

Phenyl bis(*m*-tolylamido)phosphinate

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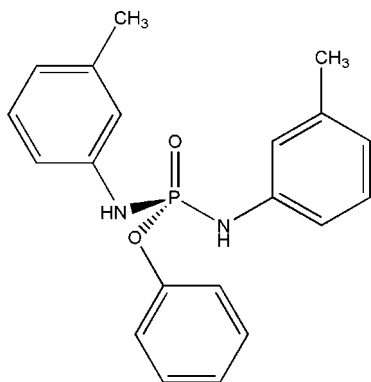
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.022; wR factor = 0.062; data-to-parameter ratio = 12.4.

The P atom of the title compound, $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_2\text{P}$, has a distorted tetrahedral configuration; the bond angles at P are in the range $96.11(6)$ – $117.32(8)^\circ$. The N atom exhibits sp^2 character. In the crystal, molecules are connected *via* N–H \cdots O hydrogen bonds into bands along the a axis, consisting of $R_2^2(8)$ rings.

Related literature

For background to compounds having a P(=O)(O)(N)(N) skeleton, see: Sabbaghi *et al.* (2010). For bond lengths in related structures, see: Ghadimi *et al.* (2009); Rudd *et al.* (1996). For graph-set notation, see Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_2\text{P}$

$M_r = 352.36$

Orthorhombic, $Pna2_1$

$a = 10.1930(2)$ Å

$b = 16.8789(3)$ Å

$c = 10.4588(3)$ Å

$V = 1799.40(7)$ Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.17$ mm⁻¹

$T = 120$ K

$0.30 \times 0.30 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur

Sapphire2 diffractometer

Absorption correction: multi-scan

(*CrysAlis RED*; Oxford

Diffraction, 2009)

$T_{\min} = 0.959$, $T_{\max} = 1.000$

20798 measured reflections

2819 independent reflections

2647 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$

$wR(F^2) = 0.062$

$S = 1.05$

2819 reflections

228 parameters

1 restraint

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.17$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Absolute structure: Flack (1983),

1140 Friedel pairs

Flack parameter: $-0.04(7)$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{i}}$	0.88	2.14	2.9917 (16)	162
$\text{N2}-\text{H2A}\cdots\text{O2}^{\text{ii}}$	0.88	2.53	3.3929 (16)	166

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2017).

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supplementary materials

Acta Cryst. (2011). E67, o1870 [doi:10.1107/S1600536811024846]

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Comment

Structure determination of the title compound, P(O)[OC₆H₅][NHC₆H₄(*m*-CH₃)₂] (Fig. 1), was performed as a part of a project in our laboratory on the synthesis of compounds having a P(=O)(O)(N)(N) skeleton (Sabbaghi *et al.*, 2010). Single crystals of title compound were obtained from a mixture of CHCl₃/CH₃OH (4:1 *v/v*) after slow evaporation at room temperature.

The P=O (1.4692 (11) Å), P—O (1.5941 (12) Å) and P—N (1.6374 (15) Å & 1.6469 (13) Å) bond lengths are usual for this category of compounds (Ghadimi *et al.*, 2009). The P atom has a distorted tetrahedral configuration (Fig. 1), as it has been noted for the other phosphoramidates and their chhalco-derivatives (Rudd *et al.*, 1996). The bond angles at the P atom vary in the range from 96.11 (6)°–117.32 (8)°. The C1—O2—P1, C7—N1—P1 and C14—N2—P1 angles are 120.49 (9)°, 127.29 (11)° and 130.85 (11)°, respectively.

Prior to the constrained refinement of the imino H atoms, we have tested a free refinement of these atoms localized from a difference Fourier map. The results suggested an almost perfect in-plane arrangement of both H atoms with the corresponding P,N,C atoms. Therefore, the imino groups were assigned a planar geometry to reduce the number of parameters and to maintain more appropriate N—H bond lengths.

One of the two N—H units adopts a *syn* orientation with respect to the phosphoryl group, while the other exists in an *anti* orientation.

In the crystal, each molecule is hydrogen-bonded to two adjacent molecules through N—H⋯O hydrogen bonds, building R₂²(8) rings (Bernstein *et al.*, 1995), and forming linear arrangements parallel to [100] (Fig. 2, Table 1).

Experimental

To a solution of phenyl dichlorophosphate (2.35 mmol) in chloroform (15 ml), a solution of *meta*-toluidine (9.40 mmol) in chloroform (30 ml) was added at 273 K. After 4 h of stirring, the solvent was evaporated in vacuum. The solid was washed with distilled water. Single crystals, suitable for crystallography, were obtained from a solution of the title compound in chloroform and methanol (4:1) after slow evaporation at room temperature. IR (KBr, cm⁻¹): 3402, 3124, 2890, 2607, 1586, 1494, 1401, 1293, 1182, 1023, 946, 757.

