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2-Nitrobenzyl diphenyl phosphate

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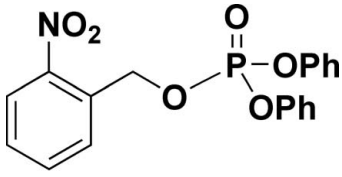
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 17.2.

In the title compound, $C_{19}H_{16}NO_6P$, the dihedral angles between the nitrobenzyl ring and the phenyl rings are 53.74 (3) and 63.30 (2)°, and that between the two phenyl rings is 88.86 (3)°. Intermolecular C—H···O hydrogen bonds result in the stabilization of the crystal structure.

Related literature

For related literature, see: Schick *et al.* (1995).



Experimental

Crystal data

$C_{19}H_{16}NO_6P$
M_r = 385.30
Monoclinic, $P2_1$
a = 6.0641 (5) Å
b = 8.9176 (8) Å
c = 16.7851 (14) Å
β = 90.727 (1)°
V = 907.62 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 295 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART 4 K CCD area-detector diffractometer
Absorption correction: none
9691 measured reflections
4190 independent reflections
3883 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.05
4190 reflections
244 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 978 Friedel pairs
Flack parameter: 0.08 (7)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···O4^{i}	0.93	2.45	3.344 (2)	162
C7—H7A···O4^{i}	0.97	2.59	3.537 (2)	164

Symmetry code: (i) $x+1, y, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

The authors are grateful to the Central China Normal University. We thank Professor Wen-Jing Xiao for fruitful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2030).

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supplementary materials

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2-Nitrobenzyl diphenyl phosphate

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Comment

Phosphates are extremely useful compounds which have received much attention as genetic materials, co-enzymes and in biochemistry in general. Phosphates have well known roles as intermediates in biochemical transformations (Schick *et al.*, 1995). The title compound (I) is formed by the reaction of diphenylphosphite with an aromatic aldehyde in presence of triethylamine. We also find that the title compound may be obtained from a hydroxyphosphonate rearrangement. In this paper, we present an X-ray crystallographic analysis of (I), shown in Fig. 1. The dihedral angles between the benzene rings A (C1—C6), B (C8—C13) and C (C14—C19) are A/B = 53.74 (3)°, A/C = 63.30 (2)° and B/C = 88.86 (3)°. In the crystal structure, intermolecular C—H···O hydrogen bonds are effective in stabilizing the structure (Fig. 2, Table 2).

Experimental

To a solution of 2-nitrobenzylaldehyde (1 mmol) in tetrahydrofuran (0.60 ml) was added diphenyl phosphite (1 mmol) at ice-bath temperature. After 15 minutes, triethylamine (0.14 ml) was added, and the reaction mixture was stirred for a further 2 h at ice-bath temperature. The resulting solution was washed with saturated $NaHCO_3$ solution, extracted with dichloromethane and dried over $MgSO_4$. The solution was then filtered and purified by column chromatography on silica gel, using ethyl acetate and petroleum as eluant, to afford compound (I).

^{1}H NMR (CDCl3, 400 MHz): 8.17 (d, J = 8.0 Hz, 1H), 7.73 (d, J = 8.0 Hz, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.51 (d, J = 8.0 Hz, 1H), 7.35 (t, J = 8.0 Hz, 4H), 7.19–7.26 (m, 6H), 5.74 (d, J = 8.0 Hz, 2H). Crystals suitable for X-ray diffraction were grown from a dichloromethane-ether solution at 298 K.

Refinement

All H atoms were initially located in a difference Fourier map. The phenyl H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93 Å. Methylene groups were treated similarly, with C—H distances of 0.97 Å.

Figures

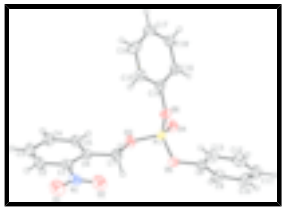

Fig. 1. View of compound (I), showing the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are represented by circles of arbitrary size.

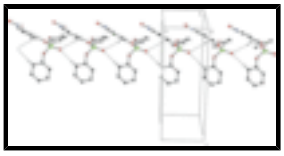

Fig. 2. Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines.

