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## 3-(3-Chloro-4-fluorophenyl)-2-(2-chlorophenoxy)-3,4-dihydro-2H-1,3,2-benzoxazaphosphorine 2-oxide

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#### Key indicators

Single-crystal X-ray study

$T = 293$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å

$R$  factor = 0.060

$wR$  factor = 0.170

Data-to-parameter ratio = 13.1

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

## 3-(3-Chloro-4-fluorophenyl)-2-(2-chloro- phenoxy)-3,4-dihydro-2H-1,3,2-benz- oxazaphosphorine 2-oxide

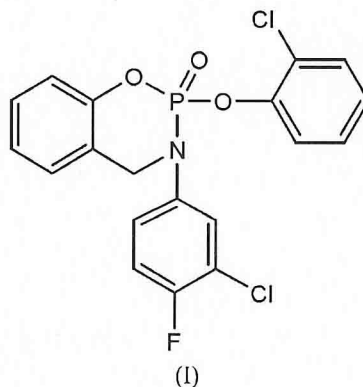
In the title compound,  $\text{C}_{19}\text{H}_{13}\text{Cl}_2\text{FNO}_3\text{P}$ , the six-membered 1,3,2-oxazaphosphorine ring adopts a screw boat conformation, with the phosphoryl O atom in an axial position. The chlorofluorobenzene and chlorophenoxy groups are nearly perpendicular to each other, with a dihedral angle of  $82.53(1)^\circ$  between them, and are equatorially oriented with dihedral angles of  $59.03(1)$  and  $26.15(2)^\circ$ , respectively, with respect to the mean plane of the heterocyclic ring.

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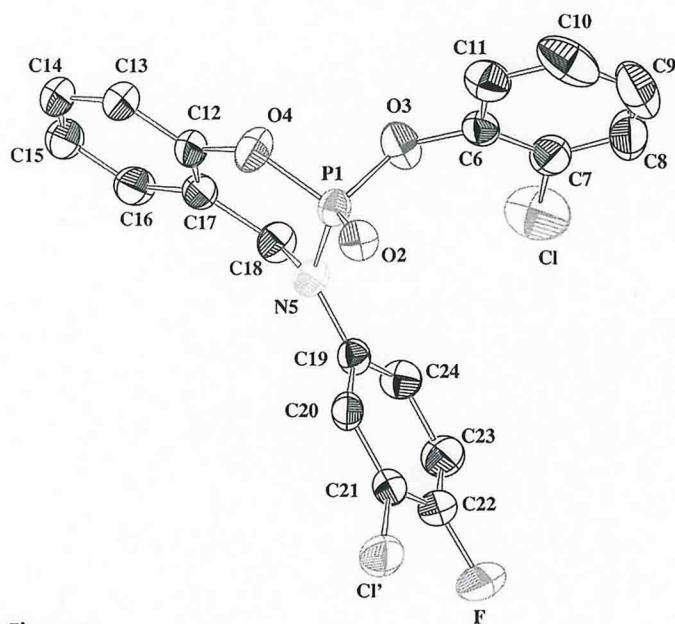
#### Comment

The oxazaphosphorine ring system has recently attracted considerable attention because of its presence in antitumour agents (Stec, 1980). The title compound, (I), was prepared and found to possess significant antimicrobial and insecticidal activity (Kiran *et al.*, 2005). Because of this activity, the X-ray crystal structure of (I) is of great interest to our continuing investigation of heterocyclic phosphorus compounds and is reported here (Fig. 1).

