organic compounds

trans-1,4-Diphenyl-1,4-diphosphacyclohexane

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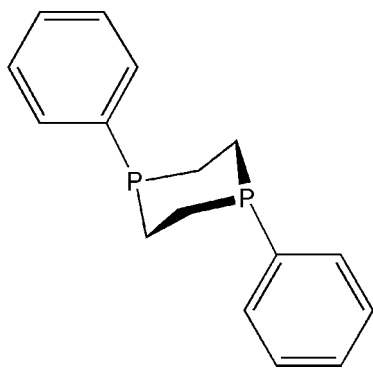Correspondence e-mail: p.healy@griffith.edu.au

The molecule of the title compound, $C_{16}H_{18}P_2$, which was prepared by the reaction of dichloroethane with lithiated bis(diphenylphosphino)ethane, is disposed about a centre of symmetry. The 1,4-diphosphacyclohexane ring adopts a chair conformation with axial phenyl groups. The planes of the phenyl rings are orthogonal to the P···P axis.

Related literature

For related literature, see: Brooks *et al.* (1989).

Experimental

Crystal data

$C_{16}H_{18}P_2$	$V = 1420.0\ (11)$ Å^3
$M_r = 272.24$	$Z = 4$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 16.585\ (6)$ Å	$\mu = 0.29$ mm^{-1}
$b = 11.584\ (6)$ Å	$T = 295$ K
$c = 7.391\ (3)$ Å	$0.40 \times 0.30 \times 0.30$ mm

Data collection

Rigaku AFC-7R diffractometer	$R_{int} = 0.029$
Absorption correction: none	3 standard reflections
1879 measured reflections	every 150 reflections
1627 independent reflections	intensity decay: 0.9%
1139 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	83 parameters
$wR(F^2) = 0.132$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{max} = 0.47$ e Å^{-3}
1627 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}

Data collection: *MSC/AFC7 Diffractometer Control Software for Windows* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC7 Diffractometer Control Software for Windows*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *TEXSAN for Windows*; program(s) used to refine structure: *TEXSAN for Windows* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows* and *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2331).

References

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supplementary materials

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Comment

The crystal structure of (I) has been previously reported as a preliminary communication without publication of the atom coordinates (Brooks *et al.*, 1989). The molecules of (I) are disposed about a crystallographic centre of symmetry. The central 1,4-diphospha-cyclohexane ring adopts a chair conformation with the phenyl groups diaxial. The planes of the phenyl rings are approximately orthogonal to the P···P axis of the 1,4-diphospha-cyclohexane ring. The P—C bond lengths are similar with P—C(aliphatic) = 1.841 (3), 1.843 (2)Å and P—C(aromatic) 1.834 (2) Å. The endocyclic C—P—C angle is 100.3 (1)° compared to the exocyclic C—P—C angles of 102.70 (9) and 102.72 (10)°.

Experimental

Bis(diphenylphosphino)ethane (dppe) (5.8 g, 0.015 mol) in dry THF (48 ml) was added dropwise over 4 hrs at 273 K to lithium (1.38 g, 0.20 mol) in dry THF (30 ml). The mixture was refluxed for 2 h. 5 ml of the lithiated dppe was then added to 1,2-dichloroethane (95 ml) at 273 K over 1 hr. The solution was stirred for 2 days, resulting in precipitation of solid material. The solvent was removed under vacuum to give a residual oil. Ethanol (50 ml) was added to this oil and the mixture placed in the freezer overnight to yield transparent well formed crystals of (I). yield 250 mg. m.p 438–443 K. Lit. (Brooks *et al.*, 1989) 441–443 K).

Refinement

Hydrogen atoms constrained in the riding model approximation, fixed to their parent C atoms with C—H set to 0.95 Å. U_{iso} values for the H atoms were set at $1.2U_{\mathrm{eq}}$ of the parent atom.
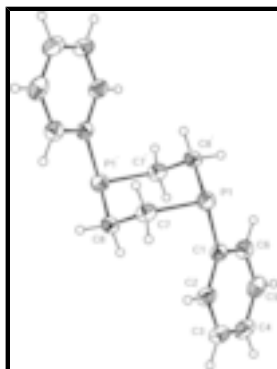
Figures

Fig. 1. View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. Symmetry code for symmetry related atoms ('): (−*x*, −*y*, 1 − *z*).