2-Nitrobenzyl diphenyl phosphate

Crystal data

$C_{19}H_{16}NO_6P$	F_{000} = 400
M_r = 385.30	D_x = 1.41 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 4390 reflections
a = 6.0641 (5) Å	θ = 2.3–27.1º
b = 8.9176 (8) Å	μ = 0.19 mm^{-1}
c = 16.7851 (14) Å	T = 295 (2) K
β = 90.7270 (10)º	Block, colorless
V = 907.62 (13) Å^3	0.20 × 0.10 × 0.10 mm
Z = 2	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	3883 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.022
Monochromator: graphite	θ_{max} = 28.0º
T = 295(2) K	θ_{min} = 2.4º
phi and ω scans	h = −7→7
Absorption correction: none	k = −11→11
9691 measured reflections	l = −21→21
4190 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.0077P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.097	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.05	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
4190 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
244 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 1868 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.08 (7)

Secondary atom site location: difference Fourier map

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	1.1681 (3)	0.2682 (3)	0.08864 (11)	0.0471 (4)
C2	1.2298 (4)	0.1344 (3)	0.05385 (12)	0.0595 (6)
H2	1.3592	0.1286	0.0248	0.071*
C3	1.0996 (4)	0.0109 (3)	0.06240 (12)	0.0631 (5)
H3	1.1400	−0.0800	0.0396	0.076*
C4	0.9084 (4)	0.0217 (3)	0.10492 (12)	0.0577 (5)
H4	0.8189	−0.0623	0.1103	0.069*
C5	0.8472 (3)	0.1545 (2)	0.13962 (11)	0.0480 (4)
H5	0.7168	0.1586	0.1681	0.058*
C6	0.9758 (3)	0.2833 (2)	0.13314 (10)	0.0416 (4)
C7	0.9080 (3)	0.4270 (2)	0.17274 (11)	0.0455 (4)
H7A	1.0211	0.4589	0.2104	0.055*
H7B	0.8881	0.5053	0.1332	0.055*
C8	0.5830 (3)	0.8082 (2)	0.26077 (10)	0.0407 (4)
C9	0.3789 (4)	0.8659 (2)	0.24225 (14)	0.0581 (5)
H9	0.2799	0.8122	0.2105	0.070*
C10	0.3237 (4)	1.0049 (3)	0.27156 (16)	0.0709 (6)
H10	0.1863	1.0457	0.2593	0.085*
C11	0.4677 (5)	1.0833 (3)	0.31827 (16)	0.0687 (7)
H11	0.4287	1.1774	0.3374	0.082*
C12	0.6692 (4)	1.0244 (3)	0.33722 (14)	0.0680 (6)
H12	0.7661	1.0780	0.3698	0.082*
C13	0.7307 (3)	0.8847 (2)	0.30818 (12)	0.0536 (5)
H13	0.8682	0.8442	0.3206	0.064*
C14	0.7906 (3)	0.42840 (18)	0.40687 (9)	0.0369 (3)
C15	0.9879 (3)	0.3524 (2)	0.41512 (12)	0.0465 (4)
H15	1.1001	0.3658	0.3785	0.056*
C16	1.0152 (4)	0.2558 (3)	0.47902 (13)	0.0575 (5)
H16	1.1469	0.2035	0.4853	0.069*
C17	0.8504 (4)	0.2363 (2)	0.53315 (13)	0.0554 (5)
H17	0.8708	0.1716	0.5761	0.066*
C18	0.6538 (3)	0.3128 (2)	0.52381 (11)	0.0525 (5)

