addenda and errata

Mustafa Odabaşoğlu,[a]* Orhan Büyükgüngör[b] and Çiğdem Albayrak[a]

[a]Department of Chemistry, Ondokuz Mayıs University, TR-55139 Samsun, Turkey, and [b]Department of Physics, Ondokuz Mayıs University, TR-55139 Samsun, Turkey

Correspondence e-mail: orhanb@omu.edu.tr

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.

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

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]
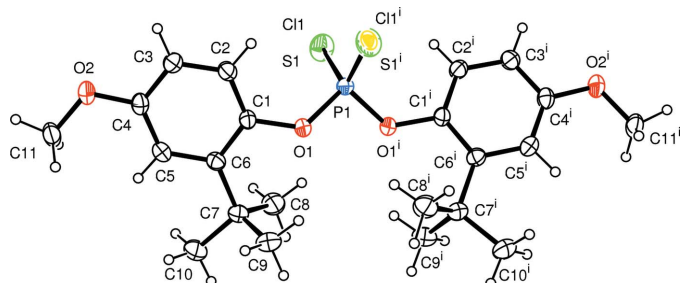

Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. [Symmetry code: (i) $-x + 1$, y, $-z + \frac{1}{2}$]
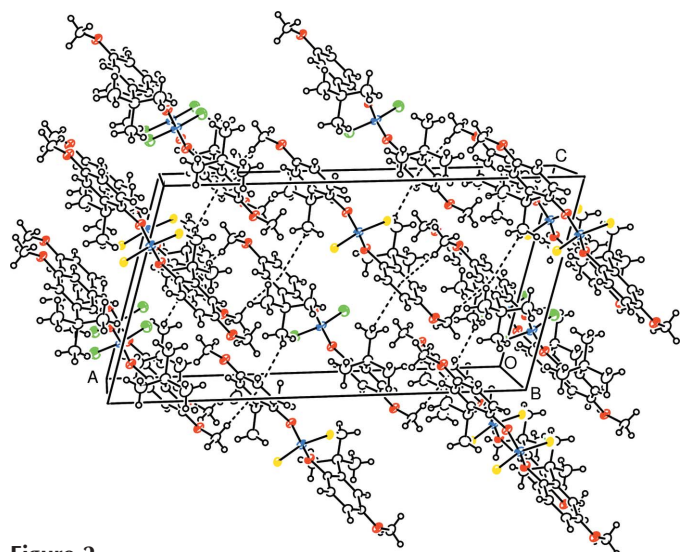

Figure 2
A view of the packing of (I); C—H$\cdots\pi$ interactions are drawn as dashed lines.

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

$C_{22}H_{30}ClO_4PS$
$M_r = 456.94$
Monoclinic, $C2/c$
$a = 23.592$ (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, colorless
$0.64 \times 0.59 \times 0.55$ 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
2327 independent reflections

2146 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.0°$
$h = -28 \rightarrow 28$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.073$
$S = 1.08$
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.

supporting information

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

Mustafa Odabaşoğlu, Orhan Büyükgüngör and Çiğdem Albayrak

S1. Comment

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: H8A···Cg1= 3.12 (2) Å and C8—H8A···Cg1 = 122.8 (2)° [Cg1 is the centroid of the C1^i–C6^i ring; and H11A···Cg2 = 2.71 (2) Å and C11—H11A···Cg2 = 142.7 (1)° (Cg2 is the centroid of the C1^{ii}–C6^{ii} ring); symmetry codes: (i) x, 1 − y, 1/2 + z; (ii) 1/2 − x, 1/2 − y, − z]. The dihedral angle between the symmetry-related benzene rings in (I) is 41.2 (2)°. Selected bond distances and angles for (I) are given in Table 1. The benzene ring is planar, with a maximum deviation from the plane defined by the six ring atoms of 0.007 (1) Å (for C3).

S2. 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%.

