

# Redetermined structure of diphenylphosphonimidotriphenylphosphorane: location of the hydrogen atoms and analysis of the intermolecular interactions

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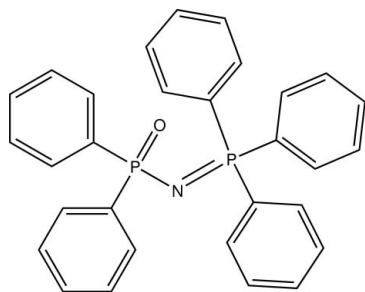
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Key indicators: single-crystal X-ray study;  $T = 200$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.027;  $wR$  factor = 0.076; data-to-parameter ratio = 14.7.

The title compound,  $\text{C}_{30}\text{H}_{25}\text{NOP}_2$ , is a bulky phosphazene derivative. Its previous crystal structure [Cameron *et al.* (1979). *Acta Cryst. B* **35**, 1373–1377] is confirmed and its H atoms have been located in the present study. The formal  $\text{P}=\text{N}$  double bond is about 0.05 Å shorter than the  $\text{P}-\text{N}$  single bond and the large  $\text{P}=\text{N}-\text{P}$  bond angle reflects the steric strain in the molecule. An intramolecular  $\text{C}-\text{H}\cdots\text{O}$  interaction occurs. In the crystal, short  $\text{C}-\text{H}\cdots\text{O}$  contacts connect the molecules into chains propagating in [011], which are cross-linked *via*  $\text{C}-\text{H}\cdots\pi$  interactions, generating a three-dimensional network. Aromatic  $\pi-\pi$  stacking also occurs [shortest centroid-centroid separation = 3.6012 (11) Å].

## Related literature

For the previous structure determination, see: Cameron *et al.* (1979). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



## Experimental

### Crystal data

$\text{C}_{30}\text{H}_{25}\text{NOP}_2$	$V = 2387.9$ (3) Å <sup>3</sup>
$M_r = 477.45$	$Z = 4$
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
$a = 17.6607$ (12) Å	$\mu = 0.21$ mm <sup>-1</sup>
$b = 15.1593$ (10) Å	$T = 200$ K
$c = 8.9192$ (6) Å	$0.88 \times 0.42 \times 0.31$ mm

### Data collection

Bruker APEXII CCD diffractometer	4498 independent reflections
12511 measured reflections	4348 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.021$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$	H-atom parameters constrained
$wR(F^2) = 0.076$	$\Delta\rho_{\text{max}} = 0.21$ e Å <sup>-3</sup>
$S = 1.11$	$\Delta\rho_{\text{min}} = -0.25$ e Å <sup>-3</sup>
4498 reflections	Absolute structure: Flack (1983),
307 parameters	1332 Friedel pairs
1 restraint	Flack parameter: $-0.03$ (6)

**Table 1**

Selected geometric parameters (Å, °).

P1–N1	1.6014 (13)	P2–N1	1.5532 (13)
P2–N1–P1	146.35 (12)		

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
C32–H32 <sup>i</sup> ⋯O1	0.95	2.34	3.257 (2)	163
C43–H43 <sup>i</sup> ⋯O1 <sup>i</sup>	0.95	2.34	3.257 (2)	162
C45–H45 <sup>i</sup> ⋯Cg1 <sup>ii</sup>	0.95	2.92	3.846 (2)	165
C55–H55 <sup>i</sup> ⋯Cg2 <sup>iii</sup>	0.95	2.73	3.644 (2)	163

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP III* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

The authors thank Mr Gerhard Schneeberger for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5826).

**References**

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**supplementary materials**

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## Redetermined structure of diphenylphosphonimidotriphenylphosphorane: location of the hydrogen atoms and analysis of the intermolecular interactions

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### Comment

For many main group elements as well as transition and rare earth metals, preferred coordination numbers in coordination compounds are apparent. While coordination numbers of 4, 6 and 8 have been found to be dominant in most cases and, as a consequence, vast structural information has been collected for such compounds in solution and in the solid state, information about other coordination numbers is comparatively limited. Especially for smaller coordination numbers the literature is scant or hitherto completely unknown for many elements. One reason for this certainly is that sometimes challenging synthesis procedures have to be followed and, thus, a general but simple synthetic protocol is desirable. Since such compounds may act as versatile and potent catalysts in many industrial processes and might even show interesting pharmacological properties, we were interested in developing an easy-access-route for their synthesis. Applying bulky ligands might open up a pathway in this aspect. In order to be able to compare metrical parameters in envisioned reaction products, we determined the crystal structure of the title compound. The latter one has already been reported earlier (Cameron *et al.* (1979)), however, no hydrogen atoms were included in the refinement thus ruling out the possibility to assess the role of C–H $\cdots$ X contacts.