Refinement

Hydrogen atoms in phenyl and NH groups were positioned geometrically and refined as riding with their U_{iso} set to 1.2 U_{eq} of the parent atoms. Hydrogen atoms in methyl groups were positioned geometrically and refined as rotating with their U_{iso} set to 1.5 U_{eq} of the parent atoms.

Figures

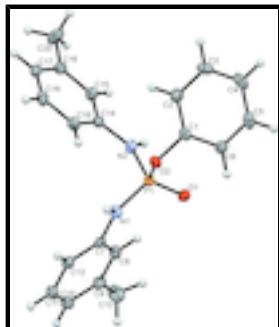


Fig. 1. The molecular structure of the title compound with ellipsoids shown at the 50% probability level.

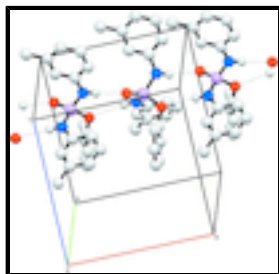


Fig. 2. Part of the crystal structure with hydrogen bonds shown as dotted lines (the C—H hydrogen atoms are omitted for clarity).

Phenyl bis(*m*-tolylamido)phosphinate

Crystal data

$C_{20}H_{21}N_2O_2P$

$M_r = 352.36$

Orthorhombic, $Pna2_1$

$a = 10.1930$ (2) Å

$b = 16.8789$ (3) Å

$c = 10.4588$ (3) Å

$V = 1799.40$ (7) Å³

$Z = 4$

$F(000) = 744$

$D_x = 1.301$ Mg m⁻³

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

Cell parameters from 13732 reflections

$\theta = 3.0$ – 27.2°

$\mu = 0.17$ mm⁻¹

$T = 120$ K

BLOCK, colourless

$0.30 \times 0.30 \times 0.20$ mm

Data collection

Oxford Diffraction Xcalibur Sapphire2 diffractometer

Radiation source: Enhance (Mo) X-ray Source graphite

Detector resolution: 8.4353 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.959$, $T_{\max} = 1.000$

20798 measured reflections

2819 independent reflections

2647 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.0^\circ$

$h = -12 \rightarrow 12$

$k = -20 \rightarrow 20$

$l = -8 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.022$	H-atom parameters constrained
$wR(F^2) = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.046P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2819 reflections	$(\Delta/\sigma)_{\max} = 0.001$
228 parameters	$\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1140 Friedel pairs Flack parameter: $-0.04 (7)$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.72349 (4)	0.24971 (2)	0.91839 (5)	0.01542 (11)
O1	0.61830 (10)	0.19795 (6)	0.96582 (11)	0.0194 (3)
O2	0.82530 (10)	0.20973 (6)	0.82235 (12)	0.0184 (3)
N1	0.83286 (12)	0.28002 (7)	1.02210 (16)	0.0179 (3)
H1B	0.9156	0.2753	0.9993	0.022*
N2	0.65260 (12)	0.32433 (7)	0.84411 (15)	0.0193 (3)
H2A	0.5663	0.3236	0.8462	0.023*
C1	0.78034 (13)	0.16622 (9)	0.71528 (18)	0.0171 (4)
C2	0.76338 (16)	0.20366 (10)	0.60039 (19)	0.0240 (4)
H2B	0.7740	0.2594	0.5937	0.029*
C3	0.73032 (17)	0.15880 (11)	0.49380 (19)	0.0269 (4)
H3A	0.7164	0.1839	0.4136	0.032*
C4	0.71767 (15)	0.07733 (11)	0.5049 (2)	0.0268 (4)
H4A	0.6978	0.0464	0.4315	0.032*
C5	0.73373 (15)	0.04090 (10)	0.6218 (2)	0.0250 (4)
H5A	0.7240	-0.0149	0.6287	0.030*
C6	0.76376 (14)	0.08496 (10)	0.72845 (18)	0.0210 (4)