S3. Refinement

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.
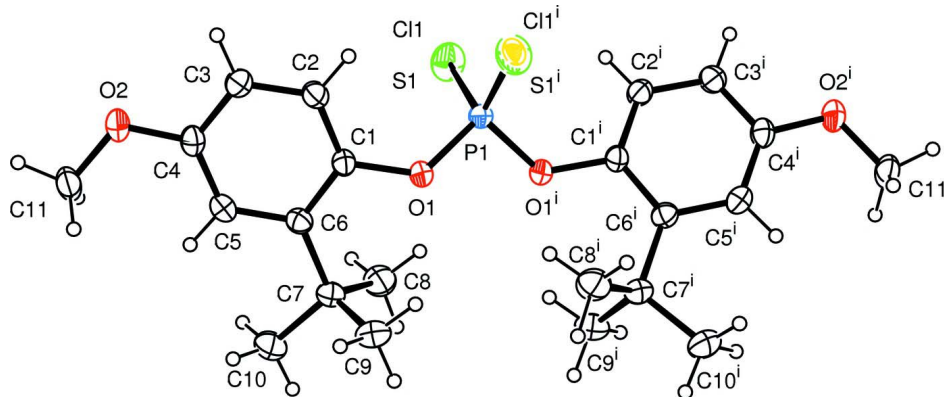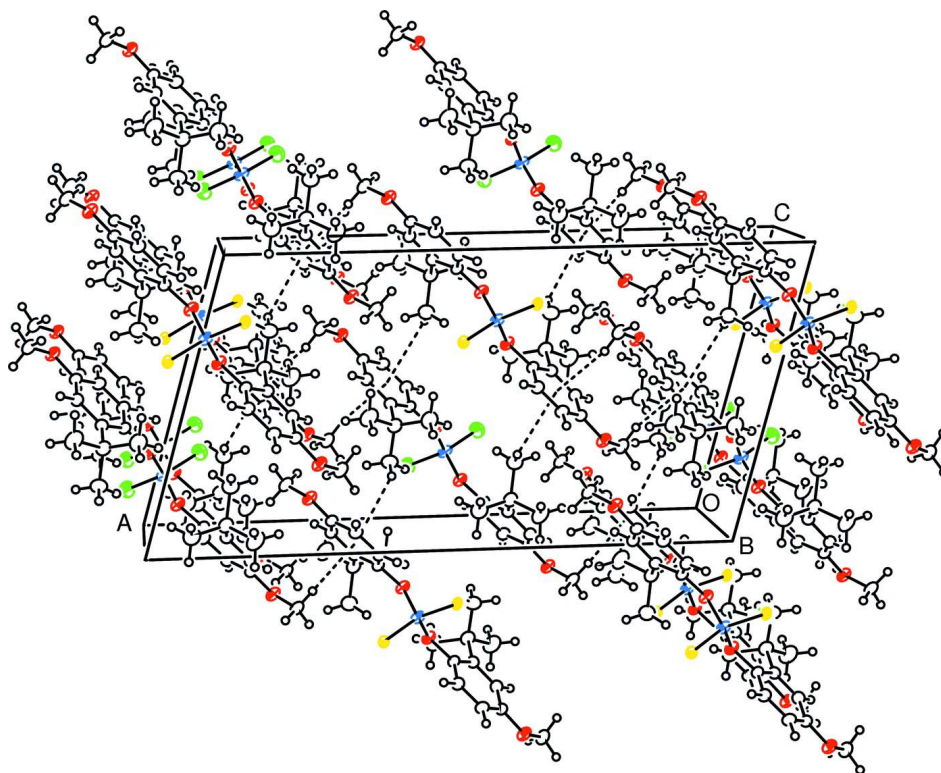

Figure 1

A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids·[Symmetry codes: (i) −*x*, *y*, −*z* + 1/2]

Figure 2

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

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

Crystal data

C$_{22}$H$_{30}$ClO$_4$PS	$F(000) = 968$
$M_r = 456.94$	$D_x = 1.286$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 16485 reflections
$a = 23.592$ (3) Å	$\theta = 1.7–26.7°$
$b = 8.3111$ (6) Å	$\mu = 0.34$ mm^{-1}
$c = 12.5067$ (14) Å	$T = 150$ K
$\beta = 105.740$ (9)°	Prism, colorless
$V = 2360.3$ (4) Å^3	$0.64 \times 0.59 \times 0.55$ mm
$Z = 4$	

