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Diphenyl chlorothiophosphonate

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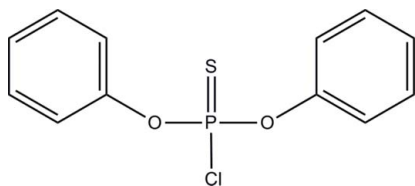
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Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.026; wR factor = 0.059; data-to-parameter ratio = 19.2.

The complete molecule of the title compound, $\text{C}_{12}\text{H}_{10}\text{ClO}_2\text{PS}$, is generated by crystallographic mirror symmetry, with the P, S and Cl atoms lying on the mirror plane. The resulting PO_2SCL tetrahedron is significantly distorted [$\text{O}-\text{P}-\text{O} = 96.79$ (9)°]. The crystal packing exhibits no directional interactions.

Related literature

For the application of related compounds as pesticides, see: Greenhalgh *et al.* (1980); Um *et al.* (2003).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{10}\text{ClO}_2\text{PS}$ $M_r = 284.68$

Orthorhombic, $Pmn2_1$
 $a = 14.9779$ (18) Å
 $b = 7.3709$ (10) Å
 $c = 5.8157$ (10) Å
 $V = 642.06$ (16) Å³

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.57$ mm⁻¹
 $T = 113$ K
 $0.26 \times 0.20 \times 0.16$ mm

Data collection

Rigaku Saturn724 CCD diffractometer
 Absorption correction: multi-scan (*CrystalClear*; Rigaku/MS, 2005)
 $T_{\min} = 0.866$, $T_{\max} = 0.914$

6462 measured reflections
 1590 independent reflections
 1422 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.059$
 $S = 1.01$
 1590 reflections
 83 parameters
 1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³
 Absolute structure: Flack (1983),
 716 Friedel pairs
 Flack parameter: -0.25 (7)

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku/MS, 2005).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6430).

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supplementary materials

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Diphenyl chlorothiophosphonate

Y.-F. Zhang, P.-H. Zhao, J.-J. Liu and G.-Z. Zhao

Experimental

Triethylamine (127.0 mmol) were added to the dichloromethane solution (80.0 ml) of phenol (120.0 mmol) while stirring. Thiophosphoryl chloride (60.0 mmol) was slowly dropwise added to the above solution, and then the reaction mixture was refluxed. After the reaction was completed, it is cooled to room temperature. The reaction mixture was washed with water and brine, respectively. The separated organic phase was dried with anhydrous sodium sulfate, and then the solvents were evaporated thoroughly *in vacuo*. The obtained crude was separated through column chromatography on silica gel to give the white product. Colourless prisms of the title compound were obtained by slow evaporation of the dichloromethane/n-hexane solutions at room temperature. ^{31}P NMR(161.9 MHz, CDCl_3 , TMS): 58.73 (s) p.p.m..

Refinement

All the H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

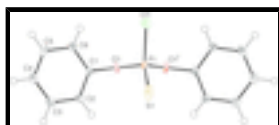


Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids. Symmetry code: (i) $-x, y, z$.

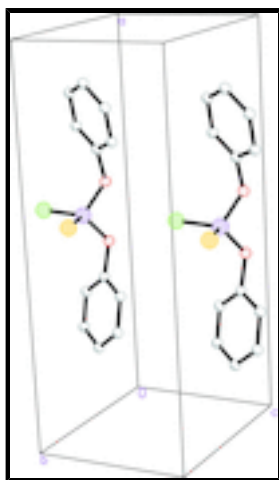


Fig. 2. The crystal packing for (I).

