Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

gem-2,2-Diamino-4,4,6,6-tetraphenoxy-1,3,5-cyclotriaza-λ^5-phosphorine

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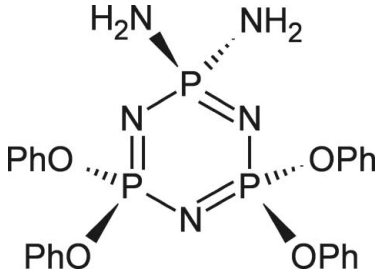
Received 25 April 2007; accepted 9 July 2007

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.058; data-to-parameter ratio = 14.6.

In the title compound, $C_{24}H_{24}N_5O_4P_3$, the molecules are linked by rather weak N—H···N hydrogen bonds into chains propagating along the c axis, their planar P_3N_3 rings being approximately coplanar within the chain.

Related literature

For general background, see: Otsuka Chemical Company (1985); Allcock & Taylor (2000); Kanebo (1991); Allcock (2003). For related structures, see: Fincham *et al.* (1985, 1988, 1986); Golinski & Jacobs (1994); Jacobs & Kirchgässner (1990); Marsh & Trotter (1971).



Experimental

Crystal data

$C_{24}H_{24}N_5O_4P_3$
M_r = 539.39
Tetragonal, $P4_1$
a = 12.9555 (18) Å
c = 15.029 (3) Å
V = 2522.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 120 (2) K
0.17 × 0.10 × 0.10 mm

Data collection

Kuma KM-4 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2005)
T_{min} = 0.924, T_{max} = 0.973
18062 measured reflections
4919 independent reflections
4714 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.058
S = 1.03
4919 reflections
337 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), with 2339 Friedel pairs
Flack parameter: −0.06 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H3···N1^{i}	0.83 (2)	2.31 (3)	3.072 (2)	153.2 (18)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors express their gratitude to the Ministry of Education for grant No. MSM0021622411.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN3046).

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  doi:10.1107/S1600536807033399

supplementary materials

Acta Cryst. (2007). E**63**, o3500 [**doi:10.1107/S1600536807033399**]

gem-2,2-Diamino-4,4,6,6-tetraphenoxy-1,3,5-cyclotriaza-λ^5-phosphorine

L. Dastychová, Z. Zák, M. Alberti and J. Taraba

Comment

Alkoxy- or aryloxy- substituted *cyclo*-triphosphazenes are generally suitable precursors for polymerization reactions in plasma. This present work is part of a study of plasma action on selected derivatives of *cyclo*-triphosphazenes. The plasma effect leads to the formation of polyorganophosphazenes, that are potentially attractive as fire retardants (Otsuka Chemical Company, 1985; Allcock & Taylor, 2000, Kanebo, 1991) and water repellents due to the presence of hydrophobic aryloxy-groups (Allcock, 2003).

Diamidotetrachloro-*cyclo*-triphosphazene, $P_3N_3(Cl)_4(NH_2)_2$ (**I**) with a geminal structure (Fincham *et al.,* 1986*)* is a common starting compound for syntheses of all known tetrasubstituted alkoxy-/aryloxy-2,2-diamino-1,3,5–2λ^5,4λ^5,6λ^5-*cyclo*-triazatriphosphorines. In a majority of cases these substitution rections lead to a migration of the $–NH_2$ group and a *non-geminaly* substituted $P_3N_3(OR)_4(NH_2)_2$: (*gem*) ≡ $P(NH_2)_2$ (*non-gem*) ≡ $P(NH_2)(OR)$ results. Thus, non-geminaly substituted *trans*-$P_3N_3(OR)_4(NH_2)_2$, (*R* = Me, Et, Pr^n, Bu^n) (Fincham *et al.*, 1985,1986) and *cis*-$P_3N_3(OR)_4(NH_2)_2$, (*R* = Me, Et, Pr^n, Bu^n) as a minor product (Fincham *et al.*, 1985,1988) were obtained. However, the only X-ray structure known is that of *trans*-$P_3N_3(OPr^n)_4(NH_2)_2$ (**II**) (Fincham *et al.*, 1985).

