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1,1′-(Diphosphene-1,2-diyl)bis(2,2,6,6-tetramethylpiperidine)

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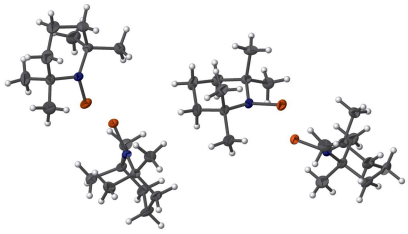
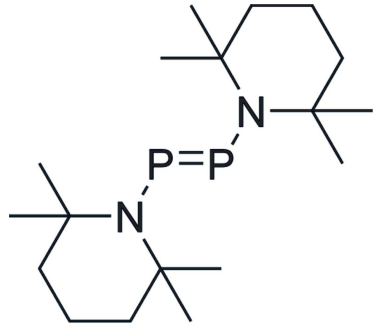
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{18}H_{36}N_2P_2$, crystallizes in the triclinic space group $P\overline{1}$ with two independent molecules in the asymmetric unit. Both molecules adopt a *trans* configuration of the tetramethylpiperidine units along the P═P axis. The crystal packing is stabilized only by van der Waals interactions.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) crystallizes in the triclinic space group $P\overline{1}$ with four molecules in the unit cell. The asymmetric unit contains two independent molecules. Both independent molecules adopt a *trans* configuration of the tetramethypiperidine (tmp) units along the P═P axis. There is no centre of inversion in the middle of the P═P bond. The tmp units are twisted against each other, with different P—N bond lengths [P1—N1 = 1.6950 (10) Å, P3—N3 = 1.6907 (11) Å, P2—N2 = 1.7508 (11) Å and P4—N4 = 1.7562 (10) Å]. All P—N bond lengths are shortened compared to the value of a P—N single bond calculated by Pyykkö (P—N = 1.82 Å; Pyykkö, 2015) and show some multiple bond character.

Whereas atoms N1 and N3 are in an almost ideal planar environment (the sums of the angles around N1 and N3 are 356.4 and 356.7°, respectively), the geometry at N2 and N4 is more trigonal pyramidal (the sums of the angles around N2 and N4 are 346.7 and 346.8°, respectively). The P—P bond lengths [P1—P2 = 2.0570 (5) Å and P3—P4 = 2.0559 (5) Å] are in agreement with the value of 2.04 Å of a P═P double bond as calculated by Pyykkö (2015) and comparable to the values found in R_2N—P═P—NR_2 diphosphenes (Schulz *et al.*, 2014; Schaffrath *et al.*, 2008; Niecke *et al.*, 1983). The *trans* bent N—P—P—N skeleton is almost planar [dihedral angle between the planes defined by N1/P1/P2 and P1/P2/N2 = 4.59 (8)° and dihedral angle between the planes defined by N3/P3/P4 and P3/P4/N4 = 0.65 (8)°]. The crystal packing is governed only by van der Waals interactions.

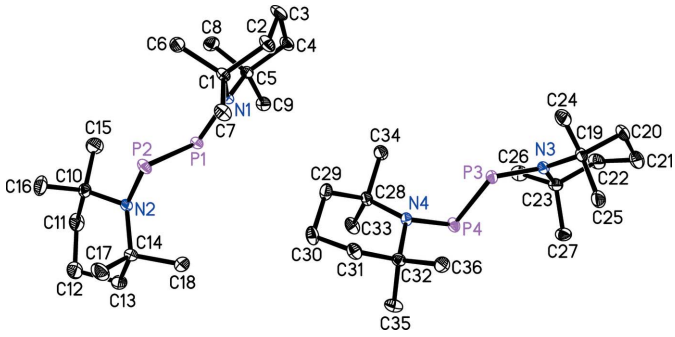


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

A search of the Cambridge Structural Database (Version 5.38, last update May 2017; Groom *et al.*, 2016) for the title compound revealed only one tungsten pentacarbonyl complex where the diphosphene is coordinated *via* one P atom (Borm *et al.*, 1987).

Synthesis and crystallization

A solution of (η^2-btmsa)Ti(Cp)$_2$ (btmsa is bis(trimethylsilyl)acetylene; 0.520 g, 1.5 mmol) in THF (10 ml) was added to a stirred solution of (tmp)PCl_2 (0.363, 1.5 mmol) in THF (10 ml). The colour changed from green to red. After 12 h of stirring, the THF was removed in a vacuum and the residue was extracted with *n*-hexane. After storing the concentrated extract for two weeks at 195 K, some red crystals could be isolated. ^{31}P NMR (121 MHz, C_6D_6, 298 K): δ 474.7. MS (CI): *m*/*z* 342, $[C_{18}H_{36}N_2P_2]^+$, 684 $[C_{36}H_{72}N_4P_2]^+$, 544 $[C_{27}H_{54}N_3P_4]^+$, 202 $[C_9H_{18}NP_2]^+$, 142 $[C_5H_6NP_2]^+$. The title compound was first synthesized in a different way by Markovski *et al.* (1983).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{36}N_2P_2$
M_r	342.43
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, *b*, *c* (Å)	11.3277 (3), 12.3257 (3), 15.5274 (4)
α, β, γ (°)	112.5896 (6), 90.7806 (7), 92.9221 (7)
V (Å^3)	1997.74 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.22
Crystal size (mm)	0.53 × 0.29 × 0.16
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.89, 0.96
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37690, 10119, 8123
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.671
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.035, 0.095, 1.03
No. of reflections	10119
No. of parameters	413
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.20

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

References

Borm, J., Huttner, G., Zsolnai, L. & Evertz, K. (1987). *J. Organomet. Chem.* **327**, 223–235.

