In the crystal structure, molecules are linked into chains by C—H···O hydrogen bonds (Table 1 and Fig. 2).

Experimental

The title compound was prepared according to the procedure of Raghu & Reddy (1996). Naphthol (0.72 g, 5 mmol), triethylamine (0.51 g, 5 mmol) and dry dichloromethane (25 ml) were placed in a 100 ml three-necked flask and a solution of 5,5-dimethyl-2-chloro-1,3,2-dioxaphosphorinane (0.93 g, 5 mmol) in dry dichloromethane (8 ml) was added dropwise over a period of 1 h at room temperature (298 K). The reaction temperature was raised to 308 K and stirring was continued for 3 h. The solvent was removed under reduced pressure and the residual mixture was washed with water, dried and recrystallized from ethanol to give compound (I). Suitable crystals were obtained from a dichloromethane solution at room temperature (m.p. 395 K).

Crystal data

$C_{15}H_{17}O_4P$
M_r = 292.26
Monoclinic, $P2_1$
a = 6.6577 (11) Å
b = 9.2835 (15) Å
c = 12.1075 (19) Å
β = 99.834 (2)°
V = 737.3 (2) Å^3
Z = 2
D_x = 1.316 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 292 (2) K
Block, colorless
0.20 × 0.20 × 0.10 mm

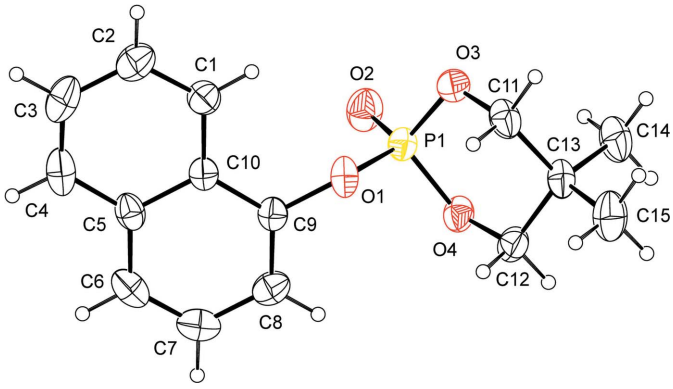


Figure 1
Molecular structure of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms.

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: none
5570 measured reflections
3257 independent reflections
3015 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.5^{\circ}$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.116$
$S = 1.12$
3257 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 0.0777P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Absolute structure: Flack (1983), 1483 Friedel pairs
Flack parameter: 0.05 (11)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2^{i}	0.93	2.53	3.307 (4)	142

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

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C)$. The methyl groups were allowed to rotate about their local threefold axes.

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

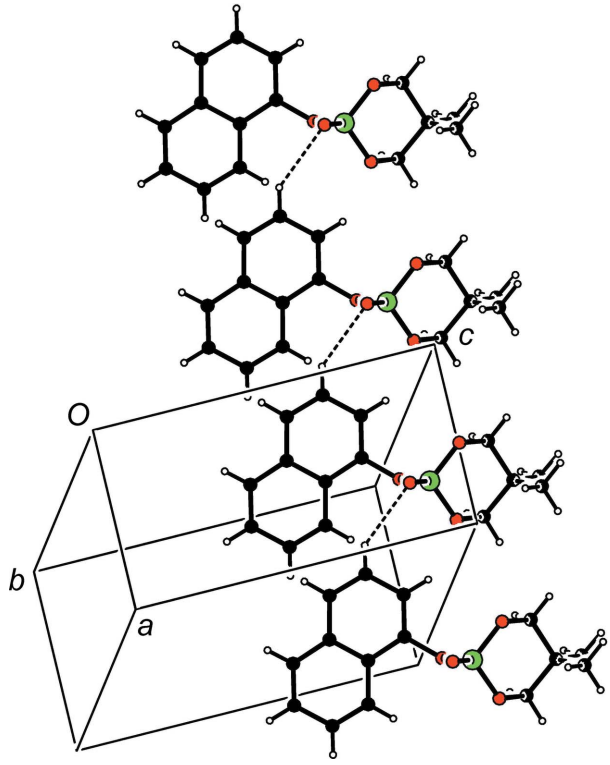


Figure 2
The crystal structure of (I), showing C—H···O hydrogen bonds as dashed lines.

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