The length of the N–P bonds deviate by 0.05 Å with the – formal – P–N-double bond found at around 1.55 Å. The P–N–P angle was measured at more than 146°. The marked widening of this angle in comparison to the value expected for a  $sp^2$ -hybridized nitrogen atom can be explained by the repulsive interaction of the phenyl-moieties on both P atoms. The phenyl groups on each phosphorus atom are approximately orientated perpendicular to each other. The least-squares planes defined by their carbon atoms intersect at an angle of 82.19 (6)° in case of the P(O)Ph<sub>2</sub>-moiety and at angles of 79.82 (5)°, 80.91 (6)° and 83.28 (6)°, respectively, in case of the PPh<sub>3</sub>-moiety. Due to the formation of an intramolecular C–H $\cdots$ O contact (*see below*), the least-squares plane defined by the P(O)–N–P motif encloses an angle of only 29.40 (9)° with one of the aromatic carbocycles on the PPh<sub>3</sub>-moiety (Fig. 1). For the same reason, both phenyl groups of the P(O)Ph<sub>2</sub>-moiety adopt a slightly eclipsed conformation with respect to the P(O) motif, the respective dihedral angles were found at about 19° and 26°.

In the crystal structure, intermolecular C–H $\cdots$ O contacts are present whose range falls by more than 0.3 Å below the sum of van-der-Waals radii of the atoms participating. These can be observed between one of the H atoms in *meta*-position of a phenyl group on the PPh<sub>3</sub>-moiety and the O atom of the P(O)Ph<sub>2</sub>-moiety and connect the molecules to infinite chains along [0 1 1] (Fig. 2). Furthermore, intramolecular C–H $\cdots$ O contacts invariably involving hydrogen atoms in *ortho*-position on one of the phenyl groups of the PPh<sub>3</sub>-moiety as well as both phenyl groups of the P(O)Ph<sub>2</sub>-moiety are present. However, the latter two ones are not very pronounced. Additionally, a set of C–H $\cdots$  $\pi$  contacts are evident involving H atoms and aromatic systems both on the PPh<sub>3</sub>-moiety as well as the P(O)Ph<sub>2</sub>-moiety. Their details are listed in Table 1 (with  $C_g(1) = C41\cdots C46$ ,  $C_g(2) = C31\cdots C36$  and  $C_g(3) = C11\cdots C16$ ). In total, the C–H $\cdots$ O contacts as well as the C–H $\cdots$  $\pi$  contacts connect the molecules to a three dimensional network. In terms of graph-set analysis (Etter *et al.* (1990); Bernstein *et al.* (1995)), the intermolecular C–H $\cdots$ O contacts can be assigned a  $C^1_1(8)$  descriptor on the unitary level while the intramolecular C–H $\cdots$ O

## supplementary materials

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contact involving the phenyl group of the PPh<sub>3</sub>-moiety necessitates a  $S^1_1(7)$  descriptor. For the other two intramolecular C—H...O contacts, a  $S^1_1(5)$  each is feasible. An analysis of  $C_g \cdots C_g$  interactions shows the closest distance between two centers of gravity to occur between a phenyl group on the PPh<sub>3</sub>-moiety and a phenyl group on the P(O)Ph<sub>2</sub>-moiety. The distance was measured at 3.6012 (11) Å.

The packing of the title compound in the crystal structure is shown in Figure 3.

### Experimental

The compound was obtained commercially (Aldrich). A colourless block suitable for the X-ray diffraction study were taken directly from the provided material.

### Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with  $U(H)$  set to  $1.2U_{eq}(C)$ .

### Figures

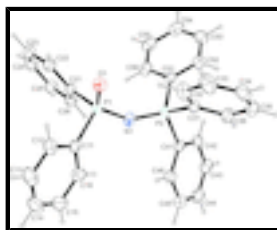


Fig. 1. The molecular structure of the title compound, with anisotropic displacement ellipsoids (drawn at 50% probability level).

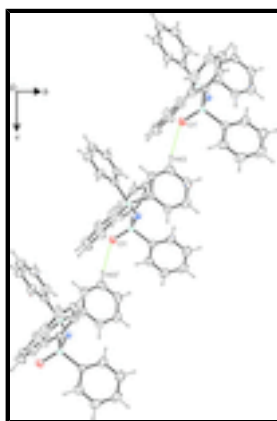


Fig. 2. Intermolecular contacts, viewed along  $[-1\ 0\ 0]$ . Symmetry operators: i  $-x + 1/2, y - 1/2, z + 1/2$ ; ii  $-x + 1/2, y + 1/2, z - 1/2$ .

