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1-Methyl-6,7-dihydropyrrolo[2,3-*c*]azepine-4,8(1*H*,5*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.088
Data-to-parameter ratio = 9.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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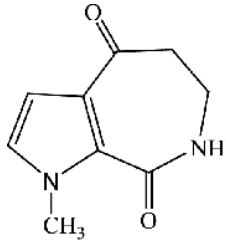
The title compound, $C_9H_{10}N_2O_2$, was synthesized by cyclization of 3-(1-methylpyrrole-2-carboxamido)propanoic acid in the presence of phosphorus oxychloride. Intermolecular N—H···O hydrogen bonds generate a one-dimensional chain in the crystal structure.

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001), and some of these compounds are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of 6,7-dihydropyrrolo[2,3-*c*]azepine-4,8(1*H*,5*H*)-diones has been synthesized by cyclization of 3-(pyrrole-2-carboxamido)propanoic acids. Pharmacological studies have shown that the title compound, (I), possesses moderately antilipoperoxidation properties. We report here its crystal structure.



(I)

The bond lengths and angles are unexceptional and are in good agreement with corresponding parameters in aldisin (Zeng *et al.*, 1991) and 2-bromoaldisin (Xu *et al.*, 2001). The conformation of the seven-membered ring can be described as follows: atoms C4, C5, N2 and C6 (and O1) are coplanar, while atoms C3, C8 and C7 deviate from the plane by 0.615 (2), 1.310 (2) and 1.240 (2) Å, respectively.

An intermolecular N—H···O hydrogen bond links the molecules into a one-dimensional chain (Table 1 and Fig. 2).

Experimental

3-(1-Methylpyrrole-2-carboxamido)propanoic acid (1.96 g, 10 mmol) was added to phosphorus oxychloride (20 ml) at about 373 K. The mixture reacted at reflux for 2 h, and was then poured into ice–water and neutralized with NaOH solution. After filtration, the aqueous solution was extracted four times with ethyl acetate (15 ml). The organic phase was dried with sodium sulfate overnight. The solvent was removed by distillation under reduced pressure, and the yellow solid residue was collected. The crude product was dissolved in MeOH at room temperature and normal pressure. Pale-yellow crystals suitable for X-ray analysis (m.p. 470 K, yield 59.5%) grew over a

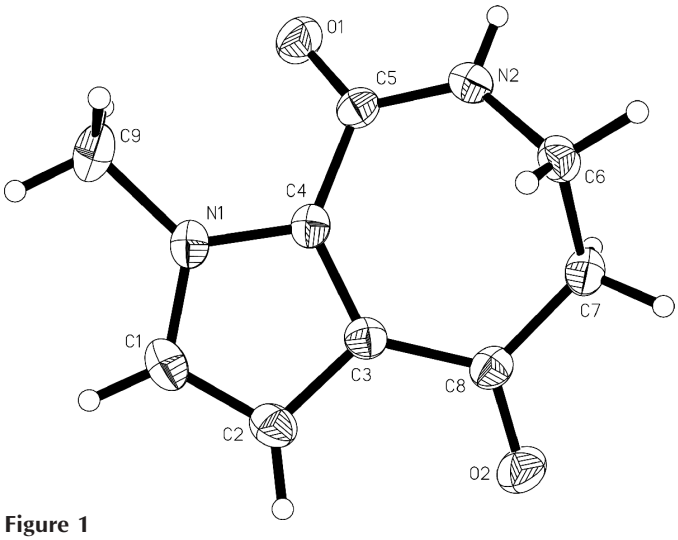


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

period of one week when the solution was exposed to air. ^{1}H NMR: 8.25 (*br s*, 1H), 7.05 (*d*, 1H), 6.48 (*d*, 1H), 3.86 (*s*, 3H), 3.32 (*m*, 2H), 2.64 (*m*, 2H); IR (KBr): 3337, 3099, 1634, 1528, 1480, 1253. Elemental analysis calculated for $C_9H_{10}N_2O_2$: C 60.66, H 5.66, N 15.72%; found: C 60.76, H 5.75, N 15.70%.

Crystal data

$C_9H_{10}N_2O_2$	Mo $K\alpha$ radiation
M_r = 178.19	Cell parameters from 863 reflections
Orthorhombic, $P2_12_12_1$	θ = 3.0–26.9°
a = 8.589 (3) Å	μ = 0.10 mm^{-1}
b = 8.686 (3) Å	T = 273 (2) K
c = 11.375 (4) Å	Block, pale yellow
V = 848.6 (5) Å^3	0.50 × 0.37 × 0.24 mm
Z = 4	
D_x = 1.395 Mg m^{-3}	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	1088 independent reflections
φ and ω scans	991 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)	R_{int} = 0.016
T_{min} = 0.953, T_{max} = 0.986	θ_{max} = 27.1°
5343 measured reflections	$h = -11 \rightarrow 10$
	$k = -11 \rightarrow 11$
	$l = -11 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.1067P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.088	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
1088 reflections	Extinction correction: none
119 parameters	
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.11	2.902 (2)	153

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

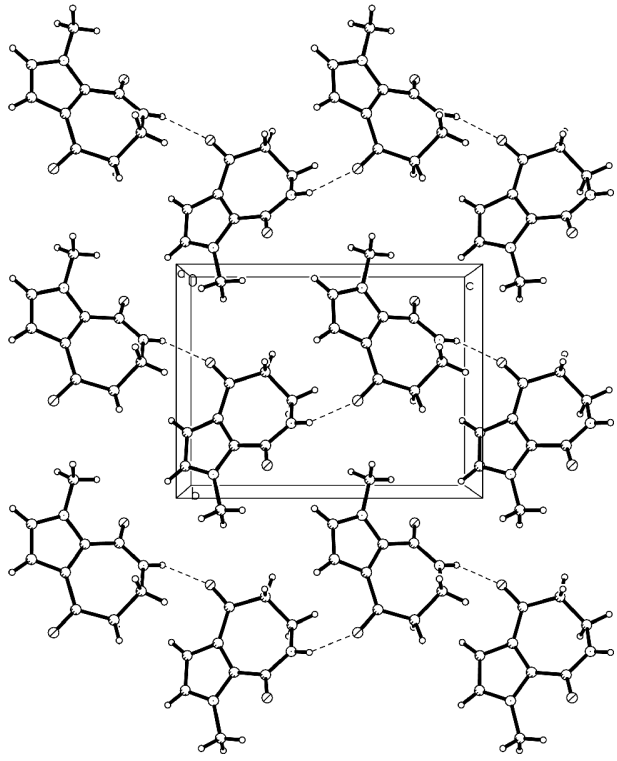


Figure 2
A view showing the one-dimensional chain formed by hydrogen bonds (dashed lines).

All H atoms were positioned geometrically (C—H = 0.97 Å for CH_2, 0.96 Å for CH_3 and 0.93 Å for CH, and N—H = 0.86 Å) and refined as riding atoms, with U_{iso}(H) = 1.2U_{eq}(C,N) [1.5U_{eq}(C) for the methyl group]. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *SMART* (Bruker,1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker,1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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