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O,*O*-Bis(2-*tert*-butyl-4-methoxyphenyl) chlorothiophosphonate

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Key indicators

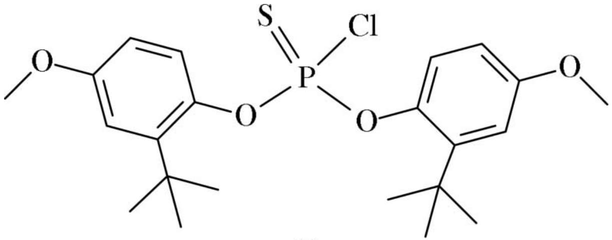
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.007 Å
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 17.1

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

The title compound, $C_{22}H_{30}ClO_4PS$, displays distorted tetrahedral geometry around the P atom. The dihedral angle between the benzene rings is 41.2 (2)°. Because of the steric effects of the *tert*-butyl groups, the aromatic rings have unusual endocyclic angles. The molecules are linked by weak C—H···π interactions.

Comment

In the course of a systematic structural investigation of thiophosphorus compounds, the structure of the title compound, (I), was determined. This investigation brings another contribution to our previous studies (Odabaşoğlu *et al.*, 1992, 1999, 2005; Yılmaz *et al.*, 1998; Büyükgüngör *et al.*, 1995; Odabaşoğlu & Gümrükçuoğlu, 1993).



(I)

An *ORTEP-3* (Farrugia, 1997) view of (I) and a packing diagram are shown in Figs. 1 and 2, respectively. Compound (I) has no classical hydrogen bonds but there are two C—H···π interactions: H31*A*···*Cg*1 = 2.726 Å and C31—H31*A*···*Cg*1 = 144° (*Cg*1 is the centroid of the $C1^i$–$C6^i$ ring), and H11*A*···*Cg*2 = 2.764 Å and C31—H31*A*···*Cg*1 = 141° [*Cg*2 is the centroid of the $C21^{ii}$–$C26^{ii}$ ring; symmetry codes: (i) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$]. The dihedral angle between the benzene rings in (I) is 41.2 (2)°. Selected bond distances and angles for (I) are given in Table 1. As in *O*,*O*-

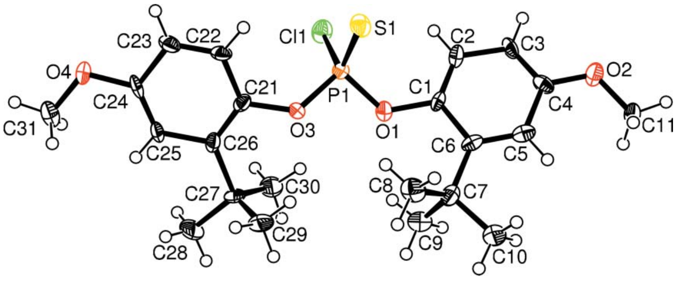


Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

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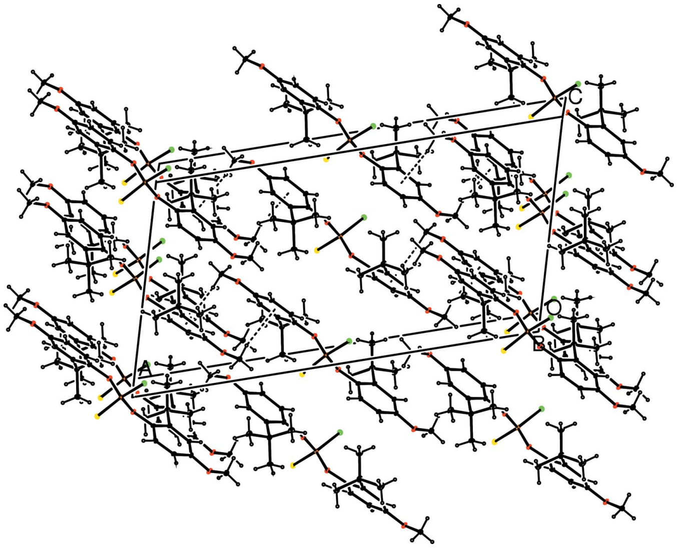

Figure 2
A view of the packing of (I); C—H···π interactions are drawn as dashed lines.

bis(2-*tert*-butyl-4-methoxylphenyl)phosphorochloridothioate (Odabaşoğlu *et al.*, 2005), the C1—C6—C5, C3—C4—C5, C21—C26—C25 and C23—C24—C25 angles and C—C bond distances (Table 1) in the benzene rings are unusual; the diversity of the angles is probably due to steric effects of the *tert*-butyl groups.

The geometry about the P atom corresponds to a distorted tetrahedron, with the O—P—O angles smaller than the ideal tetrahedral angle of 109.5° (Table 1). The P═S and P—O bond lengths agrees with those reported previously (*International Tables for X-ray Crystallography*, 1983, Vol. III; Odabaşoğlu *et al.*, 1992, 2005; Yılmaz *et al.*, 1998; Büyükgüngör *et al.*, 1995).

