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Crystal structure of 2-azido-1,3-bis(2,6-diisopropylphenyl)-1,3,2-diazaphospholidine

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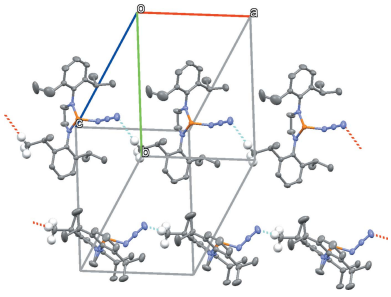
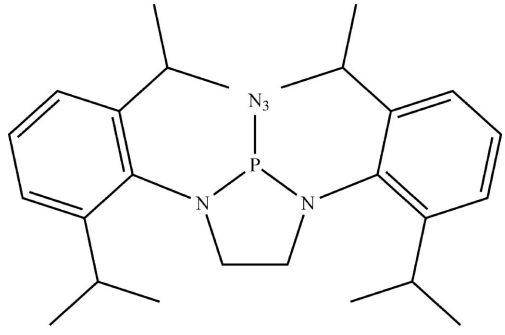
The title compound, $C_{26}H_{38}N_5P$, was synthesized by reacting 2-chloro-1,3-bis(2,6-diisopropylphenyl)-1,3,2-diazaphospholidine with sodium azide and a catalytic amount of lithium chloride in tetrahydrofuran. The title compound is the first structurally characterized 2-azido-1,3,2-diazaphospholidine and exhibits a P atom in a trigonal pyramidal geometry. The azide P—N bond length of 1.8547 (16) Å is significantly longer than the P—N separations for the chelating diamine [P—N = 1.6680 (15) and 1.6684 (14) Å]. The sterically hindered 2,6-diisopropylphenyl groups twist away from the central heterocycle, with dihedral angles between the central heteocyclic ring and benzene rings of 76.17 (10) and 79.74 (9)°. In the crystal, a weak C—H···N link to the terminal N atom of the azide group leads to [100] chains.

1. Chemical context

Phosphine azides possess at least one azide group attached to phosphorus and display a broad range of reactivity that is directly dependent on the other substituents attached to the P atom. One of the most interesting properties of these molecules is that both free and coordinated alkyl and aryl derivatives are much more reactive than their corresponding amino derivatives, as demonstrated by their lower thermal and photochemical stability. For example, the phosphinoazide complex $Ph_2P(N_3)$–$Cr(CO)_5$ readily undergoes photolysis under UV light to produce the phosphino–isocyanate complex $Ph_2P(NCO)$–$Cr(CO)_5$ (Ocando *et al.*, 1985), while the related bis(diisopropylamino) complex $(iPr_2N)_2P(N_3)$–$Cr(CO)_5$ does not (Cowley *et al.*, 1995). The crystal structure of the title compound is the first reported example of a structurally characterized 2-azido-1,3,2-diazaphospholidine; however, a few closely related compounds are known, such as those derived from 1,3,2-diazaphospholenes.





2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. It crystallizes in the monoclinic space group $P2_1/n$ with

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···N4	1.00	2.43	2.926 (2)	110
C12—H12···N4	1.00	2.44	2.913 (2)	109
C21—H21···N5	1.00	2.49	2.932 (2)	106
C24—H24···N1	1.00	2.66	3.443 (3)	136
C24—H24···N5	1.00	2.46	2.955 (2)	110
C22—H22*C*···N3^{i}	0.98	2.69	3.669 (3)	174

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

one molecule in the asymmetric unit. The bond lengths between the P atom and its flanking N atoms are similar [P1—N4 = 1.6680 (15) Å, P1—N5 = 1.6684 (14) Å and N4—P1—N5 = 91.14 (7)°], while the phosphorus centre adopts a trigonal pyramidal geometry, with the sum of the angles at phosphorus equal to 294.14 (7)°. The azide group is quasi-linear [N3—N2—N1 = 176.6 (2)°], with similar N—N bond lengths [N1—N2 = 1.168 (2) Å and N2—N3 = 1.155 (2) Å]. The phosphorus–azide bond length (P1—N1) is significantly longer [1.8547 (16) Å] than found for atoms N4 and N5. The average sum of the bond angles at the N4 and N5 positions is 359.87 (12)°, very close to an ideal trigonal planar geometry. This is a strong indication that the nominal lone pairs of atoms N4 and N5 participate in N—P···π interactions and, when coupled with the significantly longer P1—N1 bond length, suggests a partial ionic character similar to earlier reports in acyclic structures (Cowley *et al.*, 1995). The overall conformation of the C1/C2/N4/N5/P1 ring is well described as an envelope, with atom N5 deviating from the other atoms (r.m.s. deviation = 0.030 Å) by −0.274 (2) Å. The steric demands of the bulky 2,6-diisopropylphenyl groups cause the aromatic rings to twist away from the central five-membered ring, with torsion angles of 103.69 (18) and 101.83 (17)° for P1—N1—C3—C4 and P1—N2—C15—C20, respectively. The isopropyl groups are oriented away from the central five-membered ring, but the 'congested' nature of the molecule results in intramolecular short contacts between all four of the methine H atoms (H9, H12, H21 and H24) and atoms N4 and N5 (Table 1).