Data collection

Stoe IPDS-II diffractometer	2327 independent reflections
	2146 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.032$
ω scans	$\theta_{max} = 26.0°$, $\theta_{min} = 1.8°$
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)	$h = -28\rightarrow28$
	$k = -10\rightarrow10$
$T_{min} = 0.824$, $T_{max} = 0.869$	$l = -15\rightarrow15$
16485 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.028$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.073$	All H-atom parameters refined
$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 1.6163P]$
2327 reflections	where $P = (F_o^2 + 2F_c^2)/3$
192 parameters	$(\Delta/\sigma)_{max} = 0.002$
0 restraints	$\Delta\rho_{max} = 0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.34$ e Å^{-3}

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}	Occ. (<1)
C1	0.58872 (5)	0.29212 (15)	0.41994 (10)	0.0224 (3)	
C2	0.59796 (6)	0.14466 (16)	0.47516 (11)	0.0258 (3)	
C3	0.65010 (6)	0.11722 (16)	0.55649 (11)	0.0272 (3)	
C4	0.69210 (5)	0.23993 (16)	0.58337 (10)	0.0249 (3)	
C5	0.68201 (6)	0.38769 (16)	0.52737 (11)	0.0252 (3)	
C6	0.62988 (5)	0.41888 (15)	0.44326 (10)	0.0233 (3)	
C7	0.61903 (6)	0.58369 (16)	0.38318 (12)	0.0296 (3)	
C8	0.61032 (8)	0.56105 (19)	0.25687 (13)	0.0374 (3)	
C9	0.56458 (8)	0.66639 (19)	0.40646 (15)	0.0402 (4)	
C10	0.67218 (8)	0.6983 (2)	0.42394 (16)	0.0453 (4)	
C11	0.78481 (6)	0.32952 (19)	0.70159 (13)	0.0343 (3)	
O1	0.53449 (4)	0.31975 (10)	0.33826 (7)	0.0257 (2)	
O2	0.74184 (4)	0.20383 (12)	0.66690 (8)	0.0319 (2)	
P1	0.5000	0.19428 (5)	0.2500	0.02610 (13)	
Cl1	0.554354 (14)	0.06331 (4)	0.18992 (3)	0.03162 (11)	0.50
S1	0.554354 (14)	0.06331 (4)	0.18992 (3)	0.03162 (11)	0.50
H2	0.5683 (7)	0.0634 (19)	0.4571 (13)	0.032 (4)*	
H3	0.6579 (7)	0.014 (2)	0.5947 (13)	0.034 (4)*	
H5	0.7120 (7)	0.4671 (19)	0.5482 (13)	0.029 (4)*	
H8A	0.6047 (8)	0.668 (3)	0.2193 (16)	0.052 (5)*	
H8B	0.5774 (8)	0.495 (2)	0.2241 (15)	0.043 (5)*	
H8C	0.6466 (9)	0.512 (2)	0.2429 (17)	0.056 (5)*	
H9A	0.5571 (8)	0.772 (2)	0.3671 (15)	0.051 (5)*	
H9B	0.5703 (8)	0.686 (2)	0.4892 (18)	0.056 (5)*	
H9C	0.5281 (8)	0.601 (2)	0.3804 (15)	0.047 (5)*	