The only case when a substitution of Cl atoms by –OR group was not accompanied by a migration of a $–NH_2$ group is *gem*-$P_3N_3(OMe)_4(NH_2)_2$ (**III**)(Fincham *et al.*, 1988). However, this derivative was not obtained as a chemical individuum and only the structure of 1:1 mixed crystals of *cis*-$P_3N_3(OMe)_4(NH_2)_2$ (**IV**)and *gem*-$P_3N_3(OMe)_4(NH_2)_2$ (**III**) have been determined.

In the title compound (**V**), the PN ring is fairly planar with N3 being 0.25 Å out of the best plane defined by P1, N1, P2, N2 and P3 (Fig. 1) and exhibits the same lengthening of the P—N ring bonds adjacent to $P(NH_2)_2$ and a shortening of the P—N ring bonds adjacent to $P(OPh)_2$ as observed in other $P_3N_3X_4(NH_2)_2$ derivatives. In this case Δ(P—N) is 0.03 Å which is equal to the value 0.03 Å found in *gem*-$P_3N_3(OMe)_4(NH_2)_2$ (**III**)(Fincham *et al.*, 1988).

The molecules are linked by rather weak N—H···N hydrogen bonds (Table 1) into chains propagating along the axis c with approximately coplanar orientation of their PN rings. This simple system of H-bonds is in a contrast to a very complicated H-bonds system in the structure of a 1:1 mixed crystal of (**III**) and (**IV**) which in fact prevents any mutual comparison. (Fincham *et al.*, 1988).

There is a significant difference in the exocyclic P3—N4 1.646 (2) Å and P3—N5 1.628 (2) Å bond lengths, the shorter being that forming the hydrogen bond but the longer one 1.646 (2) Å equals to those found in $P_3N_3(NH_2)_6$ (av. 1.65 (2) Å) (Golinski & Jacobs,1994) and $P_3N_3(NH_2)_6$. $0.5NH_3$ (av.1.65 (1) Å) (Jacobs & Kirchgässner, 1990). On the other hand the P—O bond lengts (av. 1.594 (4) Å) are significantly longer than those found in $P_3N_3(OPh)_6$ (av. 1.582 (2) Å) (Marsh & Trotter, 1971).

Experimental

The reaction was carried out in anhydrous tetrahydrofuran (THF). 0.400 g (4.25 mmol) of PhOH was dissolved in 30 ml of THF. and 0.980 g (4.26 mmol) of Na was added to the solution, the reaction mixture was refluxed for 6 h and the PhONa was formed. 0.328 g (1.06 mmol) of (**I**) was added to the solution of PhONa. The reaction mixture was refluxed for 3.5 h and then was kept at ambient temperature for 5 days. After the reaction, the solvent was completely evaporated. 25 ml of Et_2O was added to the solid (mixture of (**V**) and NaCl), (**V**) was dissolved and insoluble NaCl was filtered off. The solvent from the solution of (**V**) was then partially evaporated under vacuum. The yield of colourless crystals of (**V**) was 0.50 g (87%). The reaction and all operations were performed in an atmosphere of dry nitrogen.

Refinement

Hydrogen atoms of phenyl rings were inserted in calculated position, those of $–NH_2$ group were found from a difference electron density map. Non-hydrogen atoms were refined anisotropically, hydrogen atoms of the phenyl rings by a ride-on approach, and $–NH_2$ group H atoms were refined isotropically with their isotropic temperature factors tied up with those of relevant nitrogen atoms.

Figures

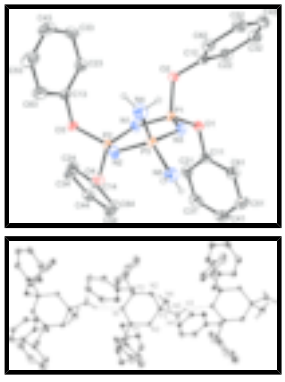

Fig. 1. Molecular structure of the title compound with the atom-labelling scheme. Ellipsoids are drawn at the 50% probability level. H atoms of the phenyl rings are omitted for clarity. Other H atoms are represented as small spheres of arbitrary radii.

