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1,1,4,4-Tetrabenzyl-1,4-diphosphinane-1,4-dium dibromide deuteriochloroform disolvate

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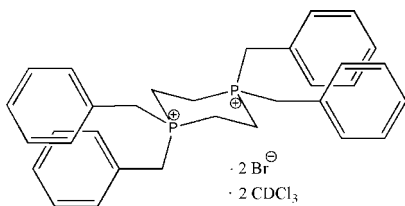
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Key indicators: single-crystal X-ray study; $T = 133$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.032; wR factor = 0.083; data-to-parameter ratio = 26.9.

In the title compound, $\text{C}_{32}\text{H}_{36}\text{P}_2^{2+} \cdot 2\text{Br}^- \cdot 2\text{CDCl}_3$, the diphosphonium ring of the centrosymmetric dication adopts a chair conformation. A network of weak $\text{C}-\text{H} \cdots \text{Cl}$ and $\text{C}-\text{H} \cdots \text{Br}$ interactions helps to establish the packing. Both bromide ions lie on crystallographic twofold rotation axes.

Related literature

For related literature, see: Brown & Trefonas (1972); Duraczynska & Nelson (2005); Hinton & Mann (1959); Matt *et al.* (1996).



Experimental

Crystal data

$\text{C}_{32}\text{H}_{36}\text{P}_2^{2+} \cdot 2\text{Br}^- \cdot 2\text{CDCl}_3$
 $M_r = 883.08$
 Monoclinic, $C2/c$
 $a = 16.2692$ (10) Å
 $b = 17.5649$ (10) Å
 $c = 13.9110$ (8) Å
 $\beta = 112.350$ (3)°

$V = 3676.7$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.75$ mm⁻¹
 $T = 133$ (2) K
 $0.38 \times 0.30 \times 0.29$ mm

Data collection

Bruker SMART1000 CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
 $T_{\min} = 0.360$, $T_{\max} = 0.502$
 (expected range = 0.323–0.450)

37985 measured reflections
 5373 independent reflections
 4684 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.083$
 $S = 1.05$
 5373 reflections

200 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.52$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.05$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{C2}-\text{H2A} \cdots \text{Cl3}^{\text{ii}}$	0.99	2.93	3.8486 (19)	155
$\text{C14}-\text{H14} \cdots \text{Cl1}^{\text{iii}}$	0.95	2.87	3.686 (2)	145
$\text{C13}-\text{H13} \cdots \text{Br1}^{\text{iv}}$	0.95	2.99	3.773 (2)	141
$\text{C2}-\text{H2B} \cdots \text{Br1}^{\text{v}}$	0.99	2.79	3.7553 (18)	166
$\text{C4}-\text{H4B} \cdots \text{Br1}^{\text{v}}$	0.99	2.75	3.6757 (18)	157
$\text{C1}-\text{H1A} \cdots \text{Br1}^{\text{vi}}$	0.99	2.86	3.7443 (19)	149

Symmetry codes: (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (iv) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (v) $-x, -y, -z + 1$; (vi) $x, y, z - 1$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2582).

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supplementary materials

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1,1,4,4-Tetrabenzyl-1,4-diphosphinane-1,4-dium dibromide deuteriochloroform disolvate

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Comment

Similar to the homologue structures known in the literature (Brown & Trefonas, 1972; Matt *et al.*, 1996; Duraczynska & Nelson, 2005) the diphosphonium ring, in the title compound, (I), which sits on a crystallographic center of symmetry, is in a chair conformation with the torsion angles of $52.58(17)^\circ$ ($C4^i-P-C3-C4$) and $-56.00(16)^\circ$ ($P-C3-C4-P^i$) ($i: -x, -y, -z$). Each phosphorus atom has two benzyl groups attached, one of which is equatorial and the other axial. The mean P—C bond distance in (I) of $1.80(18)\text{Å}$ is in accordance with the reported data for other cyclic diphosphonium salts. The P atom in (I) displays a slightly distorted tetrahedral geometry with C—P—C angles ranging from $106.61(8)^\circ$ to $113.65(8)^\circ$. The C2—P1—C1 angle is intermediate between that of phenyl (Brown & Trefonas, 1972; Matt *et al.*, 1996) and cyclohexyl substituted derivatives (Duraczynska & Nelson, 2005). The phenyl rings are tilted to each other with an angle of $47.42(10)^\circ$.

