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2-[(4-*tert*-Butylphenoxy)dichloromethyl]-phenyl dichlorophosphonate

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The reaction of phosphorus pentachloride with *p-tert*-butylphenyl salicylate yielded the title compound, $C_{17}H_{17}Cl_4O_3P$, which was originally thought to contain a P—O—C heterocycle. The true molecular structure has now been established by the determination of its crystal structure.

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Key indicators

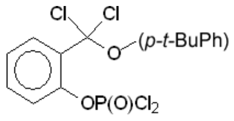
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.069
Data-to-parameter ratio = 26.1

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

Comment

It was demonstrated in a previous report (Pinkus *et al.*, 2004) that the reaction product of phosphorus pentachloride with phenyl salicylate is a non-cyclized structure containing a CCl_2 group and a terminal phosphorus oxide rather than a heterocyclic P—O—C structure, as previously believed.

The reaction of phosphorus pentachloride with *p-tert*-butylphenyl salicylate yields a related product, (1), in a similar reaction. The structure of (1) is nearly identical to that of its phenyl-substituted counterpart, with slight differences in its packing due to the increased bulk of the *tert*-butyl substituent.



(1)

The molecular structure of (1) (Fig. 1) displays bond lengths and angles that are comparable to other dichlorophosphinic acids and α-dichloroethers.

Experimental

Compound (1) was prepared by dropwise addition of *p-tert*-butylphenyl salicylate (5.41 g, 20 mmol) in dry benzene (20 ml) to a stirred solution of phosphorus pentachloride (4.17 g, 20 mmol) in benzene (25 ml). Removal of the solvent *in vacuo* after 3.5 h of stirring left a yellow oil (9.3 g), which solidified on standing. This solid was then dissolved in hot *n*-hexane and filtered through a sintered glass funnel. On standing, (1) (2.61 g, 29.6%) crystallized out of the solution (m.p. 350.0–350.6 K). Analysis calculated for $C_{17}H_{17}Cl_4$ O_3P: C 46.19, H 3.88, Cl 32.08%; found: C 45.72, H 3.81, Cl 31.86% (Cl determined by $AgNO_3$ titration of the hydrolysis product). Diffraction quality crystals of (1) were obtained by dissolving it in hot *n*-hexane, which was allowed to cool slowly.

Crystal data

$C_{17}H_{17}Cl_4O_3P$
$M_r = 442.08$
Monoclinic, $C2/c$
$a = 27.166$ (2) Å
$b = 5.9811$ (5) Å
$c = 24.158$ (2) Å
$\beta = 90.944$ (8)°
$V = 3924.7$ (6) Å^3
$Z = 8$

$D_x = 1.496$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8132 reflections
$\theta = 2.2$–30.5°
$\mu = 0.70$ mm^{-1}
$T = 273$ (2) K
Block, colorless
0.30 × 0.29 × 0.23 mm

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Data collection

Bruker X8 APEX CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1996)
$T_{\min} = 0.818$, $T_{\max} = 0.859$
60 962 measured reflections

5988 independent reflections
5358 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 30.5°$
$h = -38 \rightarrow 38$
$k = -8 \rightarrow 8$
$l = -34 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.069$
$S = 1.07$
5988 reflections
229 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0327P)^2 + 3.2995P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.56$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl2—P1	1.9784 (4)	P1—O1	1.5768 (8)
Cl1—P1	1.9880 (4)	O3—C1	1.376 (1)
P1—O2	1.4577 (9)	O3—C8	1.416 (1)
C1—O3—C8	120.05 (8)		

H atoms were included in calculated positions (C—H = 0.93 Å) and refined as riding; isotropic displacement parameters were fixed [$U_{\rm iso}({\rm H}) = 1.2U_{\rm iso}({\rm C})$].

Data collection: *APEX*2 (Bruker, 2003); cell refinement: *APEX*2; data reduction: *SAINT-Plus* (Bruker, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

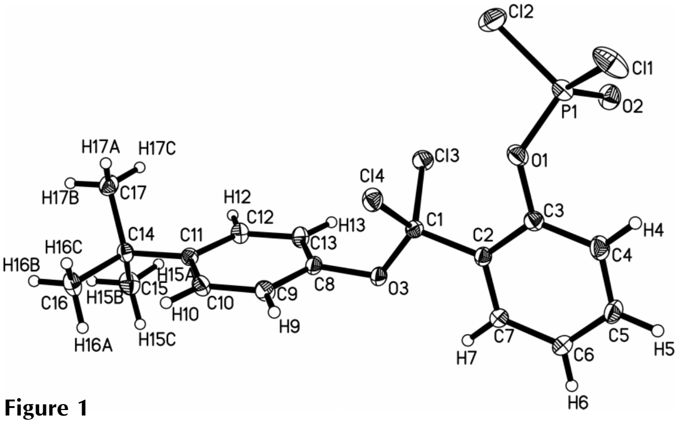

Figure 1
Molecular structure of (1), with displacement ellipsoids drawn at the 50% probability level.

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