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H6A	0.7730	0.0603	0.8096	0.025*
C7	0.80937 (15)	0.31292 (8)	1.14394 (17)	0.0167 (4)
C8	0.68462 (15)	0.31324 (9)	1.19924 (19)	0.0217 (4)
H8A	0.6124	0.2915	1.1538	0.026*
C9	0.66553 (16)	0.34526 (9)	1.32059 (18)	0.0230 (4)
C10	0.77167 (16)	0.37699 (9)	1.38620 (19)	0.0243 (4)
H10A	0.7595	0.3989	1.4690	0.029*
C11	0.89519 (16)	0.37666 (9)	1.33103 (18)	0.0240 (4)
H11A	0.9674	0.3985	1.3764	0.029*
C12	0.91464 (15)	0.34502 (9)	1.21091 (18)	0.0208 (4)
H12A	0.9998	0.3451	1.1740	0.025*
C13	0.53035 (17)	0.34519 (12)	1.3794 (2)	0.0357 (5)
H13A	0.5369	0.3315	1.4703	0.054*
H13B	0.4752	0.3061	1.3356	0.054*
H13C	0.4911	0.3979	1.3706	0.054*
C14	0.70709 (15)	0.38945 (9)	0.77755 (18)	0.0177 (4)
C15	0.62148 (15)	0.44120 (9)	0.71729 (17)	0.0198 (4)
H15A	0.5298	0.4319	0.7233	0.024*
C16	0.66624 (16)	0.50657 (9)	0.64807 (17)	0.0212 (4)
C17	0.80128 (15)	0.51933 (9)	0.64137 (18)	0.0219 (4)
H17A	0.8346	0.5635	0.5953	0.026*
C18	0.88614 (15)	0.46829 (9)	0.70120 (18)	0.0232 (4)
H18A	0.9778	0.4781	0.6959	0.028*
C19	0.84227 (15)	0.40269 (9)	0.76930 (18)	0.0201 (4)
H19A	0.9027	0.3677	0.8093	0.024*
C20	0.57089 (18)	0.55834 (10)	0.57636 (19)	0.0327 (5)
H20A	0.6048	0.6126	0.5728	0.049*
H20B	0.4860	0.5581	0.6204	0.049*
H20C	0.5598	0.5380	0.4893	0.049*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01458 (18)	0.01538 (17)	0.0163 (2)	0.00013 (14)	0.0010 (2)	-0.00022 (17)
O1	0.0181 (5)	0.0198 (5)	0.0203 (7)	-0.0003 (4)	0.0018 (5)	0.0011 (5)
O2	0.0169 (5)	0.0202 (5)	0.0180 (7)	-0.0005 (4)	0.0004 (5)	-0.0025 (5)
N1	0.0127 (6)	0.0217 (6)	0.0195 (8)	0.0011 (5)	0.0024 (6)	-0.0030 (6)
N2	0.0130 (6)	0.0196 (6)	0.0253 (9)	-0.0001 (5)	0.0007 (6)	0.0031 (6)
C1	0.0131 (7)	0.0208 (7)	0.0175 (10)	0.0007 (6)	0.0014 (7)	-0.0034 (7)
C2	0.0274 (9)	0.0203 (8)	0.0242 (12)	0.0009 (6)	0.0020 (8)	-0.0008 (8)
C3	0.0280 (9)	0.0370 (10)	0.0158 (11)	0.0028 (8)	0.0005 (8)	0.0005 (9)
C4	0.0189 (8)	0.0329 (9)	0.0287 (12)	-0.0002 (7)	-0.0021 (8)	-0.0120 (9)
C5	0.0182 (8)	0.0197 (8)	0.0372 (13)	-0.0017 (6)	0.0017 (8)	-0.0078 (9)
C6	0.0182 (8)	0.0218 (8)	0.0231 (11)	-0.0003 (6)	0.0002 (7)	0.0024 (8)
C7	0.0198 (7)	0.0129 (7)	0.0174 (10)	0.0019 (6)	0.0017 (7)	0.0026 (7)
C8	0.0191 (8)	0.0210 (7)	0.0250 (11)	-0.0026 (6)	0.0013 (7)	-0.0013 (8)
C9	0.0257 (9)	0.0214 (8)	0.0220 (10)	-0.0005 (6)	0.0066 (8)	-0.0007 (8)
C10	0.0333 (9)	0.0197 (7)	0.0197 (12)	0.0007 (7)	0.0019 (8)	-0.0024 (7)