Diphenyl chlorothiophosphonate

Crystal data

$\text{C}_{12}\text{H}_{10}\text{ClO}_2\text{PS}$

$D_x = 1.473 \text{ Mg m}^{-3}$

supplementary materials

$M_r = 284.68$

Orthorhombic, $Pmn2_1$

$a = 14.9779$ (18) Å

$b = 7.3709$ (10) Å

$c = 5.8157$ (10) Å

$V = 642.06$ (16) Å³

$Z = 2$

$F(000) = 292$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2338 reflections

$\theta = 2.7$ – 28.0°

$\mu = 0.57$ mm⁻¹

$T = 113$ K

Prism, colorless

$0.26 \times 0.20 \times 0.16$ mm

Data collection

Rigaku Saturn724 CCD
diffractometer

Radiation source: rotating anode
multilayer

Detector resolution: 14.22 pixels mm⁻¹
 ω and ϕ scans

Absorption correction: multi-scan
(*CrystalClear*; Rigaku/MS, 2005)

$T_{\min} = 0.866$, $T_{\max} = 0.914$

6462 measured reflections

1590 independent reflections

1422 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

$\theta_{\max} = 27.9^\circ$, $\theta_{\min} = 2.7^\circ$

$h = -18 \rightarrow 19$

$k = -9 \rightarrow 9$

$l = -7 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.059$

$S = 1.01$

1590 reflections

83 parameters

1 restraint

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.33$ e Å⁻³

$\Delta\rho_{\min} = -0.28$ e Å⁻³

Extinction correction: *SHELXL97* (Sheldrick, 2008),

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.034 (2)

Absolute structure: Flack (1983), **716 Friedel pairs**

Flack parameter: -0.25 (7)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -

factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R - factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.0000	0.74395 (10)	0.33956 (11)	0.01650 (17)
Cl1	0.0000	0.78689 (13)	0.00139 (10)	0.0391 (3)
S1	0.0000	0.49265 (10)	0.42063 (18)	0.0294 (2)
O1	0.07867 (7)	0.86334 (15)	0.4369 (2)	0.0162 (3)
C1	0.16873 (10)	0.8018 (2)	0.4313 (4)	0.0137 (4)
C2	0.20060 (11)	0.7047 (2)	0.6162 (3)	0.0166 (4)
H2	0.1623	0.6729	0.7400	0.020*
C3	0.29001 (12)	0.6542 (3)	0.6177 (3)	0.0196 (4)
H3	0.3137	0.5884	0.7442	0.024*
C4	0.34463 (11)	0.7004 (2)	0.4335 (4)	0.0191 (4)
H4	0.4056	0.6646	0.4337	0.023*
C5	0.31099 (12)	0.7976 (3)	0.2507 (3)	0.0203 (5)
H5	0.3491	0.8291	0.1264	0.024*
C6	0.22133 (11)	0.8504 (2)	0.2460 (3)	0.0166 (4)
H6	0.1975	0.9170	0.1204	0.020*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0102 (3)	0.0226 (4)	0.0167 (4)	0.000	0.000	-0.0060 (3)
Cl1	0.0210 (4)	0.0820 (7)	0.0144 (4)	0.000	0.000	-0.0040 (4)
S1	0.0152 (3)	0.0179 (3)	0.0549 (5)	0.000	0.000	-0.0040 (4)
O1	0.0084 (6)	0.0174 (7)	0.0228 (7)	-0.0005 (5)	-0.0024 (5)	-0.0040 (6)
C1	0.0078 (8)	0.0143 (9)	0.0188 (9)	-0.0010 (7)	-0.0003 (8)	-0.0051 (8)
C2	0.0150 (9)	0.0197 (12)	0.0151 (11)	-0.0015 (8)	0.0002 (7)	0.0007 (8)
C3	0.0181 (9)	0.0188 (11)	0.0219 (11)	0.0012 (8)	-0.0054 (8)	0.0023 (9)
C4	0.0125 (9)	0.0180 (9)	0.0267 (11)	0.0020 (7)	-0.0026 (8)	-0.0029 (9)
C5	0.0137 (10)	0.0216 (11)	0.0255 (11)	-0.0039 (8)	0.0076 (7)	-0.0015 (8)
C6	0.0162 (10)	0.0163 (10)	0.0174 (10)	-0.0016 (8)	-0.0050 (7)	0.0021 (8)