H10A	0.7097 (9)	0.650 (2)	0.4105 (16)	0.056 (6)*
H10B	0.6816 (9)	0.720 (2)	0.5075 (19)	0.059 (6)*
H10C	0.6631 (9)	0.798 (2)	0.3852 (17)	0.056 (5)*
H11A	0.8030 (8)	0.360 (2)	0.6410 (15)	0.042 (5)*
H11B	0.8151 (8)	0.282 (2)	0.7644 (15)	0.041 (4)*
H11C	0.7671 (7)	0.427 (2)	0.7281 (14)	0.035 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0207 (6)	0.0240 (6)	0.0204 (6)	0.0000 (5)	0.0020 (4)	−0.0009 (5)
C2	0.0254 (6)	0.0247 (6)	0.0254 (6)	−0.0045 (5)	0.0037 (5)	0.0004 (5)
C3	0.0293 (7)	0.0261 (7)	0.0243 (6)	−0.0001 (5)	0.0037 (5)	0.0030 (5)
C4	0.0223 (6)	0.0311 (7)	0.0198 (6)	0.0006 (5)	0.0029 (5)	−0.0023 (5)
C5	0.0241 (6)	0.0262 (6)	0.0246 (6)	−0.0044 (5)	0.0053 (5)	−0.0041 (5)
C6	0.0253 (6)	0.0223 (6)	0.0222 (6)	−0.0015 (5)	0.0064 (5)	−0.0023 (5)
C7	0.0315 (7)	0.0212 (6)	0.0333 (7)	−0.0037 (5)	0.0041 (6)	0.0019 (5)
C8	0.0447 (9)	0.0336 (8)	0.0327 (8)	−0.0051 (7)	0.0086 (7)	0.0086 (6)
C9	0.0443 (9)	0.0242 (7)	0.0498 (10)	0.0051 (6)	0.0087 (7)	−0.0019 (7)
C10	0.0439 (9)	0.0279 (8)	0.0556 (11)	−0.0124 (7)	−0.0012 (8)	0.0062 (7)
C11	0.0253 (7)	0.0395 (8)	0.0322 (7)	−0.0019 (6)	−0.0021 (6)	−0.0082 (6)
O1	0.0238 (4)	0.0220 (4)	0.0260 (5)	0.0002 (3)	−0.0022 (4)	−0.0019 (4)
O2	0.0252 (5)	0.0360 (5)	0.0283 (5)	−0.0015 (4)	−0.0033 (4)	0.0005 (4)
P1	0.0288 (2)	0.0192 (2)	0.0242 (2)	0.000	−0.00339 (18)	0.000
Cl1	0.02605 (18)	0.0368 (2)	0.0320 (2)	0.00652 (13)	0.00781 (14)	−0.00242 (13)
S1	0.02605 (18)	0.0368 (2)	0.0320 (2)	0.00652 (13)	0.00781 (14)	−0.00242 (13)

Geometric parameters (Å, º)

C1—C2	1.3946 (18)	C8—H8C	1.00 (2)
C1—C6	1.4086 (17)	C9—H9A	1.00 (2)
C1—O1	1.4226 (14)	C9—H9B	1.02 (2)
C2—C3	1.3860 (18)	C9—H9C	0.994 (19)
C2—H2	0.955 (16)	C10—H10A	1.03 (2)
C3—C4	1.3979 (18)	C10—H10B	1.02 (2)
C3—H3	0.974 (17)	C10—H10C	0.96 (2)
C4—O2	1.3759 (15)	C11—O2	1.4383 (17)
C4—C5	1.4017 (19)	C11—H11A	0.998 (18)
C5—C6	1.4088 (18)	C11—H11B	0.990 (18)
C5—H5	0.952 (16)	C11—H11C	1.009 (17)
C6—C7	1.5499 (18)	O1—P1	1.5750 (9)
C7—C10	1.5468 (19)	P1—O1ⁱ	1.5750 (9)
C7—C8	1.548 (2)	P1—S1ⁱ	1.9791 (4)
C7—C9	1.552 (2)	P1—Cl1ⁱ	1.9791 (4)
C8—H8A	1.00 (2)	P1—Cl1	1.9791 (4)
C8—H8B	0.948 (19)		
C2—C1—C6	123.25 (11)	C7—C9—H9A	109.9 (11)