Bruker (2013). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2014). *APEX2* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Markovski, L. N., Romanenko, V. D. & Kirsanov, A. V. (1983). *Phosphorus Sulfur Silicon Relat. Elem.* **18**, 31–34.

Niecke, E., Rüger, R., Lysek, M., Pohl, S. & Schoeller, W. (1983). *Angew. Chem. Int. Ed.* **22**, 486–487.

Pyykkö, P. (2015). *J. Phys. Chem. A*, **119**, 2326–2337.

Schaffrath, M., Villinger, A., Michalik, D., Rosenthal, U. & Schulz, A. (2008). *Organometallics*, **27**, 1393–1398.

Schulz, A., Villinger, A. & Westenkirchner, A. (2014). *Inorg. Chem.* **53**, 3183–3193.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2017). **2**, x170903 [https://doi.org/10.1107/S2414314617009038]

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Crystal data

$C_{18}H_{36}N_2P_2$
M_r = 342.43
Triclinic, $P\overline{1}$
a = 11.3277 (3) Å
b = 12.3257 (3) Å
c = 15.5274 (4) Å
α = 112.5896 (6)°
β = 90.7806 (7)°
γ = 92.9221 (7)°
V = 1997.74 (9) Å^3
Z = 4
$F(000)$ = 752
D_x = 1.139 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9914 reflections
θ = 2.5–28.8°
μ = 0.22 mm^{-1}
T = 150 K
Plate, red-orange
0.53 × 0.29 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.89, T_{max} = 0.96
37690 measured reflections
10119 independent reflections
8123 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 28.5°, θ_{min} = 1.8°
h = −15→15
k = −16→16
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.095
S = 1.03
10119 reflections
413 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.4629P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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}
C1	0.12200 (12)	0.41884 (11)	0.22343 (9)	0.0242 (3)
C2	0.10873 (14)	0.55084 (12)	0.24981 (10)	0.0341 (3)
H2A	0.0412	0.5750	0.2914	0.041*
H2B	0.1808	0.5947	0.2856	0.041*
C3	0.08935 (15)	0.58596 (13)	0.16821 (12)	0.0391 (4)
H3A	0.0818	0.6720	0.1904	0.047*
H3B	0.0159	0.5455	0.1326	0.047*
C4	0.19499 (13)	0.55088 (12)	0.10698 (11)	0.0327 (3)
H4A	0.2669	0.5948	0.1431	0.039*
H4B	0.1844	0.5742	0.0532	0.039*
C5	0.21403 (12)	0.41882 (11)	0.07033 (9)	0.0242 (3)
C6	0.00231 (12)	0.34924 (13)	0.19221 (10)	0.0321 (3)
H6A	−0.0349	0.3697	0.1435	0.048*
H6B	−0.0491	0.3689	0.2457	0.048*
H6C	0.0144	0.2647	0.1675	0.048*
C7	0.16625 (14)	0.40779 (12)	0.31327 (9)	0.0319 (3)
H7A	0.1593	0.3250	0.3055	0.048*
H7B	0.1185	0.4541	0.3653	0.048*
H7C	0.2493	0.4372	0.3264	0.048*
C8	0.12062 (14)	0.34995 (14)	−0.00633 (10)	0.0358 (3)
H8A	0.1277	0.2653	−0.0237	0.054*
H8B	0.1330	0.3696	−0.0611	0.054*
H8C	0.0415	0.3709	0.0169	0.054*
C9	0.33472 (13)	0.40215 (13)	0.02545 (10)	0.0308 (3)
H9A	0.3975	0.4281	0.0744	0.046*
H9B	0.3425	0.4489	−0.0132	0.046*
H9C	0.3415	0.3188	−0.0136	0.046*
C10	0.24168 (12)	−0.07528 (12)	0.05066 (10)	0.0298 (3)
C11	0.32824 (15)	−0.16386 (13)	−0.00676 (11)	0.0415 (4)
H11A	0.2827	−0.2350	−0.0502	0.050*
H11B	0.3739	−0.1291	−0.0448	0.050*
C12	0.41360 (15)	−0.20015 (13)	0.05128 (14)	0.0476 (4)
H12A	0.4684	−0.2559	0.0104	0.057*
H12B	0.3698	−0.2396	0.0870	0.057*
C13	0.48237 (14)	−0.09058 (13)	0.11755 (13)	0.0427 (4)
H13A	0.5291	−0.0550	0.0806	0.051*
H13B	0.5386	−0.1135	0.1560	0.051*
C14	0.40418 (12)	0.00193 (12)	0.18232 (10)	0.0293 (3)
C15	0.18031 (18)	−0.03158 (15)	−0.01752 (12)	0.0508 (5)
H15A	0.1137	0.0145	0.0127	0.076*
H15B	0.1511	−0.0992	−0.0731	0.076*
H15C	0.2368	0.0179	−0.0358	0.076*
C16	0.14760 (15)	−0.13645 (14)	0.08898 (14)	0.0478 (4)
H16A	0.1862	−0.1767	0.1242	0.072*
H16B	0.0994	−0.1941	0.0371	0.072*