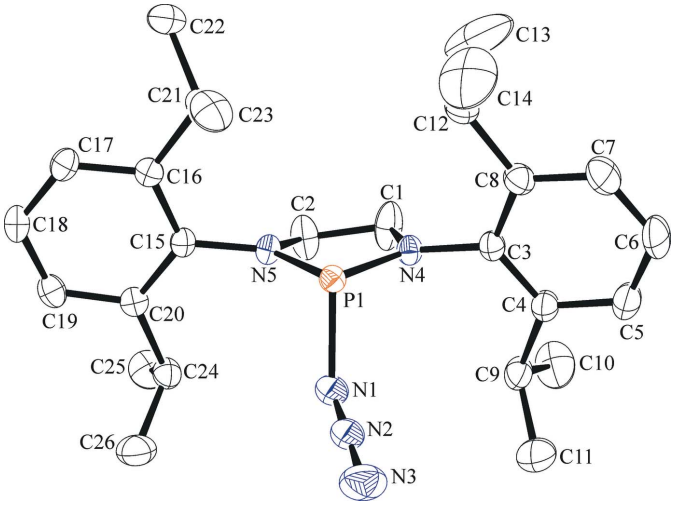


Figure 1
The molecular structure of the title compound, showing 50% displacement ellipsoids. H atoms have been omitted for clarity.

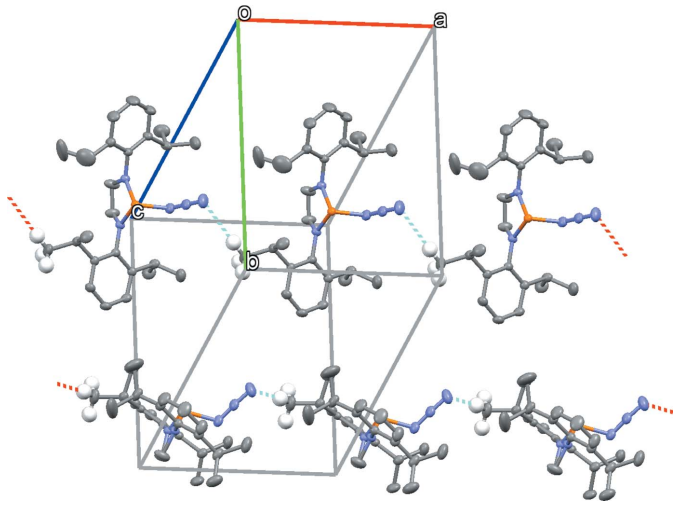


Figure 2
The packing of the title compound, showing intermolecular C—H···N interactions as dashed lines, which result in [100] chains.

3. Supramolecular features

The only significant directional interaction in the crystal of the title compound is a long [2.69 (3) Å] C—H···N hydrogen bond to the terminal N atom of the azide group, which results in [100] chains in the crystal (Fig. 2).

4. Database survey

A search of the Cambridge Structural Database (Groom *et al.*, 2016) indicated that no other 2-azido-1,3,2-(diarylamino)-phospholidine derivatives have been structurally characterized. Some structurally similar compounds were identified, however, namely 2-azido-1,3-bis(2,6-diisopropyllphenyl)-1,3,2-diazaphospholene (CSD refcode CILBAC; Gediga *et al.*, 2014) and its corresponding 2,6-dimethylphenyl derivative (GOFHAL; Burck *et al.*, 2008). Acyclic derivatives featuring bis(diisopropylamino) (PIJZAJ; Englert *et al.*, 1993) and bis(dicylohexylamino) (ZABCEK; Cowley *et al.*, 1995) ligands are known, and also 1-azido-*N*,*N*′-bis(2,4,6-tri-*tert*-butylphenyl)phosphinediamine (YABVUV; Nieger *et al.*, 2016).

