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1,2,4,5-Tetramethyl-3,6-diphenyl- 1,2,4,5-tetraaza-3,6-diphosphinane

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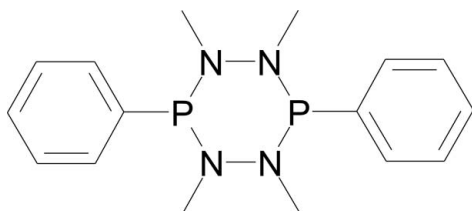
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.030; wR factor = 0.094; data-to-parameter ratio = 18.7.

The title compound, $\text{C}_{16}\text{H}_{22}\text{N}_4\text{P}_2$, crystallizes about a centre of symmetry, leading to a chair conformation of the heterocyclic ring as is commonly found for this type of compound.

Related literature

For related structures, see: Reddy *et al.* (1994, 1995).

Experimental

Crystal data

 $\text{C}_{16}\text{H}_{22}\text{N}_4\text{P}_2$ $M_r = 332.32$ Orthorhombic, $Pbca$ $a = 13.2879$ (16) Å $b = 7.5426$ (9) Å $c = 17.125$ (2) Å $V = 1716.4$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.26$ mm⁻¹ $T = 173$ (2) K $0.38 \times 0.27 \times 0.26$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: none

10040 measured reflections

1873 independent reflections

1591 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.031$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.094$ $S = 1.08$

1873 reflections

100 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2250).

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supporting information

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1,2,4,5-Tetramethyl-3,6-diphenyl-1,2,4,5-tetraaza-3,6-diphosphinane

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S1. Comment

Compound (I) has a centre of symmetry and the six-membered ring adopts a chair conformation with the phenyl groups on the phosphorous atoms being *trans* to each other. Reddy *et al.* (1994) have observed in related compounds, *e.g.* [HNP(Et)N(Me)]₂ and [HNP(Ph)N(Me)]₂, that the chair conformation was favoured over the boat conformation and was seen to readily crystallize. Bond lengths and angles in (I), with values for N—N 1.4321 (17), P—N 1.6953 (14) and 1.6984 (12), and P—C_{arom} 1.8412 (15) Å, are in the ranges observed in related structures (Reddy *et al.*, 1994, 1995).

S2. Experimental

In an attempt to crystallize bis(diphenylphosphino)dimethylhydrazine (II) (for synthesis see Reddy *et al.*, 1995), the worked-up diethylether reaction mixture was concentrated and kept at -20 °C for two days. Two small crystals were formed and on analysis of one of the crystals, (I) was identified. Analysis of the ³¹P NMR spectrum of (II) showed (I) to be present in less than 5%. Further analysis of (I) was not attempted due to the small amount of material available.

S3. Refinement

The H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with C—H = 0.93 (Ar—H) or 0.96 (CH₃) Å, and with $U_{eq} = 1.2$ (Ar—H) or 1.5 (CH₃) $U_{eq}(C)$.

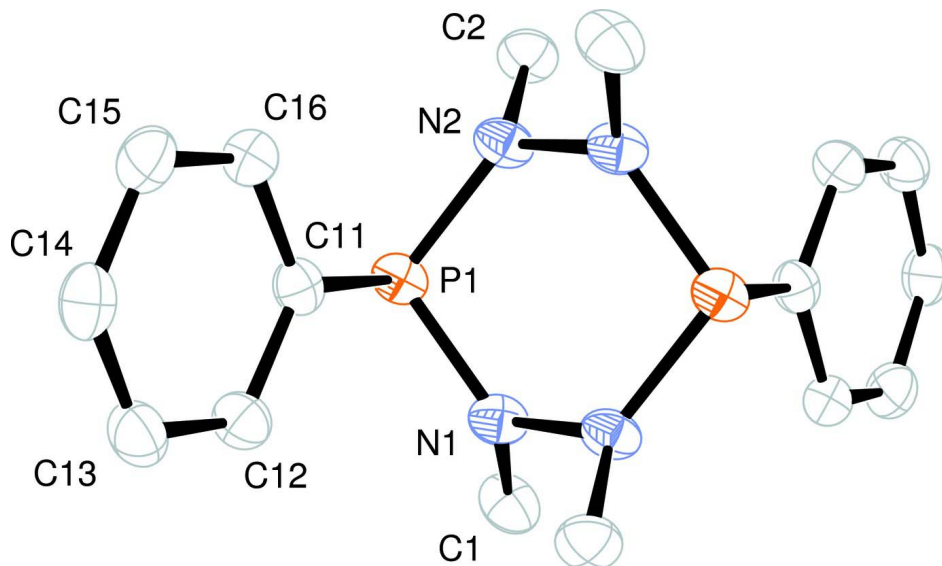


Figure 1

Molecular structure of (I) drawn with displacement ellipsoids at the 50% probability level. Hydrogen atoms have been omitted for clarity.

