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Tris(2-pyridyl)methanol

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Abstract. $C_{16}H_{13}N_3O$, $M_r = 263{\cdot}3$, monoclinic, $P2_1/n$, $a = 11{\cdot}034$ (1), $b = 8{\cdot}794$ (3), $c = 14{\cdot}007$ (2) Å, $\beta = 102{\cdot}31$ (1)°, $U = 1328$ (2) Å^3, $D_x = 1{\cdot}317$ Mg m^{-3} for $Z = 4$, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}049$ mm^{-1}, $F(000) = 552$, $T = 293$ (2) K, $R = 0{\cdot}045$ for 1811 observed reflections. Within the molecule the C—C(py) distances lie in the range 1·524 (3)–1·530 (2) Å and the maximum deviation from ideal tetrahedral geometry is seen in the O(1)—C(16)—C(11) angle of 106·1 (2)°. The dihedral angles between the N(1)—C(5), N(2)—C(10) and N(3)—C(15) rings are 89·7, 100·8 and 81·5° respectively. The disposition of the N atoms is such that two point in the opposite direction of the alcohol function while the other, N(2), lies on the same side of the molecule as the C—OH group; the O—H(16)···N(2) separation of 2·19 (3) Å may be indicative of a weak intramolecular interaction between these atoms. In the crystal lattice there is also a close contact between N(2) and the centrosymmetrically related hydroxyl proton of 2·37 (4) Å.

Table 1. *Fractional atomic coordinates and B_{eq} values* (Å^2)

$$B_{\rm eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$B_{\rm eq}$
O(1)	−0·4060 (1)	0·2963 (2)	0·0612 (1)	3·71
N(1)	−0·2377 (2)	0·2679 (2)	−0·1306 (1)	3·40
N(2)	−0·3400 (1)	0·5571 (2)	−0·0155 (1)	3·13
N(3)	−0·1071 (2)	0·1391 (2)	0·0734 (1)	3·60
C(1)	−0·3106 (2)	0·2220 (2)	−0·0715 (1)	2·72
C(2)	−0·2526 (2)	0·2002 (3)	−0·2175 (2)	4·16
C(3)	−0·3376 (3)	0·0876 (3)	−0·2494 (2)	4·49
C(4)	−0·4129 (3)	0·0425 (3)	−0·1886 (2)	4·78
C(5)	−0·4002 (2)	0·1102 (2)	−0·0987 (2)	3·99
C(6)	−0·2491 (2)	0·4613 (2)	0·0232 (1)	2·71
C(7)	−0·3102 (2)	0·7031 (2)	−0·0236 (2)	3·77
C(8)	−0·1917 (2)	0·7587 (3)	0·0061 (2)	4·08
C(9)	−0·0995 (2)	0·6597 (3)	0·0470 (2)	4·22
C(10)	−0·1281 (2)	0·5084 (3)	0·0555 (2)	3·68
C(11)	−0·2003 (2)	0·2050 (2)	0·1040 (1)	2·89
C(12)	−0·0234 (2)	0·0620 (3)	0·1395 (2)	4·01
C(13)	−0·0291 (3)	0·0484 (3)	0·2355 (2)	4·53
C(14)	−0·1245 (3)	0·1169 (4)	0·2654 (2)	5·97
C(15)	−0·2130 (3)	0·1963 (3)	0·1997 (2)	4·93
C(16)	−0·2919 (2)	0·2966 (2)	0·0287 (1)	2·79

Experimental. Transparent crystals were obtained from the slow evaporation of a benzene/373–393 K petroleum spirit solution of the compound (Wibaut, De Jonge, Van Der Voort & Otto, 1951). Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; $\omega/2\theta$ scan technique. Cell parameters on crystal 0·18 × 0·56 × 0·56 mm from least-squares procedure on 25 reflections ($13 \leq \theta \leq 19°$). Analytical absorption correction applied (Sheldrick, 1976); max./min. transmission factors 0·9919 and 0·9778. Total of 4501 reflections ($1 \leq \theta \leq 27{\cdot}5°$) measured in the range $-14 \leq h \leq 14$, $-11 \leq k \leq 0$, $-20 \leq l \leq 6$. No significant variation in the net intensities of three reference reflections (562, 253, 366) measured every 7200 s. 3049 unique reflections ($R_{\rm int}$ 0·056), 1813 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved by direct methods, full-matrix least-squares refinement of 233 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms located from difference map and refined. The reflections $\bar{2}04$ and 013 were excluded from the final refinement due to poor agreement. At convergence $R = 0{\cdot}045$, $wR = 0{\cdot}053$, $w = [\sigma^2(F) + 0{\cdot}0076F^2]^{-1}$, $S = 0{\cdot}73$, $(\Delta/\sigma)_{\max} \leq 0{\cdot}001$, $(\Delta\rho)_{\max} = 0{\cdot}16$, $(\Delta\rho)_{\min} = -0{\cdot}29$ e Å^{-3}; no extinction correction. Scattering factors for all atoms given in *SHELX*76 (Sheldrick, 1976). All calculations on VAX11/785 computer system. Atomic parameters are given in Table 1, bond distances and angles in Table 2;* the numbering scheme used is shown in Fig. 1.

