

## Diethyl [hydroxy(phenyl)methyl]-phosphonate

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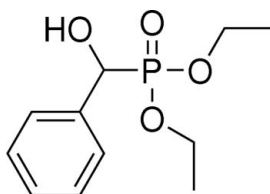
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in main residue;  $R$  factor = 0.055;  $wR$  factor = 0.175; data-to-parameter ratio = 14.0.

Molecules of the title compound,  $\text{C}_{11}\text{H}_{17}\text{O}_4\text{P}$ , are linked into chiral helical chains along the crystallographic  $b$  axis via  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds between the hydroxy group and an O atom of the phosphonate group. One ethyl group is disordered over two positions; the site occupancy factors are *ca* 0.7 and 0.3.

### Related literature

For related literature, see: Fang *et al.* (2006*a,b,c*, 2007); Kaboudin (2000); Maier & Diel (1994); Stowasser *et al.* (1992).



### Experimental

#### Crystal data

$\text{C}_{11}\text{H}_{17}\text{O}_4\text{P}$   
 $M_r = 244.22$   
 Monoclinic,  $P2_1/n$   
 $a = 9.2361$  (6) Å  
 $b = 8.0719$  (5) Å

$c = 17.4599$  (13) Å  
 $\beta = 95.096$  (5)°  
 $V = 1296.54$  (15) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.21$  mm<sup>-1</sup>  
 $T = 296$  (2) K

0.30 × 0.30 × 0.20 mm

#### Data collection

Rigaku Mercury diffractometer  
 Absorption correction: multi-scan  
 (Jacobson, 1998)  
 $T_{\min} = 0.940$ ,  $T_{\max} = 0.959$

10679 measured reflections  
 2345 independent reflections  
 1723 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.030$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$   
 $wR(F^2) = 0.174$   
 $S = 1.07$   
 2345 reflections  
 168 parameters

48 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.34$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	1.90	2.716 (3)	174

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

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2099).

### References

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**supplementary materials**

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## Diethyl [hydroxy(phenyl)methyl]phosphonate

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### Comment

The  $\alpha$ -hydroxyphosphonic esters and their derivatives have attracted considerable interest owing to their interesting biological activities, such as inhibition of inositol monophosphatase (Maier & Diel, 1994) and HIV protease (Stowasser *et al.*, 1992). These compounds are generally synthesized from aldehydes and phosphites *via* the base-catalyzed Pudovik reaction, as exemplified by diisopropyl (hydroxyphenylmethyl)phosphonate (Fang, *et al.*, 2006*a*), dimethyl [hydroxy(phenyl)methyl]phosphonate (Fang, *et al.*, 2006*b*), diphenyl (hydroxyphenylmethyl)phosphonate (Fang, *et al.*, 2006*c*) and diethyl hydroxy(4-methoxyphenyl)methylphosphonate (Fang, *et al.*, 2007). As an extension of these studies, we report herein on the structure of C<sub>11</sub>H<sub>17</sub>O<sub>4</sub>P, (**I**), (Fig. 1).

In **I**, the C10 and C11 atoms are disordered over two sets of sites with occupancies of 0.727 (7) and 0.273 (7). All bond distances and bond angles of **I** are normal and call for no further comment.

There exist a strong intermolecular H-bonding between O1—H1 $\cdots$ O2<sup>i</sup> (symmetry code: (i) 1/2-*x*, 1/2+*y*, 1/2-*z*) in **I**. Molecules of **I** are linked into chiral helical chains by this H-bonding, running parallel to the *b* axis (Fig. 2). But these chains are aligned in an antiparallel fashion to form inversion centers in the crystal, thus the whole structure is achiral (Fig.3).

### Experimental

All chemicals were obtained from commercial sources and used directly without further purification. Magnesium oxide (2 g) was added to a stirred mixture of diethyl phosphite (0.02 mol) and aldehyde (0.02 mol) at room temperature. After 2 h the mixture was washed by dichloromethane (50 ml) and dried with CaCl<sub>2</sub>; evaporation of the solvent gave the crude product. The products were crystallized from *n*-hexane (Kaboudin, 2000).

