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

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The title compound (d4pype), $C_{22}H_{20}N_4P_2$, crystallizes as a discrete molecular species disposed about a crystallographic inversion centre at the mid-point of the central C—C bond.

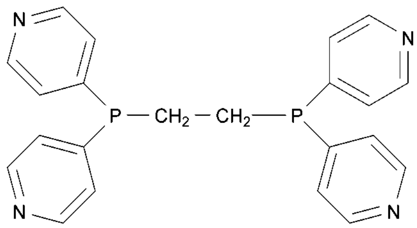
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.052
wR factor = 0.182
Data-to-parameter ratio = 18.1

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

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.



(I)

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

$C_{22}H_{20}N_4P_2$
$M_r = 402.37$
Monoclinic, $P2_1/n$
$a = 14.073$ (6) Å
$b = 8.228$ (2) Å
$c = 9.200$ (2) Å
$\beta = 108.33$ (3)°
$V = 1011.2$ (6) Å^3
$Z = 2$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 reflections
$\theta = 10.4$–17.1°
$\mu = 0.23$ mm^{-1}
$T = 295$ K
Prism, colourless
0.15 × 0.10 × 0.05 mm

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2552 measured reflections
2323 independent reflections
1170 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\rm max} = 27.5°$
$h = -7 \rightarrow 18$
$k = 0 \rightarrow 10$
$l = -11 \rightarrow 11$
3 standard reflections every 150 reflections intensity decay: 0.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.182$
$S = 1.00$
2323 reflections
128 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.016$
$\Delta\rho_{\rm max} = 0.80$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—C1	1.842 (3)	C1—C5	1.381 (5)
P1—C6	1.837 (4)	C2—C3	1.385 (6)
P1—C11	1.849 (4)	C4—C5	1.385 (6)
N1—C3	1.337 (5)	C6—C7	1.387 (6)
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$^{\rm i}$	1.526 (6)
C1—P1—C6	100.04 (15)	N1—C4—C5	124.1 (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—C7—C8	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—C2	123.8 (4)	P1—C11—C11$^{\rm i}$	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 $U_{\rm iso}$(H) values were set to $1.2U_{\rm eq}$ 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 *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows* and *PLATON* (Spek, 1980–2001).

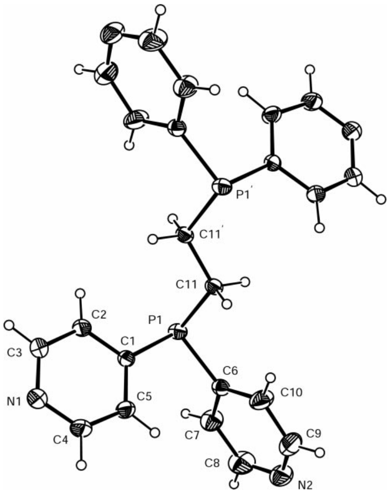


Figure 1
View of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

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