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1-Methyl-3,5-diphenyl-1*H*-1,2,4-diazaphosphole

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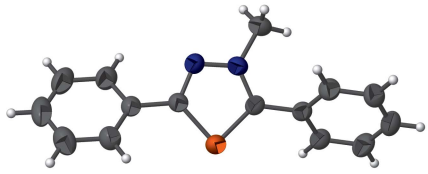
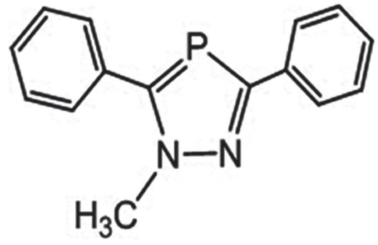
In the title *N*-substituted 1,2,4-diazaphosphole, $C_{15}H_{13}N_2P$, the phenyl rings make dihedral angles of 29.8 (3) and 55.9 (3)° with the 1,2,4-diazaphosphole ring. In the crystal, no significant intermolecular interactions are present.

3D view



Chemical scheme



Structure description

Five-membered 1,2,4-diazaphospholes are a unique type of aromatic heterocyclic compound with lone pairs of electrons on the hetero-atoms, π-electrons on the heterocyclic ring and a low-valent phosphorus ($\sigma^2\lambda^3$) atom. Recently, the investigation of 1,2,4-diazaphospholide complexes as well as symmetric and asymmetric 1,2,4-diazaphospholes have attracted considerable interest (Zheng *et al.*, 2006; Wan *et al.*, 2008; Liu *et al.*, 2014; Wang *et al.*, 2014). We have synthesized the *N*-substituted analogue, 1-methyl-3,5-diphenyl-1,2,4-diazaphosphole, $C_{15}H_{13}N_2P$, using a modified procedure of Schmidpeter & Willhalm (1984) and its crystal structure is reported herein.

In the structure of the title compound (Fig. 1), the C2- and C4-phenyl rings are inclined to the 1,2,4-diazaphosphole ring by 29.8 (3) and 55.9 (3)°, respectively. The C22—P4—C24 angle in the ring is 86.59 (11)°, comparable to those found in the other 1,2,4-diazaphospholes (Liu *et al.*, 2014; Wang *et al.*, 2014). In the crystal, no significant intermolecular interactions are present.

Synthesis and crystallization

All manipulations were carried out in an inert atmosphere of N_2 using standard Schlenk techniques in a N_2 filled glovebox. Solvents were dried over and distilled from Na/K alloy prior to use. The procedure used in the synthesis of the title compound follows that for the synthesis of other similar 1,2,4-diazaphospholes (Schmidpeter & Willhalm, 1984), by the reaction of 1,3-diphenyl-1,3-bis(dimethylamino)-2-phosphoryl chloride and pre-dried

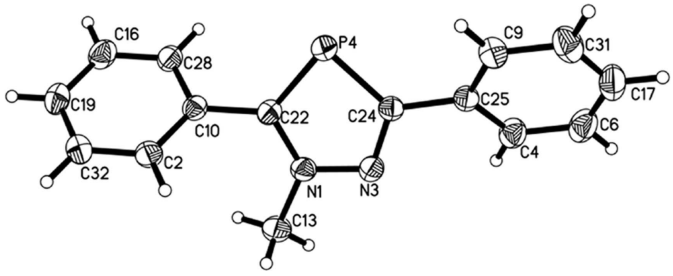


Figure 1
The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted.

methyl hydrazine. The product from the reaction was extracted with ether (2 × 10 ml) and after evaporation of the solvent over several days, gave pale-yellow crystals of the title compound [m.p. 381 K (dec.)].

^{1}H NMR (600 MHz, 298 K, $CDCl_3$): 7.95 (*d*, 2 H, Ar–H), 7.50 (*s*, 5 H, Ar–H), 7.28 (*m*, 3 H, Ar–H), 4.06 (*s*, 3 H, N–CH_3) p.p.m. ^{31}P{^{1}H} NMR (600 MHz, 298 K, $CDCl_3$): 89.22(*s*) p.p.m. Analysis calculated for $C_{15}H_{13}N_2P$: C 71.43; H 5.16; N 11.11%. Found: C 71.41; H, 5.14; N, 11.09%.