### Refinement

All H atoms were positioned geometrically and were allowed to ride on their parent atoms, with C—H distances of 0.93–0.97 Å (0.82 Å for O—H group) and  $U_{\text{iso}}(\text{H})$  values constrained to be 1.2 (1.5 for —OH and —CH<sub>3</sub> group) times  $U_{\text{eq}}$  of the carrier atom.

### Figures

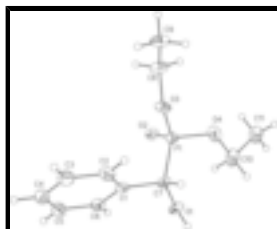


Fig. 1. A molecular structure of **I** with the atom numbering scheme. Displacement ellipsoids are drawn at 30% probability level. Only major part of disordered moiety are presented. The H atoms are drawn as a small spheres of arbitrary radius.

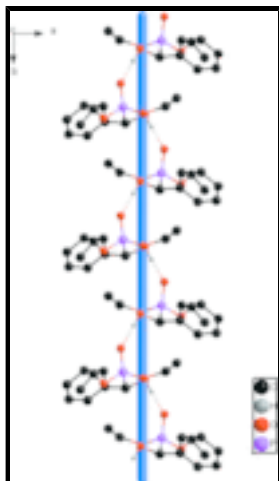


Fig. 2. The chiral helical chain constructed by O—H···O H-bonds.

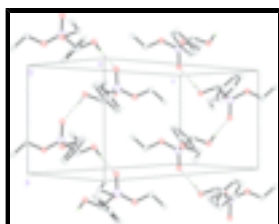


Fig. 3. Part of the crystal structure of **I**, showing chiral helical chains aligned in an antiparallel fashion. The H atoms not involved in H-bonds are omitted for clarity.

### Diethyl [hydroxy(phenyl)methyl]phosphonate

#### Crystal data

$C_{11}H_{17}O_4P$

$M_r = 244.22$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 9.2361\ (6)\ \text{\AA}$

$b = 8.0719\ (5)\ \text{\AA}$

$c = 17.4599\ (13)\ \text{\AA}$

$\beta = 95.096\ (5)^\circ$

$V = 1296.54\ (15)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 520$

$D_x = 1.251\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

$\theta = 2.3\text{--}25.2^\circ$

$\mu = 0.21\ \text{mm}^{-1}$

$T = 296\ (2)\ \text{K}$

Prism, colourless

$0.30 \times 0.30 \times 0.20\ \text{mm}$

#### Data collection

Rigaku Mercury  
diffractometer

Radiation source: Fine-focus sealed tube

Monochromator: Graphite

Detector resolution:  $7.31\ \text{pixels mm}^{-1}$

$T = 296\ (2)\ \text{K}$

$\omega$  scans

Absorption correction: multi-scan  
(Jacobson, 1998)

2345 independent reflections

1723 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\text{max}} = 25.2^\circ$

$\theta_{\text{min}} = 2.3^\circ$

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 8$

$T_{\min} = 0.940$ ,  $T_{\max} = 0.959$   
10679 measured reflections

$l = -17 \rightarrow 20$

### Refinement

Refinement on  $F^2$   
Least-squares matrix: Full  
 $R[F^2 > 2\sigma(F^2)] = 0.055$   
 $wR(F^2) = 0.174$   
 $S = 1.08$   
2345 reflections  
168 parameters  
48 restraints  
Primary atom site location: Direct

Secondary atom site location: Difmap  
Hydrogen site location: Geom  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1001P)^2 + 0.236P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.34 \text{ e } \text{\AA}^{-3}$   
Extinction correction: none