5. Synthesis and crystallization

The synthesis of the title compound was achieved using a similar method as reported in the literature for 2-azido-1,3-bis(2,6-diisopropyllphenyl)-1,3,2-diazaphospholene (Gediga *et al.*, 2014). In a 20 ml scintillation vial, 0.102 g (0.229 mmol, 1 eq.) of colourless 2-chloro-1,3-bis(2,6-diisopropylphenyl)-1,3,2-diazaphospholidine were dissolved in 1 ml of THF producing a colourless solution. To this solution, 0.016 g

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{26}H_{38}N_5P$
M_r	451.58
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	10.0148 (12), 17.343 (2), 15.6270 (19)
β (°)	105.948 (2)
V (Å^3)	2609.7 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.13
Crystal size (mm)	0.39 × 0.35 × 0.27
Data collection	
Diffractometer	Siemens/Bruker APEXII
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.718, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29851, 5708, 4350
R_{int}	0.047
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.123, 1.02
No. of reflections	5708
No. of parameters	291
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.39

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

(0.246 mmol, 1.1 eq.) of colourless sodium azide and a spatula tip (<1 mg) of lithium chloride were added to solution immediately producing a colourless mixture. The reaction mixture was left to stir for 1 d and monitored using $^{31}P\{^1H\}$ NMR spectroscopy, and once the starting material was completely consumed the reaction mixture was dried *in vacuo*. Extraction of the colourless residue with cold pentane, followed by filtration through Celite produced a colourless solution, which afforded 0.060 g (60%) of the title compound as a colourless powder after removal of the solvent. Crystals of the product were obtained by concentrating the filtrate and storing in a 238 K freezer overnight. ^1H NMR ($CDCl_3$): δ 7.31 (*t*, $^3J_{HH}$ = 7.6 Hz, 2H, *p*-Dipp), 7.24–7.17 (*m*, 4H, *m*-Dipp), 3.88–3.82 (pseudo-*q*, 2H, NHC-CH_2), 3.74 (*sept*, $^3J_{HH}$ = 6.8 Hz, 2H, iPr-CH), 3.48–3.39 (*m*, 4H, iPr-CH, NHC-CH_2), 1.33–1.25 (*m*, $^3J_{HH}$ = 6.8 Hz, 24H, iPr-CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 150.3, 148.4, 136.2, 128.1, 124.7, 124.2, 54.4, 29.0, 25.3, 24.9, 24.5. $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ 129.8. IR (KBr pellet): ν 3062 (*w*), 2963 (*s*), 2926 (*m*), 2867 (*m*), 2500 (*w*), 2125 (*m*), 2085 (s, N═N═N), 1678 (*w*), 1584 (*w*), 1462 (*s*), 1445 (*s*), 1383 (*m*), 1363 (*m*), 1324 (*m*), 1323 (*m*), 1257 (*s*), 1211 (*w*), 1185 (*w*), 1106 (*m*), 1075 (*s*), 1056 (*w*), 1043 (*w*), 980 (*w*), 946 (*w*), 935 (*w*), 852 (*w*), 806 (*s*), 761 (*s*), 730 (*w*), 688 (*w*), 651 (*w*), 602 (*w*), 583 (*w*), 550 (*w*), 542 (*w*), 470 (*s*), 437 cm^{-1} (*w*). M.p. (K): 415.4–417.6 (decomposes, gas was released).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were included in geometrically idealized positions and refined using a riding model. Dihedral angles for the methyl H atoms were allowed to refine freely. The atomic displacement parameters of atoms N1 and N2 were constrained to be approximately equal using an EADP command.