H16C	0.0969	−0.0778	0.1302	0.072*
C17	0.35612 (17)	−0.03795 (15)	0.25880 (12)	0.0465 (4)
H17A	0.2980	0.0165	0.2946	0.070*
H17B	0.4216	−0.0381	0.3007	0.070*
H17C	0.3183	−0.1175	0.2296	0.070*
C18	0.48237 (14)	0.11482 (14)	0.23090 (12)	0.0404 (4)
H18A	0.5072	0.1472	0.1849	0.061*
H18B	0.5523	0.0971	0.2598	0.061*
H18C	0.4377	0.1725	0.2791	0.061*
C19	0.73363 (13)	1.14794 (11)	0.47806 (10)	0.0291 (3)
C20	0.76170 (15)	1.26305 (13)	0.46409 (12)	0.0414 (4)
H20A	0.7006	1.2715	0.4212	0.050*
H20B	0.7576	1.3300	0.5248	0.050*
C21	0.88242 (17)	1.26920 (14)	0.42476 (14)	0.0518 (5)
H21A	0.8969	1.3455	0.4182	0.062*
H21B	0.9450	1.2617	0.4670	0.062*
C22	0.88381 (15)	1.16947 (14)	0.33088 (13)	0.0440 (4)
H22A	0.9606	1.1745	0.3026	0.053*
H22B	0.8210	1.1791	0.2899	0.053*
C23	0.86525 (12)	1.04663 (12)	0.33394 (10)	0.0296 (3)
C24	0.60186 (14)	1.14436 (13)	0.49856 (11)	0.0392 (4)
H24A	0.5546	1.1301	0.4415	0.059*
H24B	0.5831	1.2198	0.5468	0.059*
H24C	0.5836	1.0809	0.5205	0.059*
C25	0.80459 (16)	1.14766 (13)	0.56278 (11)	0.0407 (4)
H25A	0.7911	1.0712	0.5679	0.061*
H25B	0.7790	1.2100	0.6195	0.061*
H25C	0.8890	1.1616	0.5551	0.061*
C26	0.84088 (14)	0.96118 (14)	0.23293 (10)	0.0380 (3)
H26A	0.8442	0.8801	0.2290	0.057*
H26B	0.9006	0.9762	0.1932	0.057*
H26C	0.7622	0.9724	0.2118	0.057*
C27	0.97816 (13)	1.01572 (14)	0.37270 (12)	0.0387 (4)
H27A	0.9989	1.0762	0.4346	0.058*
H27B	1.0430	1.0121	0.3307	0.058*
H27C	0.9649	0.9391	0.3776	0.058*
C28	0.61407 (12)	0.59008 (11)	0.26381 (9)	0.0245 (3)
C29	0.51550 (13)	0.51827 (13)	0.28818 (10)	0.0331 (3)
H29A	0.4514	0.5704	0.3176	0.040*
H29B	0.4822	0.4558	0.2299	0.040*
C30	0.55701 (15)	0.46199 (12)	0.35352 (10)	0.0349 (3)
H30A	0.4902	0.4168	0.3672	0.042*
H30B	0.6193	0.4073	0.3243	0.042*
C31	0.60543 (13)	0.55930 (12)	0.44268 (9)	0.0305 (3)
H31A	0.6327	0.5236	0.4862	0.037*
H31B	0.5407	0.6103	0.4724	0.037*
C32	0.70809 (12)	0.63588 (11)	0.42828 (9)	0.0241 (3)
C33	0.69741 (15)	0.50609 (12)	0.19533 (9)	0.0351 (3)