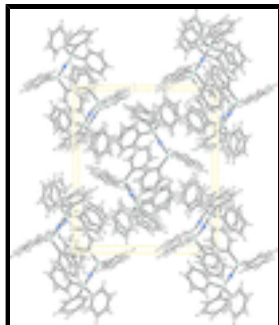


Fig. 3. Crystal packing of the title compound, viewed along [0 0 - 1] (anisotropic displacement ellipsoids drawn at 50% probability level).

### Diphenylphosphonimidotriphenylphosphorane

#### Crystal data

$C_{30}H_{25}NOP_2$	$D_x = 1.328 \text{ Mg m}^{-3}$
$M_r = 477.45$	Melting point = 442–445 K
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2c -2n	Cell parameters from 9977 reflections
$a = 17.6607 (12) \text{ \AA}$	$\theta = 2.7\text{--}28.3^\circ$
$b = 15.1593 (10) \text{ \AA}$	$\mu = 0.21 \text{ mm}^{-1}$
$c = 8.9192 (6) \text{ \AA}$	$T = 200 \text{ K}$
$V = 2387.9 (3) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.88 \times 0.42 \times 0.31 \text{ mm}$
$F(000) = 1000$	

#### Data collection

Bruker APEXII CCD diffractometer	4348 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.021$
graphite	$\theta_{\text{max}} = 28.3^\circ$ , $\theta_{\text{min}} = 2.9^\circ$
$\varphi$ and $\omega$ scans	$h = -22 \rightarrow 23$
12511 measured reflections	$k = -20 \rightarrow 16$
4498 independent reflections	$l = -7 \rightarrow 11$

#### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H-atom parameters constrained
$wR(F^2) = 0.076$	$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.5077P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
4498 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
307 parameters	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$

# supplementary materials

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1 restraint

Absolute structure: Flack (1983), 1332 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: -0.03 (6)

## *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.083912 (19)	0.16050 (2)	0.55082 (5)	0.02138 (9)
P2	0.227542 (18)	0.05280 (2)	0.53590 (6)	0.02115 (9)
O1	0.08330 (6)	0.21271 (8)	0.40946 (15)	0.0276 (3)
N1	0.15673 (7)	0.10297 (9)	0.59418 (19)	0.0272 (3)
C11	0.06899 (9)	0.23246 (11)	0.7102 (2)	0.0251 (3)
C12	0.02931 (10)	0.31087 (11)	0.6883 (3)	0.0327 (4)
H12	0.0132	0.3268	0.5903	0.039*
C13	0.01304 (11)	0.36602 (13)	0.8084 (3)	0.0420 (5)
H13	-0.0139	0.4194	0.7924	0.050*
C14	0.03602 (12)	0.34320 (14)	0.9502 (3)	0.0440 (5)
H14	0.0252	0.3813	1.0321	0.053*
C15	0.07484 (12)	0.26496 (15)	0.9752 (3)	0.0426 (5)
H15	0.0898	0.2489	1.0738	0.051*
C16	0.09162 (10)	0.21044 (13)	0.8546 (2)	0.0336 (4)
H16	0.1190	0.1573	0.8710	0.040*
C21	0.00231 (8)	0.08902 (9)	0.5576 (2)	0.0228 (3)
C22	-0.05883 (9)	0.10711 (11)	0.4654 (2)	0.0294 (4)
H22	-0.0563	0.1549	0.3968	0.035*
C23	-0.12411 (10)	0.05546 (13)	0.4728 (2)	0.0358 (4)
H23	-0.1663	0.0687	0.4106	0.043*
C24	-0.12731 (9)	-0.01467 (12)	0.5705 (2)	0.0357 (4)
H24	-0.1714	-0.0504	0.5741	0.043*
C25	-0.06683 (10)	-0.03348 (12)	0.6635 (2)	0.0331 (4)
H25	-0.0693	-0.0819	0.7309	0.040*
C26	-0.00227 (9)	0.01904 (11)	0.6576 (2)	0.0286 (4)
H26	0.0391	0.0069	0.7225	0.034*
C31	0.29089 (9)	0.11812 (11)	0.4214 (2)	0.0250 (3)
C32	0.26144 (10)	0.18326 (12)	0.3296 (2)	0.0352 (4)
H32	0.2087	0.1958	0.3315	0.042*
C33	0.30909 (12)	0.23043 (13)	0.2347 (3)	0.0421 (5)
H33	0.2887	0.2747	0.1711	0.051*
C34	0.38560 (11)	0.21312 (13)	0.2327 (2)	0.0378 (4)
H34	0.4179	0.2455	0.1676	0.045*
C35	0.41530 (10)	0.14935 (15)	0.3242 (3)	0.0404 (5)
H35	0.4681	0.1377	0.3222	0.048*
C36	0.36879 (10)	0.10175 (14)	0.4197 (2)	0.0350 (4)
H36	0.3898	0.0581	0.4839	0.042*
C41	0.28149 (8)	0.01741 (10)	0.6966 (2)	0.0222 (3)
C42	0.31729 (9)	-0.06444 (11)	0.7022 (2)	0.0288 (4)
H42	0.3146	-0.1031	0.6186	0.035*
C43	0.35690 (10)	-0.08954 (12)	0.8295 (2)	0.0340 (4)
H43	0.3809	-0.1456	0.8334	0.041*