gem-2,2-Diamino-4,4,6,6-tetraphenoxy-1,3,5–2λ^5-cyclotriazaphosphorine

Crystal data

$C_{24}H_{24}N_5O_4P_3$	$Z = 4$
$M_r = 539.39$	$F_{000} = 1120$
Tetragonal, $P4_1$	$D_x = 1.420$ Mg m^{-3}
$a = 12.9555$ (18) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 12.9555$ (18) Å	Cell parameters from 2718 reflections
$c = 15.029$ (3) Å	$\theta = 3.2–27.3°$
$\alpha = 90°$	$\mu = 0.28$ mm^{-1}
$\beta = 90°$	$T = 120$ (2) K

$\gamma = 90^\circ$	Prism, colourless
$V = 2522.5\ (7)\ Å^3$	0.17 × 0.10 × 0.10 mm

Data collection

Kuma KM-4 CCD area-detector diffractometer	4919 independent reflections
Radiation source: fine-focus sealed tube	4714 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.045$
Detector resolution: 8.4353 pixels mm^{-1}	$\theta_{max} = 26.0^\circ$
$T = 120(2)$ K	$\theta_{min} = 3.4^\circ$
ω scans	$h = -15 \rightarrow 12$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2005)	$k = -15 \rightarrow 15$
$T_{min} = 0.924$, $T_{max} = 0.973$	$l = -18 \rightarrow 17$
18062 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.058$	$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.2944P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
4919 reflections	$\Delta\rho_{max} = 0.20$ e $Å^{-3}$
337 parameters	$\Delta\rho_{min} = -0.29$ e $Å^{-3}$
1 restraint	Absolute structure: Flack (1983), with 2339 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.06 (4)

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}
P1	0.07004 (3)	0.41977 (3)	0.22059 (2)	0.01416 (9)
N1	−0.01277 (10)	0.49632 (9)	0.17903 (9)	0.0179 (3)
P2	−0.07227 (3)	0.57612 (3)	0.24143 (2)	0.01442 (9)
N2	−0.04300 (10)	0.58137 (9)	0.34272 (9)	0.0187 (3)
P3	0.04153 (3)	0.50759 (3)	0.38884 (3)	0.01613 (9)
N3	0.08060 (10)	0.41581 (10)	0.32482 (9)	0.0183 (3)
N4	0.13070 (12)	0.58672 (13)	0.42667 (10)	0.0266 (3)
H1	0.1451 (15)	0.6358 (17)	0.3909 (15)	0.032*
H2	0.1868 (16)	0.5602 (16)	0.4479 (15)	0.032*
N5	0.00452 (11)	0.45057 (12)	0.47995 (10)	0.0234 (3)
H3	0.0038 (14)	0.4852 (15)	0.5267 (17)	0.028*
H4	−0.0324 (15)	0.4041 (17)	0.4779 (14)	0.028*
O1	0.05456 (8)	0.30671 (8)	0.18133 (7)	0.0192 (2)
C11	−0.03274 (12)	0.24610 (12)	0.19850 (11)	0.0178 (3)
C21	−0.12032 (13)	0.25927 (12)	0.14673 (12)	0.0243 (3)
H21	−0.1236	0.3125	0.1034	0.029*
C31	−0.20321 (14)	0.19285 (14)	0.15963 (12)	0.0292 (4)
H31	−0.2641	0.2012	0.1252	0.035*
C41	−0.19795 (14)	0.11462 (13)	0.22217 (12)	0.0301 (4)
H41	−0.2545	0.0688	0.2298	0.036*
C51	−0.10969 (15)	0.10328 (14)	0.27371 (13)	0.0327 (4)
H51	−0.1063	0.0501	0.3172	0.039*
C61	−0.02613 (14)	0.16957 (13)	0.26189 (12)	0.0261 (4)
H61	0.0344	0.1621	0.2970	0.031*
O2	0.17700 (8)	0.44636 (8)	0.17290 (7)	0.0182 (2)
C12	0.26620 (12)	0.38844 (12)	0.19250 (10)	0.0171 (3)
C22	0.29037 (12)	0.30450 (12)	0.13938 (11)	0.0228 (3)
H22	0.2462	0.2844	0.0920	0.027*
C32	0.38075 (14)	0.25036 (14)	0.15707 (12)	0.0285 (4)
H32	0.3986	0.1928	0.1211	0.034*
C42	0.44482 (13)	0.27894 (14)	0.22592 (13)	0.0290 (4)
H42	0.5060	0.2408	0.2376	0.035*
C52	0.41943 (13)	0.36446 (14)	0.27865 (12)	0.0274 (4)
H52	0.4635	0.3846	0.3261	0.033*
C62	0.32973 (13)	0.41974 (13)	0.26141 (11)	0.0224 (3)
H62	0.3123	0.4782	0.2965	0.027*
O3	−0.06784 (8)	0.68823 (8)	0.19750 (8)	0.0194 (2)
C13	0.02350 (12)	0.74692 (12)	0.20296 (10)	0.0179 (3)
C23	0.10722 (13)	0.72322 (13)	0.14915 (12)	0.0243 (4)
H23	0.1055	0.6648	0.1110	0.029*
C33	0.19330 (13)	0.78640 (14)	0.15210 (13)	0.0305 (4)
H33	0.2507	0.7721	0.1147	0.037*
C43	0.19631 (14)	0.87029 (15)	0.20919 (14)	0.0342 (4)
H43	0.2559	0.9130	0.2113	0.041*
C53	0.11251 (15)	0.89197 (15)	0.26316 (13)	0.0365 (5)