C11	0.0246 (8)	0.0235 (8)	0.0240 (11)	-0.0019 (6)	-0.0042 (8)	-0.0030 (8)
C12	0.0190 (8)	0.0212 (7)	0.0222 (10)	-0.0005 (6)	0.0009 (7)	0.0018 (7)
C13	0.0302 (10)	0.0449 (10)	0.0322 (13)	-0.0055 (8)	0.0109 (9)	-0.0121 (9)
C14	0.0204 (8)	0.0161 (7)	0.0167 (10)	-0.0013 (6)	0.0022 (7)	-0.0024 (7)
C15	0.0159 (7)	0.0207 (7)	0.0229 (10)	0.0001 (6)	0.0026 (7)	-0.0005 (8)
C16	0.0254 (9)	0.0190 (7)	0.0190 (10)	0.0015 (6)	0.0038 (7)	-0.0015 (8)
C17	0.0269 (9)	0.0189 (8)	0.0198 (11)	-0.0057 (7)	0.0048 (8)	-0.0004 (8)
C18	0.0182 (8)	0.0232 (8)	0.0281 (12)	-0.0050 (6)	0.0036 (8)	-0.0038 (8)
C19	0.0164 (8)	0.0201 (7)	0.0240 (10)	0.0015 (6)	-0.0009 (7)	-0.0014 (7)
C20	0.0334 (10)	0.0279 (9)	0.0368 (13)	0.0033 (7)	0.0044 (9)	0.0118 (9)

Geometric parameters (Å, °)

P1—O1	1.4692 (11)	C9—C10	1.389 (2)
P1—O2	1.5941 (12)	C9—C13	1.509 (2)
P1—N1	1.6374 (15)	C10—C11	1.385 (2)
P1—N2	1.6469 (13)	C10—H10A	0.9500
O2—C1	1.416 (2)	C11—C12	1.379 (3)
N1—C7	1.410 (2)	C11—H11A	0.9500
N1—H1B	0.8800	C12—H12A	0.9500
N2—C14	1.415 (2)	C13—H13A	0.9800
N2—H2A	0.8800	C13—H13B	0.9800
C1—C2	1.369 (3)	C13—H13C	0.9800
C1—C6	1.389 (2)	C14—C15	1.386 (2)
C2—C3	1.389 (3)	C14—C19	1.399 (2)
C2—H2B	0.9500	C15—C16	1.396 (2)
C3—C4	1.386 (3)	C15—H15A	0.9500
C3—H3A	0.9500	C16—C17	1.395 (2)
C4—C5	1.378 (3)	C16—C20	1.507 (2)
C4—H4A	0.9500	C17—C18	1.372 (2)
C5—C6	1.375 (3)	C17—H17A	0.9500
C5—H5A	0.9500	C18—C19	1.390 (2)
C6—H6A	0.9500	C18—H18A	0.9500
C7—C12	1.391 (2)	C19—H19A	0.9500
C7—C8	1.397 (2)	C20—H20A	0.9800
C8—C9	1.393 (3)	C20—H20B	0.9800
C8—H8A	0.9500	C20—H20C	0.9800
O1—P1—O2	115.86 (6)	C11—C10—C9	120.06 (17)
O1—P1—N1	117.32 (8)	C11—C10—H10A	120.0
O2—P1—N1	96.11 (6)	C9—C10—H10A	120.0
O1—P1—N2	107.09 (6)	C12—C11—C10	120.77 (16)
O2—P1—N2	108.19 (7)	C12—C11—H11A	119.6
N1—P1—N2	111.85 (7)	C10—C11—H11A	119.6
C1—O2—P1	120.49 (9)	C11—C12—C7	119.90 (15)
C7—N1—P1	127.29 (11)	C11—C12—H12A	120.0
C7—N1—H1B	116.4	C7—C12—H12A	120.0
P1—N1—H1B	116.4	C9—C13—H13A	109.5
C14—N2—P1	130.85 (11)	C9—C13—H13B	109.5
C14—N2—H2A	114.6	H13A—C13—H13B	109.5

supplementary materials

P1—N2—H2A	114.6	C9—C13—H13C	109.5
C2—C1—C6	121.86 (17)	H13A—C13—H13C	109.5
C2—C1—O2	119.73 (13)	H13B—C13—H13C	109.5
C6—C1—O2	118.25 (16)	C15—C14—C19	119.44 (15)
C1—C2—C3	118.92 (15)	C15—C14—N2	117.78 (13)
C1—C2—H2B	120.5	C19—C14—N2	122.77 (15)
C3—C2—H2B	120.5	C14—C15—C16	121.87 (14)
C4—C3—C2	119.75 (18)	C14—C15—H15A	119.1
C4—C3—H3A	120.1	C16—C15—H15A	119.1
C2—C3—H3A	120.1	C17—C16—C15	118.07 (15)
C5—C4—C3	120.38 (18)	C17—C16—C20	121.46 (15)
C5—C4—H4A	119.8	C15—C16—C20	120.37 (14)
C3—C4—H4A	119.8	C18—C17—C16	120.14 (15)
C6—C5—C4	120.32 (15)	C18—C17—H17A	119.9
C6—C5—H5A	119.8	C16—C17—H17A	119.9
C4—C5—H5A	119.8	C17—C18—C19	122.08 (14)
C5—C6—C1	118.72 (17)	C17—C18—H18A	119.0
C5—C6—H6A	120.6	C19—C18—H18A	119.0
C1—C6—H6A	120.6	C18—C19—C14	118.40 (15)
C12—C7—C8	119.48 (16)	C18—C19—H19A	120.8
C12—C7—N1	118.51 (14)	C14—C19—H19A	120.8
C8—C7—N1	122.01 (15)	C16—C20—H20A	109.5
C9—C8—C7	120.39 (16)	C16—C20—H20B	109.5
C9—C8—H8A	119.8	H20A—C20—H20B	109.5
C7—C8—H8A	119.8	C16—C20—H20C	109.5
C10—C9—C8	119.41 (15)	H20A—C20—H20C	109.5
C10—C9—C13	120.67 (17)	H20B—C20—H20C	109.5
C8—C9—C13	119.92 (16)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1B \cdots O1 ⁱ	0.88	2.14	2.9917 (16)	162
N2—H2A \cdots O2 ⁱⁱ	0.88	2.53	3.3929 (16)	166

