

observed dark color of the crystals may indicate some transfer of charge. The r.m.s. deviation of C atoms from the plane of the anthracene ring system is 0.003 (1) Å, while for the dianhydride it is 0.027 Å. Atoms O(12), O(13), C(13) and C(16) deviate most from the plane.

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Structure of 4-(5-Cyano-2-furyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarbonitrile

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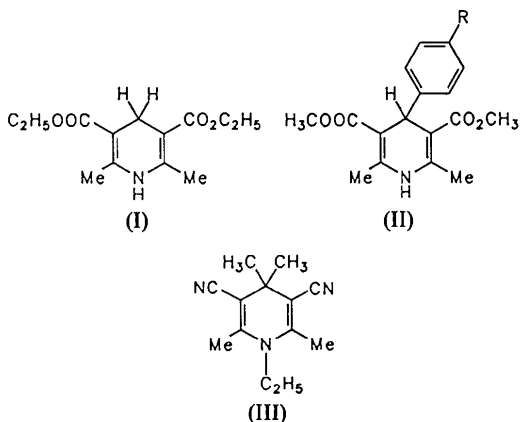
Abstract. $C_{14}H_{10}N_4O$, $M_r = 250.26$, monoclinic, $P2_1/c$, $a = 13.293$ (3), $b = 8.099$ (2), $c = 13.815$ (3) Å, $\beta = 120.474$ (5)°, $V = 1281.9$ (5) Å³, $Z = 4$, $D_m = 1.26$ (2), $D_x = 1.297$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.08$ mm⁻¹, $F(000) = 520$, $T = 299$ K, final $R = 0.0442$ for 2081 unique reflections with $F > 2\sigma(F)$. The crystal structure consists of single molecules. The substituted 1,4-dihydropyridine ring has a flat boat conformation with the N(1) and C(4) atoms displaced by 0.100 (1) and 0.141 (1) Å, respectively, from the plane through the other four C atoms which defines the base of the boat. The plane of the furyl ring makes an angle of 76.7 (1)° with the

plane of the base of the boat. No significant intermolecular interactions are observed.

Introduction. Numerous newly synthesized 1,4-dihydropyridines have been studied as models of NADH coenzymes in 'hydrogen-transfer' reactions (Eisner & Küthan, 1972). In order for a 1,4-dihydropyridine to act as a reducing agent, the H atom in the 4-position [H(3)] and that at the N atom should assume an antiperiplanar arrangement.

X-ray studies on 1,4-dihydropyridines have shown a great dependence of the geometry of the skeleton on the attached substituents, especially those in the 4

position. Thus, (I) has a planar structure (Lenstra, Petit, Dommissé & Alderweireldt, 1979), (II) has a boat conformation (Fossheim, Svarteng, Mostad, Roemming, Shefter & Triggles, 1982) while (III) is a half-boat (Huml, Ječný, Kutschabsky & Reck, 1979).



We have studied the crystal structure of 4-(5-cyano-2-furyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarbonitrile (IV) prepared by a modified Hantzsch procedure starting from 5-formyl-2-furancarboxitrile and 3-amino-2-butenitrile (Ilavský, Marchalín, Kováč, Henry-Basch & Morvan, 1987).

Experimental. A colourless well shaped crystal of dimensions $0.35 \times 0.48 \times 0.55$ mm was used. D_m was measured by flotation. Accurate unit-cell parameters were obtained by a least-squares fit of 42 reflections ($41.1 < 2\theta < 48.0^\circ$). The intensities were recorded on a Stoe-Siemens AED diffractometer for $3 < 2\theta < 50^\circ$ using graphite-monochromated $\text{Mo } K\alpha$ radiation and $\omega/2\theta$ scan mode. Index range was h 0/15, k -9/4, l -16/14. Three standard reflections (020, $32\bar{3}$, $3\bar{2}3$) were measured every 90 min; no significant systematic fluctuation was found. 3621 reflections were measured, 2250 unique, of which 2082 with $F_o > 2\sigma(F_o)$ were considered observed; $R_{\text{int}} = 0.0105$ for 1184 unique reflections. The structure was solved by direct methods using SIR88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) and refined on F by least squares with SHELX76 (Sheldrick, 1976). A difference map of the partially refined structure gave the positions of all H atoms. Refinement continued on all 213 positional and thermal parameters, anisotropic for non-H atoms and isotropic for H atoms. In the final cycle, $R = 0.0442$ and $wR = 0.0357$ for 2081 observed reflections (one of the strongest reflections, 310, was considered to suffer from extinction and was excluded from the refinement), $w^{-1} = k\sigma^2(F)$, $k = 3.1868$; max. $\Delta/\sigma = 0.056$; max. and min. heights in the final $\Delta\rho$ map 0.059 and -0.066 e \AA^{-3} . No absorption correction was applied. Secondary-extinction correction refined