H33A	0.7647	0.5519	0.1845	0.053*
H33B	0.6543	0.4613	0.1360	0.053*
H33C	0.7263	0.4516	0.2219	0.053*
C34	0.55503 (14)	0.66095 (12)	0.21461 (10)	0.0334 (3)
H34A	0.5077	0.7202	0.2591	0.050*
H34B	0.5037	0.6078	0.1627	0.050*
H34C	0.6160	0.7001	0.1906	0.050*
C35	0.82123 (14)	0.56889 (14)	0.41102 (11)	0.0358 (3)
H35A	0.8074	0.4933	0.3581	0.054*
H35B	0.8436	0.5549	0.4669	0.054*
H35C	0.8851	0.6156	0.3970	0.054*
C36	0.72659 (13)	0.74385 (12)	0.51983 (9)	0.0315 (3)
H36A	0.8004	0.7880	0.5182	0.047*
H36B	0.7310	0.7183	0.5722	0.047*
H36C	0.6602	0.7944	0.5277	0.047*
N1	0.21138 (9)	0.37439 (9)	0.14912 (7)	0.0206 (2)
N2	0.30899 (9)	0.02706 (9)	0.12518 (7)	0.0219 (2)
N3	0.76024 (9)	1.04176 (9)	0.39135 (7)	0.0233 (2)
N4	0.67588 (9)	0.67632 (9)	0.35135 (7)	0.0206 (2)
P1	0.27799 (3)	0.24572 (3)	0.12079 (2)	0.02249 (8)
P2	0.22077 (3)	0.14099 (3)	0.18980 (3)	0.02804 (9)
P3	0.69308 (3)	0.91639 (3)	0.39196 (2)	0.02235 (8)
P4	0.78561 (3)	0.76827 (3)	0.33047 (3)	0.02704 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0289 (7)	0.0201 (6)	0.0250 (6)	0.0065 (5)	0.0053 (5)	0.0092 (5)
C2	0.0421 (8)	0.0226 (7)	0.0385 (8)	0.0100 (6)	0.0118 (6)	0.0115 (6)
C3	0.0436 (9)	0.0285 (7)	0.0550 (10)	0.0148 (6)	0.0124 (7)	0.0251 (7)
C4	0.0361 (8)	0.0278 (7)	0.0430 (8)	0.0046 (6)	0.0047 (6)	0.0229 (6)
C5	0.0262 (7)	0.0253 (6)	0.0251 (6)	0.0016 (5)	−0.0012 (5)	0.0141 (5)
C6	0.0278 (7)	0.0308 (7)	0.0401 (8)	0.0045 (6)	0.0077 (6)	0.0158 (6)
C7	0.0446 (9)	0.0290 (7)	0.0213 (6)	0.0082 (6)	0.0058 (6)	0.0082 (5)
C8	0.0360 (8)	0.0443 (8)	0.0311 (7)	−0.0005 (7)	−0.0079 (6)	0.0198 (7)
C9	0.0330 (8)	0.0359 (7)	0.0287 (7)	0.0030 (6)	0.0046 (6)	0.0180 (6)
C10	0.0285 (7)	0.0217 (6)	0.0352 (7)	0.0003 (5)	−0.0008 (6)	0.0065 (5)
C11	0.0455 (9)	0.0262 (7)	0.0413 (9)	0.0040 (6)	0.0087 (7)	−0.0001 (6)
C12	0.0372 (9)	0.0262 (7)	0.0722 (12)	0.0122 (6)	0.0096 (8)	0.0093 (8)
C13	0.0269 (8)	0.0323 (8)	0.0675 (11)	0.0094 (6)	0.0003 (7)	0.0168 (8)
C14	0.0279 (7)	0.0259 (7)	0.0376 (7)	0.0029 (5)	−0.0042 (6)	0.0161 (6)
C15	0.0607 (12)	0.0363 (9)	0.0459 (10)	−0.0007 (8)	−0.0258 (8)	0.0068 (7)
C16	0.0324 (8)	0.0324 (8)	0.0706 (12)	−0.0059 (7)	0.0096 (8)	0.0117 (8)
C17	0.0625 (11)	0.0435 (9)	0.0430 (9)	0.0027 (8)	−0.0044 (8)	0.0276 (8)
C18	0.0367 (8)	0.0348 (8)	0.0479 (9)	−0.0026 (6)	−0.0128 (7)	0.0149 (7)
C19	0.0363 (8)	0.0176 (6)	0.0313 (7)	0.0016 (5)	−0.0045 (6)	0.0075 (5)
C20	0.0535 (10)	0.0208 (7)	0.0514 (9)	0.0031 (6)	−0.0084 (8)	0.0158 (7)
C21	0.0526 (11)	0.0296 (8)	0.0833 (13)	−0.0099 (7)	−0.0093 (9)	0.0349 (9)

