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Tri(2-pyridyl)methane

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Abstract. $C_{16}H_{13}N_3$, M_r = 247·3, monoclinic, $C2/c$, a = 13·998 (1), b = 8·653 (1), c = 22·368 (2) Å, β = 103·15 (1)°, U = 2638 (1) Å^3, D_x = 1·245 Mg m^{-3}, Z = 8, Mo $K\bar{\alpha}$ radiation, λ = 0·7107 Å, μ = 0·042 mm^{-1}, F(000) = 1040, T = 293 (2) K, R = 0·034 for 947 observed reflections. In the title compound, $(2\text{-py})_3CH$, the central C atom exists in a slightly distorted tetrahedral geometry [tetrahedral angles range from 105 (1) to 113·8 (2)°]. The C—C(py) distances are 1·517 (4), 1·515 (4) and 1·522 (4) Å and the dihedral angles between the three pyridyl rings are 103·6, 106·8 and 88·4°, respectively. One of the pyridyl rings is disordered with a 180° rotation about the C(methane)—C(py) bond at C(1)—C(31). In the major conformation (*ca* 58%) the two N atoms are orientated as to point in the same direction as the methane H atom.

Experimental. The compound, $(2\text{-py})_3CH$, was prepared as in the literature (Keene, Snow, Stephenson & Tiekink, 1988). Suitable crystals for X-ray study were obtained from the slow evaporation of a dichloromethane/373–393 K petroleum spirit solution of the compound. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal 0·16 × 0·20 × 0·45 mm from least-squares procedure on 25 reflections (4 ≤ θ ≤ 12°). No absorption correction applied (Sheldrick, 1976). Total of 2932 reflections (1·5 ≤ θ ≤ 25·0°) measured in the range $-16 \le h \le 16$, $0 \le k \le 10$, $-26 \le l \le 3$. No significant variation in the net intensities of two reference reflections (2$\bar{2}$5, 3$\bar{1}$1) measured every 7200 s. 2330 unique reflections (R_{int} 0·015) and 947 satisfied $I \ge 2{\cdot}5\sigma(I)$. Structure solved by direct methods (Sheldrick, 1986), full-matrix least-squares refinement of 229 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms refined with isotropic thermal parameters. The pyridyl ring C(31)–C(36) was found to be disordered over two sites such that N(32) and C(36) were interchanged by a 180° rotation about the C(1)—C(31) bond. Refinement of fractional site occupancies gave the major component of the disorder [58 (5)%] with N and C atoms in the N(32) and C(36) sites respectively and the minor component [42 (5)%] with C and N atoms in these sites respectively. At convergence R = 0·034, wR = 0·037, $w = 0{\cdot}71/[\sigma^2(F) + 0{\cdot}0018F^2]$, S = 0·84, $(\Delta/\sigma)_{\max} \le 0{\cdot}001$, $(\Delta\rho)_{\max}$ = 0·08, $(\Delta\rho)_{\min}$ = −0·13 e Å^{-3}; no extinction correction. Scattering factors for all atoms given in *SHELX76* (Sheldrick, 1976). All calculations on SUN4/280 computer system. Atomic parameters given in Table 1, selected bond distances and angles in Table 2,* the numbering scheme used is shown in Fig. 1.

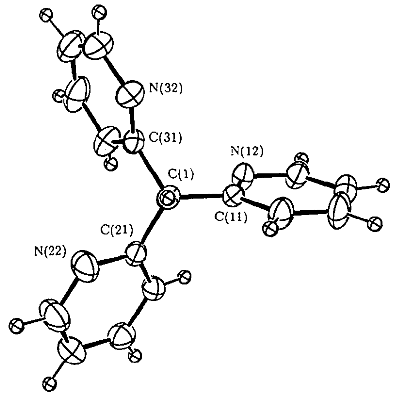

Fig. 1. Molecular structure and numbering scheme for $(2\text{-py})_3CH$ drawn at 25% probability levels (Johnson, 1971).