Table 1. Final atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) with e.s.d.'s in parentheses

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(1)	6760 (1)	618 (2)	6532 (1)	494 (6)
C(1)	5744 (2)	1446 (3)	6249 (2)	555 (10)
C(2)	5455 (2)	2460 (3)	5387 (2)	729 (12)
C(3)	6335 (2)	2274 (3)	5097 (2)	659 (10)
C(4)	7106 (1)	1164 (2)	5806 (1)	432 (8)
C(5)	5236 (2)	1079 (3)	6919 (2)	666 (11)
C(6)	8200 (1)	398 (2)	5955 (1)	412 (8)
C(7)	8692 (1)	1405 (2)	5362 (1)	416 (8)
C(8)	8540 (1)	964 (2)	4355 (1)	411 (9)
C(9)	7856 (1)	-1728 (2)	4490 (1)	411 (9)
C(10)	7979 (1)	-1353 (2)	5498 (1)	397 (7)
C(11)	7836 (2)	-2600 (2)	6143 (1)	480 (9)
C(12)	7550 (2)	-3394 (3)	3958 (2)	567 (12)
C(13)	8899 (2)	1979 (3)	3678 (2)	552 (11)
C(14)	8301 (2)	2893 (2)	5892 (1)	506 (9)
N(1)	8052 (1)	-530 (2)	3902 (1)	453 (8)
N(2)	4848 (2)	786 (3)	7468 (1)	916 (12)
N(3)	9796 (2)	4070 (2)	6336 (1)	734 (9)
N(4)	7709 (2)	-3558 (2)	6680 (1)	688 (10)

to $1.12(2) \times 10^{-6} [F_c \text{ multiplied by } (1 - xF_c^2)\sin\theta]$. The scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). No significant correlations among refined parameters were observed.

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1* and bond lengths and angles (Nardelli, 1983) in Table 2. The molecular structure with atom numbering is shown in Fig. 1 (ORTEPII; Johnson, 1971).

The structure is formed by single molecules linked via hydrogen bonds (Table 2). The furyl ring is planar within experimental error. The dihydropyridine ring adopts a flattened boat conformation. The C(6), C(7), C(10) plane makes an angle of $17.9(1)^\circ$ with the C(7), C(8), C(9), C(10) plane, and the C(8), N(1), C(9) plane forms an angle of $171.3(1)^\circ$ with the C(7), C(8), C(9), C(10) plane. The furyl ring plane forms an angle of $76.7(1)^\circ$ with plane C(7), C(8), C(9), C(10), thus confirming the $\sigma-\pi$ interaction of the p orbitals of 1,4-dihydropyridine and the σ bond C(6)(sp^3)—C(4)(sp^2). The interaction also manifests itself through the significant bathochromic shift of the longest-wavelength UV maximum. Bond distances and valence angles are normal. The 1,4-dihydropyridine cyano groups are equivalent and are involved in hydrogen bonds

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55732 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0297]

Table 2. Selected interatomic distances (Å), angles (°) and hydrogen bonds (Å, °) with e.s.d.'s in parentheses

O(1)—C(1)	1.3751 (30)	C(7)—C(8)	1.3486 (21)
O(1)—C(4)	1.3715 (23)	C(7)—C(14)	1.4292 (22)
C(1)—C(2)	1.3327 (37)	C(8)—C(13)	1.4936 (35)
C(1)—C(5)	1.4266 (47)	C(8)—N(1)	1.3656 (21)
C(2)—C(3)	1.4237 (46)	C(9)—C(10)	1.3518 (21)
C(3)—C(4)	1.3416 (25)	C(9)—C(12)	1.4910 (29)
C(4)—C(6)	1.4962 (21)	C(9)—N(1)	1.3725 (23)
C(5)—N(2)	1.1377 (41)	C(10)—C(11)	1.4219 (25)
C(6)—C(7)	1.5190 (23)	C(11)—N(4)	1.1436 (28)
C(6)—C(10)	1.5192 (23)	C(14)—N(3)	1.1439 (23)