Refinement

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

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{13}N_2P$
M_r	252.24
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, *b*, *c* (Å)	10.2774 (7), 7.4256 (5), 17.4094 (11)
β (°)	92.472 (6)
V (Å^3)	1327.38 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.19
Crystal size (mm)	0.15 × 0.12 × 0.10
Data collection	
Diffractometer	Agilent SuperNova CCD
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min}, T_{max}	0.910, 0.938
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5468, 3046, 1740
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.679
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.058, 0.122, 1.01
No. of reflections	3046
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.24

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SUPERFLIP* (Palatinus & Chapuis, 2007), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160083 [doi:10.1107/S2414314616000833]

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Crystal data

$C_{15}H_{13}N_2P$
$M_r = 252.24$
Monoclinic, $P2_1/n$
$a = 10.2774$ (7) Å
$b = 7.4256$ (5) Å
$c = 17.4094$ (11) Å
$\beta = 92.472$ (6)°
$V = 1327.38$ (15) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.262$ Mg m^{-3}
Melting point < 381 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1740 reflections
$\theta = 3.0$–$28.8°$
$\mu = 0.19$ mm^{-1}
$T = 293$ K
Block, pale yellow
0.15 × 0.12 × 0.10 mm

Data collection

Agilent SuperNova CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.910$, $T_{max} = 0.938$
5468 measured reflections
3046 independent reflections
1740 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 28.8°$, $\theta_{min} = 3.0°$
$h = -13 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -22 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.122$
$S = 1.01$
3046 reflections
164 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
P4	0.31686 (7)	0.04887 (9)	0.44478 (4)	0.0519 (2)
N1	0.15290 (19)	−0.1345 (2)	0.51899 (11)	0.0437 (5)
N3	0.18769 (19)	−0.0033 (2)	0.56999 (11)	0.0456 (5)
C10	0.1779 (2)	−0.2671 (3)	0.39049 (13)	0.0410 (6)
C22	0.2097 (2)	−0.1300 (3)	0.45053 (13)	0.0420 (6)
C24	0.2733 (2)	0.1051 (3)	0.53853 (13)	0.0400 (6)
C25	0.3189 (2)	0.2621 (3)	0.58452 (14)	0.0417 (6)
C28	0.2761 (2)	−0.3685 (3)	0.35986 (13)	0.0474 (6)
H28	0.3621	−0.3510	0.3771	0.057*
C32	0.0228 (3)	−0.4223 (3)	0.30712 (14)	0.0522 (7)
H32	−0.0628	−0.4401	0.2893	0.063*
C2	0.0506 (3)	−0.2939 (3)	0.36285 (14)	0.0493 (7)
H2	−0.0164	−0.2251	0.3820	0.059*
C4	0.3262 (3)	0.2565 (3)	0.66411 (15)	0.0564 (7)
H4	0.3051	0.1507	0.6893	0.068*
C6	0.3644 (3)	0.4059 (4)	0.70655 (16)	0.0684 (8)
H6	0.3690	0.4000	0.7600	0.082*
C9	0.3524 (3)	0.4214 (3)	0.54900 (15)	0.0539 (7)
H9	0.3497	0.4278	0.4956	0.065*
C13	0.0629 (3)	−0.2704 (3)	0.54697 (15)	0.0599 (8)
H13A	0.0790	−0.2876	0.6012	0.090*
H13B	0.0758	−0.3821	0.5206	0.090*
H13C	−0.0252	−0.2304	0.5374	0.090*
C16	0.2470 (3)	−0.4960 (3)	0.30355 (16)	0.0598 (8)
H16	0.3135	−0.5632	0.2831	0.072*
C17	0.3956 (3)	0.5628 (4)	0.6702 (2)	0.0698 (9)
H17	0.4206	0.6636	0.6990	0.084*
C19	0.1204 (3)	−0.5234 (3)	0.27784 (15)	0.0576 (7)
H19	0.1011	−0.6104	0.2407	0.069*
C31	0.3900 (3)	0.5713 (4)	0.59168 (19)	0.0674 (8)
H31	0.4114	0.6776	0.5670	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P4	0.0577 (5)	0.0558 (5)	0.0429 (4)	−0.0135 (4)	0.0110 (3)	−0.0050 (3)
N1	0.0443 (13)	0.0410 (12)	0.0462 (12)	−0.0041 (10)	0.0076 (10)	0.0010 (10)
N3	0.0493 (14)	0.0443 (12)	0.0434 (13)	−0.0012 (10)	0.0057 (10)	−0.0037 (9)
C10	0.0455 (16)	0.0400 (14)	0.0377 (13)	0.0005 (12)	0.0038 (11)	0.0014 (10)
C22	0.0446 (16)	0.0422 (14)	0.0394 (14)	0.0016 (12)	0.0047 (11)	−0.0024 (11)
C24	0.0398 (15)	0.0395 (13)	0.0407 (13)	−0.0002 (11)	0.0027 (11)	0.0016 (11)
C25	0.0378 (15)	0.0439 (15)	0.0434 (14)	0.0022 (12)	0.0006 (11)	−0.0024 (12)
C28	0.0465 (17)	0.0527 (16)	0.0432 (14)	0.0024 (13)	0.0027 (12)	−0.0036 (12)
C32	0.0521 (18)	0.0525 (17)	0.0514 (16)	−0.0041 (14)	−0.0040 (13)	−0.0028 (13)
C2	0.0419 (17)	0.0507 (16)	0.0557 (16)	0.0043 (13)	0.0049 (12)	−0.0072 (13)

