Structure of 1,4-Bis(diphenylphosphinoyl)butane

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Abstract. $C_{28}H_{28}O_2P_2$, $M_r = 458.48$, triclinic, $P\bar{1}$, $a = 5.826$ (1), $b = 8.862$ (1), $c = 12.517$ (2) Å, $\alpha = 100.29$ (1), $\beta = 102.67$ (1), $\gamma = 104.22$ (1)°, $V = 592.5$ (3) Å^3, $Z = 1$, $D_x = 1.285$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.00$ cm^{-1}, $F(000) = 242$, $T = 296$ K, final $R = 0.031$ for 1390 independent observed reflections. The —(CH$_2$)$_4$— group is essentially planar with the P atoms 0.126 (1) Å away from its calculated mean plane. Both phenyl rings are planar to within experimental accuracy. The P atom has a distorted tetrahedral configuration.

Experimental. During our studies, using {RuCl$_2$[1,4-bis(diphenylphosphino)]butane} as a starting material for reactions with bulky ligands like triethylphosphite, the title compound was obtained. Single colorless crystals were obtained from CH$_2$Cl$_2$/ether by slow evaporation at 293 K. The data collection and refinement parameters are summarized in Table 1. The structure was solved using standard direct methods and difference Fourier techniques. In final cycles of least-squares refinement, all non-H atoms were treated anisotropically, H atoms were refined isotropically. Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970); for H atoms from Stewart, Davidson & Simpson (1965). Programs used: *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

Atomic coordinates for non-H atoms are given in Table 2,* interatomic distances and angles are listed in Table 3. Fig. 1 is a projection of the title compound showing the atom-numbering scheme.

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* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54361 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Crystallographic summary*

Data collection[i]	
Mode	ω–2θ
Scan rate (° min^{-1})	1.8, 5.5
θ range (°)	0–23
Range of *hkl*	$-6 \le h \le 6$, $-9 \le k \le 9$, $0 \le l \le 13$
Total reflections measured	1848
Unique reflections	1647
R_{int}	0.01
Standard reflections (h^{-1})	1
Variation	None significant
Crystal dimensions approx. (mm)	$0.20 \times 0.20 \times 0.20$
Diffractometer	Enraf–Nonius CAD-4, graphite monochromator
Structure determination and refinement[ii,iii]	
Reflections used [$I > 3\sigma(I)$]	1390
No. of variables	202
R, wR	0.031, 0.030
w	$1/[\sigma^2(F_o) + 0.0001F_o^2]$
Max. shift/e.s.d.	0.02
Max., min. density in final difference map (e Å^{-3})	0.20, -0.22
S	1.77

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $12 < \theta < 20°$. (ii) A secondary-extinction correction was applied [$F_{corr} = F_c(1.0 \times 10^{-4}\chi F_c^2/\sin\theta)$] where χ refined to 0.009. No correction for absorption. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$.

Table 2. *Final atomic coordinates and isotropic temperature factors* (Å^2)

$$B_{iso} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{iso}
P	0.5041 (1)	0.6540 (1)	0.8270 (1)	2.60 (2)
O	0.7736 (3)	0.6842 (2)	0.8476 (1)	3.66 (4)
C(1)	0.4130 (4)	0.7703 (2)	0.9337 (2)	2.64 (6)
C(2)	0.5457 (4)	0.9493 (2)	0.9585 (2)	2.77 (6)
C(11)	0.3718 (4)	0.6998 (2)	0.6960 (2)	2.72 (5)
C(12)	0.1218 (5)	0.6681 (3)	0.6498 (2)	4.06 (7)
C(13)	0.0353 (6)	0.7013 (4)	0.5478 (2)	5.11 (8)
C(14)	0.1929 (7)	0.7678 (3)	0.4916 (2)	5.29 (9)
C(15)	0.4395 (7)	0.8018 (3)	0.5365 (2)	5.23 (9)
C(16)	0.5301 (5)	0.7678 (3)	0.6383 (2)	4.02 (7)
C(21)	0.3439 (4)	0.4456 (2)	0.8115 (2)	2.77 (5)
C(22)	0.1451 (5)	0.3960 (3)	0.8533 (2)	3.83 (7)
C(23)	0.0353 (5)	0.2344 (3)	0.8405 (3)	4.72 (8)
C(24)	0.1202 (6)	0.1216 (3)	0.7850 (2)	4.69 (7)
C(25)	0.3147 (6)	0.1684 (3)	0.7417 (2)	4.52 (7)
C(26)	0.4272 (5)	0.3297 (3)	0.7552 (2)	3.66 (7)