C44	0.36148 (10)	-0.03317 (14)	0.9506 (2)	0.0375 (4)
H44	0.3886	-0.0504	1.0378	0.045*
C45	0.32647 (11)	0.04883 (13)	0.9453 (2)	0.0372 (4)
H45	0.3301	0.0878	1.0284	0.045*
C46	0.28624 (9)	0.07380 (11)	0.8190 (2)	0.0290 (4)
H46	0.2618	0.1296	0.8161	0.035*
C51	0.20882 (9)	-0.04527 (11)	0.4262 (2)	0.0252 (3)
C52	0.25999 (10)	-0.07830 (13)	0.3230 (2)	0.0335 (4)
H52	0.3072	-0.0494	0.3088	0.040*
C53	0.24317 (12)	-0.15319 (13)	0.2399 (3)	0.0402 (5)
H53	0.2786	-0.1757	0.1695	0.048*
C54	0.17403 (12)	-0.19490 (12)	0.2607 (3)	0.0396 (5)
H54	0.1617	-0.2456	0.2030	0.047*
C55	0.12324 (11)	-0.16321 (12)	0.3645 (3)	0.0369 (4)
H55	0.0763	-0.1926	0.3792	0.044*
C56	0.14026 (9)	-0.08887 (11)	0.4474 (2)	0.0310 (4)
H56	0.1050	-0.0674	0.5192	0.037*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
P1	0.01837 (15)	0.02043 (17)	0.0254 (2)	0.00090 (11)	0.00122 (16)	0.00224 (17)
P2	0.01691 (14)	0.02080 (17)	0.0257 (2)	0.00025 (12)	-0.00016 (17)	0.00197 (17)
O1	0.0292 (6)	0.0257 (6)	0.0280 (7)	0.0001 (4)	0.0027 (5)	0.0053 (5)
N1	0.0199 (5)	0.0267 (6)	0.0351 (8)	0.0034 (5)	0.0010 (5)	0.0037 (6)
C11	0.0199 (6)	0.0245 (8)	0.0308 (9)	-0.0035 (6)	0.0018 (6)	-0.0028 (7)
C12	0.0298 (8)	0.0289 (8)	0.0395 (11)	0.0029 (6)	0.0048 (8)	0.0001 (8)
C13	0.0428 (10)	0.0290 (9)	0.0542 (14)	0.0017 (8)	0.0125 (10)	-0.0085 (9)
C14	0.0450 (10)	0.0417 (11)	0.0452 (13)	-0.0110 (8)	0.0144 (10)	-0.0188 (10)
C15	0.0421 (10)	0.0524 (12)	0.0333 (11)	-0.0127 (9)	-0.0004 (9)	-0.0061 (9)
C16	0.0309 (8)	0.0361 (9)	0.0338 (10)	-0.0025 (7)	-0.0022 (8)	-0.0020 (8)
C21	0.0199 (6)	0.0213 (6)	0.0271 (9)	0.0013 (5)	0.0022 (6)	-0.0025 (7)
C22	0.0264 (7)	0.0307 (8)	0.0312 (9)	0.0017 (6)	-0.0034 (7)	-0.0001 (7)
C23	0.0232 (7)	0.0444 (10)	0.0398 (11)	0.0007 (7)	-0.0051 (7)	-0.0080 (9)
C24	0.0265 (7)	0.0418 (9)	0.0388 (11)	-0.0100 (6)	0.0079 (7)	-0.0124 (8)
C25	0.0346 (8)	0.0314 (8)	0.0332 (10)	-0.0077 (7)	0.0073 (8)	0.0006 (8)
C26	0.0258 (7)	0.0317 (8)	0.0282 (9)	-0.0022 (6)	0.0002 (7)	0.0028 (7)
C31	0.0232 (7)	0.0257 (7)	0.0262 (8)	-0.0035 (6)	0.0031 (6)	-0.0017 (7)
C32	0.0318 (8)	0.0326 (9)	0.0411 (12)	0.0050 (7)	0.0094 (8)	0.0071 (8)
C33	0.0481 (10)	0.0323 (9)	0.0460 (12)	0.0030 (8)	0.0174 (10)	0.0110 (9)
C34	0.0417 (10)	0.0398 (10)	0.0320 (10)	-0.0152 (8)	0.0124 (8)	-0.0041 (8)
C35	0.0238 (8)	0.0626 (13)	0.0348 (11)	-0.0106 (8)	0.0025 (8)	-0.0013 (10)
C36	0.0243 (7)	0.0480 (10)	0.0328 (10)	-0.0023 (7)	-0.0012 (7)	0.0056 (8)
C41	0.0186 (6)	0.0234 (7)	0.0247 (8)	-0.0013 (5)	-0.0001 (6)	0.0030 (6)
C42	0.0257 (7)	0.0273 (8)	0.0335 (10)	0.0035 (6)	-0.0004 (7)	-0.0010 (7)
C43	0.0290 (8)	0.0324 (8)	0.0407 (11)	0.0042 (6)	-0.0041 (8)	0.0091 (8)
C44	0.0316 (8)	0.0503 (11)	0.0305 (10)	0.0008 (7)	-0.0061 (7)	0.0094 (9)
C45	0.0346 (9)	0.0451 (10)	0.0319 (10)	-0.0010 (7)	-0.0040 (8)	-0.0076 (9)



