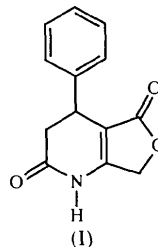


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5-Oxo-4-phenyl-1,2,3,4,5,7-hexahydrofuro- [3,4-*b*]-2(1*H*)-pyridone†

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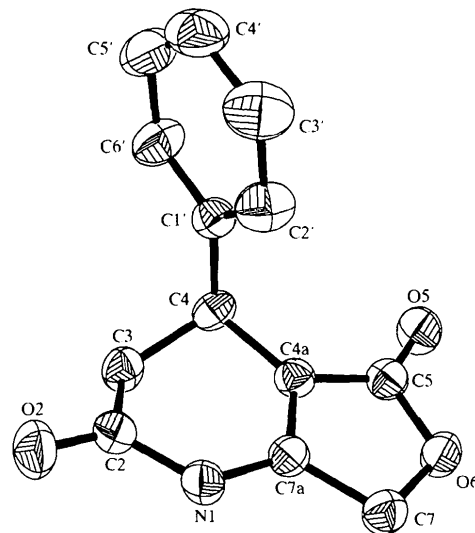


Fig. 1. A perspective view of the molecule with the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Abstract

The six-membered ring in the title compound, C₁₃H₁₁NO₃, adopts a twist conformation and the lactone ring a planar conformation. The aryl group occupies a pseudo-axial position and is orthogonal to the plane through the pyridone ring. The molecules are linked by an intermolecular hydrogen bond between the N atom and the carbonyl-O atom of a neighbouring molecule [$N1 \cdots O5(x, y-1, z) = 2.820(5) \text{ \AA}$].

Comment

Research on the 1,4-dihydropyridine (1,4-DHP) system is of current interest because of its properties as a calcium channel antagonist (Bossert & Vater, 1989). Substitution on the 1,4-DHP ring has been studied (Kuthan & Kurfurst, 1982) because of the effect of the substituent on the biological properties. It has been found that cyclohexanone and γ -lactone rings fused to the 1,4-DHP moiety result in a striking effect on the entry of calcium ions into the intracellular space (calcium-agonist effect) (Meyer *et al.*, 1976).

The six-membered ring of the title compound, (I), is best described as having a twist conformation; its puckering parameters (Nardelli, 1983a) are $Q = 0.422(4) \text{ \AA}$, $\theta = 120.6(5)^\circ$ and $\varphi = -39.9(7)^\circ$ with $\Delta C_2(N1-C7a) = 0.039(2)$ and $\Delta C_2(C2) = 0.098(1)$.

† Alternative name: 4-phenyl-1,2,3,4,5,7-hexahydrofuro[3,4-*b*]pyridine-2,5-dione.

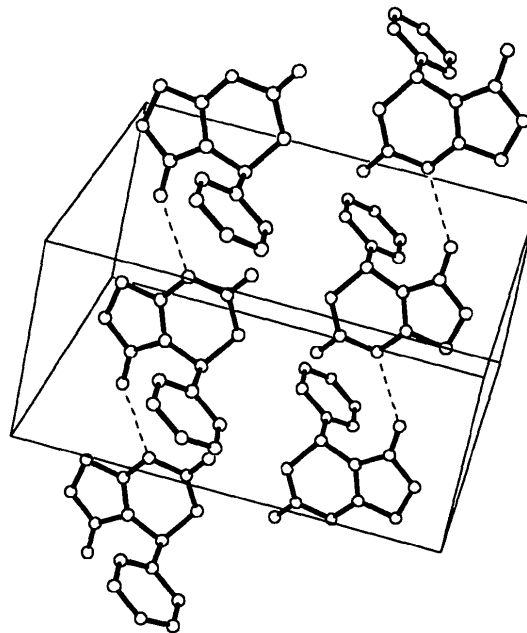


Fig. 2. Crystal structure viewed along the *b* axis. Hydrogen bonds are represented as dashed lines.

Experimental

6-Methyl-5-methoxycarbonyl-4-phenyl-3,4-dihydro-2(1H)-pyridone (1.15 g, 5 mmol) (Morales *et al.*, 1996) and *N*-bromosuccinimide (0.89 g, 5 mmol) in 10 ml of dry chloroform were refluxed for 12 h. The reaction mixture was cooled and the solid that precipitated was collected by filtration. Further purification was accomplished by recrystallization from ethanol; m.p. 512–513 K. ^1H NMR (DMSO- d_6): δ 10.74 (*s*, 1H, NH); 7.35–7.18 (*m*, 5H, aryl protons), 4.91(*dd*, 2H, OCH₂), 4.01 (*dd*, 1H, H-4 $J = 8.9$ Hz, $J = 3.8$ Hz, X part of ABX), 3.12 (*dd*, 1H, H-3a, $J = 16.7$ Hz, $J = 8.9$ Hz, A part of ABX), 2.57 (*dd*, H-3b, $J = 16.6$ Hz, $J = 3.7$ Hz, B part of ABX); ^{13}C NMR (DMSO) δ 170.9 (C2), 169.6 (C5), 160.7 (C7a), 144.8, 128.7 (2C), 126.9, 126.5 (2C) (aryl), 101.7 (C4a), 65.3 (C7), 38.5 (C3), 33.3 (C4).