C(1)—O(1)—C(4)	106.3 (2)	C(6)—C(7)—C(8)	122.3 (1)
O(1)—C(1)—C(5)	115.7 (2)	C(8)—C(7)—C(14)	120.1 (1)
O(1)—C(1)—C(2)	110.6 (3)	C(7)—C(8)—N(1)	119.7 (1)
C(2)—C(1)—C(5)	133.7 (3)	C(7)—C(8)—C(13)	124.9 (2)
C(1)—C(2)—C(3)	106.3 (2)	C(13)—C(8)—N(1)	115.4 (1)
C(2)—C(3)—C(4)	107.3 (2)	C(12)—C(9)—N(1)	115.7 (2)
O(1)—C(4)—C(3)	109.6 (2)	C(10)—C(9)—N(1)	119.4 (1)
C(3)—C(4)—C(6)	135.1 (2)	C(10)—C(9)—C(12)	124.9 (2)
O(1)—C(4)—C(6)	115.4 (1)	C(6)—C(10)—C(9)	122.3 (1)
C(1)—C(5)—N(2)	178.9 (3)	C(9)—C(10)—C(11)	120.1 (1)
C(4)—C(6)—C(10)	111.1 (1)	C(6)—C(10)—C(11)	117.5 (1)
C(4)—C(6)—C(7)	111.0 (1)	C(10)—C(11)—N(4)	177.4 (2)
C(7)—C(6)—C(10)	108.8 (1)	C(7)—C(14)—N(3)	178.7 (1)
C(6)—C(7)—C(14)	117.6 (1)	C(8)—N(1)—C(9)	122.9 (1)

D—H...A	D...A	H...A	D—H...A
N(1)—H(4)...N(4 ⁱⁱ)	2.959 (2)	2.10 (2)	177 (2)
C(13)—H(8)...O(1 ⁱⁱ)	3.480 (2)	2.72 (2)	138 (2)
C(6)—H(3)...N(3 ⁱⁱⁱ)	3.485 (2)	2.57 (1)	158 (1)

Symmetry code: (i) $x, -\frac{1}{2}-y, -\frac{1}{2}+z$; (ii) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (iii) $2-x, -\frac{1}{2}+y, \frac{3}{2}-z$.

(Table 2). Double-bond lengths in the furyl ring differ very slightly [C(1)=C(2) = 1.333 (4) and C(3)=C(4) = 1.342 (3) Å] owing to the conjugation in the N(2)(*sp*)-C(5)(*sp*)-C(1)(*sp*²)-C(2)(*sp*²)-C(3)(*sp*²)-C(4)(*sp*²) unit. The replacement of 4-methylpyridine in position 5 of the furan ring (Lokaj, Vrabel, Sivý, Kettmann, Ilavský & Ječný, 1991) by the cyano group has resulted in increased conjugation, leading to a decrease in the C(2)—C(3) bond length from 1.441 (3) to 1.424 (5) Å.

Atomic thermal motion increases significantly with distance from the molecular centre. The U_{eq} values range from <0.042 Å² for dihydropyridine to

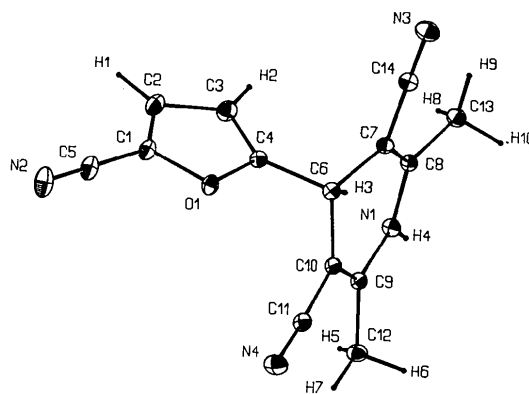


Fig. 1. The molecular structure of the title compound showing the atom numbering.

0.073 Å² in the furyl ring and up to 0.092 (1) Å² for the outer N(2) atom. The ratio of the shortest to longest principal axis increases likewise, with a maximum asymmetry of 2.98 for N(4).

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Structure of 12b-Hydroxy-5-methyl-1,2,3,4,4a,5,6,12b-octahydro-12H-benzo[*a*]xanthen-12-one

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Abstract. C₁₈H₂₀O₃, $M_r = 284.35$, triclinic, $P\bar{1}$, $a = 7.505$ (2), $b = 8.805$ (2), $c = 11.443$ (4) Å, $\alpha =$

101.53 (3), $\beta = 99.57$ (3), $\gamma = 95.43$ (2)°, $V = 724.3$ (9) Å³, $Z = 2$, $D_x = 1.299$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.816$ cm⁻¹, $F(000) = 304$, $T = 296$ K, $R = 0.045$ for 1900 independent reflections.

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