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2,6-Bis[(2-phenyl-1,3-dioxolan-2-yl)methyl]pyridine

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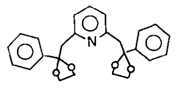
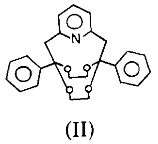
Abstract. $\mathrm{C_{25}H_{25}NO_4}$, M_r = 403·5, monoclinic, $C2/c$, a = 21·192 (6), b = 6·276 (2), c = 15·845 (5) Å, β = 95·29 (3)°, V = 2098 (2) Å^3, Z = 4, D_x = 1·277 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·81 cm^{-1}, F(000) = 856·0, T = 296 K, R = 0·041 for 1273 observations. The molecule has crystallographic symmetry 2. The planar pyridine and phenyl rings [maximum deviations 0·005 (2) Å for each] form a dihedral angle of 55·0 (2)°, and the acetal ring is in the envelope conformation with an O atom at the flap. The crystal packing is dominated by the parallel stacking of aromatic rings.

Introduction. The title compound was prepared during a study of the chelation properties of a series of bis(ethyleneglycol) acetals of 2,6-diacylpyridine. The synthetic procedure afforded one of two possible isomers: the bis(acetal) (I) or the cryptand (II).



(I)



(II)

Standard spectral means (NMR, IR, MS) were insufficient to distinguish between the two structures, and thus a crystal-structure determination was conducted. This investigation proved isomer (I) to be correct.

Experimental. Yellow crystal, dimensions 0·20 × 0·23 × 0·60 mm, cut from a needle, space-group determination from systematic absences hkl with $h + k$ odd, $h0l$ with l odd and successful refinement in centrosymmetric space group. Cell dimensions from least squares of setting angles of 25 reflections, $13 < \theta < 14°$. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator. Intensity data by ω–2θ scans designed to yield $I = 50\sigma(I)$, scan rates varied 0·26–5·0° min^{-1}. Data having $1 < \theta < 25°$, $h + k$ even, $0 \le h \le 26$, $0 \le k \le 7$, $-18 \le l \le 18$ measured, corrected for Lorentz, polarization, background. Absorption and decay insignificant (600, 020, 0,0,10 standard reflections, 3·2% variation). Equivalent data averaged, $R_{\rm int}$ = 0·027, 1841 unique data. Structure solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refined by full-matrix least squares based on F using 1273 data with $I > 3\sigma(I)$, $w = [\sigma^2(|F|) + 0{\cdot}02|F|^2]^{-1}$, using *SHELX*76 (Sheldrick, 1976) and Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974). Non-H atoms anisotropic; H atoms located by ΔF map, refined isotropically (those of acetal groups: fixed contributions, C—H 0·95 Å, B = 8·0 Å^2). Final R = 0·041 (0·074 all data), wR = 0·060, S = 1·994 for 172 variables. Max. Δ/σ <0·01 in final cycle, max. residual density 0·16 e Å^{-3}, min. −0·18 e Å^{-3}, extinction g = 1·3 (2) × 10^{-6} [$|F_c| = |F_o|(1 + gI_c)$].

Discussion. Atomic coordinates and equivalent isotropic thermal parameters for non-H atoms are given in Table 1.* Bis(acetal) (I) possesses crystallographic C_2 symmetry. Both pyridine and phenyl rings are planar [maximum deviations 0·005 (2) Å for each], and they form a dihedral angle of 55·0 (2)°. The conformation of (I) in the crystal is illustrated in Fig. 1 and is specified by torsion angles N—C(3)—C(4)—C(5) −113·8 (3),

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42290 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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C(3)—C(4)—C(5)—C(6) 58·9 (3), C(4)—C(5)—C(6)—C(7) −87·3 (3)°, as well as the endocyclic torsion angles of the acetal ring: −0·7 (3) about O(1)—C(12); −18·1 (3) C(12)—C(13); 19·4 (3) O(1)—C(5); 30·1 (3) C(13)—O(2); −31·3 (3)° O(2)—C(5). The latter angles reveal that the acetal ring is in the envelope conformation with O(2) at the flap and asymmetry parameter $\Delta C_s(5) = 1{\cdot}4°$ (Duax & Norton, 1975).

Bond distances and angles (Table 2) are normal, except for C(12)—C(13), which exhibits shortening due to large thermal parameters in this part of the acetal ring. The high degree of anisotropy in C(12) and C(13) may reflect unresolved disorder of these atoms. Aromatic C—C bonds average 1·380 [8] Å, C—O bonds average 1·422 [3] Å, and C—H bonds involving refined H atoms range 0·92 (2)–1·02 (2) Å and average 0·97 [3] Å. The crystal packing is viewed down the symmetry axis in Fig. 2, and is dominated by the parallel stacking of aromatic rings. No intermolecular contacts of less than 3·4 Å between heavy atoms exist.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\beta_{12}\cos\gamma + ac\beta_{13}\cos\beta + bc\beta_{23}\cos\alpha)$.