### Special details

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.0039 (3)	0.8100 (3)	0.27207 (16)	0.0699 (7)	
C2	-0.1128 (4)	0.9141 (5)	0.2543 (2)	0.0945 (10)	
H2	-0.1080	0.9916	0.2152	0.113*	
C3	-0.2344 (5)	0.9061 (6)	0.2925 (3)	0.1194 (14)	
H3	-0.3115	0.9773	0.2789	0.143*	
C4	-0.2447 (5)	0.7966 (7)	0.3498 (3)	0.1225 (15)	
H4	-0.3278	0.7927	0.3761	0.147*	
C5	-0.1305 (5)	0.6901 (6)	0.3689 (2)	0.1170 (14)	
H5	-0.1370	0.6138	0.4084	0.140*	
C6	-0.0049 (4)	0.6957 (4)	0.3296 (2)	0.0888 (10)	
H6	0.0716	0.6229	0.3423	0.107*	
C7	0.1365 (3)	0.8216 (3)	0.22835 (17)	0.0700 (7)	
H7	0.1517	0.9376	0.2146	0.084*	
C8	-0.0806 (5)	0.7118 (5)	0.0237 (2)	0.1202 (15)	
H8A	-0.0072	0.6701	-0.0078	0.144*	
H8B	-0.1403	0.6191	0.0370	0.144*	

## supplementary materials

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C9	-0.1684 (5)	0.8298 (5)	-0.0188 (2)	0.1294 (16)	
H9A	-0.2427	0.8689	0.0118	0.194*	
H9B	-0.2128	0.7791	-0.0648	0.194*	
H9C	-0.1093	0.9213	-0.0322	0.194*	
C10	0.3970 (12)	0.693 (3)	0.1245 (10)	0.128 (2)	0.273 (7)
H10A	0.3922	0.6073	0.1630	0.153*	0.273 (7)
H10B	0.4512	0.7864	0.1477	0.153*	0.273 (7)
C11	0.4683 (18)	0.629 (2)	0.0560 (10)	0.131 (2)	0.273 (7)
H11A	0.4753	0.7168	0.0194	0.197*	0.273 (7)
H11B	0.4110	0.5404	0.0325	0.197*	0.273 (7)
H11C	0.5638	0.5889	0.0725	0.197*	0.273 (7)
C10'	0.3723 (5)	0.6344 (7)	0.0883 (5)	0.122 (2)	0.727 (7)
H10C	0.3621	0.5770	0.0394	0.147*	0.727 (7)
H10D	0.3777	0.5529	0.1293	0.147*	0.727 (7)
C11'	0.5025 (6)	0.7386 (9)	0.0942 (5)	0.136 (2)	0.727 (7)
H11D	0.4976	0.8149	0.0518	0.204*	0.727 (7)
H11E	0.5872	0.6700	0.0928	0.204*	0.727 (7)
H11F	0.5081	0.7992	0.1416	0.204*	0.727 (7)
O1	0.2648 (2)	0.7606 (3)	0.26977 (14)	0.0876 (7)	
H1	0.3101	0.8380	0.2908	0.131*	
O2	0.1031 (2)	0.5201 (2)	0.15362 (11)	0.0787 (6)	
O3	-0.0104 (2)	0.7844 (3)	0.09335 (11)	0.0889 (7)	
O4	0.2499 (3)	0.7456 (3)	0.09497 (16)	0.1077 (8)	
P1	0.11894 (8)	0.69802 (9)	0.14126 (4)	0.0710 (3)	

