

Tris(2-pyridyl)phosphine

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Abstract. $C_{15}H_{12}N_3P$, $M_r = 265.3$, monoclinic, $P2_1/c$, $a = 9.162$ (1), $b = 9.163$ (1), $c = 16.071$ (2) Å, $\beta = 100.92$ (1)°, $U = 1325$ (2) Å³, D_m (by flotation) = 1.33 (1), $D_x = 1.330$ Mg m⁻³ for $Z = 4$, Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 0.153$ mm⁻¹, $F(000) = 552$, $T = 293$ (2) K, $R = 0.046$ for 2041 observed reflections. The P-atom environment is pyramidal with P–C bond distances of 1.834 (3), 1.824 (3) and 1.826 (2) Å and C–P–C angles of 102.7 (1), 101.9 (1) and 101.0 (1)°. The orientations of the rings are such that there is a significant deviation from C_{3v} symmetry; one of the N atoms is directed to the same side of the molecule as the P^{III} lone pair of electrons, while the remaining two N atoms point in the opposite direction. The P atom is almost coplanar with each of the rings [max. deviation 0.0420 (6) Å] and the dihedral angles between the planes through the N(1)–C(5), N(2)–C(10) and N(3)–C(15) groups are 87.8, 97.0 and 97.5° respectively.

Experimental. Transparent crystals were obtained from the slow evaporation of a benzene/373–393 K petroleum spirit solution of the compound (Hafeli, Keene, Stephenson & Wright, 1986). Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; $\omega:2\theta$ scan technique. Cell parameters on crystal 0.88 × 0.48 × 0.34 mm from least-squares procedure on 25 reflections ($8 \leq \theta \leq 14^\circ$). Analytical absorption correction applied (Sheldrick, 1976); max./min. transmission factors 0.9596 and 0.8797. Total of 5159 reflections ($1 \leq \theta \leq 27.5^\circ$) measured in the range $-11 \leq h \leq 11$, $-11 \leq k \leq 0$, $-20 \leq l \leq 9$. No significant variation in the net intensities of three reference reflections (415, 227, 524) measured every 3600 s. 3029 unique reflections ($R_{int} 0.057$), 2041 satisfied $I \geq 2.5\sigma(I)$. Structure solved from interpretation of Patterson map, full-matrix least-squares refinement of 173 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included in the model at their calculated positions. The

Table 1. Fractional atomic coordinates and B_{eq} values (Å²)

	$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$			
	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
P(1)	0.11758 (7)	0.28688 (7)	0.37503 (4)	3.08
N(1)	0.2973 (3)	0.1143 (3)	0.4927 (2)	4.19
N(2)	0.1697 (2)	0.5576 (2)	0.3332 (1)	3.50
N(3)	0.3809 (2)	0.2597 (2)	0.3161 (1)	3.34
C(1)	0.1700 (3)	0.1890 (3)	0.4759 (2)	3.17
C(2)	0.3292 (4)	0.0429 (3)	0.5673 (2)	4.56
C(3)	0.2360 (4)	0.0433 (3)	0.6257 (2)	4.47
C(4)	0.1068 (4)	0.1183 (4)	0.6072 (2)	5.13
C(5)	0.0721 (3)	0.1928 (3)	0.5310 (2)	4.47
C(6)	0.2046 (3)	0.4648 (3)	0.3991 (2)	3.02
C(7)	0.2231 (3)	0.6934 (3)	0.3435 (2)	4.42
C(8)	0.3128 (3)	0.7428 (3)	0.4169 (2)	4.47
C(9)	0.3486 (4)	0.6483 (3)	0.4832 (2)	4.81
C(10)	0.2925 (3)	0.5058 (3)	0.4752 (2)	3.85
C(11)	0.2417 (2)	0.2052 (2)	0.3115 (1)	2.67
C(12)	0.4634 (3)	0.1977 (3)	0.2657 (2)	3.88
C(13)	0.4189 (3)	0.0810 (3)	0.2123 (2)	3.93
C(14)	0.2792 (3)	0.0279 (3)	0.2089 (2)	3.74
C(15)	0.1879 (3)	0.0904 (3)	0.2582 (2)	3.21

Table 2. Interatomic distances (Å) and bond angles (°)