C22	0.0391 (9)	0.0415 (9)	0.0683 (11)	−0.0008 (7)	0.0049 (8)	0.0402 (9)
C23	0.0231 (7)	0.0324 (7)	0.0423 (8)	0.0009 (5)	0.0027 (6)	0.0243 (6)
C24	0.0407 (9)	0.0295 (7)	0.0407 (8)	0.0106 (6)	0.0077 (7)	0.0048 (6)
C25	0.0583 (11)	0.0258 (7)	0.0340 (8)	0.0001 (7)	−0.0128 (7)	0.0080 (6)
C26	0.0362 (8)	0.0490 (9)	0.0375 (8)	0.0059 (7)	0.0119 (6)	0.0254 (7)
C27	0.0228 (7)	0.0420 (8)	0.0622 (10)	0.0000 (6)	−0.0003 (7)	0.0323 (8)
C28	0.0332 (7)	0.0191 (6)	0.0205 (6)	−0.0002 (5)	0.0000 (5)	0.0073 (5)
C29	0.0373 (8)	0.0289 (7)	0.0307 (7)	−0.0091 (6)	−0.0049 (6)	0.0105 (6)
C30	0.0471 (9)	0.0262 (7)	0.0331 (7)	−0.0071 (6)	0.0034 (6)	0.0144 (6)
C31	0.0396 (8)	0.0296 (7)	0.0273 (7)	0.0023 (6)	0.0060 (6)	0.0162 (6)
C32	0.0277 (7)	0.0238 (6)	0.0215 (6)	0.0047 (5)	0.0014 (5)	0.0092 (5)
C33	0.0550 (10)	0.0254 (7)	0.0229 (7)	0.0068 (6)	0.0066 (6)	0.0064 (5)
C34	0.0445 (9)	0.0282 (7)	0.0272 (7)	0.0001 (6)	−0.0082 (6)	0.0110 (6)
C35	0.0369 (8)	0.0384 (8)	0.0361 (8)	0.0162 (6)	0.0045 (6)	0.0170 (7)
C36	0.0385 (8)	0.0312 (7)	0.0223 (6)	0.0028 (6)	−0.0026 (6)	0.0075 (6)
N1	0.0228 (5)	0.0192 (5)	0.0217 (5)	0.0036 (4)	0.0007 (4)	0.0098 (4)
N2	0.0219 (5)	0.0182 (5)	0.0257 (5)	0.0042 (4)	0.0020 (4)	0.0080 (4)
N3	0.0240 (5)	0.0199 (5)	0.0276 (5)	0.0004 (4)	0.0002 (4)	0.0112 (4)
N4	0.0247 (5)	0.0177 (5)	0.0188 (5)	−0.0005 (4)	0.0013 (4)	0.0065 (4)
P1	0.02445 (17)	0.02026 (15)	0.02379 (16)	0.00495 (12)	0.00272 (12)	0.00913 (13)
P2	0.03202 (19)	0.02171 (16)	0.03365 (19)	0.00838 (14)	0.01169 (14)	0.01312 (14)
P3	0.02274 (17)	0.01921 (15)	0.02477 (16)	0.00041 (12)	0.00330 (12)	0.00812 (13)
P4	0.02562 (18)	0.02122 (16)	0.03441 (19)	0.00226 (13)	0.00918 (14)	0.01051 (14)

Geometric parameters (Å, º)

C1—N1	1.5032 (16)	C19—C20	1.5341 (19)
C1—C7	1.5329 (18)	C20—C21	1.515 (3)
C1—C2	1.5337 (17)	C20—H20A	0.9900
C1—C6	1.5341 (19)	C20—H20B	0.9900
C2—C3	1.505 (2)	C21—C22	1.504 (3)
C2—H2A	0.9900	C21—H21A	0.9900
C2—H2B	0.9900	C21—H21B	0.9900
C3—C4	1.514 (2)	C22—C23	1.5369 (19)
C3—H3A	0.9900	C22—H22A	0.9900
C3—H3B	0.9900	C22—H22B	0.9900
C4—C5	1.5321 (18)	C23—N3	1.5085 (17)
C4—H4A	0.9900	C23—C26	1.528 (2)
C4—H4B	0.9900	C23—C27	1.5325 (19)
C5—N1	1.5204 (15)	C24—H24A	0.9800
C5—C9	1.5302 (19)	C24—H24B	0.9800
C5—C8	1.5345 (19)	C24—H24C	0.9800
C6—H6A	0.9800	C25—H25A	0.9800
C6—H6B	0.9800	C25—H25B	0.9800
C6—H6C	0.9800	C25—H25C	0.9800
C7—H7A	0.9800	C26—H26A	0.9800
C7—H7B	0.9800	C26—H26B	0.9800
C7—H7C	0.9800	C26—H26C	0.9800

