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6-Chloro-2-(4-chlorophenoxy)-2-thioxo-4-trichloromethyl-4*H*-1,3,2-benzodioxaphosphorine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.049
wR factor = 0.118
Data-to-parameter ratio = 12.8

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

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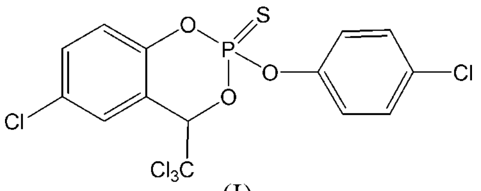
In the title compound, $C_{14}H_8Cl_5O_3PS$, the six-membered dioxaphosphorine ring exhibits a boat conformation with the S atom in an equatorial and the trichloromethyl group in an axial position. The benzene rings are oriented at dihedral angles of 17.77 (15) and 19.47 (19)° with respect to the mean plane of the heterocyclic ring.

Comment

The title compound, (I), has been studied as part of our continuing program of structural studies on organophosphorus compounds (Radha Krishna *et al.*, 2006; Krishnaiah *et al.*, 2005). Various organophosphorus derivatives are used as pesticides (Fest & Schmidt, 1982), insecticides, bactericides, flame retardants, lubricants (Ismail, 1975), antioxidants, and stabilizers in polymers and oils (Spivack, 1982). The title compound also exhibits antifungal activity against *Curvularia lunata*, *Fusarium culmorum* and *Helminthosporium oryzae* (Sankar Reddy *et al.*, 1995).



(I)

In (I), the dioxaphosphorine ring adopts a boat conformation with atoms P2, O3, C9 and C10 coplanar [maximum deviation 0.025 (5) Å for C9 and C10] and with atoms O1 and C4 displaced in the same direction by 0.430 (3) and 0.324 (5) Å, respectively. The S atom at P2 is equatorial, whereas the trichloromethyl group at C4 is in an axial position. The dihedral angles formed by the mean plane through the heterocyclic ring with the C5–C10 and C12–C17 benzene rings are 17.77 (15) and 19.47 (19)°, respectively. Bond lengths and angles of the dioxaphosphorine ring are comparable to those observed in similar compounds (Ślepokura & Lis, 2004; Hommer *et al.*, 1998; Miller *et al.*, 1981; Grand & Robert, 1978). The geometry around the phosphorus atom (Table 1) is in agreement with that found in the literature (Pop *et al.*, 2002; Devendranath Reddy *et al.*, 1995; Jagadeesh Kumar, 1992). The significant difference between the two endocyclic C—O bond distances and P—O—C angles may probably be ascribed to the presence of the fused benzene ring and the CCl_3 group.

The molecular packing exhibits no unusual features, all intermolecular non-bonding contacts being normal van der Waals distances.

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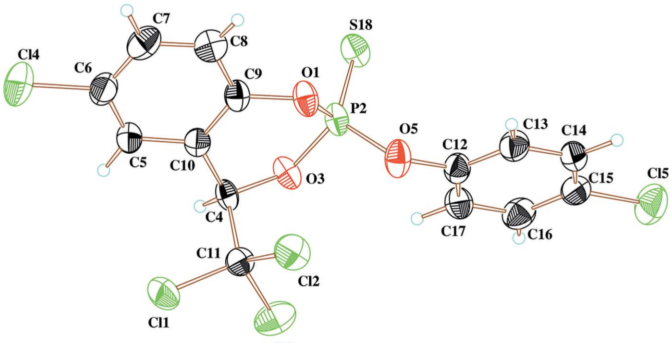

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level. H atoms are represented by circles of arbitrary radii.

Experimental

A solution of 4-chlorophenyl phosphorodichloridate (2.25 g, 0.01 mol) in dry toluene (20 ml) was added dropwise to a stirred solution of 2-(2,2,2-trichloro-1-hydroxyethyl)-4-chlorophenol (2.50 g, 0.01 ml) and triethylamine (2.02 g, 0.02 mol) in dry toluene (80 ml) at room temperature. After completion of the addition, stirring was continued for 5 h at 323–333 K. The formation of the product was ascertained by thin-layer chromatography analysis. Solid triethylamine hydrochloride was separated by filtration and the solvent was evaporated from the filtrate under reduced pressure. The residue obtained was washed with water and recrystallized from dichloromethane to afford the pure title compound (m.p. 442–443 K).

Crystal data

$C_{14}H_8Cl_5O_3PS$
M_r = 464.48
Triclinic, $P\overline{1}$
a = 9.4022 (9) Å
b = 10.6065 (10) Å
c = 10.6577 (9) Å
α = 100.694 (10)°
β = 101.516 (8)°
γ = 112.166 (10)°
V = 923.75 (18) Å^3
Z = 2
D_x = 1.670 Mg m^{-3}
D_m = 1.67 Mg m^{-3}
D_m measured by aqueous KI flotation
Mo $K\alpha$ radiation
μ = 1.00 mm^{-1}
T = 293 (2) K
Block, colourless
0.28 × 0.12 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min}$ = 0.803, $T_{\max}$ = 0.905
3412 measured reflections
3192 independent reflections
1981 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.050
$\theta_{\max}$ = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.118
S = 1.03
3192 reflections
249 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.3459P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P2—O5	1.571 (3)	O3—C4	1.443 (5)
P2—O1	1.584 (3)	O5—C12	1.400 (5)
P2—O3	1.585 (3)	C4—C10	1.493 (6)
P2—S18	1.9025 (18)	C9—C10	1.390 (6)
O1—C9	1.396 (5)		
O5—P2—O3	106.47 (19)	O3—C4—C10	111.7 (4)
O1—P2—O3	104.42 (16)	O3—C4—C11	109.3 (3)
O5—P2—S18	116.56 (13)	C10—C4—C11	114.9 (4)
O1—P2—S18	117.45 (15)	C10—C9—O1	119.8 (4)
C9—O1—P2	119.9 (3)	C9—C10—C4	120.7 (4)
C12—O5—P2	124.6 (3)		
O3—P2—O1—C9	−35.4 (4)	P2—O1—C9—C10	37.7 (6)
O1—P2—O3—C4	1.2 (4)	O1—C9—C10—C4	−1.3 (7)
P2—O3—C4—C10	29.0 (5)	O3—C4—C10—C9	−30.3 (6)

All H atoms were located in difference Fourier syntheses and refined isotropically.

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, 1997); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *PARST* (Nardelli, 1995).

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