Acknowledgements

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supporting information

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Crystal structure of 2-azido-1,3-bis(2,6-diisopropylphenyl)-1,3,2-diazaphospholidine

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Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

2-Azido-1,3-bis(2,6-diisopropylphenyl)-1,3,2-diazaphospholidine

Crystal data

$C_{26}H_{38}N_5P$
$M_r = 451.58$
Monoclinic, $P2_1/n$
$a = 10.0148$ (12) Å
$b = 17.343$ (2) Å
$c = 15.6270$ (19) Å
$\beta = 105.948$ (2)°
$V = 2609.7$ (5) Å^3
$Z = 4$
$F(000) = 976$
$D_x = 1.149$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5265 reflections
$\theta = 2.2$–25.2°
$\mu = 0.13$ mm^{-1}
$T = 150$ K
Block, colourless
0.39 × 0.35 × 0.27 mm

Data collection

Siemens/Bruker APEXII diffractometer
Detector resolution: 66 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
$T_{min} = 0.718$, $T_{max} = 0.746$
29851 measured reflections
5708 independent reflections
4350 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.8°$
$h = -12 \rightarrow 12$
$k = -22 \rightarrow 22$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.123$
$S = 1.02$
5708 reflections
291 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 1.4293P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.59945 (4)	0.54203 (3)	0.26470 (3)	0.02022 (12)
N1	0.79052 (16)	0.53002 (10)	0.29154 (11)	0.0320 (3)
N2	0.84392 (16)	0.55461 (10)	0.23965 (11)	0.0320 (3)
N3	0.9030 (2)	0.57761 (13)	0.19098 (13)	0.0507 (5)
N4	0.54371 (15)	0.45109 (8)	0.24970 (9)	0.0219 (3)
N5	0.57675 (15)	0.54345 (8)	0.36653 (9)	0.0213 (3)
C1	0.5191 (3)	0.41282 (12)	0.32687 (13)	0.0425 (6)
H1A	0.574471	0.364849	0.340333	0.051*
H1B	0.419615	0.399379	0.315259	0.051*
C2	0.5609 (2)	0.46709 (11)	0.40234 (13)	0.0364 (5)
H2A	0.489221	0.468627	0.435128	0.044*
H2B	0.649678	0.450354	0.443932	0.044*
C3	0.51337 (18)	0.41222 (10)	0.16538 (11)	0.0231 (4)
C4	0.60771 (19)	0.35813 (11)	0.14988 (12)	0.0274 (4)
C5	0.5759 (2)	0.32088 (13)	0.06777 (14)	0.0413 (5)
H5	0.638572	0.283914	0.055981	0.050*
C6	0.4543 (2)	0.33718 (15)	0.00350 (14)	0.0492 (6)
H6	0.434281	0.311734	−0.052431	0.059*
C7	0.3621 (2)	0.38992 (13)	0.01970 (13)	0.0420 (5)
H7	0.278444	0.400226	−0.025191	0.050*
C8	0.38869 (19)	0.42858 (11)	0.10071 (12)	0.0286 (4)
C9	0.7420 (2)	0.33751 (12)	0.21946 (13)	0.0336 (5)
H9	0.750243	0.371828	0.272092	0.040*
C10	0.7379 (3)	0.25436 (14)	0.25043 (17)	0.0561 (7)
H10A	0.731285	0.219361	0.200199	0.084*
H10B	0.656980	0.247168	0.273363	0.084*
H10C	0.822820	0.243131	0.297620	0.084*
C11	0.8689 (2)	0.35145 (16)	0.18521 (17)	0.0519 (6)
H11A	0.865868	0.316548	0.135398	0.078*
H11B	0.953597	0.341772	0.233196	0.078*
H11C	0.868644	0.404983	0.165078	0.078*
C12	0.2812 (2)	0.48372 (12)	0.11812 (14)	0.0367 (5)
H12	0.326243	0.513951	0.172950	0.044*
C13	0.1612 (3)	0.43896 (19)	0.1354 (3)	0.1000 (14)
H13A	0.093897	0.475020	0.148196	0.150*
H13B	0.196137	0.404586	0.186406	0.150*
H13C	0.116090	0.408329	0.082660	0.150*
C14	0.2272 (4)	0.53980 (17)	0.0432 (2)	0.0820 (10)
H14A	0.177132	0.511669	−0.010362	0.123*