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0760 (17)	0.0661 (16)	0.0641 (15)	0.0012 (13)	-0.0139 (13)	-0.0117 (12)
C2	0.097 (2)	0.101 (2)	0.084 (2)	0.0294 (19)	0.0000 (18)	-0.0047 (17)
C3	0.100 (3)	0.146 (4)	0.111 (3)	0.030 (3)	0.002 (2)	-0.027 (3)
C4	0.107 (3)	0.144 (4)	0.119 (3)	-0.012 (3)	0.027 (3)	-0.048 (3)
C5	0.143 (4)	0.119 (3)	0.091 (3)	-0.029 (3)	0.022 (3)	-0.005 (2)
C6	0.102 (2)	0.080 (2)	0.082 (2)	-0.0091 (17)	-0.0077 (19)	0.0029 (16)
C7	0.0726 (16)	0.0543 (14)	0.0788 (17)	0.0010 (12)	-0.0172 (14)	0.0031 (12)
C8	0.125 (3)	0.118 (3)	0.108 (3)	0.018 (2)	-0.044 (3)	-0.014 (2)
C9	0.148 (4)	0.129 (3)	0.100 (3)	0.009 (3)	-0.050 (3)	-0.001 (2)
C10	0.093 (3)	0.113 (5)	0.179 (6)	0.013 (4)	0.031 (4)	0.015 (4)
C11	0.098 (4)	0.117 (5)	0.181 (6)	0.016 (4)	0.031 (4)	0.012 (4)
C10'	0.091 (3)	0.107 (4)	0.173 (6)	0.014 (2)	0.038 (3)	0.016 (3)
C11'	0.101 (3)	0.121 (4)	0.188 (6)	0.007 (3)	0.023 (3)	0.012 (4)
O1	0.0732 (12)	0.0743 (12)	0.1085 (16)	0.0046 (10)	-0.0301 (12)	-0.0079 (12)
O2	0.0826 (13)	0.0656 (12)	0.0845 (13)	0.0092 (9)	-0.0106 (10)	-0.0046 (9)
O3	0.1065 (16)	0.0916 (15)	0.0645 (11)	0.0306 (11)	-0.0157 (11)	-0.0027 (9)
O4	0.1062 (16)	0.0990 (16)	0.1221 (19)	0.0288 (14)	0.0325 (15)	0.0324 (15)
P1	0.0741 (5)	0.0677 (5)	0.0694 (5)	0.0125 (3)	-0.0035 (4)	0.0054 (3)

Geometric parameters (Å, °)

C1—C6	1.371 (4)	C9—H9B	0.9600
C1—C2	1.380 (4)	C9—H9C	0.9600
C1—C7	1.503 (4)	C10—O4	1.471 (8)
C2—C3	1.358 (5)	C10—C11	1.509 (10)
C2—H2	0.9300	C10—H10A	0.9700
C3—C4	1.344 (7)	C10—H10B	0.9700
C3—H3	0.9300	C11—H11A	0.9600
C4—C5	1.378 (7)	C11—H11B	0.9600
C4—H4	0.9300	C11—H11C	0.9600
C5—C6	1.401 (5)	C10'—O4	1.456 (5)
C5—H5	0.9300	C10'—C11'	1.464 (7)
C6—H6	0.9300	C10'—H10C	0.9700
C7—O1	1.420 (3)	C10'—H10D	0.9700
C7—P1	1.814 (3)	C11'—H11D	0.9600
C7—H7	0.9800	C11'—H11E	0.9600
C8—C9	1.418 (5)	C11'—H11F	0.9600
C8—O3	1.450 (4)	O1—H1	0.82
C8—H8A	0.9700	O2—P1	1.462 (2)
C8—H8B	0.9700	O3—P1	1.561 (2)
C9—H9A	0.9600	O4—P1	1.562 (3)
C6—C1—C2	118.6 (3)	H9A—C9—H9B	109.5
C6—C1—C7	121.2 (3)	C8—C9—H9C	109.5
C2—C1—C7	120.2 (3)	H9A—C9—H9C	109.5
C3—C2—C1	121.6 (4)	H9B—C9—H9C	109.5
C3—C2—H2	119.2	O4—C10—C11	105.9 (11)
C1—C2—H2	119.2	O4—C10—H10A	110.5
C4—C3—C2	120.9 (4)	C11—C10—H10A	110.5
C4—C3—H3	119.6	O4—C10—H10B	110.5
C2—C3—H3	119.6	C11—C10—H10B	110.5
C3—C4—C5	119.1 (4)	H10A—C10—H10B	108.7
C3—C4—H4	120.4	O4—C10'—C11'	106.2 (5)
C5—C4—H4	120.4	O4—C10'—H10C	110.5
C4—C5—C6	120.7 (4)	C11'—C10'—H10C	110.5
C4—C5—H5	119.6	O4—C10'—H10D	110.5
C6—C5—H5	119.6	C11'—C10'—H10D	110.5
C1—C6—C5	119.1 (4)	H10C—C10'—H10D	108.7
C1—C6—H6	120.5	C10'—C11'—H11D	109.5
C5—C6—H6	120.5	C10'—C11'—H11E	109.5
O1—C7—C1	113.6 (2)	H11D—C11'—H11E	109.5
O1—C7—P1	104.17 (19)	C10'—C11'—H11F	109.5
C1—C7—P1	112.03 (18)	H11D—C11'—H11F	109.5
O1—C7—H7	109.0	H11E—C11'—H11F	109.5
C1—C7—H7	109.0	C7—O1—H1	109.5
P1—C7—H7	109.0	C8—O3—P1	122.2 (2)
C9—C8—O3	111.1 (3)	C10'—O4—P1	122.1 (3)
C9—C8—H8A	109.4	C10—O4—P1	118.8 (8)

