1,2-bis(di-4-pyridylphosphino)ethane (d4pype)

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1,2-Bis(di-4-pyridylphosphino)ethane (d4pype)

The title compound (d4pype), C$_{22}$H$_{20}$N$_{4}$P$_{2}$, crystallizes as a discrete molecular species disposed about a crystallographic inversion centre at the mid-point of the central C—C bond.

Comment

Bidentate tertiary phosphine ligands with pyridyl substituents, such as the title compound, (I), are of interest because a number of studies have shown that metal complexes with these ligands exhibit selective anti-tumour properties (Berners-Price et al., 1999; McKeage et al., 2000). During the course of our work in this area, we obtained crystals of (I) which were suitable for X-ray diffraction studies.

![Structure of 1,2-Bis(di-4-pyridylphosphino)ethane (d4pype)](image)

Compound (I) crystallizes in space group $P2_1/n$ as discrete molecules disposed in a trans configuration about a crystallographic inversion centre at the mid-point of the central C—C bond (Fig. 1). Noteworthy features of the geometric parameters in the structure are the P—C(py) bond lengths of 1.842 (3) and 1.837 (4) Å, which are similar to values of 1.846 (3) and 1.850 (3) Å for the structure of 1,2-bis(di-2-pyridylphosphino)ethane (Jones et al., 1999) but longer than bond lengths of 1.818 (4) and 1.829 (3) Å observed for the P—C(Ph) bonds in 1,2-bis(diphenylphosphino)ethane (Pelizzi & Pelizzi, 1979). The C—N bond lengths in the pyridyl rings range between 1.317 (5) and 1.337 (5) Å, which are characteristic for analogous bond lengths in other pyridine and pyridyl systems (e.g. Brammer & Zhao, 1995; Jones et al., 1999). All other bond lengths and angles are in accord with expected values.

Experimental

1,2-Bis(di-4-pyridylphosphino)ethane was prepared according to published procedures (Bowen et al., 1998). Single crystals suitable for X-ray crystallographic analysis were obtained as a by-product of slow evaporation of a solution of copper(I) chloride and (I) (molar ratio 1:2) in an acetonitrile/dichloromethane mixture.
Crystal data

C₂₂H₂₀N₄P₂

Mᵣ = 402.37

Monoclinic, P2₁/n

a = 14.073 (6) Å

b = 8.228 (2) Å
c = 9.200 (2) Å

β = 108.33 (3)°

V = 1011.2 (6) Å³

Z = 2

Dᵣ = 1.321 Mg m⁻³

Mo Kα radiation

Cell parameters from 16 reflections

θ = 10.4–17.1°

μ = 0.23 mm⁻¹

T = 295 K

Prism, colourless

0.15 × 0.10 × 0.05 mm

Data collection

Rigaku AFC-7R diffractometer

ω/2θ scans

Absorption correction: none

2332 independent reflections

1170 reflections with I > 2σ(I)

R(int) = 0.038

Refinement

Refinement on F²

R(F²) = 0.052

wR(F²) = 0.182

S = 1.00

2332 reflections

128 parameters

H-atoms: Rmax = 0.80 e Å⁻³

Δρmax = 0.26 e Å⁻³

Selected geometric parameters (Å, °)

Table 1

P₁—C₁ 1.842 (3) C₁—C₁ 1.381 (5)
P₁—C₆ 1.837 (4) C₂—C₃ 1.385 (6)
P₁—C₁₁ 1.849 (4) C₃—C₄ 1.387 (6)
N₁—C₃ 1.337 (5) C₄—C₅ 1.385 (6)
N₁—C₅ 1.381 (5) C₅—C₆ 1.381 (5)
N₁—C₄ 1.328 (5) C₆—C₁₀ 1.367 (6)
N₂—C₈ 1.317 (7) C₇—C₈ 1.383 (7)
N₂—C₉ 1.318 (7) C₉—C₁₀ 1.386 (7)
C₁—C₂ 1.388 (5) C₁₁—C₁₁ 1.386 (5)

C₁—P₁—C₆ 100.04 (15) N₁—C₄—C₅ 122.1 (4)
C₁—P₁—C₁₁ 102.74 (16) C₁—C₅—C₄ 120.0 (4)
C₆—P₁—C₁₁ 102.61 (18) P₁—C₆—C₇ 117.4 (3)
C₃—N₁—C₄ 115.9 (4) P₁—C₆—C₁₀ 126.0 (3)
C₈—N₂—C₉ 114.9 (5) C₇—C₆—C₁₀ 116.5 (4)
P₁—C₁—C₂ 118.3 (3) C₆—C₅—C₄ 119.1 (4)
P₁—C₁—C₅ 125.3 (3) N₂—C₈—C₇ 125.0 (5)
C₂—C₁—C₅ 116.3 (3) N₂—C₉—C₁₀ 125.0 (5)
C₁—C₂—C₃ 119.8 (4) C₆—C₁₀—C₉ 119.4 (4)
N₁—C₃—C₄ 123.8 (4) P₁—C₁₁—C₁₁ 112.4 (3)

Symmetry code: (i) 2−x, 1−y, 2−z.

H atoms were constrained in the riding model approximation, fixed to their parent C atoms at a C–H distance of 0.95 Å, and U(eq)(H) values were set to 1.2U(eq) of the parent atom.

Data collection: MSC/AFC-7 Diffractometer Control Software for Windows (Molecular Structure Corporation, 1999); cell refinement: MSC/AFC-7 Diffractometer Control Software for Windows; data reduction: TEXSAN for Windows (Molecular Structure Corporation, 1997–2001); program(s) used to solve structure: TEXSAN for Windows; program(s) used to refine structure: TEXSAN for Windows and SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: TEXSAN for Windows and PLATON (Spek, 1980–2001).

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References