Geometric parameters (\AA , $^\circ$)

P1—O1 ⁱ	1.5758 (12)	C2—H2	0.9500
P1—O1	1.5758 (12)	C3—C4	1.390 (3)
P1—S1	1.9114 (11)	C3—H3	0.9500
P1—Cl1	1.9920 (9)	C4—C5	1.378 (3)
O1—C1	1.4234 (17)	C4—H4	0.9500
C1—C2	1.377 (3)	C5—C6	1.398 (2)
C1—C6	1.382 (3)	C5—H5	0.9500
C2—C3	1.390 (2)	C6—H6	0.9500
O1 ⁱ —P1—O1	96.79 (9)	C2—C3—C4	119.77 (17)
O1 ⁱ —P1—S1	116.91 (6)	C2—C3—H3	120.1

supplementary materials

O1—P1—S1	116.91 (6)	C4—C3—H3	120.1
O1 ⁱ —P1—C11	105.42 (6)	C5—C4—C3	120.46 (16)
O1—P1—C11	105.42 (6)	C5—C4—H4	119.8
S1—P1—C11	113.42 (6)	C3—C4—H4	119.8
C1—O1—P1	121.50 (11)	C4—C5—C6	120.71 (18)
C2—C1—C6	123.07 (15)	C4—C5—H5	119.6
C2—C1—O1	118.43 (16)	C6—C5—H5	119.6
C6—C1—O1	118.41 (17)	C1—C6—C5	117.43 (17)
C1—C2—C3	118.56 (16)	C1—C6—H6	121.3
C1—C2—H2	120.7	C5—C6—H6	121.3
C3—C2—H2	120.7		
O1 ⁱ —P1—O1—C1	169.66 (11)	C1—C2—C3—C4	-0.7 (3)
S1—P1—O1—C1	44.80 (16)	C2—C3—C4—C5	0.7 (3)
C11—P1—O1—C1	-82.25 (14)	C3—C4—C5—C6	-0.5 (3)
P1—O1—C1—C2	-90.03 (19)	C2—C1—C6—C5	-0.3 (3)
P1—O1—C1—C6	93.23 (17)	O1—C1—C6—C5	176.31 (15)
C6—C1—C2—C3	0.5 (3)	C4—C5—C6—C1	0.2 (3)
O1—C1—C2—C3	-176.05 (16)		

Symmetry codes: (i) $-x, y, z$.

Fig. 1

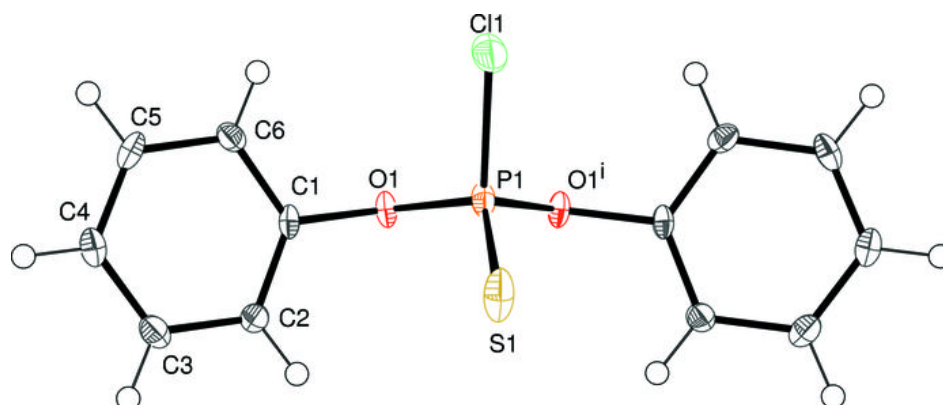


Fig. 2