## supplementary materials

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O3—C8—H8A	109.4	O2—P1—O3	115.86 (12)
C9—C8—H8B	109.4	O2—P1—O4	114.24 (13)
O3—C8—H8B	109.4	O3—P1—O4	101.79 (14)
H8A—C8—H8B	108.0	O2—P1—C7	114.79 (12)
C8—C9—H9A	109.5	O3—P1—C7	102.18 (12)
C8—C9—H9B	109.5	O4—P1—C7	106.42 (15)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ O2 <sup>i</sup>	0.82	1.90	2.716 (3)	174

Symmetry codes: (i)  $-x+1/2, y+1/2, -z+1/2$ .



Fig. 1

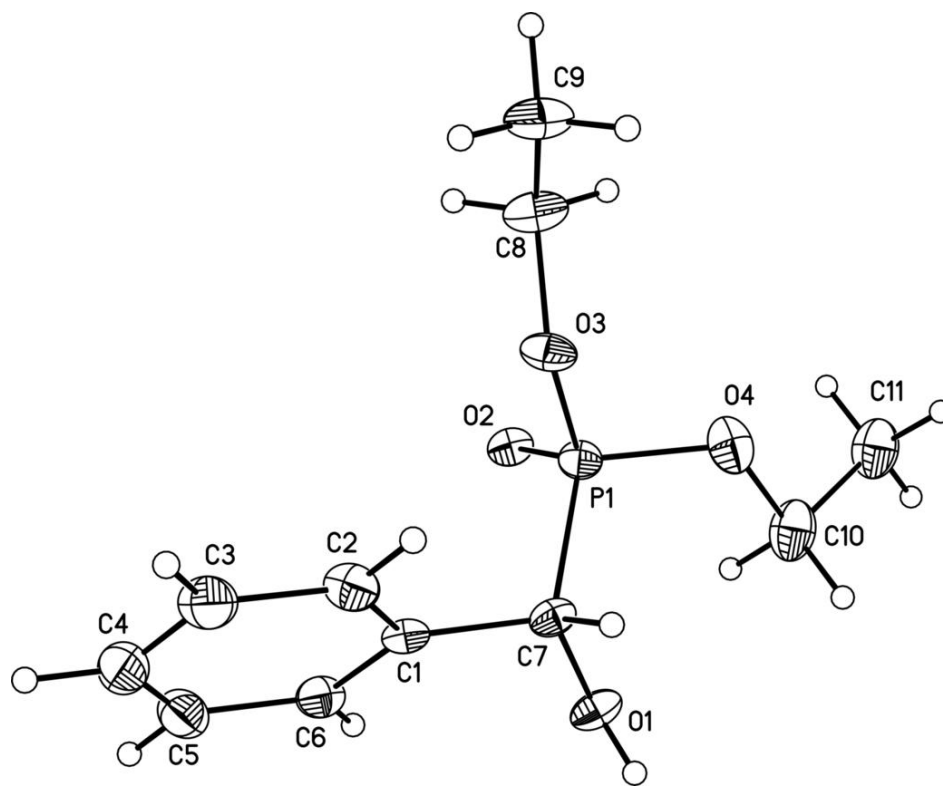


Fig. 2

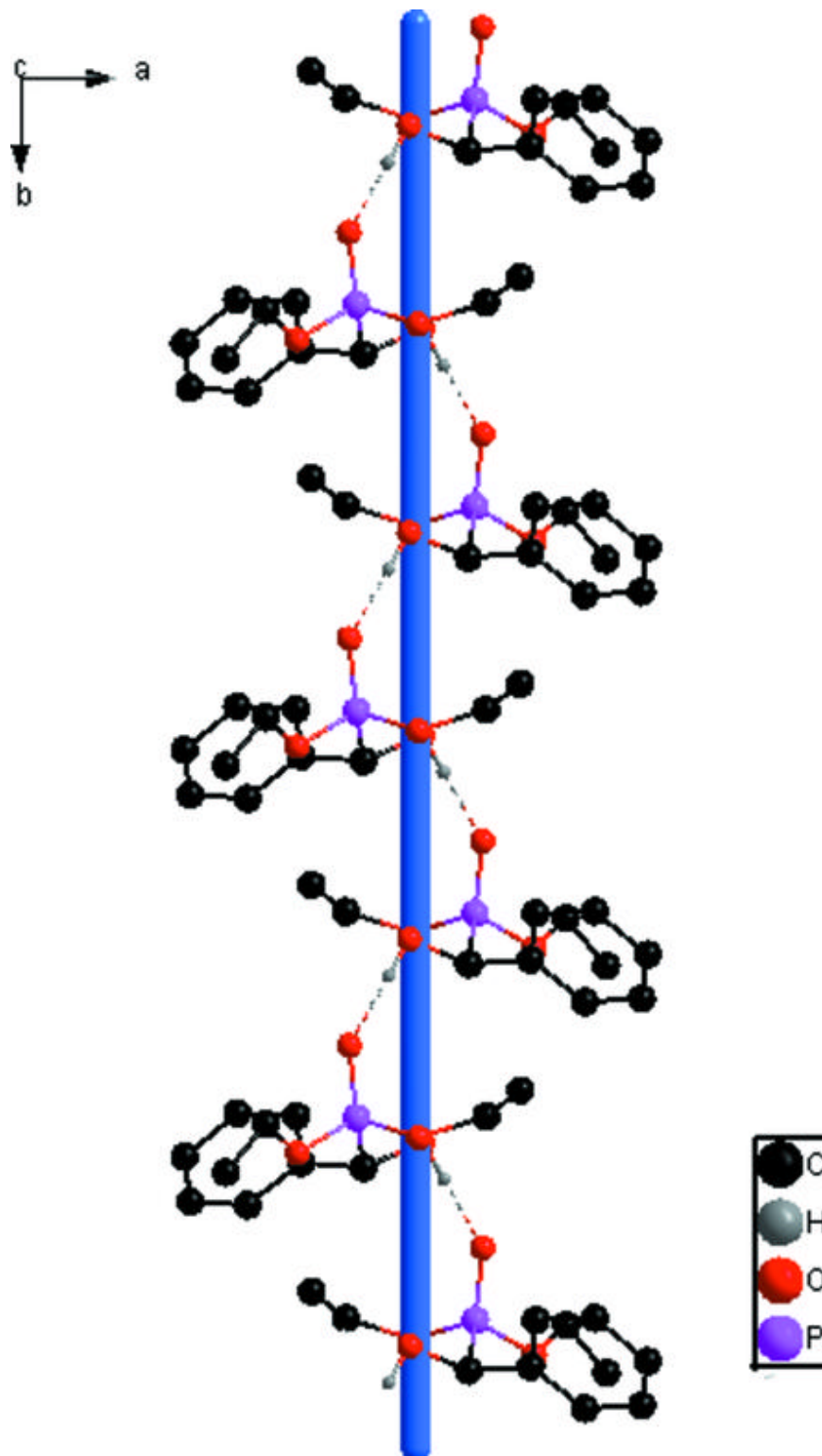


Fig. 3