Table 3. *Interatomic distances (Å) and angles (°)*

P—O	1·482 (2)	P—C(1)	1·793 (2)
P—C(11)	1·812 (2)	P—C(21)	1·807 (2)
C(1)—C(2)	1·525 (3)	C(2)—C(2')	1·521 (3)
C(11)—C(12)	1·381 (4)	C(11)—C(16)	1·382 (4)
C(12)—C(13)	1·377 (4)	C(13)—C(14)	1·361 (5)
C(14)—C(15)	1·358 (5)	C(15)—C(16)	1·383 (4)
C(21)—C(22)	1·381 (4)	C(21)—C(26)	1·387 (3)
C(22)—C(23)	1·382 (4)	C(23)—C(24)	1·368 (4)
C(24)—C(25)	1·367 (5)	C(25)—C(26)	1·381 (4)

O—P—C(1)	114·9 (1)	O—P—C(11)	111·00 (9)
O—P—C(21)	112·45 (9)	C(1)—P—C(11)	105·6 (1)
C(1)—P—C(21)	106·9 (1)	C(11)—P—C(21)	105·3 (1)
P—C(1)—C(2)	112·0 (2)	C(1)—C(2)—C(2')	113·1 (2)
P—C(11)—C(12)	123·7 (2)	P—C(11)—C(16)	118·1 (2)
C(12)—C(11)—C(16)	118·3 (2)	C(11)—C(12)—C(13)	120·1 (2)
C(12)—C(13)—C(14)	121·1 (3)	C(13)—C(14)—C(15)	119·6 (3)
C(14)—C(15)—C(16)	120·2 (3)	C(11)—C(16)—C(15)	120·7 (3)
P—C(21)—C(22)	123·6 (2)	P—C(21)—C(26)	117·9 (2)
C(22)—C(21)—C(26)	118·5 (2)	C(21)—C(22)—C(23)	120·4 (2)
C(22)—C(23)—C(24)	120·4 (3)	C(23)—C(24)—C(25)	120·0 (3)
C(24)—C(25)—C(26)	119·9 (3)	C(21)—C(26)—C(25)	120·8 (2)

Fig. 1. Perspective view of the molecule showing the atom labeling.

Related literature. The molecule is sited on a crystallographic center of symmetry which relates one half of the molecule to the other. Related structures have been described by Oliva, Castellano & De Carvalho (1981) and Rivera, Gómez C, Rodulfo de Gil & Suarez (1988).

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Structure of *endo*-(5R*,6R*,11R*,12S*)-5,6,11,12-Tetrahydro-4,11,12-trimethoxy-9,13,13-trimethyl-5-(triethylsiloxy)-6,10-methano-8(7H)-benzocyclodecenone

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Abstract. $C_{27}H_{42}O_5Si$, $M_r = 474.71$, monoclinic, $P2_1/n$, $a = 9.389$ (5), $b = 35.542$ (7), $c = 8.899$ (4) Å, $\beta = 114.25$ (3)°, $V = 2707$ (2) Å³, $Z = 4$, $D_x = 1.164$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71069$ Å, $\mu = 1.14$ cm⁻¹, $F(000) = 1032$, $T = 298$ K, final $R = 0.049$ for 2926 unique reflections $[I > 3.0\sigma(I)]$. This C-aromatic taxane-like compound contains a C=C double bond [C(9)=C(10)] at the bridgehead site [C(10)] and, consequently, atoms C(8), C(11), C(13) and C(16) bonded to this C=C bond are twisted

from coplanarity. The largest torsion angle, C(8)—C(9)—C(10)—C(11), is −158.3 (3)°.

Experimental. Colorless plate-like crystals grown from hexane. Crystal size $0.40 \times 0.40 \times 0.50$ mm, Rigaku AFC-5R diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω scan with scan speed $16°$ min⁻¹ in ω, scan width $(1.34 + 0.35\tan\theta)°$. Range of indices, $0 < h < 11$, $0 < k < 42$, $-9 < l < 9$ $(2\theta < 50°)$. Three standard reflections ($\overline{3}3\overline{3}$, $50\overline{5}$, $4,\overline{16},2$) monitored every 100 reflections with random variation of 2.7% over data collection. Lattice-

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