Experimental

Compound (I) was prepared by the method of Odabaşoğlu *et al.* (2005), using 2-*tert*-butyl-4-methoxyphenol 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 77%, m.p. 423 K). Analysis calculated: C 57.83, H 6.57%; found: C 57.36, H 6.38%.

Crystal data

$C_{22}H_{30}ClO_4PS$
$M_r = 456.94$
Monoclinic, *Cc*
$a = 23.592$ (3) Å
$b = 8.3111$ (6) Å
$c = 12.5067$ (14) Å
$\beta = 105.740$ (9)°
$V = 2360.3$ (4) Å^3
$Z = 4$
$D_x = 1.286$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16485 reflections
$\theta = 1.7$–26.7°
$\mu = 0.34$ mm^{-1}
$T = 150$ K
Prism, colourless
0.64 × 0.59 × 0.55 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.824$, $T_{\max} = 0.869$
16485 measured reflections
4630 independent reflections
4160 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 26.0$°
$h = -28 \rightarrow 28$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.074$
$S = 1.12$
4630 reflections
270 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 1.103P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0038 (3)
Absolute structure: Flack (1983), with 2303 Friedel pairs
Flack parameter = 0.51 (1)

Table 1
Selected geometric parameters (Å, °).

C1—C6	1.373 (6)	C22—C23	1.465 (6)
C1—O1	1.402 (5)	C23—C24	1.330 (6)
C1—C2	1.446 (5)	C24—C25	1.457 (5)
C2—C3	1.347 (6)	C25—C26	1.355 (6)
C3—C4	1.463 (6)	O1—P1	1.583 (3)
C4—C5	1.338 (6)	O3—P1	1.564 (3)
C5—C6	1.412 (5)	P1—S1	1.9740 (18)
C21—C22	1.372 (5)	P1—Cl1	1.9848 (18)
C21—O3	1.451 (5)		
C6—C1—O1	121.8 (4)	C26—C25—C24	118.0 (4)
C6—C1—C2	121.0 (4)	C25—C26—C21	119.3 (4)
O1—C1—C2	117.2 (4)	C21—O3—P1	126.4 (3)
C3—C2—C1	117.9 (4)	C24—O4—C31	115.9 (3)
C2—C3—C4	120.7 (4)	O3—P1—O1	97.13 (5)
C5—C4—C3	120.1 (4)	O3—P1—S1	112.66 (14)
C4—C5—C6	120.3 (4)	O1—P1—S1	112.02 (15)
C1—C6—C5	119.9 (4)	O3—P1—Cl1	110.44 (15)
C22—C21—O3	118.0 (3)	O1—P1—Cl1	110.26 (14)
C23—C24—C25	120.7 (4)	S1—P1—Cl1	113.24 (2)

All H atoms were refined using a riding model, with C—H = 0.93 Å [$U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom)] for aromatic H atoms and C—H = 0.96 Å [$U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(parent atom)] for methyl H atoms. The crystal investigated was an inversion twin. The ratio of the two twin components refined to 0.51 (1):0.49 (1).

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).

References

Büyükgüngör, O., Odabaşoğlu, M., Gümrükçüoğlu, İ. E., Eichhorn, E. & Mattern, G. (1995). *Acta Cryst.* C**51**, 1207–1209.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Odabaşoğlu, M., Büyükgüngör, O. & Albayrak, Ç. (2005). *Acta Cryst.* E**61**, o2525–o2527.
Odabaşoğlu, M. & Gümrükçüoğlu, İ. E. (1993). *Doğa Turk. J. Chem.* **17**, 29–32.
Odabaşoğlu, M., Gümrükçüoğlu, İ. E. & Taprama, R. (1999). *Phosphorus, Sulfur and Silicon*, **152**, 27–34.

Odabaşoğlu, M., Gümrükçüoğlu, İ. E., Yılmaz, V. T. & Howie, R. A. (1992). *Doğa Turk. J. Chem.* **16**, 293–298.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

Yılmaz, V. T., Odabaşoğlu, M. & Howie, R. A. (1998). *Doğa Turk. J. Chem.* **22**, 409–413.

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O,*O*-Bis(2-*tert*-butyl-4-methoxyphenyl) chlorothiophosphonate. Corrigendum.

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.028
wR factor = 0.073
Data-to-parameter ratio = 12.1

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

In the original report by Odabaşoğlu, Büyükgüngör & Albayrak [*Acta Cryst*. (2005), E**61**, o2528–o2530], the structure was reported in the incorrect space group *Cc*. The structure is now reported as disordered in the correct space group *C*2/*c*. The P atom lies on a twofold rotation axis.A revised description of the hydrogen bonding is also given.