H53	0.1151	0.9489	0.3029	0.044*
C63	0.02444 (14)	0.83060 (13)	0.25939 (12)	0.0286 (4)
H63	−0.0340	0.8462	0.2951	0.034*
O4	−0.19345 (8)	0.55725 (8)	0.23680 (7)	0.0185 (2)
C14	−0.25038 (11)	0.53720 (12)	0.15887 (10)	0.0175 (3)
C24	−0.25405 (13)	0.60670 (13)	0.08943 (11)	0.0236 (4)
H24	−0.2147	0.6686	0.0914	0.028*
C34	−0.31641 (13)	0.58452 (14)	0.01644 (12)	0.0270 (4)
H34	−0.3197	0.6317	−0.0319	0.032*
C44	−0.37402 (13)	0.49399 (13)	0.01364 (11)	0.0249 (4)
H44	−0.4162	0.4792	−0.0365	0.030*
C54	−0.36950 (13)	0.42588 (13)	0.08406 (11)	0.0234 (4)
H54	−0.4088	0.3640	0.0823	0.028*
C64	−0.30775 (12)	0.44703 (12)	0.15784 (11)	0.0218 (3)
H64	−0.3051	0.4004	0.2065	0.026*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01532 (18)	0.01371 (18)	0.0135 (2)	0.00091 (13)	0.00172 (15)	−0.00008 (14)
N1	0.0201 (7)	0.0186 (7)	0.0148 (6)	0.0016 (5)	−0.0004 (5)	0.0001 (5)
P2	0.01389 (18)	0.01381 (18)	0.0156 (2)	0.00082 (14)	0.00005 (14)	0.00037 (15)
N2	0.0212 (7)	0.0162 (7)	0.0188 (7)	0.0039 (5)	−0.0013 (5)	−0.0037 (5)
P3	0.01789 (19)	0.01769 (19)	0.01279 (18)	0.00177 (15)	−0.00055 (15)	−0.00132 (15)
N3	0.0234 (7)	0.0164 (6)	0.0151 (7)	0.0042 (5)	0.0007 (5)	0.0004 (5)
N4	0.0255 (8)	0.0260 (8)	0.0284 (8)	0.0002 (6)	−0.0069 (6)	−0.0031 (6)
N5	0.0293 (8)	0.0248 (7)	0.0160 (7)	0.0012 (6)	0.0024 (6)	−0.0001 (6)
O1	0.0183 (6)	0.0169 (5)	0.0224 (6)	−0.0011 (4)	0.0046 (4)	−0.0048 (4)
C11	0.0179 (8)	0.0161 (8)	0.0195 (8)	−0.0015 (6)	0.0059 (6)	−0.0052 (6)
C21	0.0274 (9)	0.0189 (8)	0.0265 (9)	0.0013 (6)	−0.0010 (7)	−0.0042 (7)
C31	0.0250 (9)	0.0293 (9)	0.0332 (10)	−0.0014 (7)	−0.0028 (7)	−0.0144 (8)
C41	0.0277 (9)	0.0278 (9)	0.0349 (10)	−0.0083 (7)	0.0098 (8)	−0.0088 (8)
C51	0.0387 (11)	0.0297 (9)	0.0295 (10)	−0.0077 (8)	0.0092 (8)	0.0050 (8)
C61	0.0254 (9)	0.0291 (9)	0.0239 (9)	−0.0020 (7)	−0.0005 (7)	0.0009 (7)
O2	0.0173 (5)	0.0186 (5)	0.0187 (6)	0.0005 (4)	0.0036 (4)	0.0033 (4)
C12	0.0150 (7)	0.0191 (8)	0.0171 (8)	−0.0002 (6)	0.0036 (6)	0.0057 (6)
C22	0.0231 (8)	0.0268 (8)	0.0184 (8)	0.0013 (7)	0.0025 (7)	−0.0014 (7)
C32	0.0307 (9)	0.0274 (9)	0.0273 (9)	0.0061 (7)	0.0087 (7)	−0.0001 (7)
C42	0.0203 (8)	0.0338 (9)	0.0331 (9)	0.0060 (7)	0.0045 (7)	0.0124 (8)
C52	0.0203 (8)	0.0348 (10)	0.0271 (9)	−0.0021 (7)	−0.0042 (7)	0.0054 (7)
C62	0.0233 (8)	0.0224 (8)	0.0215 (8)	−0.0023 (7)	0.0015 (6)	0.0002 (6)
O3	0.0152 (5)	0.0166 (5)	0.0264 (6)	−0.0018 (4)	−0.0029 (4)	0.0037 (4)
C13	0.0145 (7)	0.0194 (8)	0.0196 (8)	−0.0030 (6)	−0.0039 (6)	0.0062 (6)
C23	0.0250 (8)	0.0244 (8)	0.0234 (9)	0.0016 (7)	0.0010 (7)	0.0030 (7)
C33	0.0197 (8)	0.0367 (10)	0.0350 (10)	0.0010 (7)	0.0055 (7)	0.0122 (8)
C43	0.0249 (9)	0.0359 (10)	0.0419 (11)	−0.0135 (8)	−0.0077 (8)	0.0099 (8)
C53	0.0407 (11)	0.0319 (10)	0.0368 (11)	−0.0138 (8)	−0.0033 (9)	−0.0067 (8)
C63	0.0279 (9)	0.0271 (9)	0.0308 (10)	−0.0059 (7)	0.0053 (7)	−0.0026 (7)