1,2,4,5-Tetramethyl-3,6-diphenyl-1,2,4,5-tetraaza-3,6-diphosphinane*Crystal data*C₁₆H₂₂N₄P₂ $M_r = 332.32$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 13.2879$ (16) Å $b = 7.5426$ (9) Å $c = 17.125$ (2) Å $V = 1716.4$ (4) Å³ $Z = 4$ $F(000) = 704$ $D_x = 1.286$ Mg m⁻³Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 899 reflections

 $\theta = 3.0$ – 28.2° $\mu = 0.26$ mm⁻¹ $T = 173$ K

Plates, colourless

 $0.38 \times 0.27 \times 0.26$ mm*Data collection*Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

10040 measured reflections

1873 independent reflections

1591 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.031$ $\theta_{\text{max}} = 27.0^\circ$, $\theta_{\text{min}} = 2.4^\circ$ $h = -15 \rightarrow 16$ $k = -7 \rightarrow 9$ $l = -21 \rightarrow 21$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.094$ $S = 1.08$

1873 reflections

100 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.5379P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.005$ $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.25$ 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.

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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.08579 (13)	0.3217 (2)	-0.05297 (9)	0.0359 (4)
H1A	-0.0364	0.4122	-0.0624	0.054*
H1B	-0.1442	0.3737	-0.0292	0.054*
H1C	-0.1044	0.2675	-0.1016	0.054*
C2	0.17104 (11)	-0.0731 (2)	-0.08775 (8)	0.0336 (3)
H2A	0.2138	0.0277	-0.0961	0.050*

H2B	0.1272	-0.0878	-0.1319	0.050*
H2C	0.2116	-0.1775	-0.0815	0.050*
C11	0.09375 (10)	0.14618 (18)	0.11718 (8)	0.0257 (3)
C12	0.03662 (11)	0.25669 (19)	0.16564 (8)	0.0295 (3)
H12	-0.0127	0.3288	0.1438	0.035*
C13	0.05251 (13)	0.2604 (2)	0.24603 (9)	0.0339 (3)
H13	0.0135	0.3338	0.2775	0.041*
C14	0.12622 (12)	0.1549 (2)	0.27919 (9)	0.0362 (4)
H14	0.1359	0.1554	0.3330	0.043*
C15	0.18563 (12)	0.0482 (2)	0.23183 (9)	0.0372 (4)
H15	0.2359	-0.0215	0.2539	0.045*
C16	0.17020 (11)	0.0452 (2)	0.15125 (9)	0.0307 (3)
H16	0.2112	-0.0248	0.1199	0.037*
N1	-0.04376 (10)	0.18785 (15)	-0.00091 (7)	0.0277 (3)
N2	0.11088 (9)	-0.04525 (16)	-0.01743 (7)	0.0277 (3)
P1	0.08233 (3)	0.16498 (5)	0.01035 (2)	0.02618 (14)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0509 (10)	0.0263 (8)	0.0304 (8)	0.0060 (7)	-0.0051 (7)	0.0052 (6)
C2	0.0319 (7)	0.0386 (8)	0.0302 (7)	0.0019 (6)	0.0104 (6)	-0.0022 (6)
C11	0.0285 (7)	0.0237 (7)	0.0250 (7)	-0.0039 (5)	0.0019 (5)	0.0006 (5)
C12	0.0330 (7)	0.0262 (7)	0.0292 (7)	0.0013 (6)	0.0007 (6)	0.0004 (6)
C13	0.0375 (8)	0.0345 (8)	0.0298 (8)	-0.0014 (6)	0.0017 (6)	-0.0076 (7)
C14	0.0391 (9)	0.0413 (9)	0.0283 (7)	-0.0057 (7)	-0.0076 (6)	-0.0036 (6)
C15	0.0339 (8)	0.0385 (9)	0.0392 (8)	0.0016 (7)	-0.0129 (6)	-0.0017 (7)
C16	0.0269 (7)	0.0306 (7)	0.0346 (7)	-0.0002 (6)	-0.0009 (6)	-0.0042 (6)
N1	0.0323 (6)	0.0226 (6)	0.0282 (6)	0.0037 (5)	0.0036 (5)	0.0076 (5)
N2	0.0314 (6)	0.0252 (6)	0.0265 (6)	0.0025 (5)	0.0099 (5)	0.0015 (5)
P1	0.0307 (2)	0.0230 (2)	0.0248 (2)	0.00033 (14)	0.00652 (14)	0.00337 (13)