H14B	0.305181	0.567885	0.031406	0.123*
H14C	0.164104	0.576421	0.059862	0.123*
C15	0.58170 (17)	0.61147 (10)	0.42027 (11)	0.0207 (3)
C16	0.46359 (18)	0.65928 (10)	0.40491 (11)	0.0219 (4)
C17	0.46834 (19)	0.72263 (10)	0.46045 (12)	0.0274 (4)
H17	0.389882	0.755641	0.450633	0.033*
C18	0.5844 (2)	0.73839 (11)	0.52929 (12)	0.0305 (4)
H18	0.585062	0.781626	0.566744	0.037*
C19	0.6994 (2)	0.69150 (10)	0.54386 (12)	0.0281 (4)
H19	0.779039	0.703016	0.591430	0.034*
C20	0.70149 (18)	0.62737 (10)	0.49017 (11)	0.0231 (4)
C21	0.33327 (18)	0.64485 (11)	0.32984 (12)	0.0277 (4)
H21	0.331695	0.589036	0.313540	0.033*
C22	0.2000 (2)	0.66235 (13)	0.35634 (17)	0.0453 (6)
H22A	0.191522	0.718159	0.363168	0.068*
H22B	0.203732	0.636691	0.412819	0.068*
H22C	0.119651	0.643396	0.309992	0.068*
C23	0.3355 (2)	0.69207 (13)	0.24749 (14)	0.0412 (5)
H23A	0.254521	0.678656	0.198252	0.062*
H23B	0.420602	0.680639	0.230557	0.062*
H23C	0.332647	0.747141	0.260929	0.062*
C24	0.83013 (19)	0.57663 (11)	0.51126 (12)	0.0285 (4)
H24	0.815047	0.535143	0.465149	0.034*
C25	0.8521 (2)	0.53809 (13)	0.60211 (14)	0.0442 (5)
H25A	0.769090	0.508486	0.602823	0.066*
H25B	0.869085	0.577684	0.648599	0.066*
H25C	0.932307	0.503398	0.613113	0.066*
C26	0.9599 (2)	0.62141 (13)	0.50874 (16)	0.0432 (5)
H26A	0.946650	0.644086	0.449550	0.065*
H26B	1.039822	0.586419	0.521593	0.065*
H26C	0.976794	0.662518	0.553468	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0210 (2)	0.0213 (2)	0.0179 (2)	−0.00023 (17)	0.00470 (16)	0.00068 (17)
N1	0.0236 (6)	0.0394 (7)	0.0296 (6)	0.0011 (5)	0.0017 (4)	−0.0038 (5)
N2	0.0236 (6)	0.0394 (7)	0.0296 (6)	0.0011 (5)	0.0017 (4)	−0.0038 (5)
N3	0.0390 (11)	0.0744 (15)	0.0439 (11)	−0.0083 (10)	0.0201 (9)	−0.0003 (10)
N4	0.0268 (8)	0.0214 (7)	0.0180 (7)	−0.0014 (6)	0.0070 (6)	−0.0002 (6)
N5	0.0271 (8)	0.0186 (7)	0.0191 (7)	0.0007 (6)	0.0078 (6)	0.0005 (6)
C1	0.0801 (17)	0.0260 (10)	0.0300 (11)	−0.0088 (10)	0.0293 (11)	−0.0024 (8)
C2	0.0631 (14)	0.0236 (10)	0.0229 (9)	−0.0099 (9)	0.0125 (9)	0.0016 (8)
C3	0.0262 (9)	0.0233 (9)	0.0200 (8)	−0.0019 (7)	0.0067 (7)	−0.0023 (7)
C4	0.0286 (10)	0.0290 (10)	0.0239 (9)	0.0018 (8)	0.0060 (7)	−0.0042 (7)
C5	0.0395 (12)	0.0490 (13)	0.0339 (11)	0.0079 (10)	0.0077 (9)	−0.0162 (10)
C6	0.0501 (14)	0.0641 (16)	0.0275 (11)	0.0046 (12)	0.0009 (10)	−0.0212 (11)
C7	0.0367 (12)	0.0535 (14)	0.0277 (10)	0.0043 (10)	−0.0050 (9)	−0.0066 (9)