## supplementary materials

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C46	0.0255 (7)	0.0279 (8)	0.0335 (10)	0.0004 (6)	-0.0005 (7)	-0.0030 (8)
C51	0.0232 (7)	0.0239 (7)	0.0284 (9)	0.0016 (5)	-0.0052 (6)	0.0014 (6)
C52	0.0289 (8)	0.0379 (9)	0.0339 (11)	0.0013 (7)	-0.0019 (7)	-0.0064 (8)
C53	0.0417 (10)	0.0382 (10)	0.0407 (12)	0.0087 (8)	-0.0027 (9)	-0.0109 (9)
C54	0.0471 (10)	0.0272 (8)	0.0443 (12)	0.0033 (7)	-0.0163 (9)	-0.0032 (8)
C55	0.0346 (8)	0.0282 (8)	0.0480 (12)	-0.0054 (7)	-0.0104 (9)	0.0039 (8)
C56	0.0265 (7)	0.0286 (8)	0.0379 (10)	-0.0019 (6)	-0.0023 (7)	0.0012 (8)

### *Geometric parameters (Å, °)*

P1—O1	1.4887 (13)	C32—C33	1.392 (3)
P1—N1	1.6014 (13)	C32—H32	0.9500
P1—C21	1.8040 (15)	C33—C34	1.377 (3)
P1—C11	1.8109 (18)	C33—H33	0.9500
P2—N1	1.5532 (13)	C34—C35	1.370 (3)
P2—C41	1.8027 (17)	C34—H34	0.9500
P2—C31	1.8099 (17)	C35—C36	1.386 (3)
P2—C51	1.8099 (17)	C35—H35	0.9500
C11—C16	1.389 (3)	C36—H36	0.9500
C11—C12	1.394 (2)	C41—C46	1.389 (2)
C12—C13	1.389 (3)	C41—C42	1.394 (2)
C12—H12	0.9500	C42—C43	1.387 (3)
C13—C14	1.373 (4)	C42—H42	0.9500
C13—H13	0.9500	C43—C44	1.380 (3)
C14—C15	1.388 (3)	C43—H43	0.9500
C14—H14	0.9500	C44—C45	1.389 (3)
C15—C16	1.388 (3)	C44—H44	0.9500
C15—H15	0.9500	C45—C46	1.384 (3)
C16—H16	0.9500	C45—H45	0.9500
C21—C22	1.385 (2)	C46—H46	0.9500
C21—C26	1.388 (2)	C51—C52	1.384 (3)
C22—C23	1.395 (2)	C51—C56	1.392 (2)
C22—H22	0.9500	C52—C53	1.388 (3)
C23—C24	1.376 (3)	C52—H52	0.9500
C23—H23	0.9500	C53—C54	1.387 (3)
C24—C25	1.382 (3)	C53—H53	0.9500
C24—H24	0.9500	C54—C55	1.375 (3)
C25—C26	1.392 (2)	C54—H54	0.9500
C25—H25	0.9500	C55—C56	1.381 (3)
C26—H26	0.9500	C55—H55	0.9500
C31—C32	1.384 (3)	C56—H56	0.9500
C31—C36	1.398 (2)		
O1—P1—N1	120.00 (8)	C31—C32—C33	119.94 (17)
O1—P1—C21	110.00 (8)	C31—C32—H32	120.0
N1—P1—C21	107.83 (7)	C33—C32—H32	120.0
O1—P1—C11	110.08 (7)	C34—C33—C32	120.2 (2)
N1—P1—C11	104.80 (8)	C34—C33—H33	119.9
C21—P1—C11	102.66 (7)	C32—C33—H33	119.9
N1—P2—C41	107.77 (8)	C35—C34—C33	120.16 (18)