H18	0.5415	0.2987	0.5603	0.063*
C19	0.6229 (3)	0.4103 (2)	0.46058 (10)	0.0446 (4)
H19	0.4912	0.4627	0.4544	0.054*
N1	1.3160 (3)	0.3970 (2)	0.07554 (10)	0.0561 (4)
O1	1.4834 (3)	0.3768 (3)	0.03849 (12)	0.0898 (6)
O2	1.2634 (3)	0.5195 (2)	0.10143 (10)	0.0750 (4)
O3	0.7011 (2)	0.39994 (15)	0.21402 (8)	0.0467 (3)
O4	0.3803 (2)	0.48193 (15)	0.29503 (8)	0.0497 (3)
O5	0.6448 (2)	0.66933 (14)	0.22804 (7)	0.0467 (3)
O6	0.7736 (2)	0.53291 (14)	0.34462 (7)	0.0445 (3)
P1	0.60419 (7)	0.51722 (5)	0.27268 (2)	0.03749 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0406 (9)	0.0635 (12)	0.0372 (9)	0.0015 (8)	0.0018 (7)	0.0020 (8)
C2	0.0499 (12)	0.0801 (16)	0.0487 (10)	0.0117 (11)	0.0109 (9)	−0.0105 (11)
C3	0.0671 (13)	0.0659 (13)	0.0565 (11)	0.0134 (13)	0.0070 (9)	−0.0201 (11)
C4	0.0644 (12)	0.0523 (10)	0.0566 (10)	−0.0039 (11)	0.0058 (9)	−0.0096 (11)
C5	0.0475 (10)	0.0521 (10)	0.0446 (9)	0.0000 (9)	0.0082 (7)	−0.0051 (8)
C6	0.0389 (9)	0.0503 (10)	0.0357 (8)	0.0020 (7)	0.0016 (6)	−0.0002 (7)
C7	0.0444 (10)	0.0475 (10)	0.0450 (9)	−0.0043 (8)	0.0106 (8)	−0.0042 (8)
C8	0.0481 (10)	0.0353 (8)	0.0387 (8)	0.0004 (7)	0.0039 (7)	0.0063 (7)
C9	0.0539 (12)	0.0493 (11)	0.0709 (13)	0.0040 (9)	−0.0128 (10)	0.0028 (10)
C10	0.0636 (13)	0.0541 (13)	0.0950 (17)	0.0157 (12)	0.0072 (12)	0.0089 (13)
C11	0.0900 (18)	0.0394 (10)	0.0775 (15)	−0.0010 (11)	0.0233 (13)	−0.0038 (10)
C12	0.0841 (16)	0.0547 (12)	0.0652 (12)	−0.0249 (14)	−0.0009 (11)	−0.0077 (13)
C13	0.0475 (11)	0.0555 (12)	0.0577 (11)	−0.0097 (9)	−0.0047 (9)	0.0046 (9)
C14	0.0406 (9)	0.0314 (8)	0.0386 (7)	−0.0039 (6)	−0.0019 (6)	−0.0029 (6)
C15	0.0350 (9)	0.0470 (10)	0.0576 (10)	−0.0027 (8)	0.0038 (8)	0.0022 (8)
C16	0.0446 (11)	0.0514 (11)	0.0761 (15)	0.0056 (9)	−0.0096 (10)	0.0092 (11)
C17	0.0690 (14)	0.0455 (10)	0.0513 (11)	0.0000 (9)	−0.0125 (10)	0.0092 (9)
C18	0.0586 (12)	0.0555 (11)	0.0435 (9)	−0.0019 (9)	0.0080 (8)	0.0018 (9)
C19	0.0430 (10)	0.0440 (9)	0.0470 (9)	0.0055 (7)	0.0054 (7)	−0.0015 (8)
N1	0.0447 (9)	0.0784 (13)	0.0453 (8)	−0.0064 (9)	0.0064 (7)	0.0105 (9)
O1	0.0597 (10)	0.1117 (16)	0.0989 (13)	−0.0036 (10)	0.0374 (10)	0.0141 (12)
O2	0.0738 (10)	0.0738 (10)	0.0778 (10)	−0.0244 (10)	0.0200 (8)	−0.0061 (10)
O3	0.0446 (7)	0.0426 (7)	0.0532 (7)	−0.0040 (5)	0.0128 (6)	−0.0068 (6)
O4	0.0392 (7)	0.0580 (9)	0.0520 (7)	0.0011 (5)	0.0053 (5)	0.0027 (6)
O5	0.0587 (8)	0.0401 (6)	0.0414 (6)	0.0050 (6)	0.0087 (5)	0.0037 (5)
O6	0.0471 (6)	0.0403 (6)	0.0460 (6)	−0.0067 (6)	−0.0018 (5)	0.0079 (6)
P1	0.0378 (2)	0.0362 (2)	0.0386 (2)	0.00156 (19)	0.00463 (15)	0.00116 (18)

Geometric parameters (Å, °)

C1—C2	1.382 (3)	C11—H11	0.9300
C1—C6	1.399 (2)	C12—C13	1.390 (4)
C1—N1	1.476 (3)	C12—H12	0.9300
C2—C3	1.364 (4)	C13—H13	0.9300

