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3-(3-Methylphenoxy)-1,5-dihydro-2,4,3-benzodioxaphosphepin-3-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.117
Data-to-parameter ratio = 10.7

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

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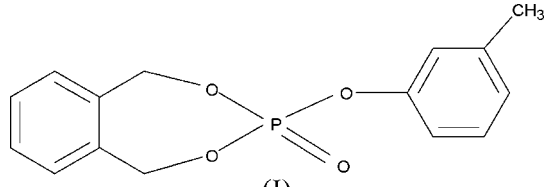
In the title compound, $C_{15}H_{15}O_4P$, the seven-membered phosphepine ring exhibits a twist–chair conformation, with the phosphoryl O atom in an equatorial and the methylphenoxy group in an axial position. The crystal structure is stabilized by both intra- and intermolecular C—H···O hydrogen bonds.

Comment

Much interest in recent years has been shown in structural studies of organophosphorus heterocycles in general and dioxaphosphepine derivatives in particular, because of their applications as insecticides, bactericides, fungicides (Ismail, 1975), pesticides (Fest & Schmidt, 1982) and anticancer agents (Papanastassiou & Bardos, 1962). The title compound, (I) (Fig. 1), is reported here as a continuation of our studies of oxazaphosphorine and dioxaphosphepine compounds (Krishnaiah *et al.*, 2007, 2005; Krishna *et al.*, 2006) to evaluate the effects of substituents on the conformation of the seven-membered ring.



(I)

In compound (I), the dioxaphosphepine ring adopts a twist–chair conformation, with atoms C1/O2/O4/C5 nearly coplanar, and atoms C10/C11 and P3 displaced in opposite directions. The phosphoryl atom O20 occupies an equatorial position and atom O12 an axial position on the phosphepine ring.

The bond lengths and angles in the two P—O—CH_2—C fragments are equal within the limits of error. The endocyclic distances and angles for the dioxaphosphepine ring are consistent with the literature values (Krishna *et al.*, 2006; Grand & Robert, 1978). The exocyclic P—O distances and P—O—C angles are considerably larger than the average endocyclic values. These variations may be due to the bulky methylphenoxy group attached to the phosphepine ring.

In the crystal structure of (I), C18—H18···O20^{i} hydrogen bonds (Fig. 2, Table 2) form parallel chains down the *a* axis.

Experimental

A solution of 3-methylphenylphosphorodichloridate (0.48 g, 2 mmol) in dry tetrahydrofuran (20 ml) was added dropwise over a period of

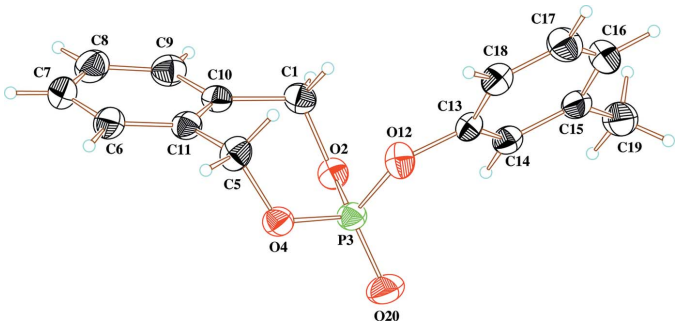


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by circles of arbitrary radii.

20 min at 273 K to a stirred solution of 1,2-benzenedimethanol (0.27 g, 2 mmol) and triethylamine (0.404 g, 4 mmol) in dry tetrahydrofuran (30 ml). After completion of the addition, the temperature was slowly raised to room temperature and the reaction mixture stirred for 4 h. The progress of the reaction was monitored by thin-layer chromatographic analysis (ethyl acetate–hexane 1:2 *v*/*v*). The precipitated triethylamine hydrochloride was filtered off and the filtrate evaporated under vacuum. The residue obtained was washed with water and recrystallized from ethanol to afford 0.38 g (62%) of the pure title compound.

Crystal data

$C_{15}H_{15}O_4P$
M_r = 290.24
Monoclinic, $P2_1/n$
a = 9.4405 (10) Å
b = 15.2017 (16) Å
c = 9.7458 (10) Å
β = 95.782 (2)°
V = 1391.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 293 (2) K
0.38 × 0.34 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.836, T_{max} = 0.959
16173 measured reflections
2457 independent reflections
2304 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.117
S = 1.18
2457 reflections
229 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O12	0.99 (3)	2.57 (2)	3.025 (3)	108.0 (18)
C5—H5B···O12	1.00 (3)	2.48 (3)	2.932 (3)	106.8 (19)
C18—H18···O20^{i}	0.93 (3)	2.51 (3)	3.233 (3)	134 (2)

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

All H atoms were clearly identified in difference Fourier syntheses and refined isotropically (the C—H bond lengths are in the range 0.93 (3)–1.00 (3) Å.), except H atoms of the methyl group, which were positioned geometrically and refined using a riding model, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C).

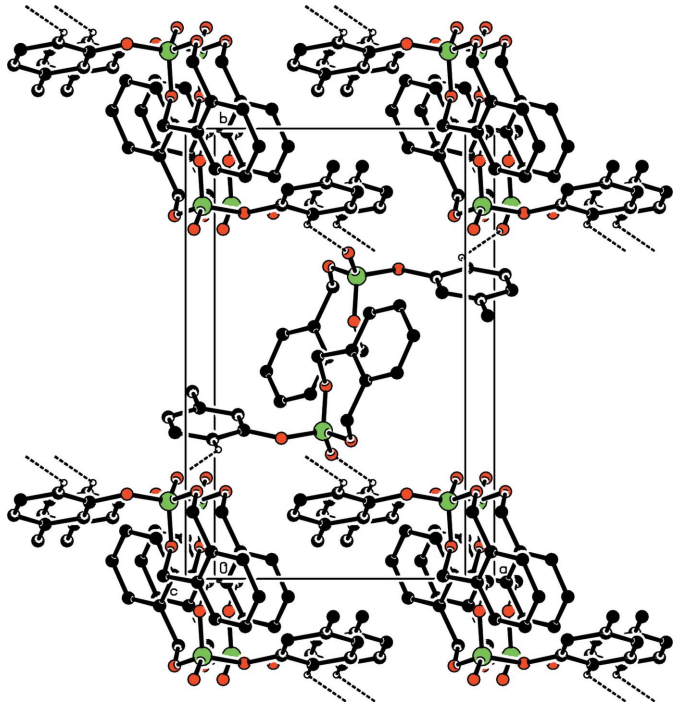


Figure 2
The molecular packing of (I), viewed down the *c* axis. Hydrogen bonds are drawn as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1998); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *PARST* (Nardelli, 1995).

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