Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

O,*O*-Bis(2-*tert*-butylphenyl) chlorothiophosphonate

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Received 27 November 2006
Accepted 20 December 2006

The title compound, $C_{20}H_{26}ClO_2PS$, displays distorted tetrahedral geometry around the P atom, which lies on a crystallographic twofold axis. The S and Cl substituents on the P atom are disordered about the twofold axis. The internal angles in the aromatic rings vary significantly due to the steric influence of the *tert*-butyl groups. Weak intramolecular C—H···O hydrogen bonds generate two *S*(6) motifs. The crystal structure is stabilized only by van der Waals interactions.

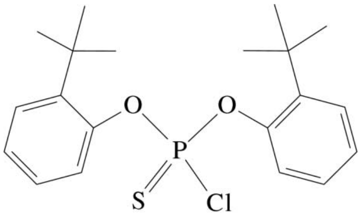
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.087
Data-to-parameter ratio = 16.2

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

Comment

In the course of a systematic structural investigation of thiophosphorus compounds (Odabaşoğlu *et al.*, 2005*a*,*b*, 1999, 1992; Yılmaz *et al.*, 1998; Büyükgüngör *et al.*, 1995; Odabaşoğlu & Gümrükçüoğlu, 1993), the structure of the title compound, (I), was determined (Fig. 1).



(I)

Atom P1, which lies on a crystallographic twofold axis, adopts a distorted tetrahedral geometry. The S1 and Cl1 substituents on P1 are disordered about the twofold axis. The P═S double-bond distance [2.002 (8) Å] is in the range of equivalent distances reported by Schomaker & Stevenson (1941) for PSF_3 (1.85 Å) and for $PSCl_3$ (1.91 Å). The P—O bond length [1.5626 (10) Å] is similar to that of the P—OAr bond for (2*S*)-2-[1-(2,4-difluoro-5-iodophenyl)-2-deoxy-β-D-ribofuranos-5-yloxy]-8-methyl-4*H*-1,3,2-benzodioxa-phosphole 2-oxide [1.565 (4) Å], (Ohkura *et al.*, 2004).

The internal angles in the aromatic rings vary significantly, from 114.18 (14) to 123.90 (13)°. This presumably reflects the steric influence of the *tert*-butyl groups. The rings are nonetheless planar, with a maximum deviation from the plane defined by the six ring atoms of 0.011 (1) Å (for C1). The dihedral angle between symmetry-related benzene rings in (I) is 45.02 (6)°, compared with 41.2 (2)° in *O*,*O*-bis(2-*tert*-butyl-4-methoxyphenyl)chlorothiophosphonate (Odabaşoğlu *et al.*, 2005*a*) and 43.8 (2)° in *O*,*O*-bis(2-*tert*-butyl-4-methylphenyl)-phosphorochlorodithioate (Odabaşoğlu *et al.*,2005*b*). The crystal structure of (I) is stabilized only by van der Waals interactions. There are weak intramolecular hydrogen bonds (Table 1).

doi:10.1107/S1600536806055371

Experimental

Compound (I) was prepared by the method described previously by Odabaşoğlu & Gümrükçüoğlu (1993), using 2-*tert*-butylphenol and $PSCl_3$ as starting materials. Crystals of (I) suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in *n*-hexane (yield 64%; m.p. 383 K). Elemental analysis, calculated: C 60.53, H 6.85%; found: C 60.59, H 6.70%.

Crystal data

$C_{20}H_{26}ClO_2PS$
$M_r = 396.89$
Monoclinic, $C2/c$
$a = 17.8761\ (16)$ Å
$b = 8.2053\ (5)$ Å
$c = 14.5700\ (14)$ Å
$\beta = 105.863\ (7)^\circ$
$V = 2055.7\ (3)$ Å^3
$Z = 4$
$D_x = 1.282$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.38$ mm^{-1}
$T = 296$ K
Prism, colourless
$0.78 \times 0.65 \times 0.45$ mm

Data collection

Stoe IPDSII diffractometer
ω scans
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)
$T_{\min} = 0.771$, $T_{\max} = 0.814$
16049 measured reflections
2010 independent reflections
1796 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$
$\theta_{\max} = 26.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.087$
$S = 1.06$
2010 reflections
124 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.6643P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.11$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C9—H9$A\cdots$O1	0.96	2.37	3.011 (2)	124
C10—H10$B\cdots$O1	0.96	2.36	3.0041 (19)	124

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for aromatic H, and with C—H = 0.96Å and $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(C) for methyl H.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

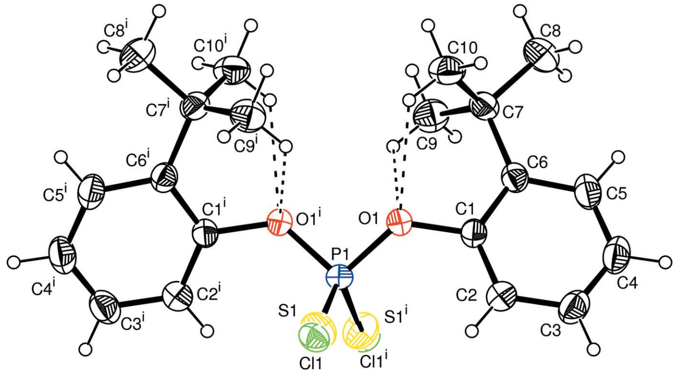


Figure 1
The molecular structure of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids. Intramolecular hydrogen bonds are drawn as dashed lines. [Symmetry code: (i) $-x, y, -z + \frac{1}{2}$].

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant F.279 of the University Research Fund).

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