O4	0.0162 (5)	0.0214 (5)	0.0180 (5)	−0.0004 (4)	0.0015 (4)	−0.0003 (5)
C14	0.0132 (7)	0.0212 (8)	0.0181 (8)	0.0026 (6)	0.0013 (6)	−0.0035 (6)
C24	0.0211 (8)	0.0230 (8)	0.0268 (9)	−0.0045 (7)	−0.0021 (7)	0.0031 (7)
C34	0.0262 (9)	0.0286 (9)	0.0262 (9)	−0.0031 (7)	−0.0055 (7)	0.0077 (7)
C44	0.0189 (8)	0.0317 (9)	0.0240 (8)	−0.0002 (7)	−0.0045 (6)	−0.0045 (7)
C54	0.0221 (8)	0.0215 (8)	0.0267 (9)	−0.0045 (7)	0.0004 (7)	−0.0042 (7)
C64	0.0228 (8)	0.0208 (8)	0.0219 (8)	−0.0004 (6)	0.0013 (7)	0.0023 (6)

Geometric parameters (Å, °)

P1—N3	1.5732 (14)	C22—H22	0.9500
P1—N1	1.5889 (13)	C32—C42	1.377 (3)
P1—O1	1.5918 (11)	C32—H32	0.9500
P1—O2	1.5977 (11)	C42—C52	1.401 (3)
N1—P2	1.5945 (13)	C42—H42	0.9500
P2—N2	1.5704 (14)	C52—C62	1.389 (2)
P2—O4	1.5904 (11)	C52—H52	0.9500
P2—O3	1.5965 (11)	C62—H62	0.9500
N2—P3	1.6104 (14)	O3—C13	1.4089 (18)
P3—N3	1.6111 (13)	C13—C63	1.376 (2)
P3—N5	1.6281 (15)	C13—C23	1.387 (2)
P3—N4	1.6459 (16)	C23—C33	1.384 (2)
N4—H1	0.85 (2)	C23—H23	0.9500
N4—H2	0.86 (2)	C33—C43	1.385 (3)
N5—H3	0.83 (2)	C33—H33	0.9500
N5—H4	0.77 (2)	C43—C53	1.384 (3)
O1—C11	1.4008 (18)	C43—H43	0.9500
C11—C61	1.378 (2)	C53—C63	1.392 (3)
C11—C21	1.386 (2)	C53—H53	0.9500
C21—C31	1.390 (2)	C63—H63	0.9500
C21—H21	0.9500	O4—C14	1.4082 (18)
C31—C41	1.384 (3)	C14—C24	1.379 (2)
C31—H31	0.9500	C14—C64	1.385 (2)
C41—C51	1.389 (3)	C24—C34	1.392 (2)
C41—H41	0.9500	C24—H24	0.9500
C51—C61	1.393 (2)	C34—C44	1.391 (2)
C51—H51	0.9500	C34—H34	0.9500
C61—H61	0.9500	C44—C54	1.379 (2)
O2—C12	1.4091 (19)	C44—H44	0.9500
C12—C62	1.384 (2)	C54—C64	1.394 (2)
C12—C22	1.385 (2)	C54—H54	0.9500
C22—C32	1.391 (2)	C64—H64	0.9500
N3—P1—N1	118.06 (7)	C32—C22—H22	120.8
N3—P1—O1	110.49 (6)	C42—C32—C22	121.03 (16)
N1—P1—O1	110.09 (7)	C42—C32—H32	119.5
N3—P1—O2	112.23 (7)	C22—C32—H32	119.5
N1—P1—O2	105.94 (6)	C32—C42—C52	119.73 (16)
O1—P1—O2	98.13 (6)	C32—C42—H42	120.1
P1—N1—P2	120.00 (9)	C52—C42—H42	120.1