N1—P2—C31	114.75 (7)	C35—C34—H34	119.9
C41—P2—C31	106.55 (7)	C33—C34—H34	119.9
N1—P2—C51	115.84 (7)	C34—C35—C36	120.47 (17)
C41—P2—C51	106.37 (8)	C34—C35—H35	119.8
C31—P2—C51	104.91 (8)	C36—C35—H35	119.8
P2—N1—P1	146.35 (12)	C35—C36—C31	119.81 (18)
C16—C11—C12	118.64 (17)	C35—C36—H36	120.1
C16—C11—P1	122.75 (13)	C31—C36—H36	120.1
C12—C11—P1	118.51 (15)	C46—C41—C42	119.50 (16)
C13—C12—C11	120.6 (2)	C46—C41—P2	118.28 (12)
C13—C12—H12	119.7	C42—C41—P2	122.22 (14)
C11—C12—H12	119.7	C43—C42—C41	120.15 (18)
C14—C13—C12	119.86 (19)	C43—C42—H42	119.9
C14—C13—H13	120.1	C41—C42—H42	119.9
C12—C13—H13	120.1	C44—C43—C42	120.04 (17)
C13—C14—C15	120.6 (2)	C44—C43—H43	120.0
C13—C14—H14	119.7	C42—C43—H43	120.0
C15—C14—H14	119.7	C43—C44—C45	120.09 (18)
C14—C15—C16	119.3 (2)	C43—C44—H44	120.0
C14—C15—H15	120.3	C45—C44—H44	120.0
C16—C15—H15	120.3	C46—C45—C44	120.07 (18)
C15—C16—C11	120.92 (18)	C46—C45—H45	120.0
C15—C16—H16	119.5	C44—C45—H45	120.0
C11—C16—H16	119.5	C45—C46—C41	120.15 (16)
C22—C21—C26	119.16 (14)	C45—C46—H46	119.9
C22—C21—P1	118.95 (13)	C41—C46—H46	119.9
C26—C21—P1	121.82 (12)	C52—C51—C56	119.09 (16)
C21—C22—C23	120.35 (17)	C52—C51—P2	122.51 (13)
C21—C22—H22	119.8	C56—C51—P2	118.39 (14)
C23—C22—H22	119.8	C51—C52—C53	120.76 (17)
C24—C23—C22	119.84 (17)	C51—C52—H52	119.6
C24—C23—H23	120.1	C53—C52—H52	119.6
C22—C23—H23	120.1	C54—C53—C52	119.33 (19)
C23—C24—C25	120.51 (15)	C54—C53—H53	120.3
C23—C24—H24	119.7	C52—C53—H53	120.3
C25—C24—H24	119.7	C55—C54—C53	120.31 (18)
C24—C25—C26	119.50 (18)	C55—C54—H54	119.8
C24—C25—H25	120.3	C53—C54—H54	119.8
C26—C25—H25	120.3	C54—C55—C56	120.23 (18)
C21—C26—C25	120.63 (16)	C54—C55—H55	119.9
C21—C26—H26	119.7	C56—C55—H55	119.9
C25—C26—H26	119.7	C55—C56—C51	120.27 (18)
C32—C31—C36	119.36 (16)	C55—C56—H56	119.9
C32—C31—P2	119.46 (12)	C51—C56—H56	119.9
C36—C31—P2	121.14 (14)		
C41—P2—N1—P1	173.79 (15)	C41—P2—C31—C36	29.82 (17)
C31—P2—N1—P1	55.30 (19)	C51—P2—C31—C36	-82.72 (17)
C51—P2—N1—P1	-67.26 (19)	C36—C31—C32—C33	1.3 (3)
O1—P1—N1—P2	-25.4 (2)	P2—C31—C32—C33	-176.30 (16)