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* Lists of structure factors, thermal parameters, H-atom parameters, all bond distances and angles, and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52830 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates* (× 10⁴) *and* B_{eq} *values* (Å²)

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

	x	*y*	*z*	B_{eq}
C(1)	5100 (2)	2765 (3)	3498 (1)	4·15
C(11)	5843 (2)	1858 (3)	3968 (1)	4·11
N(12)	5568 (2)	1297 (3)	4456 (1)	5·18
C(13)	6225 (2)	495 (4)	4865 (2)	5·54
C(14)	7151 (3)	217 (4)	4808 (2)	6·46
C(15)	7436 (3)	805 (5)	4313 (2)	7·98
C(16)	6779 (2)	1641 (4)	3879 (2)	6·41
C(21)	5021 (2)	4449 (3)	3664 (1)	4·26
N(22)	4619 (2)	5353 (4)	3188 (1)	7·14
C(23)	4537 (3)	6856 (5)	3293 (2)	8·51
C(24)	4826 (3)	7520 (5)	3852 (2)	6·96
C(25)	5229 (3)	6599 (4)	4339 (2)	6·03
C(26)	5327 (2)	5043 (4)	4239 (2)	4·76
C(31)	4095 (2)	1999 (3)	3358 (2)	4·39
N(32)*	3898 (3)	1010 (4)	2873 (1)	6·43
C(33)	2979 (4)	334 (5)	2738 (3)	7·96
C(34)	2304 (4)	656 (6)	3060 (3)	8·51
C(35)	2538 (3)	1660 (7)	3543 (2)	8·42
C(36)*	3439 (2)	2343 (6)	3690 (2)	6·49

* Site occupancy factor 0·58 (5).

Related literature. The title compound was examined as part of a wider study of compounds of this type including the ligands $(2\text{-py})_3P$ (Keene, Snow & Tiekink, 1988*a*) and $(2\text{-py})_3COH$ (Keene, Snow & Tiekink, 1988*b*), and their complexes (Keene *et al.*, 1988). In complexes both bidentate and tridentate coordination have been shown by X-ray crystallography in $[AuMe_2\{(2\text{-py})_3CH\}]NO_3$ (Canty, Minchin, Healy & White, 1982) and $[Ru\{(2\text{-py})_3CH\}_2]$(tosylate)$_2$ (Keene *et al.*, 1988), respectively.

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

C(1)—C(11)	1·517 (4)	C(1)—C(21)	1·515 (4)
C(1)—C(31)	1·522 (4)	C(1)—H(1)	1·11 (2)
C(11)—N(12)	1·329 (3)	N(12)—C(13)	1·335 (4)
C(21)—N(22)	1·338 (4)	N(22)—C(23)	1·332 (5)
C(31)—C(32)*	1·361 (5)	N(32)—C(33)	1·383 (6)
C(31)—C(36)*	1·338 (5)	C(36)—C(35)	1·363 (6)

C(11)—C(1)—C(21)	113·8 (2)	C(11)—C(1)—C(31)	111·9 (2)
C(11)—C(1)—H(1)	109 (1)	C(21)—C(1)—C(31)	110·7 (2)
C(21)—C(1)—H(1)	106 (1)	C(31)—C(1)—H(1)	105 (1)
C(1)—C(11)—N(12)	118·5 (2)	C(1)—C(11)—C(16)	119·6 (3)
C(1)—C(21)—N(22)	114·1 (3)	C(1)—C(21)—C(26)	124·8 (3)
C(1)—C(31)—N(32)	116·6 (3)	C(1)—C(31)—C(36)	120·5 (3)
C(11)—N(12)—C(13)	118·0 (3)	C(21)—N(22)—C(23)	117·9 (3)
C(31)—N(32)—C(33)	116·7 (4)		

* N(32) and C(36) have site occupancies 0·58 (5).

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Structure of (±)-Encecanescin

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Abstract. (R^*,R^*)-(±)-6,6′-(Oxydiethylidine)bis[7-methoxy-2,2-dimethyl-2*H*-1-benzopyran] [compound (1), (±)-encecanescin], $C_{28}H_{34}O_5$, $M_r = 450{\cdot}57$, monoclinic, $P2_1/n$, $a = 27{\cdot}35$ (1), $b = 8{\cdot}293$ (2), $c = 22{\cdot}133$ (7) Å, $\beta = 94{\cdot}68$ (4)°, $V = 5003$ (3) Å³, $Z = 8$ (two molecules, *A* and *B*, in the asymmetric unit), $D_x = 1{\cdot}196$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å), $\mu = 0{\cdot}755$ cm^{-1}, $F(000) = 1936$, $T = 163$ K. Block-matrix least-squares refinement of (1) converged to $R = 0{\cdot}048$ and $wR = 0{\cdot}042$ using 4015 reflections with $I > 3\sigma(I)$. The crystal is a racemic mixture of both enantiomers, namely the 11-α-Me,11′-β-Me and the 11-β-Me,11′-α-Me. The two molecules in the asymmetric unit are related by a pseudo inversion center,

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