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Diphenyl chlorothiophosphonate

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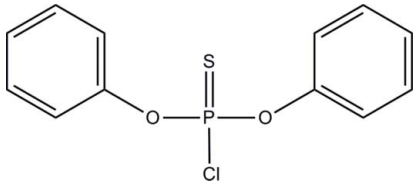
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.059; data-to-parameter ratio = 19.2.

The complete molecule of the title compound, $C_{12}H_{10}ClO_2PS$, 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 [O—P—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

$C_{12}H_{10}ClO_2PS$ 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) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.57 mm^{-1}
T = 113 K
0.26 × 0.20 × 0.16 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.866, T_{max} = 0.914
6462 measured reflections
1590 independent reflections
1422 reflections with $I > 2\sigma(I)$
R_{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
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 716 Friedel pairs
Flack parameter: −0.25 (7)

Data collection: *CrystalClear* (Rigaku/MSC, 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/MSC, 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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S1. Experimental

Triethylamine (127.0 mmol) were added to the dichloromethane solution (80.0 ml) of phenol (120.0 mmol) while stirring. Thiophosphory chloride (60.0 mmol) was slowly dropwise added to the above solution, and then the rection 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 evporated 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..

S2. Refinement

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

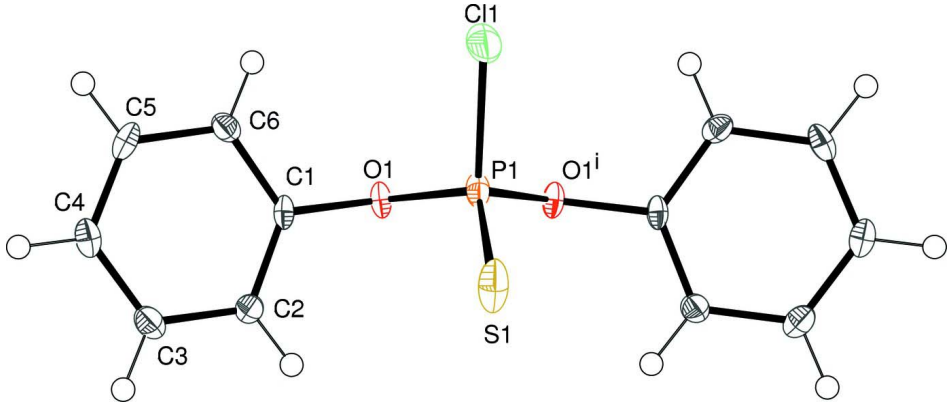

Figure 1

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

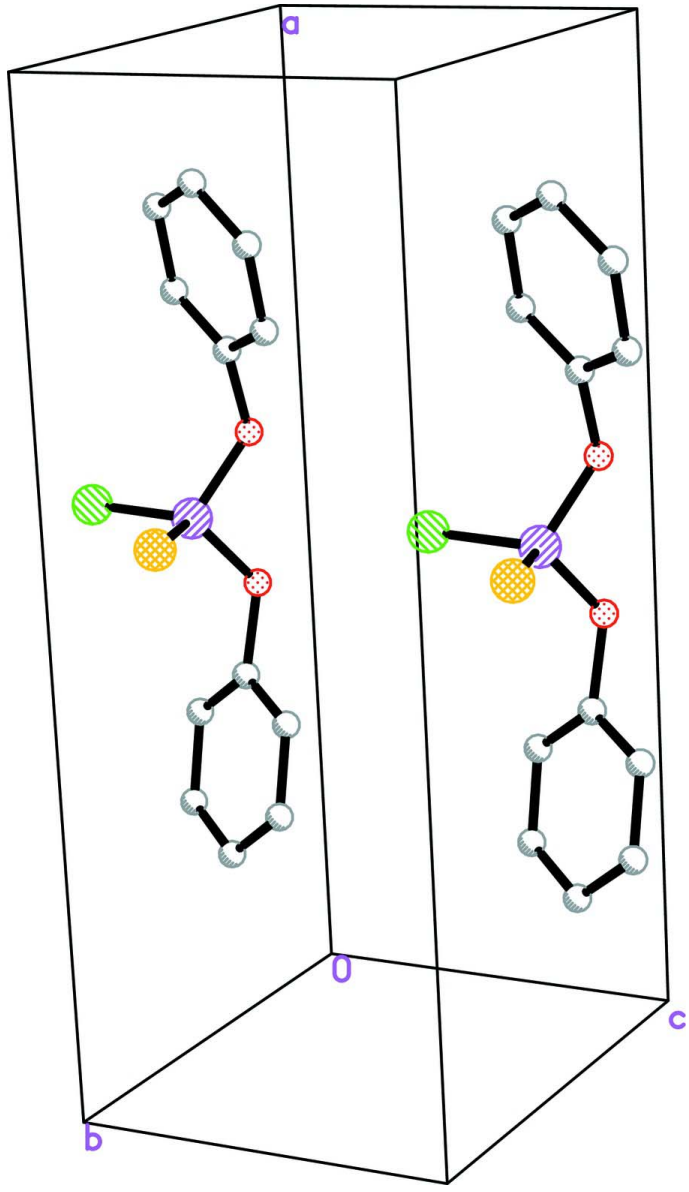

Figure 2

The crystal packing for (I).

Diphenyl chlorothiophosphonate

Crystal data

$C_{12}H_{10}ClO_2PS$
$M_r = 284.68$
Orthorhombic, $Pmn2_1$
$a = 14.9779\ (18)$ Å
$b = 7.3709\ (10)$ Å
$c = 5.8157\ (10)$ Å
$V = 642.06\ (16)$ Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.473$ Mg m^{-3}
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 × 0.20 × 0.16 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.22 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.866, T_{max} = 0.914
6462 measured reflections
1590 independent reflections
1422 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.9°, θ_{min} = 2.7°
h = −18→19
k = −9→9
l = −7→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), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.034 (2)
Absolute structure: Flack (1983), **716 Friedel pairs**
Absolute structure 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 (Å^2)

	x	y	z	U_{iso}*/U_{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 (Å²)

	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 (Å, º)

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
O1—P1—S1	116.91 (6)	C4—C3—H3	120.1
O1^{i}—P1—Cl1	105.42 (6)	C5—C4—C3	120.46 (16)
O1—P1—Cl1	105.42 (6)	C5—C4—H4	119.8
S1—P1—Cl1	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)
Cl1—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 code: (i) $-x, y, z$.