C8	0.0265 (9)	0.0280 (10)	0.0285 (10)	0.0009 (7)	0.0029 (8)	−0.0011 (8)
C9	0.0325 (11)	0.0347 (11)	0.0301 (10)	0.0094 (8)	0.0027 (8)	−0.0077 (8)
C10	0.0661 (17)	0.0387 (13)	0.0538 (15)	0.0155 (12)	−0.0002 (13)	0.0041 (11)
C11	0.0322 (12)	0.0669 (17)	0.0542 (15)	0.0099 (11)	0.0081 (10)	−0.0126 (12)
C12	0.0262 (10)	0.0320 (11)	0.0448 (12)	0.0055 (8)	−0.0022 (9)	−0.0070 (9)
C13	0.073 (2)	0.065 (2)	0.192 (4)	0.0142 (17)	0.086 (3)	0.009 (2)
C14	0.098 (3)	0.0568 (19)	0.082 (2)	0.0371 (17)	0.0091 (19)	0.0182 (16)
C15	0.0260 (9)	0.0196 (8)	0.0177 (8)	−0.0012 (7)	0.0080 (7)	0.0004 (6)
C16	0.0245 (9)	0.0198 (8)	0.0223 (9)	−0.0013 (7)	0.0079 (7)	0.0033 (7)
C17	0.0320 (10)	0.0206 (9)	0.0304 (10)	0.0035 (7)	0.0100 (8)	0.0000 (7)
C18	0.0400 (11)	0.0231 (9)	0.0285 (10)	−0.0017 (8)	0.0093 (8)	−0.0069 (8)
C19	0.0321 (10)	0.0280 (10)	0.0219 (9)	−0.0041 (8)	0.0035 (7)	−0.0014 (7)
C20	0.0261 (9)	0.0222 (9)	0.0212 (8)	−0.0006 (7)	0.0068 (7)	0.0025 (7)
C21	0.0247 (9)	0.0230 (9)	0.0329 (10)	−0.0002 (7)	0.0036 (8)	−0.0010 (8)
C22	0.0250 (11)	0.0445 (13)	0.0645 (15)	−0.0016 (9)	0.0090 (10)	−0.0103 (11)
C23	0.0414 (12)	0.0395 (12)	0.0344 (11)	0.0035 (9)	−0.0038 (9)	0.0086 (9)
C24	0.0284 (10)	0.0263 (9)	0.0272 (10)	0.0026 (8)	0.0015 (7)	−0.0014 (8)
C25	0.0451 (13)	0.0438 (13)	0.0366 (12)	0.0078 (10)	−0.0009 (10)	0.0118 (10)
C26	0.0291 (11)	0.0429 (13)	0.0576 (14)	0.0040 (9)	0.0120 (10)	0.0013 (11)

Geometric parameters (Å, º)

P1—N4	1.6680 (15)	C12—H12	1.0000
P1—N5	1.6684 (14)	C13—H13A	0.9800
P1—N1	1.8547 (16)	C13—H13B	0.9800
N1—N2	1.168 (2)	C13—H13C	0.9800
N2—N3	1.155 (2)	C14—H14A	0.9800
N4—C3	1.436 (2)	C14—H14B	0.9800
N4—C1	1.456 (2)	C14—H14C	0.9800
N5—C15	1.441 (2)	C15—C16	1.410 (2)
N5—C2	1.463 (2)	C15—C20	1.410 (2)
C1—C2	1.477 (3)	C16—C17	1.393 (2)
C1—H1A	0.9900	C16—C21	1.517 (2)
C1—H1B	0.9900	C17—C18	1.377 (3)
C2—H2A	0.9900	C17—H17	0.9500
C2—H2B	0.9900	C18—C19	1.377 (3)
C3—C4	1.399 (2)	C18—H18	0.9500
C3—C8	1.403 (2)	C19—C20	1.397 (2)
C4—C5	1.393 (3)	C19—H19	0.9500
C4—C9	1.521 (3)	C20—C24	1.520 (2)
C5—C6	1.378 (3)	C21—C23	1.531 (3)
C5—H5	0.9500	C21—C22	1.532 (3)
C6—C7	1.372 (3)	C21—H21	1.0000
C6—H6	0.9500	C22—H22A	0.9800
C7—C8	1.392 (3)	C22—H22B	0.9800
C7—H7	0.9500	C22—H22C	0.9800
C8—C12	1.519 (3)	C23—H23A	0.9800
C9—C10	1.525 (3)	C23—H23B	0.9800

C9—C11	1.528 (3)	C23—H23C	0.9800
C9—H9	1.0000	C24—C26	1.524 (3)
C10—H10A	0.9800	C24—C25	1.529 (3)
C10—H10B	0.9800	C24—H24	1.0000
C10—H10C	0.9800	C25—H25A	0.9800
C11—H11A	0.9800	C25—H25B	0.9800
C11—H11B	0.9800	C25—H25C	0.9800
C11—H11C	0.9800	C26—H26A	0.9800
C12—C14	1.503 (3)	C26—H26B	0.9800
C12—C13	1.516 (4)	C26—H26C	0.9800