C4	0.062 (2)	0.0616 (18)	0.0450 (16)	−0.0014 (14)	−0.0018 (13)	−0.0023 (13)
C6	0.063 (2)	0.089 (2)	0.0525 (17)	0.0070 (18)	−0.0053 (15)	−0.0200 (18)
C9	0.0567 (18)	0.0507 (16)	0.0540 (16)	−0.0032 (14)	−0.0017 (13)	−0.0006 (14)
C13	0.067 (2)	0.0576 (17)	0.0569 (17)	−0.0163 (15)	0.0161 (14)	0.0028 (13)
C16	0.061 (2)	0.0613 (18)	0.0582 (18)	0.0120 (15)	0.0090 (14)	−0.0128 (13)
C17	0.0509 (19)	0.071 (2)	0.087 (2)	0.0057 (16)	−0.0084 (17)	−0.0338 (19)
C19	0.072 (2)	0.0525 (16)	0.0485 (16)	−0.0003 (16)	0.0015 (15)	−0.0105 (12)
C31	0.065 (2)	0.0474 (17)	0.089 (2)	−0.0029 (14)	−0.0044 (18)	−0.0060 (16)

Geometric parameters (Å, º)

P4—C22	1.731 (2)	C17—C31	1.367 (5)
P4—C24	1.761 (2)	C19—C32	1.369 (4)
N1—N3	1.356 (2)	C24—C25	1.479 (3)
N1—C13	1.466 (3)	C2—H2	0.9300
N1—C22	1.350 (3)	C4—H4	0.9300
N3—C24	1.328 (3)	C6—H6	0.9300
C2—C10	1.389 (4)	C9—H9	0.9300
C2—C32	1.381 (3)	C13—H13A	0.9600
C4—C6	1.381 (4)	C13—H13B	0.9600
C4—C25	1.385 (4)	C13—H13C	0.9600
C6—C17	1.371 (4)	C16—H16	0.9300
C9—C25	1.385 (3)	C17—H17	0.9300
C9—C31	1.384 (4)	C19—H19	0.9300
C10—C22	1.485 (3)	C28—H28	0.9300
C10—C28	1.384 (3)	C31—H31	0.9300
C16—C19	1.373 (4)	C32—H32	0.9300
C16—C28	1.386 (3)		
C22—P4—C24	86.59 (11)	C10—C2—H2	120.00
N3—N1—C13	115.43 (18)	C32—C2—H2	120.00
N3—N1—C22	116.65 (17)	C6—C4—H4	120.00
C13—N1—C22	127.83 (18)	C25—C4—H4	120.00
N1—N3—C24	109.04 (18)	C4—C6—H6	120.00
C10—C2—C32	120.4 (3)	C17—C6—H6	120.00
C6—C4—C25	120.9 (2)	C25—C9—H9	120.00
C4—C6—C17	120.2 (3)	C31—C9—H9	119.00
C25—C9—C31	121.1 (3)	N1—C13—H13A	110.00
C2—C10—C22	121.2 (2)	N1—C13—H13B	109.00