Related literature. Several crystal structures have been determined for metal complexes containing the title compound, $(py)_3COH$, as a ligand. Thus symmetric

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* 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 44671 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

N(1)–C(1)	1·333 (3)	N(1)–C(2)	1·334 (3)
N(2)–C(6)	1·333 (2)	N(2)–C(7)	1·336 (3)
N(3)–C(11)	1·329 (3)	N(3)–C(12)	1·343 (3)
C(2)–C(3)	1·372 (4)	C(3)–C(4)	1·368 (4)
C(4)–C(5)	1·374 (4)	C(5)–C(1)	1·389 (3)
C(7)–C(8)	1·375 (3)	C(8)–C(9)	1·368 (4)
C(9)–C(10)	1·378 (3)	C(10)–C(6)	1·379 (3)
C(12)–C(13)	1·364 (4)	C(13)–C(14)	1·354 (4)
C(14)–C(15)	1·380 (4)	C(15)–C(11)	1·380 (3)
C(16)–O(1)	1·427 (2)	C(16)–C(1)	1·524 (3)
C(16)–C(6)	1·530 (2)	C(16)–C(11)	1·526 (3)
O(1)–H(16)	0·88 (3)		

C(1)–N(1)–C(2)	117·3 (2)	C(6)–N(2)–C(7)	117·2 (2)
C(11)–N(3)–C(12)	117·6 (2)	N(1)–C(1)–C(5)	122·2 (2)
N(1)–C(1)–C(16)	117·4 (2)	C(5)–C(1)–C(16)	120·4 (2)
N(1)–C(2)–C(3)	124·2 (3)	C(2)–C(3)–C(4)	118·1 (2)
C(3)–C(4)–C(5)	119·2 (2)	C(4)–C(5)–C(1)	119·0 (2)
N(2)–C(6)–C(10)	122·3 (2)	N(2)–C(6)–C(16)	113·9 (1)
C(10)–C(6)–C(16)	123·8 (2)	N(2)–C(7)–C(8)	123·4 (2)
C(7)–C(8)–C(9)	118·4 (2)	C(8)–C(9)–C(10)	119·1 (2)
C(9)–C(10)–C(6)	119·0 (2)	N(3)–C(11)–C(15)	122·3 (2)
N(3)–C(11)–C(16)	116·9 (2)	C(15)–C(11)–C(16)	120·8 (2)
N(3)–C(12)–C(13)	123·7 (2)	C(12)–C(13)–C(14)	117·9 (2)
C(13)–C(14)–C(15)	120·3 (2)	C(14)–C(15)–C(11)	118·2 (2)
O(1)–C(16)–C(1)	109·8 (1)	O(1)–C(16)–C(6)	108·7 (1)
O(1)–C(16)–C(11)	106·1 (2)	C(1)–C(16)–C(6)	110·1 (1)
C(1)–C(16)–C(11)	110·4 (2)	C(6)–C(16)–C(11)	111·6 (1)
C(16)–O(1)–H(16)	106 (2)		

tridentate N,N′,N″ coordination has been noted in the cations $[M\{(py)_3COH\}_2]^{2+}$, M = Co (Szalda & Keene, 1986) and for one ligand in M = Ru (Keene, Szalda & Wilson, 1987). Asymmetric tridentate coordination has been found in $[CH_3Hg(py)_3COH)][NO_3]$ (Canty, Chaichit, Gatehouse & George, 1981) with Hg—N bond distances in the range 2·28 (1) to 2·53 (1) Å. Tridentate coordination *via* two N atoms and the hydroxyl O atom has also been observed in one of the ligands in $[Ru\{(py)_3COH\}_2]^{2+}$ (Keene, Szalda & Wilson, 1987) and in $[Ru(NH_3)_3\{(py)_3COH\}]^{2+}$ (Diamantis, Keene, Moritz, Snow & Tiekink, 1988).

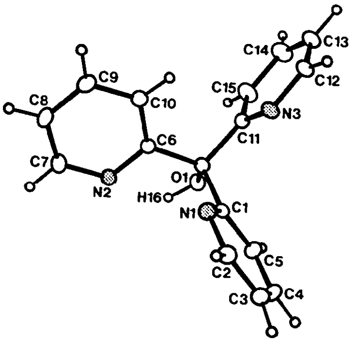

Fig. 1. The molecular structure and numbering scheme for $(C_5H_4N)_3COH$. Hydrogen atoms are numbered according to the C atom to which they are bonded (Johnson, 1971).

When deprotonated, the ligand also coordinates *via* two N atoms and the O atom as found in $[Ru\{(py)_3COH\}\{(py)_3CO\}]^+$ (Keene, Szalda & Wilson, 1987).

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Structure of 4*β*-Hydroxy-6-methoxymethoxy-*N*,*N*-dimethyl-1,2,3,4α,4a,9b-hexahydro-9b*β*-dibenzofuranacetamide

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Abstract. $C_{18}H_{25}NO_5$, $M_r = 335{\cdot}4$, triclinic, $P\bar{1}$, $a = 11{\cdot}155\,(4)$, $b = 9{\cdot}732\,(3)$, $c = 9{\cdot}150\,(3)$ Å, $\alpha = 110{\cdot}81\,(3)$, $\beta = 79{\cdot}86\,(2)$, $\gamma = 112{\cdot}11\,(3)°$, $V = 859{\cdot}21$ Å^3, $Z = 2$, $D_x = 1{\cdot}30$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}87$ cm^{-1}, $F(000) = 360$, room temperature, final $R = 0{\cdot}068$ for 2683 observed reflections. The title compound is an intermediate in the synthesis of a morphinic analogue. The relative configuration was established. There are no unusual bond distances or angles.

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