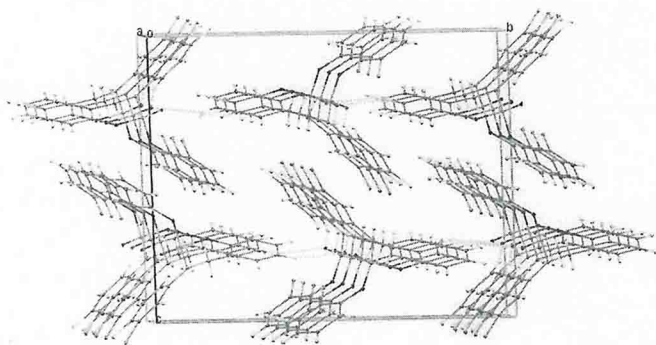


The oxazaphosphorine ring of (I) adopts a screw boat conformation, with atoms O4, C12, C17 and C18 coplanar and atoms P1 and N5 displaced away from this plane in the same direction by  $0.187(1)$  and  $0.750(3)$  Å, respectively. The phosphoryl atom O2 occupies an axial position and atom O3 an equatorial position on the phosphorine ring. The conformation of the phosphorine ring is influenced by the steric interaction between the phosphoryl atom O2 and the chlorofluorobenzene ring. The  $\text{O2}\cdots\text{C20}$  distance is  $3.072(4)$  Å and the aromatic ring is rotated such that the angle between the C12/C17/C18/O4 and chlorofluorobenzene planes is  $59.03(1)^\circ$ .

The  $\text{P}=\text{O}$  distance of  $1.446(2)$  Å and the endo- and exocyclic distances and angles for the oxazaphosphorine unit are consistent with the literature values (Nuti *et al.*, 1988; Subramanian *et al.*, 1989; Selladurai & Subramanian, 1990,



**Figure 1**  
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.



**Figure 2**  
The packing of the molecules of (I) in the unit cell.

1992; Selladurai *et al.*, 1991; Angelov *et al.*, 2002). The C—F [1.360 (4) Å] and average C—Cl [1.729 (3) Å] bond lengths are in good agreement with the values reported by Allen *et al.* (1987).

The chlorofluorobenzene and chlorophenoxy groups are in equatorial positions on the phosphorine ring. These ring planes are perpendicular to each other [dihedral angle 82.53 (1)°] and make dihedral angles of 26.15 (2) and 59.03 (1)°, respectively, with the C12/C17/C18/O4 plane.

In the crystal structure of (I), C15—H15···O2<sup>i</sup> hydrogen bonds link the molecules into chains along *b*, while C24—H24···O2 interactions form an additional, approximately orthogonal, set of chains along *a* (Fig. 2; symmetry codes as in Table 1).

## Experimental

A solution of 2-chlorophenylphosphorodichloridate (0.02 mol) in dry toluene (10 ml) was added dropwise to a stirred solution of 2-(3-chloro-4-fluorophenylamino)methylphenol (0.02 mol), triethylamine

(0.04 mol) and a catalytic amount of dimethylaminopyridine dissolved in dry toluene (20 ml) at 273 K over a period of 20 min. After completion of the addition, the reaction temperature was slowly raised to 323–328 K and maintained at this temperature for 6–7 h. On separation of the triethylamine hydrochloride by filtration, and evaporation of the filtrate under reduced pressure, a solid residue was obtained. This was washed with water and recrystallized from chloroform to afford yellowish transparent single crystals of (I) suitable for X-ray diffraction.

## Crystal data

C<sub>19</sub>H<sub>13</sub>Cl<sub>2</sub>FNO<sub>3</sub>P  
*M<sub>r</sub>* = 424.17  
 Monoclinic, *P*<sub>2</sub><sub>1</sub>/*n*  
*a* = 6.731 (2) Å  
*b* = 18.652 (3) Å  
*c* = 15.045 (3) Å  
 $\beta$  = 101.98 (3)°

*V* = 1847.7 (8) Å<sup>3</sup>  
*Z* = 4  
 Mo *K*α radiation  
 $\mu$  = 0.47 mm<sup>-1</sup>  
*T* = 293 (2) K  
 0.30 × 0.25 × 0.14 mm

## Data collection

Siemens SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 2001)  
*T*<sub>min</sub> = 0.911, *T*<sub>max</sub> = 0.937

4357 measured reflections  
 3221 independent reflections  
 2887 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.039

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$   
 $wR(F^2) = 0.170$   
*S* = 1.09  
 3221 reflections

245 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.73 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.66 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C24—H24···O2 <sup>i</sup>	0.93	2.36	3.201 (4)	151
C15—H15···O2 <sup>ii</sup>	0.93	2.65	3.435 (4)	143

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aromatic H and C—H = 0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for CH<sub>2</sub> groups.

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: ZORTEPII (Zsolnai, 1998); software used to prepare material for publication: enCIFer (Allen *et al.*, 2004) and PARST (Nardelli, 1995).

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