Geometric parameters (Å, °)

C1—N1	1.4583 (18)	C12—H12	0.9300
C1—H1A	0.9600	C13—C14	1.384 (2)
C1—H1B	0.9600	C13—H13	0.9300
C1—H1C	0.9600	C14—C15	1.389 (2)
C2—N2	1.4605 (17)	C14—H14	0.9300
C2—H2A	0.9600	C15—C16	1.395 (2)
C2—H2B	0.9600	C15—H15	0.9300
C2—H2C	0.9600	C16—H16	0.9300
C11—C16	1.398 (2)	N1—N2 ⁱ	1.4321 (17)
C11—C12	1.400 (2)	N1—P1	1.6953 (14)
C11—P1	1.8412 (15)	N2—N1 ⁱ	1.4321 (17)
C12—C13	1.393 (2)	N2—P1	1.6984 (12)
N1—C1—H1A	109.5	C12—C13—H13	120.0

N1—C1—H1B	109.5	C13—C14—C15	119.73 (14)
H1A—C1—H1B	109.5	C13—C14—H14	120.1
N1—C1—H1C	109.5	C15—C14—H14	120.1
H1A—C1—H1C	109.5	C14—C15—C16	120.24 (14)
H1B—C1—H1C	109.5	C14—C15—H15	119.9
N2—C2—H2A	109.5	C16—C15—H15	119.9
N2—C2—H2B	109.5	C15—C16—C11	120.72 (14)
H2A—C2—H2B	109.5	C15—C16—H16	119.6
N2—C2—H2C	109.5	C11—C16—H16	119.6
H2A—C2—H2C	109.5	N2 ⁱ —N1—C1	114.56 (12)
H2B—C2—H2C	109.5	N2 ⁱ —N1—P1	120.94 (9)
C16—C11—C12	118.12 (13)	C1—N1—P1	121.23 (10)
C16—C11—P1	121.12 (11)	N1 ⁱ —N2—C2	114.43 (11)
C12—C11—P1	119.90 (11)	N1 ⁱ —N2—P1	120.04 (9)
C13—C12—C11	121.04 (14)	C2—N2—P1	119.18 (10)
C13—C12—H12	119.5	N1—P1—N2	106.49 (6)
C11—C12—H12	119.5	N1—P1—C11	101.67 (6)
C14—C13—C12	120.08 (14)	N2—P1—C11	100.84 (6)
C14—C13—H13	120.0		
C16—C11—C12—C13	-2.7 (2)	N2 ⁱ —N1—P1—C11	-66.99 (11)
P1—C11—C12—C13	-172.18 (11)	C1—N1—P1—C11	134.55 (11)
C11—C12—C13—C14	0.5 (2)	N1 ⁱ —N2—P1—N1	-37.75 (13)
C12—C13—C14—C15	1.3 (2)	C2—N2—P1—N1	112.73 (11)
C13—C14—C15—C16	-1.0 (2)	N1 ⁱ —N2—P1—C11	68.00 (11)
C14—C15—C16—C11	-1.3 (2)	C2—N2—P1—C11	-141.52 (11)
C12—C11—C16—C15	3.1 (2)	C16—C11—P1—N1	148.76 (12)
P1—C11—C16—C15	172.42 (12)	C12—C11—P1—N1	-42.10 (12)
N2 ⁱ —N1—P1—N2	38.17 (13)	C16—C11—P1—N2	39.20 (13)
C1—N1—P1—N2	-120.30 (12)	C12—C11—P1—N2	-151.65 (11)

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