C8—H8A	0.9800	C27—H27A	0.9800
C8—H8B	0.9800	C27—H27B	0.9800
C8—H8C	0.9800	C27—H27C	0.9800
C9—H9A	0.9800	C28—N4	1.5021 (16)
C9—H9B	0.9800	C28—C29	1.5301 (18)
C9—H9C	0.9800	C28—C34	1.5336 (18)
C10—N2	1.5059 (17)	C28—C33	1.5411 (19)
C10—C11	1.528 (2)	C29—C30	1.514 (2)
C10—C16	1.529 (2)	C29—H29A	0.9900
C10—C15	1.533 (2)	C29—H29B	0.9900
C11—C12	1.509 (2)	C30—C31	1.514 (2)
C11—H11A	0.9900	C30—H30A	0.9900
C11—H11B	0.9900	C30—H30B	0.9900
C12—C13	1.514 (2)	C31—C32	1.5332 (18)
C12—H12A	0.9900	C31—H31A	0.9900
C12—H12B	0.9900	C31—H31B	0.9900
C13—C14	1.531 (2)	C32—N4	1.5074 (15)
C13—H13A	0.9900	C32—C35	1.5308 (19)
C13—H13B	0.9900	C32—C36	1.5316 (18)
C14—N2	1.5050 (16)	C33—H33A	0.9800
C14—C18	1.5294 (19)	C33—H33B	0.9800
C14—C17	1.545 (2)	C33—H33C	0.9800
C15—H15A	0.9800	C34—H34A	0.9800
C15—H15B	0.9800	C34—H34B	0.9800
C15—H15C	0.9800	C34—H34C	0.9800
C16—H16A	0.9800	C35—H35A	0.9800
C16—H16B	0.9800	C35—H35B	0.9800
C16—H16C	0.9800	C35—H35C	0.9800
C17—H17A	0.9800	C36—H36A	0.9800
C17—H17B	0.9800	C36—H36B	0.9800
C17—H17C	0.9800	C36—H36C	0.9800
C18—H18A	0.9800	N1—P1	1.6950 (10)
C18—H18B	0.9800	N2—P2	1.7508 (11)
C18—H18C	0.9800	N3—P3	1.6907 (11)
C19—N3	1.5212 (17)	N4—P4	1.7562 (10)
C19—C24	1.532 (2)	P1—P2	2.0570 (5)
C19—C25	1.533 (2)	P3—P4	2.0559 (5)

N1—C1—C7	109.91 (10)	C19—C20—H20A	108.9
N1—C1—C2	110.44 (10)	C21—C20—H20B	108.9
C7—C1—C2	105.26 (11)	C19—C20—H20B	108.9
N1—C1—C6	111.20 (10)	H20A—C20—H20B	107.7
C7—C1—C6	108.94 (11)	C22—C21—C20	107.51 (14)
C2—C1—C6	110.92 (12)	C22—C21—H21A	110.2
C3—C2—C1	114.64 (12)	C20—C21—H21A	110.2
C3—C2—H2A	108.6	C22—C21—H21B	110.2
C1—C2—H2A	108.6	C20—C21—H21B	110.2
C3—C2—H2B	108.6	H21A—C21—H21B	108.5

C1—C2—H2B	108.6	C21—C22—C23	114.19 (13)
H2A—C2—H2B	107.6	C21—C22—H22A	108.7
C2—C3—C4	107.61 (12)	C23—C22—H22A	108.7
C2—C3—H3A	110.2	C21—C22—H22B	108.7
C4—C3—H3A	110.2	C23—C22—H22B	108.7
C2—C3—H3B	110.2	H22A—C22—H22B	107.6
C4—C3—H3B	110.2	N3—C23—C26	109.48 (11)
H3A—C3—H3B	108.5	N3—C23—C27	111.38 (11)
C3—C4—C5	113.65 (11)	C26—C23—C27	109.69 (13)
C3—C4—H4A	108.8	N3—C23—C22	110.55 (12)
C5—C4—H4A	108.8	C26—C23—C22	106.03 (12)
C3—C4—H4B	108.8	C27—C23—C22	109.57 (12)
C5—C4—H4B	108.8	C19—C24—H24A	109.5
H4A—C4—H4B	107.7	C19—C24—H24B	109.5
N1—C5—C9	110.53 (10)	H24A—C24—H24B	109.5
N1—C5—C4	110.97 (10)	C19—C24—H24C	109.5
C9—C5—C4	106.70 (11)	H24A—C24—H24C	109.5
N1—C5—C8	110.70 (10)	H24B—C24—H24C	109.5
C9—C5—C8	107.44 (11)	C19—C25—H25A	109.5
C4—C5—C8	110.37 (11)	C19—C25—H25B	109.5
C1—C6—H6A	109.5	H25A—C25—H25B	109.5
C1—C6—H6B	109.5	C19—C25—H25C	109.5
H6A—C6—H6B	109.5	H25A—C25—H25C	109.5
C1—C6—H6C	109.5	H25B—C25—H25C	109.5
H6A—C6—H6C	109.5	C23—C26—H26A	109.5
H6B—C6—H6C	109.5	C23—C26—H26B	109.5
C1—C7—H7A	109.5	H26A—C26—H26B	109.5
C1—C7—H7B	109.5	C23—C26—H26C	109.5
H7A—C7—H7B	109.5	H26A—C26—H26C	109.5
C1—C7—H7C	109.5	H26B—C26—H26C	109.5
H7A—C7—H7C	109.5	C23—C27—H27A	109.5
H7B—C7—H7C	109.5	C23—C27—H27B	109.5
C5—C8—H8A	109.5	H27A—C27—H27B	109.5
C5—C8—H8B	109.5	C23—C27—H27C	109.5
H8A—C8—H8B	109.5	H27A—C27—H27C	109.5
C5—C8—H8C	109.5	H27B—C27—H27C	109.5
H8A—C8—H8C	109.5	N4—C28—C29	110.14 (10)
H8B—C8—H8C	109.5	N4—C28—C34	107.63 (10)
C5—C9—H9A	109.5	C29—C28—C34	107.12 (12)
C5—C9—H9B	109.5	N4—C28—C33	113.73 (11)
H9A—C9—H9B	109.5	C29—C28—C33	109.53 (11)
C5—C9—H9C	109.5	C34—C28—C33	108.44 (11)
H9A—C9—H9C	109.5	C30—C29—C28	113.23 (12)
H9B—C9—H9C	109.5	C30—C29—H29A	108.9
N2—C10—C11	109.58 (11)	C28—C29—H29A	108.9
N2—C10—C16	113.54 (12)	C30—C29—H29B	108.9
C11—C10—C16	110.12 (12)	C28—C29—H29B	108.9
N2—C10—C15	109.13 (11)	H29A—C29—H29B	107.7