Unlike the phenyl analog (Matt *et al.*, 1996), the packing for (I) is consolidated by weak C—H \cdots Cl and C—H \cdots Br interactions (Table 1), linking the dication, the anions, and the deuterated solvent molecule.

Experimental

The title compound was been prepared from the corresponding diphosphine, $(\text{PhCH}_2)_2\text{PCH}_2\text{CH}_2\text{P}(\text{CH}_2\text{Ph})_2$, and 1,2-dibromoethane according to the procedure given by Hinton & Mann (1959). Colourless blocks of (I) were obtained from deuterated chloroform by slow evaporation at room temperature.

Figures

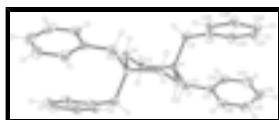


Fig. 1. The molecular structure of the dication in (I), with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level. The unlabelled atoms are generated by the symmetry operation $(-x, -y, -z)$.

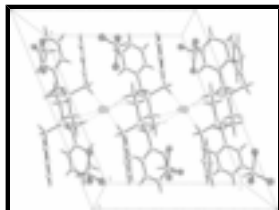


Fig. 2. A packing diagram for (I), viewed along the a axis with the H bonds indicated by dashed lines.

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Crystal data

$\text{C}_{32}\text{H}_{36}\text{P}_2^{2+}\cdot 2\text{Br}^- \cdot 2\text{CDCl}_3$

$M_r = 883.08$

$F_{000} = 1776$

$D_x = 1.595 \text{ Mg m}^{-3}$

supplementary materials

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 16.2692$ (10) Å

$b = 17.5649$ (10) Å

$c = 13.9110$ (8) Å

$\beta = 112.350$ (3)°

$V = 3676.7$ (4) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 8675 reflections

$\theta = 2.3$ – 30.4 °

$\mu = 2.75$ mm⁻¹

$T = 133$ (2) K

Block, colourless

$0.38 \times 0.30 \times 0.29$ mm

Data collection

Bruker SMART1000 CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 8.192 pixels mm⁻¹

$T = 133$ (2) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 1999)

$T_{\min} = 0.360$, $T_{\max} = 0.502$

37985 measured reflections

5373 independent reflections

4684 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 30.0$ °

$\theta_{\text{min}} = 1.8$ °

$h = -22 \rightarrow 22$

$k = -24 \rightarrow 24$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.083$

$S = 1.05$

5373 reflections

200 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 6.1465P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 1.52$ e Å⁻³