C2—C10—C28	118.7 (2)	N1—C13—H13C	109.00
C22—C10—C28	120.13 (19)	H13A—C13—H13B	110.00
C19—C16—C28	120.3 (2)	H13A—C13—H13C	109.00
C6—C17—C31	120.0 (3)	H13B—C13—H13C	109.00
C16—C19—C32	119.8 (2)	C19—C16—H16	120.00
P4—C22—N1	111.74 (16)	C28—C16—H16	120.00
P4—C22—C10	127.33 (17)	C6—C17—H17	120.00
N1—C22—C10	120.93 (19)	C31—C17—H17	120.00
P4—C24—N3	115.97 (16)	C16—C19—H19	120.00

P4—C24—C25	126.96 (17)	C32—C19—H19	120.00
N3—C24—C25	117.0 (2)	C10—C28—H28	120.00
C4—C25—C9	117.9 (2)	C16—C28—H28	120.00
C4—C25—C24	121.3 (2)	C9—C31—H31	120.00
C9—C25—C24	120.7 (2)	C17—C31—H31	120.00
C10—C28—C16	120.4 (2)	C2—C32—H32	120.00
C9—C31—C17	119.9 (3)	C19—C32—H32	120.00
C2—C32—C19	120.5 (3)		

C24—P4—C22—N1	−0.38 (17)	C4—C6—C17—C31	−0.5 (5)
C24—P4—C22—C10	179.4 (2)	C31—C9—C25—C4	−1.0 (4)
C22—P4—C24—N3	0.70 (18)	C31—C9—C25—C24	177.3 (2)
C22—P4—C24—C25	−177.4 (2)	C25—C9—C31—C17	0.6 (5)
C13—N1—N3—C24	177.24 (19)	C2—C10—C22—P4	−122.8 (2)
C22—N1—N3—C24	0.5 (3)	C2—C10—C22—N1	57.0 (3)
N3—N1—C22—P4	0.0 (2)	C28—C10—C22—P4	55.9 (3)
N3—N1—C22—C10	−179.74 (18)	C28—C10—C22—N1	−124.4 (2)
C13—N1—C22—P4	−176.26 (19)	C2—C10—C28—C16	−1.0 (3)
C13—N1—C22—C10	4.0 (3)	C22—C10—C28—C16	−179.7 (2)
N1—N3—C24—P4	−0.8 (2)	C28—C16—C19—C32	1.0 (4)
N1—N3—C24—C25	177.49 (18)	C19—C16—C28—C10	−0.3 (4)
C32—C2—C10—C22	−179.9 (2)	C6—C17—C31—C9	0.2 (5)
C32—C2—C10—C28	1.5 (3)	C16—C19—C32—C2	−0.6 (4)
C10—C2—C32—C19	−0.7 (4)	P4—C24—C25—C4	−152.0 (2)
C25—C4—C6—C17	0.1 (5)	P4—C24—C25—C9	29.8 (3)
C6—C4—C25—C9	0.7 (4)	N3—C24—C25—C4	30.0 (3)
C6—C4—C25—C24	−177.6 (2)	N3—C24—C25—C9	−148.3 (2)