## supplementary materials

C21—P1—N1—P2	101.52 (17)	C31—C32—C33—C34	-0.6 (3)
C11—P1—N1—P2	-149.62 (16)	C32—C33—C34—C35	0.0 (3)
O1—P1—C11—C16	-157.41 (13)	C33—C34—C35—C36	-0.1 (3)
N1—P1—C11—C16	-27.08 (16)	C34—C35—C36—C31	0.8 (3)
C21—P1—C11—C16	85.50 (15)	C32—C31—C36—C35	-1.4 (3)
O1—P1—C11—C12	26.37 (15)	P2—C31—C36—C35	176.14 (16)
N1—P1—C11—C12	156.70 (13)	N1—P2—C41—C46	-38.07 (14)
C21—P1—C11—C12	-90.72 (14)	C31—P2—C41—C46	85.57 (13)
C16—C11—C12—C13	0.3 (2)	C51—P2—C41—C46	-162.90 (13)
P1—C11—C12—C13	176.66 (14)	N1—P2—C41—C42	141.06 (13)
C11—C12—C13—C14	-0.2 (3)	C31—P2—C41—C42	-95.30 (14)
C12—C13—C14—C15	-0.6 (3)	C51—P2—C41—C42	16.23 (16)
C13—C14—C15—C16	1.2 (3)	C46—C41—C42—C43	0.5 (2)
C14—C15—C16—C11	-1.1 (3)	P2—C41—C42—C43	-178.67 (13)
C12—C11—C16—C15	0.3 (3)	C41—C42—C43—C44	-0.5 (3)
P1—C11—C16—C15	-175.88 (14)	C42—C43—C44—C45	0.0 (3)
O1—P1—C21—C22	-18.58 (16)	C43—C44—C45—C46	0.7 (3)
N1—P1—C21—C22	-151.12 (14)	C44—C45—C46—C41	-0.8 (3)
C11—P1—C21—C22	98.56 (15)	C42—C41—C46—C45	0.2 (2)
O1—P1—C21—C26	164.52 (14)	P2—C41—C46—C45	179.36 (14)
N1—P1—C21—C26	31.99 (17)	N1—P2—C51—C52	155.63 (15)
C11—P1—C21—C26	-78.33 (15)	C41—P2—C51—C52	-84.66 (17)
C26—C21—C22—C23	-0.1 (3)	C31—P2—C51—C52	28.00 (17)
P1—C21—C22—C23	-177.12 (14)	N1—P2—C51—C56	-24.50 (18)
C21—C22—C23—C24	-1.0 (3)	C41—P2—C51—C56	95.21 (15)
C22—C23—C24—C25	1.2 (3)	C31—P2—C51—C56	-152.12 (14)
C23—C24—C25—C26	-0.1 (3)	C56—C51—C52—C53	0.8 (3)
C22—C21—C26—C25	1.2 (3)	P2—C51—C52—C53	-179.31 (16)
P1—C21—C26—C25	178.09 (14)	C51—C52—C53—C54	0.3 (3)
C24—C25—C26—C21	-1.1 (3)	C52—C53—C54—C55	-1.1 (3)
N1—P2—C31—C32	-33.42 (18)	C53—C54—C55—C56	0.9 (3)
C41—P2—C31—C32	-152.60 (15)	C54—C55—C56—C51	0.2 (3)
C51—P2—C31—C32	94.86 (16)	C52—C51—C56—C55	-1.0 (3)
N1—P2—C31—C36	148.99 (15)	P2—C51—C56—C55	179.07 (15)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C32—H32 $\cdots$ O1	0.95	2.34	3.257 (2)	163
C43—H43 $\cdots$ O1 <sup>i</sup>	0.95	2.34	3.257 (2)	162
C45—H45 $\cdots$ Cg1 <sup>ii</sup>	0.95	2.92	3.846 (2)	165
C55—H55 $\cdots$ Cg2 <sup>iii</sup>	0.95	2.73	3.644 (2)	163