$\Delta\rho_{\text{min}} = -1.05$ e Å⁻³

Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ is used only for calculat-

ing 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.0000	0.121056 (14)	0.7500	0.02374 (7)
Br2	0.0000	0.209800 (15)	0.2500	0.03136 (8)
P1	0.08262 (3)	0.06365 (2)	0.06148 (3)	0.01914 (9)
C1	0.12293 (12)	0.15385 (10)	0.03500 (15)	0.0246 (3)
H1A	0.0881	0.1678	-0.0382	0.030*
H1B	0.1117	0.1932	0.0795	0.030*
C11	0.22023 (12)	0.15556 (10)	0.05260 (14)	0.0238 (3)
C12	0.27929 (14)	0.19798 (11)	0.13390 (16)	0.0291 (4)
H12	0.2580	0.2269	0.1773	0.035*
C13	0.36960 (15)	0.19821 (13)	0.15203 (19)	0.0380 (5)
H13	0.4098	0.2274	0.2076	0.046*
C14	0.40056 (16)	0.15622 (14)	0.0894 (2)	0.0421 (5)
H14	0.4624	0.1551	0.1032	0.050*
C15	0.34194 (17)	0.11551 (13)	0.0062 (2)	0.0401 (5)
H15	0.3633	0.0875	-0.0379	0.048*
C16	0.25188 (15)	0.11585 (12)	-0.01245 (17)	0.0316 (4)
H16	0.2116	0.0887	-0.0702	0.038*
C2	0.13706 (12)	0.03240 (10)	0.19539 (14)	0.0223 (3)
H2A	0.1500	0.0777	0.2411	0.027*
H2B	0.0952	-0.0001	0.2132	0.027*
C21	0.22303 (11)	-0.01158 (10)	0.21883 (13)	0.0217 (3)
C22	0.30491 (13)	0.02553 (12)	0.25009 (15)	0.0279 (4)
H22	0.3076	0.0793	0.2580	0.033*
C23	0.38249 (13)	-0.01563 (13)	0.26965 (17)	0.0331 (4)
H23	0.4379	0.0101	0.2912	0.040*
C24	0.37956 (13)	-0.09415 (13)	0.25799 (16)	0.0323 (4)
H24	0.4327	-0.1220	0.2704	0.039*
C25	0.29897 (14)	-0.13164 (12)	0.22824 (16)	0.0293 (4)
H25	0.2968	-0.1854	0.2204	0.035*
C26	0.22100 (12)	-0.09085 (11)	0.20968 (14)	0.0247 (3)
H26	0.1660	-0.1171	0.1907	0.030*
C3	-0.03415 (11)	0.07968 (10)	0.03025 (14)	0.0222 (3)
H3A	-0.0403	0.1155	0.0821	0.027*
H3B	-0.0593	0.1050	-0.0384	0.027*
C4	-0.09037 (11)	0.00863 (10)	0.02692 (14)	0.0214 (3)
H4A	-0.1533	0.0240	0.0059	0.026*
H4B	-0.0708	-0.0133	0.0977	0.026*
C11	-0.10604 (4)	0.26705 (3)	-0.07399 (5)	0.03723 (12)
C12	-0.09062 (6)	0.41535 (4)	0.01860 (5)	0.05499 (18)
C13	-0.26168 (4)	0.33812 (4)	-0.06513 (5)	0.04406 (15)
C99	-0.14538 (14)	0.32753 (12)	0.00011 (16)	0.0311 (4)
H99	-0.1329	0.3035	0.0694	0.037*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02504 (12)	0.02263 (12)	0.02072 (12)	0.000	0.00553 (9)	0.000
Br2	0.04348 (16)	0.02395 (13)	0.02526 (14)	0.000	0.01151 (11)	0.000
P1	0.01808 (19)	0.01810 (19)	0.0191 (2)	-0.00022 (15)	0.00459 (15)	-0.00208 (14)
C1	0.0265 (8)	0.0204 (8)	0.0244 (9)	-0.0010 (6)	0.0069 (7)	0.0010 (6)
C11	0.0285 (8)	0.0206 (8)	0.0227 (8)	-0.0028 (6)	0.0101 (7)	0.0015 (6)
C12	0.0354 (10)	0.0241 (8)	0.0280 (10)	-0.0065 (7)	0.0121 (8)	-0.0021 (7)
C13	0.0337 (10)	0.0371 (11)	0.0396 (12)	-0.0147 (9)	0.0099 (9)	0.0022 (9)
C14	0.0327 (11)	0.0446 (13)	0.0539 (15)	-0.0049 (9)	0.0220 (10)	0.0125 (11)
C15	0.0479 (13)	0.0375 (11)	0.0479 (14)	-0.0006 (10)	0.0329 (11)	0.0038 (10)
C16	0.0393 (11)	0.0315 (10)	0.0281 (10)	-0.0047 (8)	0.0174 (8)	-0.0017 (7)
C2	0.0225 (8)	0.0241 (8)	0.0193 (8)	0.0018 (6)	0.0069 (6)	-0.0002 (6)
C21	0.0218 (8)	0.0252 (8)	0.0176 (8)	0.0008 (6)	0.0067 (6)	0.0024 (6)
C22	0.0262 (9)	0.0288 (9)	0.0260 (9)	-0.0028 (7)	0.0069 (7)	0.0036 (7)
C23	0.0228 (9)	0.0431 (11)	0.0335 (11)	-0.0030 (8)	0.0107 (8)	0.0043 (9)
C24	0.0273 (9)	0.0427 (11)	0.0294 (10)	0.0093 (8)	0.0138 (8)	0.0051 (8)
C25	0.0331 (10)	0.0286 (9)	0.0261 (9)	0.0054 (8)	0.0113 (7)	0.0024 (7)
C26	0.0238 (8)	0.0261 (8)	0.0225 (8)	-0.0006 (7)	0.0067 (6)	0.0009 (6)
C3	0.0202 (7)	0.0193 (8)	0.0243 (8)	0.0026 (6)	0.0053 (6)	-0.0025 (6)
C4	0.0213 (8)	0.0214 (8)	0.0208 (8)	0.0012 (6)	0.0070 (6)	-0.0020 (6)
Cl1	0.0334 (2)	0.0369 (3)	0.0452 (3)	0.0021 (2)	0.0192 (2)	0.0015 (2)
Cl2	0.0842 (5)	0.0376 (3)	0.0367 (3)	-0.0212 (3)	0.0157 (3)	-0.0044 (2)
Cl3	0.0367 (3)	0.0499 (3)	0.0516 (3)	0.0161 (2)	0.0235 (2)	0.0209 (3)
C99	0.0363 (10)	0.0303 (10)	0.0264 (10)	-0.0011 (8)	0.0115 (8)	0.0040 (7)