C11—C10—C15	105.99 (13)	C31—C30—C29	107.93 (11)
C16—C10—C15	108.20 (14)	C31—C30—H30A	110.1
C12—C11—C10	113.89 (13)	C29—C30—H30A	110.1
C12—C11—H11A	108.8	C31—C30—H30B	110.1
C10—C11—H11A	108.8	C29—C30—H30B	110.1
C12—C11—H11B	108.8	H30A—C30—H30B	108.4
C10—C11—H11B	108.8	C30—C31—C32	113.98 (11)
H11A—C11—H11B	107.7	C30—C31—H31A	108.8
C11—C12—C13	108.20 (13)	C32—C31—H31A	108.8
C11—C12—H12A	110.1	C30—C31—H31B	108.8
C13—C12—H12A	110.1	C32—C31—H31B	108.8
C11—C12—H12B	110.1	H31A—C31—H31B	107.7
C13—C12—H12B	110.1	N4—C32—C35	113.36 (10)
H12A—C12—H12B	108.4	N4—C32—C36	108.77 (10)
C12—C13—C14	113.66 (13)	C35—C32—C36	108.03 (11)
C12—C13—H13A	108.8	N4—C32—C31	110.12 (10)
C14—C13—H13A	108.8	C35—C32—C31	109.73 (11)
C12—C13—H13B	108.8	C36—C32—C31	106.57 (11)
C14—C13—H13B	108.8	C28—C33—H33A	109.5
H13A—C13—H13B	107.7	C28—C33—H33B	109.5
N2—C14—C18	108.35 (11)	H33A—C33—H33B	109.5
N2—C14—C13	109.41 (12)	C28—C33—H33C	109.5
C18—C14—C13	107.43 (12)	H33A—C33—H33C	109.5
N2—C14—C17	113.71 (12)	H33B—C33—H33C	109.5
C18—C14—C17	107.50 (13)	C28—C34—H34A	109.5
C13—C14—C17	110.21 (13)	C28—C34—H34B	109.5
C10—C15—H15A	109.5	H34A—C34—H34B	109.5
C10—C15—H15B	109.5	C28—C34—H34C	109.5
H15A—C15—H15B	109.5	H34A—C34—H34C	109.5
C10—C15—H15C	109.5	H34B—C34—H34C	109.5
H15A—C15—H15C	109.5	C32—C35—H35A	109.5
H15B—C15—H15C	109.5	C32—C35—H35B	109.5
C10—C16—H16A	109.5	H35A—C35—H35B	109.5
C10—C16—H16B	109.5	C32—C35—H35C	109.5
H16A—C16—H16B	109.5	H35A—C35—H35C	109.5
C10—C16—H16C	109.5	H35B—C35—H35C	109.5
H16A—C16—H16C	109.5	C32—C36—H36A	109.5
H16B—C16—H16C	109.5	C32—C36—H36B	109.5
C14—C17—H17A	109.5	H36A—C36—H36B	109.5
C14—C17—H17B	109.5	C32—C36—H36C	109.5
H17A—C17—H17B	109.5	H36A—C36—H36C	109.5
C14—C17—H17C	109.5	H36B—C36—H36C	109.5
H17A—C17—H17C	109.5	C1—N1—C5	119.26 (9)
H17B—C17—H17C	109.5	C1—N1—P1	125.11 (8)
C14—C18—H18A	109.5	C5—N1—P1	112.03 (8)
C14—C18—H18B	109.5	C14—N2—C10	118.53 (10)
H18A—C18—H18B	109.5	C14—N2—P2	114.13 (8)
C14—C18—H18C	109.5	C10—N2—P2	114.07 (8)

