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1,2-Bis(ditolylphosphino)ethane

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Key indicators

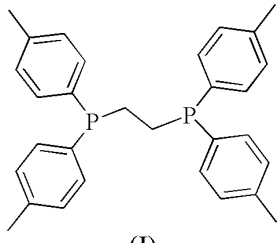
Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.123
Data-to-parameter ratio = 21.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the solid state, the title compound, 1,2-{(*p*-CH_3–C_6H_4)$_2$P}$_2$$C_2H_4$ or $C_{30}H_{32}P_2$, exhibits *trans* geometry. There is an inversion center at the mid-point of the central C—C bond.

Comment

In the course of our work on molybdenum and tungsten phosphine complexes, we synthesized and isolated the title compound, 1,2-bis(ditolylphosphino)ethane, (I). It crystallizes in the space group $P2_1c$, with two molecules in the unit cell. The P atoms are in a *trans* configuration with respect to each other, with an inversion center located at the mid-point of the central C—C bond. One of the tolyl rings lies approximately in the P—C—C—P plane, with atoms C21/P1/C1/C1^i/P1^i/C21^i forming a zigzag chain [symmetry code: (i) $1-x, 2-y, 1-z$]. The corresponding dihedral angle C1^i—C1—P1—C21 is 7.70 (19)°. The other tolyl ring is oriented roughly perpendicular to the P—C—C—P plane, the dihedral angle C1^i—C1—P1—C11 being 113.0 (2)°. All other bond lengths and angles are in the expected ranges.



(I)

Experimental

Bis(ditolylphosphino)ethane was synthesized by the reaction of 1,2-bis(dichlorophosphino)ethane with 4.5 equivalents of the Grignard reagent derived from 4-bromotoluene in tetrahydrofuran (THF). After hydrolysis with 10% aqueous ammonium chloride solution, extraction with diethyl ether, washing with water and drying with magnesium sulfate, the compound was crystallized from a toluene–hexane mixture at 277 K, giving single crystals suitable for X-ray structural analysis.

Crystal data

$C_{30}H_{32}P_2$
M_r = 454.50
Monoclinic, $P2_1/c$
a = 9.5469 (5) Å
b = 11.8565 (6) Å
c = 14.3645 (7) Å
β = 128.731 (1)°
V = 1268.40 (11) Å^3
Z = 2
D_x = 1.190 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9472 reflections
θ = 2.2–28.3°
μ = 0.19 mm^{-1}
T = 90 (2) K
Block, colorless
0.62 × 0.52 × 0.25 mm

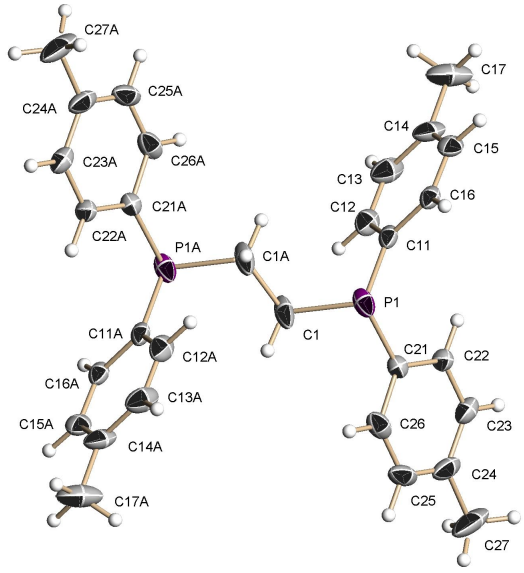


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. The suffix A corresponds to symmetry code (i) in the text and Table 1.

Data collection

Bruker AXS SMART APEX CCD diffractometer	$R_{int} = 0.046$
ω scans	$\theta_{max} = 28.3^\circ$
12 864 measured reflections	$h = -12 \rightarrow 12$
3137 independent reflections	$k = -15 \rightarrow 15$
2920 reflections with $I > 2\sigma(I)$	$l = -19 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.6662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.123$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.38$ e Å^{-3}
3137 reflections	
147 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

P1—C11	1.8215 (15)	C12—C13	1.377 (3)
P1—C21	1.8247 (13)	C25—C26	1.381 (2)
P1—C1	1.8489 (15)	C25—C24	1.385 (3)
C11—C16	1.3911 (17)	C23—C24	1.384 (2)
C11—C12	1.392 (2)	C14—C15	1.385 (2)
C21—C26	1.3929 (18)	C14—C13	1.390 (2)
C21—C22	1.3933 (17)	C14—C17	1.503 (2)
C22—C23	1.3853 (18)	C1—C1^{i}	1.529 (3)
C16—C15	1.385 (2)	C24—C27	1.508 (2)
C11—P1—C21	102.99 (6)	C21—P1—C1	98.67 (6)
C11—P1—C1	101.86 (8)	C1^{i}—C1—P1	111.25 (13)

Symmetry code: (i) $1-x, 2-y, 1-z$.

H atoms were positioned geometrically, with fixed C—H distances of 0.95 Å (methylene and aromatic) and 0.98 Å (methyl). Isotropic displacement parameters were set at 1.2 (methylene and aromatic) and 1.5 (methyl) times the U_{eq}(C). The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

Data collection: *SMART for Windows NT*/2000 (Bruker 1997–2000); cell refinement: *SAINT-Plus* (Bruker, 1997–1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXL*97; software used to prepare material for publication: *SHELXL*97.

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