Geometric parameters (\AA , $^\circ$)

P1—C3	1.8027 (18)	Cl2—C99	1.751 (2)
P1—C4 ⁱ	1.8050 (18)	Cl3—C99	1.770 (2)
P1—C1	1.8053 (18)	C1—H1A	0.9900
P1—C2	1.8170 (18)	C1—H1B	0.9900
C1—C11	1.507 (3)	C12—H12	0.9500
C11—C16	1.387 (3)	C13—H13	0.9500
C11—C12	1.389 (3)	C14—H14	0.9500
C12—C13	1.393 (3)	C15—H15	0.9500
C13—C14	1.375 (4)	C16—H16	0.9500
C14—C15	1.386 (4)	C2—H2A	0.9900
C15—C16	1.387 (3)	C2—H2B	0.9900
C2—C21	1.521 (2)	C22—H22	0.9500
C21—C22	1.396 (3)	C23—H23	0.9500
C21—C26	1.397 (3)	C24—H24	0.9500
C22—C23	1.389 (3)	C25—H25	0.9500
C23—C24	1.387 (3)	C26—H26	0.9500
C24—C25	1.383 (3)	C3—H3A	0.9900
C25—C26	1.393 (3)	C3—H3B	0.9900

C3—C4	1.538 (2)	C4—H4A	0.9900
C4—P1 ⁱ	1.8049 (18)	C4—H4B	0.9900
C11—C99	1.761 (2)	C99—H99	1.0000
C3—P1—C4 ⁱ	106.61 (8)	C14—C13—H13	120.0
C3—P1—C1	104.19 (8)	C12—C13—H13	120.0
C4 ⁱ —P1—C1	110.70 (9)	C13—C14—H14	119.9
C3—P1—C2	110.63 (9)	C15—C14—H14	119.9
C4 ⁱ —P1—C2	110.66 (8)	C14—C15—H15	120.1
C1—P1—C2	113.65 (8)	C16—C15—H15	120.1
C11—C1—P1	115.15 (13)	C11—C16—H16	119.7
C16—C11—C12	119.16 (19)	C15—C16—H16	119.7
C16—C11—C1	120.75 (17)	C21—C2—H2A	108.5
C12—C11—C1	120.09 (17)	P1—C2—H2A	108.5
C11—C12—C13	120.3 (2)	C21—C2—H2B	108.5
C14—C13—C12	119.9 (2)	P1—C2—H2B	108.5
C13—C14—C15	120.3 (2)	H2A—C2—H2B	107.5
C14—C15—C16	119.8 (2)	C23—C22—H22	119.8
C11—C16—C15	120.5 (2)	C21—C22—H22	119.8
C21—C2—P1	115.04 (12)	C24—C23—H23	119.8
C22—C21—C26	118.72 (17)	C22—C23—H23	119.8
C22—C21—C2	121.37 (17)	C25—C24—H24	120.2
C26—C21—C2	119.91 (16)	C23—C24—H24	120.2
C23—C22—C21	120.44 (19)	C24—C25—H25	119.9
C24—C23—C22	120.42 (19)	C26—C25—H25	119.9
C25—C24—C23	119.67 (18)	C25—C26—H26	119.8
C24—C25—C26	120.25 (19)	C21—C26—H26	119.8
C25—C26—C21	120.48 (17)	C4—C3—H3A	108.2
C4—C3—P1	116.16 (12)	P1—C3—H3A	108.2
C3—C4—P1 ⁱ	113.36 (12)	C4—C3—H3B	108.2
C12—C99—C11	109.85 (12)	P1—C3—H3B	108.2
C12—C99—C13	111.58 (12)	H3A—C3—H3B	107.4
C11—C99—C13	108.59 (12)	C3—C4—H4A	108.9
C11—C1—H1A	108.5	P1 ⁱ —C4—H4A	108.9
P1—C1—H1A	108.5	C3—C4—H4B	108.9
C11—C1—H1B	108.5	P1 ⁱ —C4—H4B	108.9
P1—C1—H1B	108.5	H4A—C4—H4B	107.7
H1A—C1—H1B	107.5	C12—C99—H99	108.9
C11—C12—H12	119.9	C11—C99—H99	108.9
C13—C12—H12	119.9	C13—C99—H99	108.9
C3—P1—C1—C11	-178.60 (14)	C1—P1—C2—C21	-86.12 (15)
C4 ⁱ —P1—C1—C11	-64.34 (16)	P1—C2—C21—C22	89.50 (19)
C2—P1—C1—C11	60.91 (16)	P1—C2—C21—C26	-90.81 (18)
P1—C1—C11—C16	69.5 (2)	C26—C21—C22—C23	1.2 (3)
P1—C1—C11—C12	-111.24 (18)	C2—C21—C22—C23	-179.06 (18)
C16—C11—C12—C13	-2.4 (3)	C21—C22—C23—C24	0.3 (3)
C1—C11—C12—C13	178.39 (18)	C22—C23—C24—C25	-1.0 (3)
C11—C12—C13—C14	-0.1 (3)	C23—C24—C25—C26	0.2 (3)