N4—P1—N5	91.14 (7)	C12—C13—H13B	109.5
N4—P1—N1	102.14 (8)	H13A—C13—H13B	109.5
N5—P1—N1	100.86 (7)	C12—C13—H13C	109.5
N2—N1—P1	116.29 (14)	H13A—C13—H13C	109.5
N3—N2—N1	176.6 (2)	H13B—C13—H13C	109.5
C3—N4—C1	120.31 (14)	C12—C14—H14A	109.5
C3—N4—P1	123.43 (11)	C12—C14—H14B	109.5
C1—N4—P1	116.17 (12)	H14A—C14—H14B	109.5
C15—N5—C2	120.53 (14)	C12—C14—H14C	109.5
C15—N5—P1	125.17 (11)	H14A—C14—H14C	109.5
C2—N5—P1	114.15 (12)	H14B—C14—H14C	109.5
N4—C1—C2	107.20 (16)	C16—C15—C20	120.81 (15)
N4—C1—H1A	110.3	C16—C15—N5	119.29 (15)
C2—C1—H1A	110.3	C20—C15—N5	119.82 (15)
N4—C1—H1B	110.3	C17—C16—C15	118.32 (16)
C2—C1—H1B	110.3	C17—C16—C21	119.13 (16)
H1A—C1—H1B	108.5	C15—C16—C21	122.54 (15)
N5—C2—C1	107.95 (15)	C18—C17—C16	121.37 (17)
N5—C2—H2A	110.1	C18—C17—H17	119.3
C1—C2—H2A	110.1	C16—C17—H17	119.3
N5—C2—H2B	110.1	C17—C18—C19	120.01 (17)
C1—C2—H2B	110.1	C17—C18—H18	120.0
H2A—C2—H2B	108.4	C19—C18—H18	120.0
C4—C3—C8	121.38 (16)	C18—C19—C20	121.38 (17)
C4—C3—N4	119.40 (15)	C18—C19—H19	119.3
C8—C3—N4	119.21 (15)	C20—C19—H19	119.3
C5—C4—C3	118.43 (17)	C19—C20—C15	118.11 (16)
C5—C4—C9	118.87 (17)	C19—C20—C24	118.84 (16)
C3—C4—C9	122.69 (16)	C15—C20—C24	123.02 (15)
C6—C5—C4	120.59 (19)	C16—C21—C23	110.73 (15)
C6—C5—H5	119.7	C16—C21—C22	112.73 (16)
C4—C5—H5	119.7	C23—C21—C22	109.64 (17)
C7—C6—C5	120.47 (19)	C16—C21—H21	107.9
C7—C6—H6	119.8	C23—C21—H21	107.9
C5—C6—H6	119.8	C22—C21—H21	107.9
C6—C7—C8	121.22 (19)	C21—C22—H22A	109.5
C6—C7—H7	119.4	C21—C22—H22B	109.5

C8—C7—H7	119.4	H22A—C22—H22B	109.5
C7—C8—C3	117.90 (17)	C21—C22—H22C	109.5
C7—C8—C12	119.86 (17)	H22A—C22—H22C	109.5
C3—C8—C12	122.18 (17)	H22B—C22—H22C	109.5
C4—C9—C10	110.70 (18)	C21—C23—H23A	109.5
C4—C9—C11	111.68 (17)	C21—C23—H23B	109.5
C10—C9—C11	110.99 (19)	H23A—C23—H23B	109.5
C4—C9—H9	107.8	C21—C23—H23C	109.5
C10—C9—H9	107.8	H23A—C23—H23C	109.5
C11—C9—H9	107.8	H23B—C23—H23C	109.5
C9—C10—H10A	109.5	C20—C24—C26	112.19 (16)
C9—C10—H10B	109.5	C20—C24—C25	110.59 (16)
H10A—C10—H10B	109.5	C26—C24—C25	109.90 (17)
C9—C10—H10C	109.5	C20—C24—H24	108.0
H10A—C10—H10C	109.5	C26—C24—H24	108.0
H10B—C10—H10C	109.5	C25—C24—H24	108.0
C9—C11—H11A	109.5	C24—C25—H25A	109.5
C9—C11—H11B	109.5	C24—C25—H25B	109.5
H11A—C11—H11B	109.5	H25A—C25—H25B	109.5
C9—C11—H11C	109.5	C24—C25—H25C	109.5
H11A—C11—H11C	109.5	H25A—C25—H25C	109.5
H11B—C11—H11C	109.5	H25B—C25—H25C	109.5
C14—C12—C13	109.7 (2)	C24—C26—H26A	109.5
C14—C12—C8	112.9 (2)	C24—C26—H26B	109.5
C13—C12—C8	110.17 (19)	H26A—C26—H26B	109.5
C14—C12—H12	108.0	C24—C26—H26C	109.5
C13—C12—H12	108.0	H26A—C26—H26C	109.5
C8—C12—H12	108.0	H26B—C26—H26C	109.5
C12—C13—H13A	109.5		