H18A—C18—H18C	109.5	C23—N3—C19	120.57 (10)
H18B—C18—H18C	109.5	C23—N3—P3	124.62 (9)
N3—C19—C24	110.36 (11)	C19—N3—P3	111.55 (8)
N3—C19—C25	110.11 (11)	C28—N4—C32	119.12 (9)
C24—C19—C25	108.19 (13)	C28—N4—P4	113.39 (8)
N3—C19—C20	111.04 (12)	C32—N4—P4	114.29 (8)
C24—C19—C20	106.82 (12)	N1—P1—P2	115.56 (4)
C25—C19—C20	110.23 (12)	N2—P2—P1	92.65 (4)
C21—C20—C19	113.33 (13)	N3—P3—P4	114.77 (4)
C21—C20—H20A	108.9	N4—P4—P3	93.06 (4)

N1—C1—C2—C3	49.82 (16)	C18—C14—N2—C10	163.34 (12)
C7—C1—C2—C3	168.41 (13)	C13—C14—N2—C10	46.50 (15)
C6—C1—C2—C3	−73.92 (16)	C17—C14—N2—C10	−77.21 (15)
C1—C2—C3—C4	−59.70 (17)	C18—C14—N2—P2	−57.89 (13)
C2—C3—C4—C5	59.26 (17)	C13—C14—N2—P2	−174.73 (9)
C3—C4—C5—N1	−49.56 (16)	C17—C14—N2—P2	61.56 (13)
C3—C4—C5—C9	−170.04 (12)	C11—C10—N2—C14	−46.29 (16)
C3—C4—C5—C8	73.53 (15)	C16—C10—N2—C14	77.29 (15)
N2—C10—C11—C12	51.57 (17)	C15—C10—N2—C14	−161.94 (13)
C16—C10—C11—C12	−74.01 (17)	C11—C10—N2—P2	174.92 (10)
C15—C10—C11—C12	169.19 (14)	C16—C10—N2—P2	−61.50 (14)
C10—C11—C12—C13	−58.32 (18)	C15—C10—N2—P2	59.27 (14)
C11—C12—C13—C14	58.50 (19)	C26—C23—N3—C19	−152.83 (11)
C12—C13—C14—N2	−51.92 (18)	C27—C23—N3—C19	85.68 (14)
C12—C13—C14—C18	−169.33 (14)	C22—C23—N3—C19	−36.39 (16)
C12—C13—C14—C17	73.82 (18)	C26—C23—N3—P3	49.33 (14)
N3—C19—C20—C21	−48.35 (17)	C27—C23—N3—P3	−72.15 (14)
C24—C19—C20—C21	−168.74 (14)	C22—C23—N3—P3	165.78 (10)
C25—C19—C20—C21	73.95 (18)	C24—C19—N3—C23	155.00 (12)
C19—C20—C21—C22	60.97 (18)	C25—C19—N3—C23	−85.63 (14)
C20—C21—C22—C23	−61.35 (18)	C20—C19—N3—C23	36.73 (16)
C21—C22—C23—N3	48.60 (18)	C24—C19—N3—P3	−44.51 (13)
C21—C22—C23—C26	167.17 (14)	C25—C19—N3—P3	74.86 (13)
C21—C22—C23—C27	−74.53 (18)	C20—C19—N3—P3	−162.77 (10)
N4—C28—C29—C30	−51.98 (15)	C29—C28—N4—C32	44.24 (15)
C34—C28—C29—C30	−168.76 (12)	C34—C28—N4—C32	160.71 (11)
C33—C28—C29—C30	73.82 (15)	C33—C28—N4—C32	−79.14 (14)
C28—C29—C30—C31	59.64 (16)	C29—C28—N4—P4	−176.83 (9)
C29—C30—C31—C32	−58.67 (16)	C34—C28—N4—P4	−60.37 (12)
C30—C31—C32—N4	49.73 (15)	C33—C28—N4—P4	59.79 (12)
C30—C31—C32—C35	−75.73 (15)	C35—C32—N4—C28	80.36 (14)
C30—C31—C32—C36	167.54 (12)	C36—C32—N4—C28	−159.45 (11)
C7—C1—N1—C5	−155.66 (11)	C31—C32—N4—C28	−43.01 (14)
C2—C1—N1—C5	−39.96 (15)	C35—C32—N4—P4	−58.22 (13)
C6—C1—N1—C5	83.63 (13)	C36—C32—N4—P4	61.97 (12)
C7—C1—N1—P1	47.47 (14)	C31—C32—N4—P4	178.42 (8)
C2—C1—N1—P1	163.17 (10)	C1—N1—P1—P2	4.11 (11)

C6—C1—N1—P1	−73.24 (13)	C5—N1—P1—P2	−154.19 (7)
C9—C5—N1—C1	158.52 (11)	C14—N2—P2—P1	115.47 (8)
C4—C5—N1—C1	40.35 (15)	C10—N2—P2—P1	−103.90 (9)
C8—C5—N1—C1	−82.55 (14)	C23—N3—P3—P4	12.90 (11)
C9—C5—N1—P1	−41.76 (12)	C19—N3—P3—P4	−146.66 (8)
C4—C5—N1—P1	−159.93 (9)	C28—N4—P4—P3	112.83 (8)
C8—C5—N1—P1	77.17 (12)	C32—N4—P4—P3	−106.20 (8)