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

Fig. 1

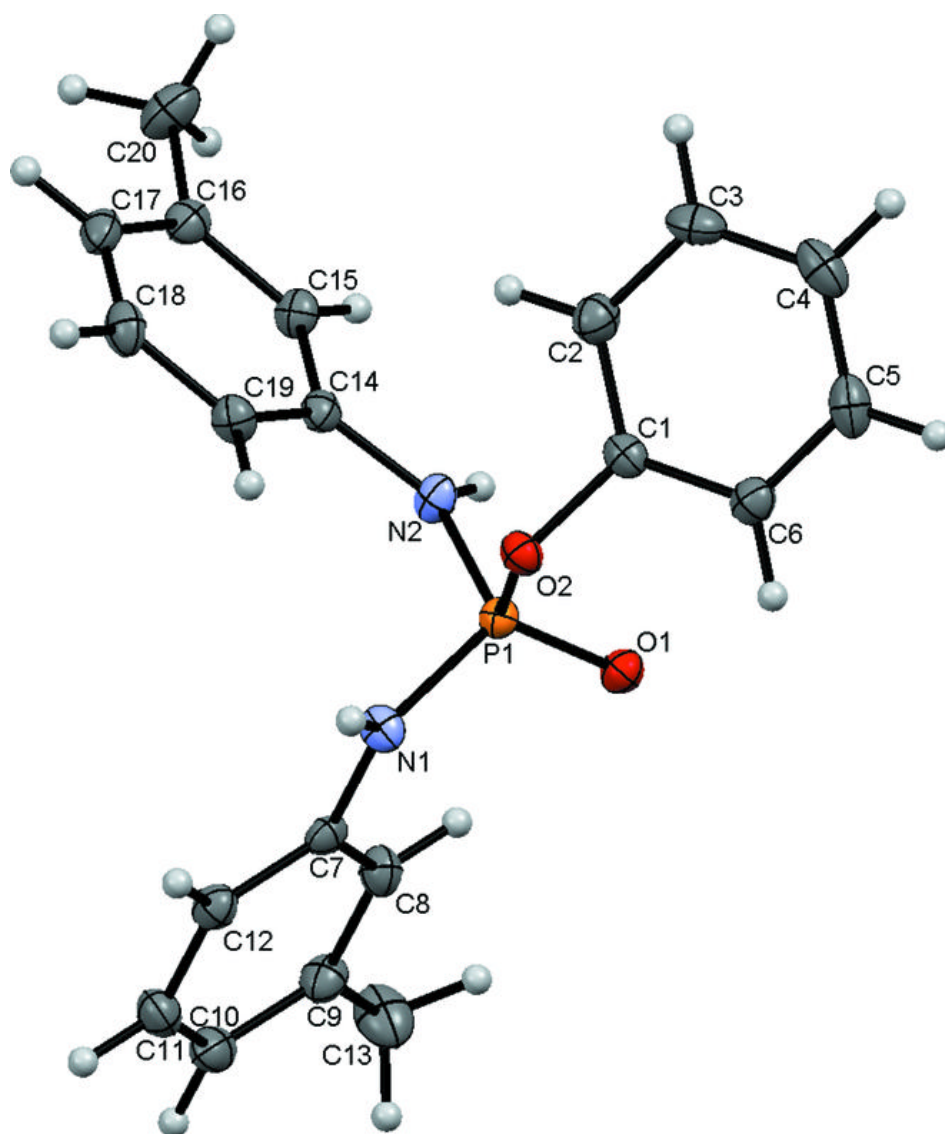


Fig. 2