N2—P2—O4	106.70 (7)	C62—C52—C42	119.89 (16)
N2—P2—N1	118.78 (7)	C62—C52—H52	120.1
O4—P2—N1	110.60 (6)	C42—C52—H52	120.1
N2—P2—O3	110.65 (7)	C12—C62—C52	119.08 (16)
O4—P2—O3	99.05 (6)	C12—C62—H62	120.5
N1—P2—O3	109.23 (7)	C52—C62—H62	120.5
P2—N2—P3	123.76 (8)	C13—O3—P2	119.81 (9)
N2—P3—N3	113.25 (7)	C63—C13—C23	121.77 (15)
N2—P3—N5	115.53 (7)	C63—C13—O3	117.93 (14)
N3—P3—N5	105.07 (7)	C23—C13—O3	120.21 (14)
N2—P3—N4	104.85 (8)	C33—C23—C13	118.71 (16)
N3—P3—N4	116.45 (8)	C33—C23—H23	120.6
N5—P3—N4	101.46 (9)	C13—C23—H23	120.6
P1—N3—P3	122.90 (8)	C23—C33—C43	120.44 (17)
P3—N4—H1	113.5 (14)	C23—C33—H33	119.8
P3—N4—H2	118.0 (14)	C43—C33—H33	119.8
H1—N4—H2	110 (2)	C53—C43—C33	120.01 (16)
P3—N5—H3	117.9 (14)	C53—C43—H43	120.0
P3—N5—H4	120.3 (16)	C33—C43—H43	120.0
H3—N5—H4	117 (2)	C43—C53—C63	120.22 (17)
C11—O1—P1	123.31 (9)	C43—C53—H53	119.9
C61—C11—C21	121.84 (15)	C63—C53—H53	119.9
C61—C11—O1	118.73 (14)	C13—C63—C53	118.83 (17)
C21—C11—O1	119.23 (14)	C13—C63—H63	120.6
C11—C21—C31	118.55 (16)	C53—C63—H63	120.6
C11—C21—H21	120.7	C14—O4—P2	125.58 (10)
C31—C21—H21	120.7	C24—C14—C64	121.58 (15)
C41—C31—C21	120.68 (17)	C24—C14—O4	121.80 (14)
C41—C31—H31	119.7	C64—C14—O4	116.50 (14)
C21—C31—H31	119.7	C14—C24—C34	118.78 (15)
C31—C41—C51	119.78 (16)	C14—C24—H24	120.6
C31—C41—H41	120.1	C34—C24—H24	120.6
C51—C41—H41	120.1	C44—C34—C24	120.61 (16)
C41—C51—C61	120.23 (17)	C44—C34—H34	119.7
C41—C51—H51	119.9	C24—C34—H34	119.7
C61—C51—H51	119.9	C54—C44—C34	119.58 (15)
C11—C61—C51	118.92 (16)	C54—C44—H44	120.2
C11—C61—H61	120.5	C34—C44—H44	120.2
C51—C61—H61	120.5	C44—C54—C64	120.58 (15)
C12—O2—P1	120.17 (9)	C44—C54—H54	119.7
C62—C12—C22	121.81 (15)	C64—C54—H54	119.7
C62—C12—O2	119.23 (14)	C14—C64—C54	118.86 (15)
C22—C12—O2	118.89 (14)	C14—C64—H64	120.6
C12—C22—C32	118.44 (16)	C54—C64—H64	120.6
C12—C22—H22	120.8		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N5—H3···N1^{i}	0.83 (2)	2.31 (3)	3.072 (2)	153.2 (18)