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

Fig. 1

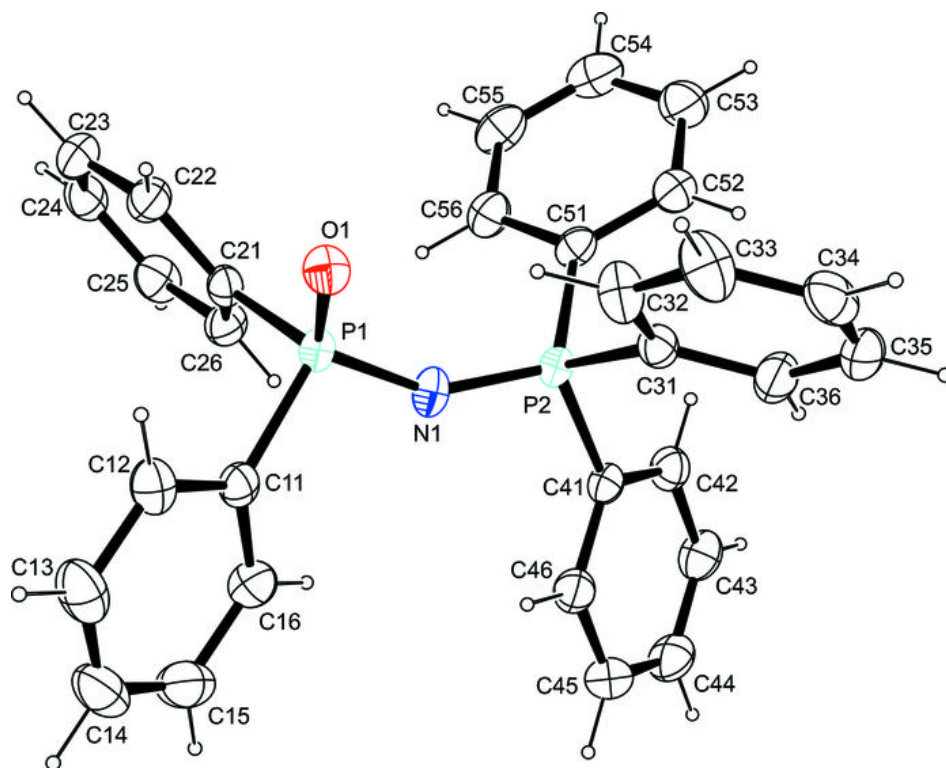


Fig. 2

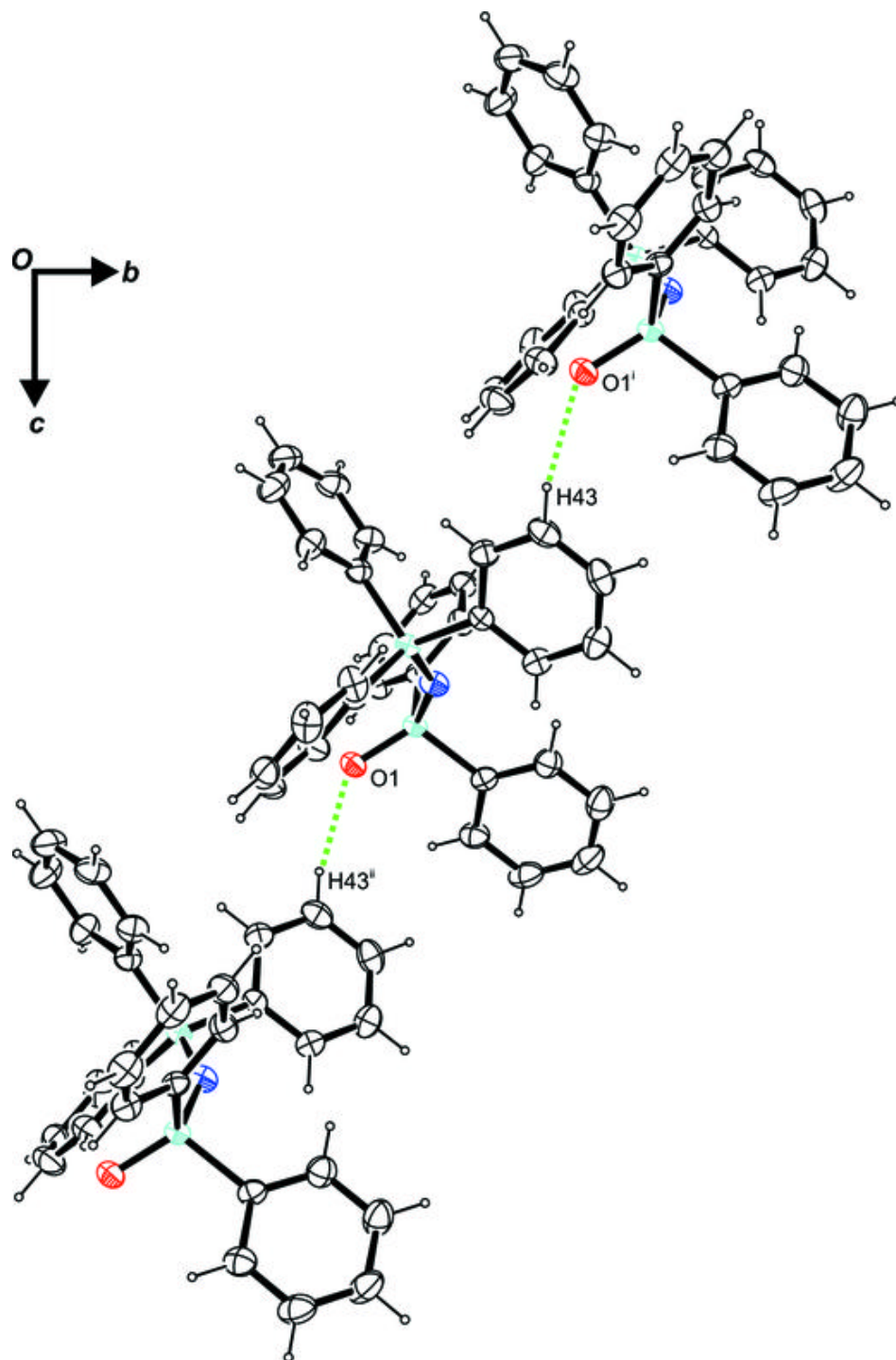


Fig. 3