C2—H2	0.9300	C14—C19	1.377 (2)
C3—C4	1.372 (3)	C14—C15	1.381 (3)
C3—H3	0.9300	C14—O6	1.403 (2)
C4—C5	1.373 (3)	C15—C16	1.384 (3)
C4—H4	0.9300	C15—H15	0.9300
C5—C6	1.394 (3)	C16—C17	1.370 (3)
C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.503 (3)	C17—C18	1.381 (3)
C7—O3	1.461 (2)	C17—H17	0.9300
C7—H7A	0.9700	C18—C19	1.383 (3)
C7—H7B	0.9700	C18—H18	0.9300
C8—C13	1.372 (3)	C19—H19	0.9300
C8—C9	1.373 (3)	N1—O1	1.211 (2)
C8—O5	1.408 (2)	N1—O2	1.219 (3)
C9—C10	1.376 (3)	O3—P1	1.5568 (13)
C9—H9	0.9300	O4—P1	1.4475 (14)
C10—C11	1.360 (4)	O5—P1	1.5706 (13)
C10—H10	0.9300	O6—P1	1.5815 (13)
C11—C12	1.363 (4)		
C2—C1—C6	122.76 (19)	C11—C12—C13	120.4 (2)
C2—C1—N1	116.14 (17)	C11—C12—H12	119.8
C6—C1—N1	121.10 (19)	C13—C12—H12	119.8
C3—C2—C1	119.48 (18)	C8—C13—C12	118.2 (2)
C3—C2—H2	120.3	C8—C13—H13	120.9
C1—C2—H2	120.3	C12—C13—H13	120.9
C2—C3—C4	119.5 (2)	C19—C14—C15	121.53 (17)
C2—C3—H3	120.3	C19—C14—O6	121.17 (16)
C4—C3—H3	120.3	C15—C14—O6	117.15 (15)
C3—C4—C5	121.1 (2)	C14—C15—C16	118.56 (17)
C3—C4—H4	119.5	C14—C15—H15	120.7
C5—C4—H4	119.5	C16—C15—H15	120.7
C4—C5—C6	121.55 (18)	C17—C16—C15	120.8 (2)
C4—C5—H5	119.2	C17—C16—H16	119.6
C6—C5—H5	119.2	C15—C16—H16	119.6
C5—C6—C1	115.66 (17)	C16—C17—C18	119.84 (19)
C5—C6—C7	120.78 (15)	C16—C17—H17	120.1
C1—C6—C7	123.56 (17)	C18—C17—H17	120.1
O3—C7—C6	108.07 (14)	C17—C18—C19	120.42 (18)
O3—C7—H7A	110.1	C17—C18—H18	119.8
C6—C7—H7A	110.1	C19—C18—H18	119.8
O3—C7—H7B	110.1	C14—C19—C18	118.83 (18)
C6—C7—H7B	110.1	C14—C19—H19	120.6
H7A—C7—H7B	108.4	C18—C19—H19	120.6
C13—C8—C9	121.7 (2)	O1—N1—O2	122.8 (2)
C13—C8—O5	119.27 (17)	O1—N1—C1	118.4 (2)
C9—C8—O5	119.02 (17)	O2—N1—C1	118.81 (16)
C8—C9—C10	118.6 (2)	C7—O3—P1	121.49 (11)
C8—C9—H9	120.7	C8—O5—P1	121.98 (10)
C10—C9—H9	120.7	C14—O6—P1	123.41 (11)

C11—C10—C9	120.8 (2)	O4—P1—O3	112.38 (8)
C11—C10—H10	119.6	O4—P1—O5	117.69 (8)
C9—C10—H10	119.6	O3—P1—O5	102.46 (7)
C10—C11—C12	120.2 (2)	O4—P1—O6	115.06 (7)
C10—C11—H11	119.9	O3—P1—O6	107.19 (8)
C12—C11—H11	119.9	O5—P1—O6	100.58 (7)
C6—C1—C2—C3	0.2 (3)	C15—C16—C17—C18	0.5 (3)
N1—C1—C2—C3	−179.07 (19)	C16—C17—C18—C19	−0.6 (3)
C1—C2—C3—C4	0.5 (3)	C15—C14—C19—C18	−0.4 (3)
C2—C3—C4—C5	−0.6 (3)	O6—C14—C19—C18	−175.85 (17)
C3—C4—C5—C6	0.0 (3)	C17—C18—C19—C14	0.6 (3)
C4—C5—C6—C1	0.6 (3)	C2—C1—N1—O1	−2.7 (3)
C4—C5—C6—C7	−178.93 (18)	C6—C1—N1—O1	177.95 (18)
C2—C1—C6—C5	−0.8 (3)	C2—C1—N1—O2	176.26 (19)
N1—C1—C6—C5	178.51 (17)	C6—C1—N1—O2	−3.1 (3)
C2—C1—C6—C7	178.76 (19)	C6—C7—O3—P1	169.83 (12)
N1—C1—C6—C7	−2.0 (3)	C13—C8—O5—P1	89.90 (19)
C5—C6—C7—O3	0.0 (2)	C9—C8—O5—P1	−92.19 (19)
C1—C6—C7—O3	−179.49 (16)	C19—C14—O6—P1	−67.5 (2)
C13—C8—C9—C10	0.8 (3)	C15—C14—O6—P1	116.86 (16)
O5—C8—C9—C10	−177.09 (18)	C7—O3—P1—O4	168.89 (14)
C8—C9—C10—C11	−0.3 (4)	C7—O3—P1—O5	41.62 (15)
C9—C10—C11—C12	−0.5 (4)	C7—O3—P1—O6	−63.76 (15)
C10—C11—C12—C13	0.9 (4)	C8—O5—P1—O4	56.83 (16)
C9—C8—C13—C12	−0.4 (3)	C8—O5—P1—O3	−179.38 (14)
O5—C8—C13—C12	177.48 (16)	C8—O5—P1—O6	−68.94 (14)
C11—C12—C13—C8	−0.5 (3)	C14—O6—P1—O4	47.55 (15)
C19—C14—C15—C16	0.2 (3)	C14—O6—P1—O3	−78.22 (14)
O6—C14—C15—C16	175.83 (17)	C14—O6—P1—O5	175.07 (13)
C14—C15—C16—C17	−0.2 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15···O4^{i}	0.93	2.45	3.344 (2)	162
C7—H7A···O4^{i}	0.97	2.59	3.537 (2)	164