supplementary materials

trans-1,4-Diphenyl-1,4-diphosphacyclohexane

Crystal data

$C_{16}H_{18}P_2$	$F_{000} = 576$
$M_r = 272.24$	$D_x = 1.273$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 25 reflections
$a = 16.585$ (6) Å	$\theta = 12.5–16.6º$
$b = 11.584$ (6) Å	$\mu = 0.29$ mm^{-1}
$c = 7.391$ (3) Å	$T = 295$ K
$V = 1420.0$ (11) Å^3	Prism, colourless
$Z = 4$	$0.40 \times 0.30 \times 0.30$ mm

Data collection

Rigaku AFC-7R diffractometer	$R_{int} = 0.029$
Radiation source: Rigaku rotating anode	$\theta_{max} = 27.5º$
Monochromator: graphite	$\theta_{min} = 3.0º$
$T = 295$ K	$h = 0 \rightarrow 21$
ω–2/q scans	$k = -6 \rightarrow 15$
Absorption correction: none	$l = -9 \rightarrow 3$
1879 measured reflections	3 standard reflections
1627 independent reflections	every 150 reflections
1139 reflections with $I > 2\sigma(I)$	intensity decay: 0.9%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0715P)^2 + 0.24P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.47$ e Å^{-3}
1627 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}
83 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $FC^* = KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.007 (2)
Secondary atom site location: difference Fourier map	

Special details

Experimental. The scan width was (1.68 + 0.30tanθ)° with an ω scan speed of 16° per minute (up to 4 scans to achieve I/σ(I) > 10). Stationary background counts were recorded at each end of the scan, and the scan time:background time ratio was 2:1.

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All e.s.d.'s are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2sigma(F^2)$ 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	0.01337 (3)	−0.07964 (5)	0.29128 (8)	0.0474 (2)
C1	0.12165 (13)	−0.10065 (18)	0.3298 (3)	0.0423 (6)
C2	0.17770 (15)	−0.0227 (2)	0.2600 (3)	0.0530 (7)
C3	0.25994 (15)	−0.0433 (3)	0.2746 (4)	0.0622 (8)
C4	0.28740 (13)	−0.1423 (3)	0.3555 (3)	0.0603 (8)
C5	0.23294 (15)	−0.2210 (2)	0.4236 (3)	0.0586 (8)
C6	0.15063 (13)	−0.20060 (19)	0.4110 (3)	0.0499 (7)
C7	0.00128 (12)	0.0773 (2)	0.3197 (3)	0.0481 (7)
C8	0.03159 (13)	0.13119 (18)	0.4954 (3)	0.0491 (7)
H2	0.15770	0.04710	0.20000	0.0620*
H3	0.29830	0.01270	0.22970	0.0730*
H4	0.34470	−0.15830	0.36490	0.0710*
H5	0.25200	−0.29160	0.47890	0.0660*
H6	0.11180	−0.25750	0.45820	0.0550*
H71	−0.05410	0.09350	0.30670	0.0540*
H72	0.03030	0.11240	0.22130	0.0540*
H81	0.08900	0.11660	0.50640	0.0460*
H82	0.02070	0.21320	0.49270	0.0550*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0446 (3)	0.0532 (4)	0.0444 (4)	−0.0003 (2)	−0.0041 (2)	−0.0051 (3)
C1	0.0439 (10)	0.0463 (11)	0.0366 (10)	0.0020 (8)	0.0029 (8)	−0.0028 (8)
C2	0.0527 (13)	0.0528 (12)	0.0536 (13)	0.0047 (10)	0.0136 (10)	0.0066 (10)
C3	0.0489 (13)	0.0720 (16)	0.0658 (15)	−0.0036 (12)	0.0183 (12)	−0.0055 (13)
C4	0.0424 (11)	0.0850 (18)	0.0534 (13)	0.0088 (12)	0.0021 (10)	−0.0107 (13)
C5	0.0620 (14)	0.0655 (15)	0.0484 (12)	0.0194 (11)	−0.0010 (11)	0.0000 (11)
C6	0.0536 (12)	0.0507 (12)	0.0454 (11)	0.0038 (10)	0.0024 (9)	0.0023 (9)
C7	0.0455 (11)	0.0506 (12)	0.0482 (11)	0.0066 (9)	0.0042 (9)	0.0144 (9)
C8	0.0439 (11)	0.0389 (11)	0.0644 (14)	−0.0044 (9)	0.0070 (10)	0.0034 (10)