Symmetry codes: (i) −*x*, −*y*+1, *z*+1/2.

Fig. 1

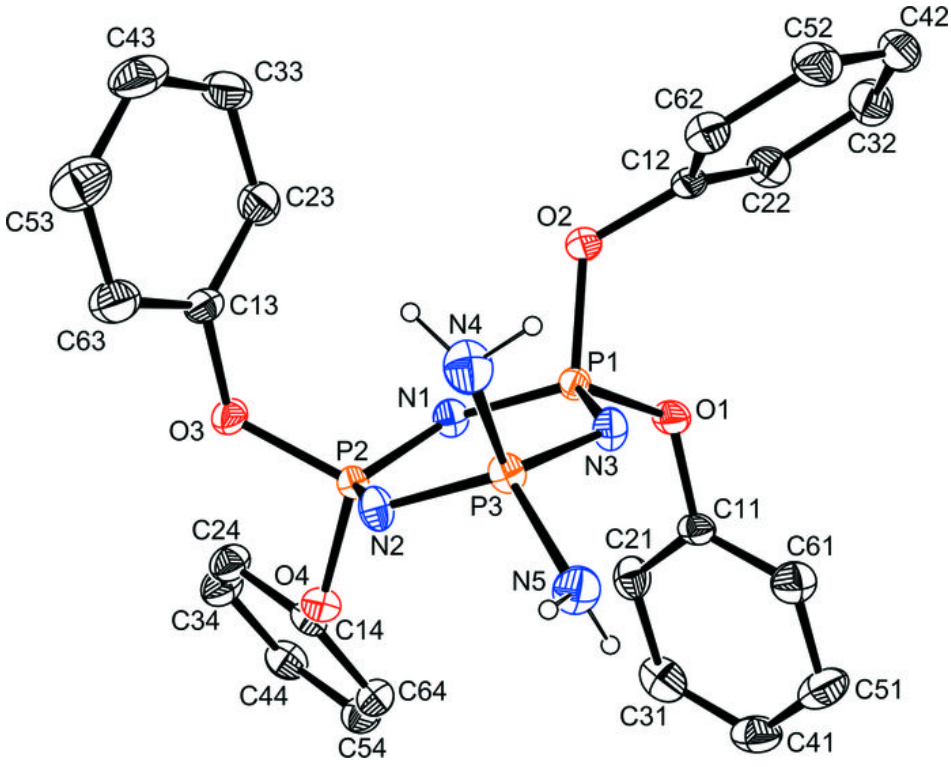

Fig. 2