	x	y	z	B_{eq}(Å^2)
O(1)	0·78699 (6)	0·1281 (3)	0·17589 (10)	6·07 (4)
O(2)	0·81877 (6)	0·4705 (2)	0·15962 (9)	5·24 (3)
N	1	0·2555 (3)	0·25	3·31 (4)
C(1)	1	0·6984 (4)	0·25	4·23 (5)
C(2)	0·94413 (8)	0·5857 (3)	0·2498 (1)	4·08 (4)
C(3)	0·94543 (8)	0·3656 (3)	0·2490 (1)	3·36 (3)
C(4)	0·88552 (8)	0·2367 (3)	0·2470 (1)	4·12 (4)
C(5)	0·84105 (7)	0·2574 (3)	0·1659 (1)	4·10 (4)
C(6)	0·87186 (7)	0·1923 (3)	0·0869 (1)	3·86 (4)
C(7)	0·90573 (9)	0·3404 (3)	0·0444 (1)	4·50 (4)
C(8)	0·93505 (10)	0·2819 (4)	−0·0269 (1)	5·29 (5)
C(9)	0·93109 (9)	0·0762 (4)	−0·0561 (1)	5·30 (5)
C(10)	0·89844 (9)	−0·0713 (4)	−0·0141 (1)	5·51 (5)
C(11)	0·86898 (9)	−0·0161 (3)	0·0570 (1)	4·87 (4)
C(12)	0·73270 (10)	0·2509 (5)	0·1470 (2)	8·16 (8)
C(13)	0·75586 (10)	0·4588 (5)	0·1203 (2)	6·93 (6)

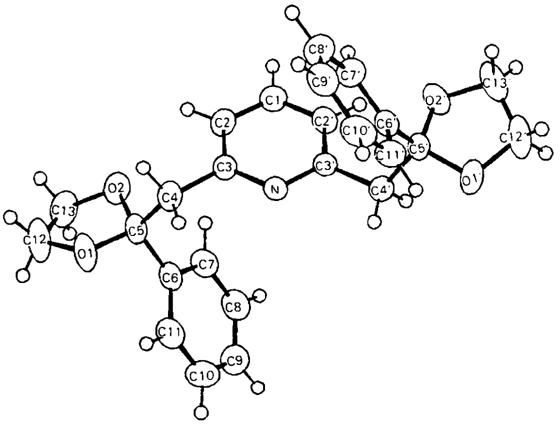

Fig. 1. Numbering scheme of the title compound (*ORTEP*, Johnson, 1976).

Table 2. *Bond distances* (Å) *and angles* (°)

O(1)—C(5)	1·425 (2)	C(5)—C(6)	1·521 (2)
O(1)—C(12)	1·425 (2)	C(6)—C(7)	1·386 (2)
O(2)—C(5)	1·419 (2)	C(6)—C(11)	1·390 (2)
O(2)—C(13)	1·421 (2)	C(7)—C(8)	1·388 (2)
N—C(3)	1·346 (1)	C(8)—C(9)	1·372 (2)
C(1)—C(2)	1·379 (2)	C(9)—C(10)	1·364 (3)
C(2)—C(3)	1·382 (2)	C(10)—C(11)	1·382 (2)
C(3)—C(4)	1·503 (2)	C(12)—C(13)	1·469 (3)
C(4)—C(5)	1·527 (2)		
C(5)—O(1)—C(12)	106·9 (2)	O(2)—C(5)—C(6)	111·1 (1)
C(5)—O(2)—C(13)	106·0 (2)	C(4)—C(5)—C(6)	113·0 (1)
C(3)—N—C(3′)	118·2 (1)	C(5)—C(6)—C(7)	120·1 (2)
C(2)—C(1)—C(2′)	118·3 (1)	C(5)—C(6)—C(11)	121·6 (2)
C(1)—C(2)—C(3)	119·7 (2)	C(7)—C(6)—C(11)	118·3 (2)
N—C(3)—C(2)	122·1 (2)	C(6)—C(7)—C(8)	120·6 (2)
N—C(3)—C(4)	116·6 (2)	C(7)—C(8)—C(9)	120·3 (2)
C(2)—C(3)—C(4)	121·4 (2)	C(8)—C(9)—C(10)	119·5 (2)
C(3)—C(4)—C(5)	115·3 (1)	C(9)—C(10)—C(11)	121·0 (2)
O(1)—C(5)—O(2)	106·2 (1)	C(6)—C(11)—C(10)	120·3 (2)
O(1)—C(5)—C(4)	107·4 (1)	O(1)—C(12)—C(13)	107·0 (2)
O(1)—C(5)—C(6)	110·4 (1)	O(2)—C(13)—C(12)	104·2 (2)
O(2)—C(5)—C(4)	108·4 (1)		

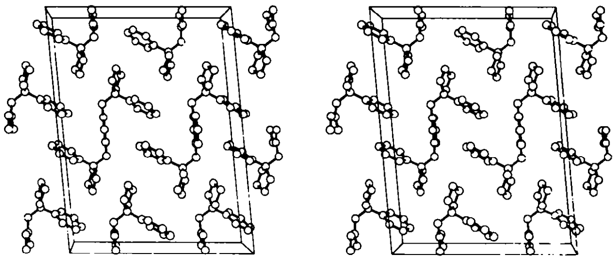

Fig. 2. Unit cell viewed down the symmetry axis, with the c axis horizontal (*PLUTO*, Motherwell & Clegg, 1978).

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