supplementary materials

C12—C13—C14—C15	2.1 (3)	C24—C25—C26—C21	1.3 (3)
C13—C14—C15—C16	-1.5 (4)	C22—C21—C26—C25	-2.0 (3)
C12—C11—C16—C15	3.0 (3)	C2—C21—C26—C25	178.29 (17)
C1—C11—C16—C15	-177.79 (19)	C4 ⁱ —P1—C3—C4	52.58 (17)
C14—C15—C16—C11	-1.1 (3)	C1—P1—C3—C4	169.70 (13)
C3—P1—C2—C21	157.08 (13)	C2—P1—C3—C4	-67.81 (15)
C4 ⁱ —P1—C2—C21	39.15 (16)	P1—C3—C4—P1 ⁱ	-56.00 (16)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2A \cdots Cl3 ⁱⁱ	0.99	2.93	3.8486 (19)	155
C14—H14 \cdots Cl1 ⁱⁱⁱ	0.95	2.87	3.686 (2)	145
C13—H13 \cdots Br1 ^{iv}	0.95	2.99	3.773 (2)	141
C2—H2B \cdots Br1 ^v	0.99	2.79	3.7553 (18)	166
C4—H4B \cdots Br1 ^v	0.99	2.75	3.6757 (18)	157
C1—H1A \cdots Br1 ^{vi}	0.99	2.86	3.7443 (19)	149

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

Fig. 1

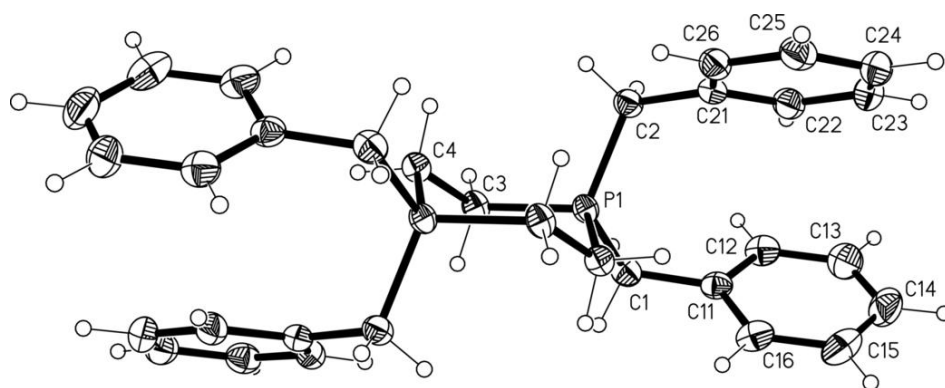


Fig. 2