Geometric parameters (Å, °)

P1—C1	1.834 (2)	C2—H2	0.9800
P1—C7	1.841 (3)	C3—H3	0.9673
P1—C8[i]	1.843 (2)	C4—H4	0.9707
C1—C2	1.395 (3)	C5—H5	0.9674
C1—C6	1.390 (3)	C6—H6	0.9853
C2—C3	1.389 (4)	C7—H71	0.9424
C3—C4	1.371 (5)	C7—H72	0.9622
C4—C5	1.379 (4)	C8—H81	0.9704
C5—C6	1.389 (3)	C8—H82	0.9672
C7—C8	1.526 (3)		
P1···P1[i]	3.622 (2)	C8···H6[i]	2.8132
P1···H4[ii]	3.1604	H2···C7	2.7632
C2···C8	3.475 (4)	H2···H72	2.2498
C2···C4[iii]	3.595 (4)	H2···C4[iii]	2.9203
C4···C2[iv]	3.595 (4)	H4···P1[vi]	3.1604
C6···C7[i]	3.514 (4)	H5···C3[vii]	2.9072
C7···C6[i]	3.514 (4)	H5···C4[vii]	2.9459
C8···C2	3.475 (4)	H6···C8[i]	2.8132
C1···H71[i]	2.9120	H6···H82[i]	2.2856
C1···H81	2.8862	H71···C1[i]	2.9120
C2···H81	2.8433	H71···C6[i]	2.9079
C2···H72	2.9167	H72···C2	2.9167
C3···H5[v]	2.9072	H72···H2	2.2498
C4···H5[v]	2.9459	H81···C1	2.8862
C4···H2[iv]	2.9203	H81···C2	2.8433
C6···H71[i]	2.9079	H82···C6[i]	2.9329
C6···H82[i]	2.9329	H82···H6[i]	2.2856
C7···H2	2.7632		
C1—P1—C7	102.70 (9)	C3—C4—H4	121.07
C1—P1—C8[i]	102.72 (10)	C5—C4—H4	119.28
C7—P1—C8[i]	100.27 (10)	C4—C5—H5	119.95
P1—C1—C2	120.60 (17)	C6—C5—H5	119.57
P1—C1—C6	121.07 (16)	C1—C6—H6	118.95
C2—C1—C6	117.9 (2)	C5—C6—H6	120.33
C1—C2—C3	121.0 (2)	P1—C7—H71	106.93
C2—C3—C4	120.2 (3)	P1—C7—H72	106.05
C3—C4—C5	119.7 (2)	C8—C7—H71	109.05
C4—C5—C6	120.5 (2)	C8—C7—H72	107.79
C1—C6—C5	120.7 (2)	H71—C7—H72	109.04
P1—C7—C8	117.72 (15)	C7—C8—H81	108.86
P1[i]—C8—C7	117.52 (15)	C7—C8—H82	108.83
C1—C2—H2	118.42	H81—C8—H82	110.85

C3—C2—H2	120.62	P1^i—C8—H81	105.59
C2—C3—H3	120.27	P1^i—C8—H82	105.09
C4—C3—H3	119.48		
C7—P1—C1—C2	−40.7 (2)	P1—C1—C6—C5	173.61 (17)
C7—P1—C1—C6	146.59 (19)	C2—C1—C6—C5	0.7 (3)
C8^i—P1—C1—C2	−144.49 (18)	C6—C1—C2—C3	−1.3 (3)
C8^i—P1—C1—C6	42.8 (2)	C1—C2—C3—C4	1.2 (4)
C1—P1—C7—C8	−54.41 (18)	C2—C3—C4—C5	−0.5 (4)
C8^i—P1—C7—C8	51.27 (17)	C3—C4—C5—C6	−0.1 (4)
C1—P1—C8^i—C7^i	54.53 (18)	C4—C5—C6—C1	0.0 (3)
C7—P1—C8^i—C7^i	−51.14 (18)	P1—C7—C8—P1^i	−59.9 (2)
P1—C1—C2—C3	−174.2 (2)		

Symmetry codes: (i) −x, −y, −z+1; (ii) x−1/2, y, −z+1/2; (iii) −x+1/2, −y, z−1/2; (iv) −x+1/2, −y, z+1/2; (v) x, −y−1/2, z−1/2; (vi) x+1/2, y, −z+1/2; (vii) x, −y−1/2, z+1/2.

Fig. 1