Symmetry codes: (i) *x*+1, *y*, *z*.

Fig. 1

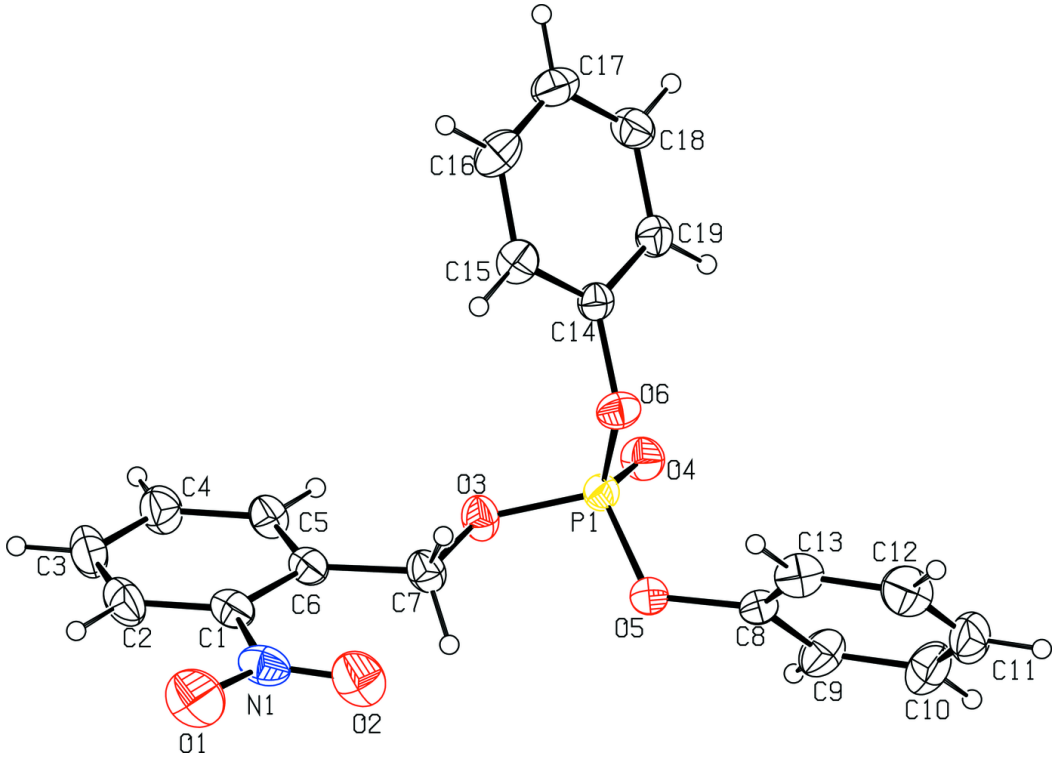
C17
C18
C16
C19
C15
C14
O6
O4
O3
P1
C4
C5
C3
C6
C7
C13
C12
O5
C8
C2
C1
C9
C11
C10
N1
O2
O1

Fig. 2