N4—P1—N1—N2	113.83 (16)	C3—C4—C9—C10	113.2 (2)
N5—P1—N1—N2	−152.62 (15)	C5—C4—C9—C11	58.4 (3)
N5—P1—N4—C3	169.76 (14)	C3—C4—C9—C11	−122.6 (2)
N1—P1—N4—C3	−88.87 (14)	C7—C8—C12—C14	−48.6 (3)
N5—P1—N4—C1	−6.93 (16)	C3—C8—C12—C14	134.2 (2)
N1—P1—N4—C1	94.44 (16)	C7—C8—C12—C13	74.3 (3)
N4—P1—N5—C15	−168.59 (14)	C3—C8—C12—C13	−102.9 (3)
N1—P1—N5—C15	88.82 (14)	C2—N5—C15—C16	−103.4 (2)
N4—P1—N5—C2	15.88 (14)	P1—N5—C15—C16	81.30 (19)
N1—P1—N5—C2	−86.71 (14)	C2—N5—C15—C20	73.4 (2)
C3—N4—C1—C2	179.70 (17)	P1—N5—C15—C20	−101.83 (17)
P1—N4—C1—C2	−3.5 (2)	C20—C15—C16—C17	0.1 (2)
C15—N5—C2—C1	163.95 (17)	N5—C15—C16—C17	176.90 (15)
P1—N5—C2—C1	−20.3 (2)	C20—C15—C16—C21	179.24 (16)
N4—C1—C2—N5	14.1 (3)	N5—C15—C16—C21	−3.9 (2)
C1—N4—C3—C4	−79.8 (2)	C15—C16—C17—C18	−0.5 (3)
P1—N4—C3—C4	103.69 (18)	C21—C16—C17—C18	−179.74 (17)
C1—N4—C3—C8	99.4 (2)	C16—C17—C18—C19	0.6 (3)

P1—N4—C3—C8	−77.2 (2)	C17—C18—C19—C20	−0.2 (3)
C8—C3—C4—C5	0.6 (3)	C18—C19—C20—C15	−0.2 (3)
N4—C3—C4—C5	179.73 (18)	C18—C19—C20—C24	−178.33 (17)
C8—C3—C4—C9	−178.35 (18)	C16—C15—C20—C19	0.3 (2)
N4—C3—C4—C9	0.8 (3)	N5—C15—C20—C19	−176.51 (15)
C3—C4—C5—C6	0.1 (3)	C16—C15—C20—C24	178.31 (16)
C9—C4—C5—C6	179.1 (2)	N5—C15—C20—C24	1.5 (2)
C4—C5—C6—C7	−0.6 (4)	C17—C16—C21—C23	84.6 (2)
C5—C6—C7—C8	0.4 (4)	C15—C16—C21—C23	−94.5 (2)
C6—C7—C8—C3	0.3 (3)	C17—C16—C21—C22	−38.6 (2)
C6—C7—C8—C12	−177.1 (2)	C15—C16—C21—C22	142.23 (18)
C4—C3—C8—C7	−0.8 (3)	C19—C20—C24—C26	−58.9 (2)
N4—C3—C8—C7	−179.90 (18)	C15—C20—C24—C26	123.11 (19)
C4—C3—C8—C12	176.48 (18)	C19—C20—C24—C25	64.2 (2)
N4—C3—C8—C12	−2.6 (3)	C15—C20—C24—C25	−113.80 (19)
C5—C4—C9—C10	−65.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···N4	1.00	2.43	2.926 (2)	110
C12—H12···N4	1.00	2.44	2.913 (2)	109
C21—H21···N5	1.00	2.49	2.932 (2)	106
C24—H24···N1	1.00	2.66	3.443 (3)	136
C24—H24···N5	1.00	2.46	2.955 (2)	110
C22—H22*C*···N3^{i}	0.98	2.69	3.669 (3)	174

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