Comment

An *ORTEP-3* (Farrugia, 1997) view of (I) and a packing diagram are shown in Figs. 1 and 2, respectively. The P atom lies on a twofold rotation axis, eading to disorder of the Cl and S atoms. Compound (I) has no classical hydrogen bonds, but there are two C—H$\cdots\pi$ interactions: H8$A\cdots Cg$1= 3.12 (2) Å and C8—H8$A\cdots Cg$1 = 122.8 (2)° (Cg1 is the centroid of the C1^{ii}–C6^{ii} ring), and H11$A\cdots Cg$2 = 2.71 (2) Å and C11—H11$A\cdots Cg$2 = 142.7 (1)° (Cg2 is the centroid of the C1^{iii}–C6^{iii}

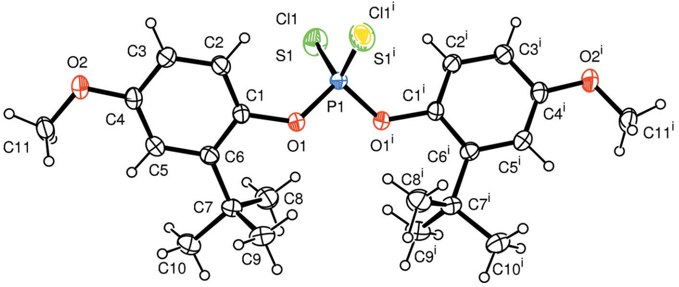

Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids·[Symmetry code: (i) $-x + 1$, y, $-z + \frac{1}{2}$]

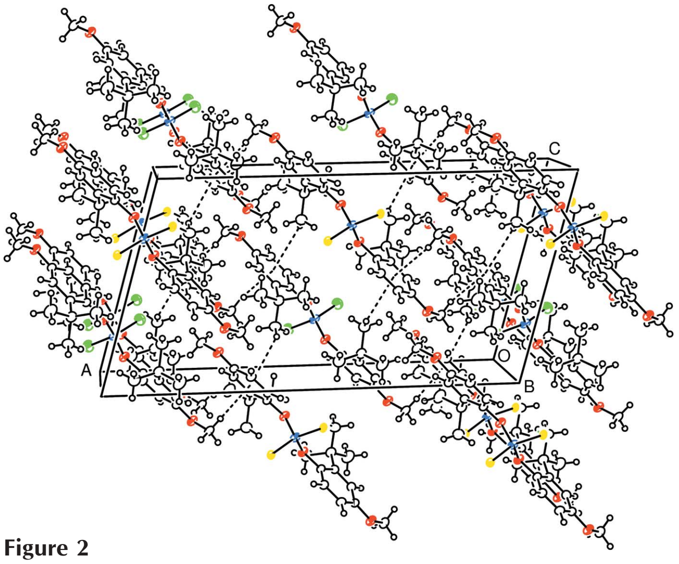

Figure 2
A view of the packing of (I); C—H$\cdots\pi$ interactions are drawn as dashed lines.

e4 Odabaşoğlu *et al.* • $C_{22}H_{30}ClO_4PS$ doi:10.1107/S1600536805027789 *Acta Cryst.* (2005). E**61**, e4–e5

ring) [symmetry codes: (ii) $x, 1-y, \frac{1}{2}+z$; (iii) $\frac{1}{2}-x, \frac{1}{2}-y, -z$]. The dihedral angle between the symmetry-related benzene rings is 41.2 (2)°. Selected bond distances and angles are given in Table 1.

Experimental

Crystal data

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$M_r = 456.94$
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Cell parameters from 16485 reflections
$\theta = 1.7$–26.7°
$\mu = 0.34$ mm^{-1}
$T = 150$ K
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$R_{\rm int} = 0.032$
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$h = -28 \rightarrow 28$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.073$
$S = 1.08$
2327 reflections
192 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 1.6163P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.3946 (18)	C4—C5	1.4017 (19)
C1—C6	1.4086 (17)	C5—C6	1.4088 (18)
C1—O1	1.4226 (14)	O1—P1	1.5750 (9)
C2—C3	1.3860 (18)	P1—S1^{i}	1.9791 (4)
C3—C4	1.3979 (18)	P1—Cl1	1.9791 (4)
C2—C1—C6	123.25 (11)	C4—C5—C6	121.99 (12)
C2—C1—O1	119.13 (11)	C1—C6—C5	115.48 (11)
C6—C1—O1	117.58 (11)	O1—P1—O1^{i}	97.08 (7)
C3—C2—C1	119.77 (12)	O1—P1—S1^{i}	111.27 (4)
C2—C3—C4	119.11 (12)	O1—P1—Cl1	111.44 (3)
C3—C4—C5	120.39 (11)	S1^{i}—P1—Cl1	113.26 (3)

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

All H atoms were refined freely. Atoms S1 and Cl1 were assigned to the same atomic site and refined freely with the same atomic coordinates and with fixed site-occupancy factors of 0.5.

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).

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

Acta Cryst. (2005). E**61**, e4–e5 Odabaşoğlu *et al.* • $C_{22}H_{30}ClO_4PS$ **e5**