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Key indicators

Single-crystal X-ray study
 $T = 100\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.037
 wR factor = 0.104
 Data-to-parameter ratio = 14.8

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

Bis[1,2-bis(*p*-ethylbenzene)phosphino]benzene

In the solid state, the title compound, $1,2\text{-}\{(p\text{-Et-C}_6\text{H}_4)_2\text{P}\}_2\text{-}(\text{C}_6\text{H}_4)$ or $\text{C}_{38}\text{H}_{40}\text{P}_2$, does not exhibit its molecular C_{2v} symmetry. It crystallizes in the space group $P\bar{1}$, with a full molecule in the asymmetric unit.

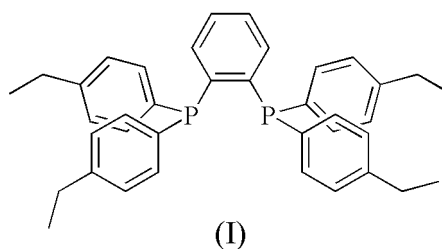
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Comment

In the course of our work on molybdenum and tungsten complexes with rigid phosphine ligands, we synthesized and isolated the title compound, bis[1,2-bis(*p*-ethylbenzene)phosphino]benzene, (I). It crystallizes in space group $P\bar{1}$, with $Z = 2$, and the inversion center is located between the two molecules. The C_{2v} symmetry found in solution is lost in the solid state.



The Ar_2P groups are rotated with respect to the plane of the central $\text{P}-(\text{C}_6\text{H}_4)-\text{P}$ fragment, resulting in a loss of the C_2 axis and the mirror planes. The torsion angles involving atom

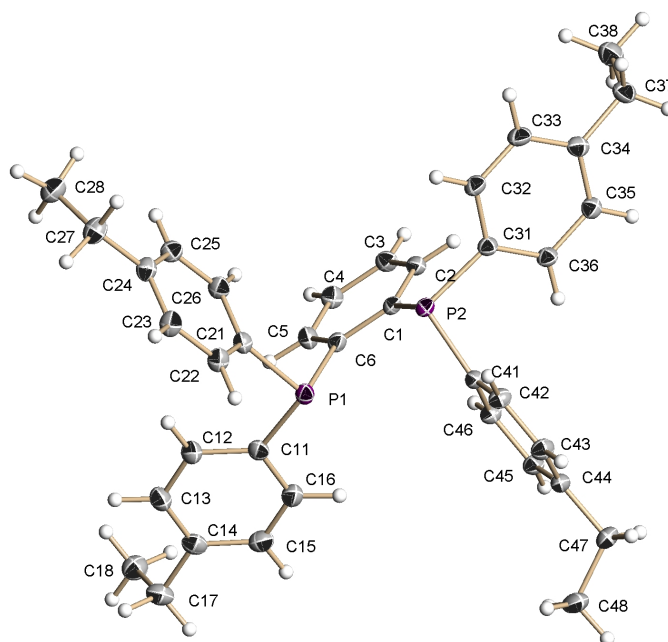


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids.

P1 are 96.06 (9) (C1–C6–P1–C21) and -158.86 (8) $^\circ$ (C1–C6–P1–C11), and those involving P2 are 83.75 (9) (C6–C1–P2–C31) and -173.49 (8) $^\circ$ (C6–C1–P2–C41). The rotation angle of the Ar₂P units from the mirror symmetric position can thus be estimated to be 31.4 $^\circ$ for P1 and 44.9 $^\circ$ for P2. Atom P1 is nearly perfectly coplanar with the annelated benzene ring. In contrast, atom P2 deviates from the plane formed by the six C atoms by 0.1602 (14) Å. All other bond lengths and angles are in the expected ranges.

Experimental

1,2-Bis(dichlorophosphino)benzene was prepared from 1,2-diphosphinobenzene (Kyba *et al.*, 1983) by slow addition of two equivalents of diphosgene (trichloromethylchloroformate) in THF. The temperature was raised to 323 K for 2 h, followed by removal of solvent and distillation in an oil pump vacuum. The title compound was prepared by the reaction of 1,2-bis(dichlorophosphino)benzene with 4.5 equivalents of the Grignard reagent derived from 1-bromo-4-ethylbenzene 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 THF–diethyl ether mixture at 258 K. This yielded single crystals suitable for X-ray structural analysis.

Crystal data

C₃₈H₄₀P₂
M_r = 558.64
 Triclinic, *P* $\bar{1}$
a = 11.0970 (7) Å
b = 11.6441 (7) Å
c = 13.5466 (8) Å
 α = 69.279 (1) $^\circ$
 β = 72.818 (1) $^\circ$
 γ = 89.604 (1) $^\circ$
V = 1554.77 (16) Å³

Z = 2
D_x = 1.193 Mg m⁻³
 Mo *K* α radiation
 Cell parameters from 6851 reflections
 θ = 2.5–28.3 $^\circ$
 μ = 0.17 mm⁻¹
T = 100 (2) K
 Block, colorless
 0.5 × 0.5 × 0.4 mm

Data collection

Bruker AXS SMART APEX CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS in S_{AINT-Plus}; Bruker 1997–1999)
T_{min} = 0.919, *T_{max}* = 0.936
 16 351 measured reflections

7697 independent reflections
 7138 reflections with *I* > 2 σ (*I*)
R_{int} = 0.021
 θ_{\max} = 28.3 $^\circ$
h = –14 → 14
k = –15 → 15
l = –18 → 18

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.037
wR(*F*²) = 0.104
S = 1.05
 7697 reflections
 521 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.6662P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.44 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.27 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, $^\circ$).

P2–C41	1.8322 (10)	C1–C2	1.3994 (14)
P2–C31	1.8359 (11)	C1–C6	1.4126 (14)
P2–C1	1.8478 (10)	C2–C3	1.3920 (15)
P1–C21	1.8316 (11)	C6–C5	1.3960 (15)
P1–C11	1.8331 (11)	C5–C4	1.3932 (15)
P1–C6	1.8436 (11)	C4–C3	1.3875 (16)
C2–C1–C6	118.54 (9)	C5–C6–P1	121.71 (8)
C2–C1–P2	123.08 (8)	C1–C6–P1	118.74 (8)
C6–C1–P2	118.16 (8)	C4–C5–C6	121.06 (10)
C3–C2–C1	121.33 (10)	C3–C4–C5	119.59 (10)
C5–C6–C1	119.55 (9)	C4–C3–C2	119.92 (10)
C6–C1–C2–C3	0.42 (15)	C1–C6–C5–C4	–0.77 (16)
P2–C1–C2–C3	–174.09 (8)	P1–C6–C5–C4	178.46 (8)
C2–C1–C6–C5	–0.01 (15)	C6–C5–C4–C3	1.15 (17)
P2–C1–C6–C5	174.77 (8)	C5–C4–C3–C2	–0.74 (17)
C2–C1–C6–P1	–179.27 (8)	C1–C2–C3–C4	–0.05 (16)
P2–C1–C6–P1	–4.49 (11)		

All H atoms were located in a difference Fourier map and were refined isotropically. The C–H bond lengths are in the ranges 0.928 (16)–0.990 (17) Å (aliphatic) and 0.947 (19)–1.035 (18) Å (aromatic). 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: *S_{AINT-Plus}* (Bruker, 1997–1999); data reduction: *S_{AINT-Plus}*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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