C2—C1—O1	119.13 (11)	C7—C9—H9B	111.8 (11)
C6—C1—O1	117.58 (11)	H9A—C9—H9B	108.6 (16)
C3—C2—C1	119.77 (12)	C7—C9—H9C	112.5 (11)
C3—C2—H2	120.3 (9)	H9A—C9—H9C	107.2 (15)
C1—C2—H2	119.9 (9)	H9B—C9—H9C	106.6 (15)
C2—C3—C4	119.11 (12)	C7—C10—H10A	111.4 (11)
C2—C3—H3	121.1 (9)	C7—C10—H10B	112.1 (11)
C4—C3—H3	119.8 (9)	H10A—C10—H10B	106.1 (16)
O2—C4—C3	115.24 (12)	C7—C10—H10C	108.5 (12)
O2—C4—C5	124.38 (11)	H10A—C10—H10C	109.9 (16)
C3—C4—C5	120.39 (11)	H10B—C10—H10C	108.8 (16)
C4—C5—C6	121.99 (12)	O2—C11—H11A	111.5 (10)
C4—C5—H5	117.3 (9)	O2—C11—H11B	104.1 (10)
C6—C5—H5	120.7 (9)	H11A—C11—H11B	109.7 (14)
C1—C6—C5	115.48 (11)	O2—C11—H11C	111.3 (9)
C1—C6—C7	123.14 (11)	H11A—C11—H11C	110.4 (14)
C5—C6—C7	121.37 (11)	H11B—C11—H11C	109.7 (13)
C10—C7—C8	106.40 (13)	C1—O1—P1	126.11 (8)
C10—C7—C6	111.83 (11)	C4—O2—C11	117.18 (11)
C8—C7—C6	110.24 (11)	O1—P1—O1^i	97.08 (7)
C10—C7—C9	107.68 (13)	O1—P1—S1^i	111.27 (4)
C8—C7—C9	110.96 (12)	O1^i—P1—S1^i	111.44 (3)
C6—C7—C9	109.67 (12)	O1—P1—Cl1^i	111.27 (4)
C7—C8—H8A	109.7 (11)	O1^i—P1—Cl1^i	111.44 (3)
C7—C8—H8B	112.2 (11)	S1^i—P1—Cl1^i	0.00 (2)
H8A—C8—H8B	108.9 (16)	O1—P1—Cl1	111.44 (3)
C7—C8—H8C	110.0 (12)	O1^i—P1—Cl1	111.27 (4)
H8A—C8—H8C	106.8 (16)	S1^i—P1—Cl1	113.26 (3)
H8B—C8—H8C	109.0 (16)	Cl1^i—P1—Cl1	113.26 (3)
C6—C1—C2—C3	−0.8 (2)	C5—C6—C7—C10	2.35 (18)
O1—C1—C2—C3	−178.73 (11)	C1—C6—C7—C8	−60.70 (16)
C1—C2—C3—C4	1.4 (2)	C5—C6—C7—C8	120.51 (13)
C2—C3—C4—O2	178.39 (11)	C1—C6—C7—C9	61.76 (16)
C2—C3—C4—C5	−1.14 (19)	C5—C6—C7—C9	−117.03 (14)
O2—C4—C5—C6	−179.21 (11)	C2—C1—O1—P1	−40.07 (15)
C3—C4—C5—C6	0.28 (19)	C6—C1—O1—P1	141.89 (10)
C2—C1—C6—C5	−0.05 (18)	C3—C4—O2—C11	−176.65 (12)
O1—C1—C6—C5	177.90 (10)	C5—C4—O2—C11	2.86 (18)
C2—C1—C6—C7	−178.90 (12)	C1—O1—P1—O1^i	−155.66 (11)
O1—C1—C6—C7	−0.95 (18)	C1—O1—P1—S1^i	88.01 (9)
C4—C5—C6—C1	0.31 (18)	C1—O1—P1—Cl1^i	88.01 (9)
C4—C5—C6—C7	179.19 (12)	C1—O1—P1—Cl1	−39.46 (10)
C1—C6—C7—C10	−178.86 (13)		

Symmetry code: (i) −x+1, y, −z+1/2.