P(1)–C(1)	1.834 (3)	P(1)–C(6)	1.824 (3)
P(1)–C(11)	1.826 (2)	N(1)–C(1)	1.335 (3)
N(1)–C(2)	1.347 (3)	N(2)–C(6)	1.348 (3)
N(2)–C(7)	1.337 (4)	N(3)–C(11)	1.358 (3)
N(3)–C(12)	1.335 (3)	C(1)–C(5)	1.376 (4)
C(2)–C(3)	1.384 (4)	C(3)–C(4)	1.352 (4)
C(4)–C(5)	1.385 (4)	C(6)–C(10)	1.384 (3)
C(7)–C(8)	1.380 (4)	C(8)–C(9)	1.364 (5)
C(9)–C(10)	1.401 (4)	C(11)–C(15)	1.387 (3)
C(12)–C(13)	1.383 (4)	C(13)–C(14)	1.361 (4)
C(14)–C(15)	1.381 (4)		
C(1)–P(1)–C(6)	102.7 (1)	C(1)–P(1)–C(11)	101.9 (1)
C(6)–P(1)–C(11)	101.0 (1)	C(1)–N(1)–C(2)	117.6 (2)
C(6)–N(2)–C(7)	117.6 (2)	C(11)–N(3)–C(12)	116.4 (2)
P(1)–C(1)–N(1)	120.2 (2)	P(1)–C(1)–C(5)	117.6 (2)
N(1)–C(1)–C(5)	122.2 (3)	N(1)–C(2)–C(3)	123.0 (3)
C(2)–C(3)–C(4)	118.6 (3)	C(3)–C(4)–C(5)	119.3 (3)
C(1)–C(5)–C(4)	119.2 (3)	P(1)–C(6)–N(2)	111.6 (2)
P(1)–C(6)–C(10)	126.0 (2)	N(2)–C(6)–C(10)	122.4 (2)
N(2)–C(7)–C(8)	123.8 (3)	C(7)–C(8)–C(9)	118.4 (3)
C(8)–C(9)–C(10)	119.4 (3)	C(6)–C(10)–C(9)	118.4 (3)
P(1)–C(11)–N(3)	119.8 (2)	P(1)–C(11)–C(15)	118.1 (2)
N(3)–C(11)–C(15)	122.1 (2)	N(3)–C(12)–C(13)	124.8 (2)
C(12)–C(13)–C(14)	117.8 (2)	C(13)–C(14)–C(15)	119.7 (2)
C(11)–C(15)–C(14)	119.1 (2)		

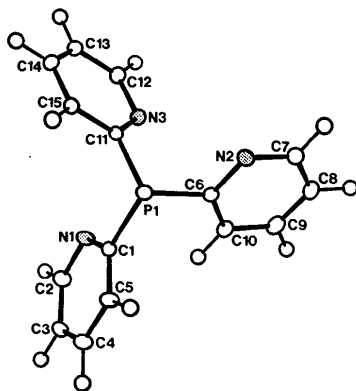


Fig. 1. Molecular structure and numbering scheme for tris-(2-pyridyl)phosphine. Hydrogen atoms are numbered according to the C atom to which they are bonded (Johnson, 1971).

reflections $\bar{5}14$, $\bar{6}14$ and $\bar{7}14$ were excluded from the final refinement owing to poor agreement. At convergence $R = 0.046$, $wR = 0.056$, $w = [\sigma^2(F) + 0.0028F^2]^{-1}$, $S = 1.47$, $(\Delta/\sigma)_{\max} \leq 0.001$, $(\Delta\rho)_{\max} = 0.94$, $(\Delta\rho)_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$; no extinction correction. Scattering factors for all atoms given in *SHELX76* (Sheldrick, 1976). All calculations on VAX11/785 computer system. Atomic parameters given in Table 1, bond distances and angles in Table 2;* the numbering scheme used is shown in Fig. 1.

Related literature. The title compound, $(2\text{-py})_3\text{P}$, has been shown by spectroscopic methods to function

* Lists of structure factors, thermal parameters, H-atom parameters and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44593 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

primarily as a tridentate ligand through the three N donor atoms, e.g. $[M\{(2\text{-py})_3\text{P}\}_2](\text{ClO}_4)_2$ for $M = \text{Mn}$, Co, Ni, Cu and Zn (Bogges & Zatkan, 1975), for $M = \text{Co}$ (Bogges & Zatkan, 1976; Hafeli & Keene, 1987) and for $M = \text{Fe}$ (Bogges, Hughes, Chew & Kemper, 1981). However, both monodentate P and bidentate P,N coordination have been reported for the $(2\text{-py})_3\text{P}$ ligand in a series of Ru and Rh compounds, e.g. $\text{RuHCl}\{(2\text{-py})_3\text{P}\}_3$ and $\text{RhCl}\{(2\text{-py})_3\text{P}\}_2$ (Kurtev, Ribola, Jones, Cole-Hamilton & Wilkinson, 1980). The only crystallographic characterization of a compound containing $(2\text{-py})_3\text{P}$ is that for the $[\text{Ru}\{(2\text{-py})_3\text{P}\}_2]^{2+}$ cation in which $(2\text{-py})_3\text{P}$ adopts the more common tridentate mode of coordination (Stephenson, Keene, Snow & Tiekink, 1988).

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Propane-1,2,3-tricarboxylic Acid (Tricarballic Acid)

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Abstract. $\text{C}_6\text{H}_8\text{O}_6$, $M_r = 176.12$, orthorhombic, *Pbca*, $a = 11.89$ (2), $b = 23.96$ (3), $c = 5.38$ (2) \AA , $V = 1532.68 \text{ \AA}^3$, $Z = 8$, $D_x = 1.53 \text{ Mg m}^{-3}$, $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ \AA}$, $\mu = 1.118 \text{ mm}^{-1}$, $F(000) = 736$, $T = 293 \text{ K}$, $R = 0.062$ for 603 reflexions. The molecule has no

unusual bond lengths or angles. Molecules are linked into double sheets by hydrogen bonding similar to that found in citric acid although the molecular conformations are different.

Experimental. Colourless crystals from water, very few of adequate diffraction quality. Preliminary

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