Crystal data

C₁₃H₁₁NO₃
 $M_r = 229.24$
 Monoclinic
 $P2_1/c$
 $a = 11.297$ (1) Å
 $b = 6.949$ (1) Å
 $c = 13.732$ (2) Å
 $\beta = 91.95$ (1)°
 $V = 1077.4$ (2) Å³
 $Z = 4$
 $D_x = 1.413$ Mg m⁻³
 D_m not measured

Data collection

Enraf–Nonius CAD-4
 diffractometer
 ω -2 θ scans
 Absorption correction: none
 3367 measured reflections
 2546 independent reflections
 821 reflections with
 $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R(F)[F > 4\sigma(F)] = 0.054$
 $wR(F^2)[F^2 > 2\sigma(F^2)] = 0.088$
 $S(F^2) = 1.162$
 2546 reflections
 198 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0471P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

Mo $K\alpha$ radiation
 $\lambda = 0.71069$ Å
 Cell parameters from 25 reflections
 $\theta = 2$ –25°
 $\mu = 0.1015$ mm⁻¹
 $T = 293$ K
 Prismatic
 0.18 × 0.10 × 0.10 mm
 Colourless

$R_{\text{int}} = 0.070$
 $\theta_{\text{max}} = 27.96^\circ$
 $h = -14 \rightarrow 14$
 $k = 0 \rightarrow 9$
 $l = 0 \rightarrow 18$
 2 standard reflections
 frequency: 100 min
 intensity decay: <3%

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.199$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.269$ e Å⁻³
 Extinction correction: none
 Scattering factors from
International Tables for X-ray Crystallography
 (Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Selected geometric parameters (Å, °)

N1—C7a	1.368 (6)	C2—C3	1.505 (7)
N1—C2	1.380 (6)	C3—C4	1.541 (7)
O6—C7	1.440 (6)	C4—C1'	1.527 (6)
O6—C5	1.370 (6)	C1'—C2'	1.372 (7)
O2—C2	1.212 (6)	C1'—C6'	1.380 (7)
O5—C5	1.210 (6)	C2'—C3'	1.391 (8)
C7—C7a	1.483 (7)	C3'—C4'	1.36 (1)

C7a—C4a	1.336 (6)	C4'—C5'	1.37 (1)
C4a—C5	1.441 (7)	C5'—C6'	1.384 (9)
C4a—C4	1.499 (6)		
C7a—N1—C2	120.4 (4)	N1—C2—C3	115.1 (4)
C7—O6—C5	109.3 (4)	C2—C3—C4	114.5 (4)
O6—C7—C7a	103.3 (4)	C4a—C4—C3	105.8 (4)
N1—C7a—C7	125.5 (4)	C3—C4—C1'	110.7 (4)
C7—C7a—C4a	110.7 (4)	C4a—C4—C1'	113.7 (4)
N1—C7a—C4a	123.8 (4)	C4—C1'—C6'	118.1 (4)
C7a—C4a—C4	121.9 (4)	C4—C1'—C2'	123.1 (4)
C7a—C4a—C5	107.5 (4)	C2'—C1'—C6'	118.7 (5)
C5—C4a—C4	130.6 (4)	C1'—C2'—C3'	120.5 (5)
O5—C5—C4a	131.6 (5)	C2'—C3'—C4'	120.1 (6)
O6—C5—C4a	109.2 (4)	C3'—C4'—C5'	120.3 (6)
O6—C5—O5	119.2 (4)	C4'—C5'—C6'	119.8 (6)
N1—C2—O2	119.8 (4)	C1'—C6'—C5'	120.7 (5)
O2—C2—C3	124.9 (4)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...O5'	0.87 (5)	1.96 (5)	2.820 (5)	170 (4)

Symmetry code: (i) $x, y - 1, z$.

A poor quality crystal of dimensions 0.18 × 0.10 × 0.10 mm was the only one available for analysis, despite repeated attempts at recrystallization. The poor crystal quality resulted in just one third of the data having $I > 2\sigma(I)$.

All H atoms were located by difference Fourier synthesis and were refined isotropically.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1992). Cell refinement: *CRYSDA (DIRDIF; Beurskens et al., 1992)*. Data reduction: *REFLEX* (local program). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *PARST* (Nardelli, 1983*b*); *PARSTCIF* (Nardelli, 1992).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1036). Services for accessing these data are described at the back of the journal.

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5-Methoxycarbonyl-6-methyl-4-phenyl-3,4-dihydro-2(1*H*)-pyridone†

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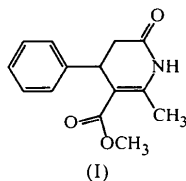
Abstract

Crystals of the title compound, C₁₄H₁₅NO₃ are stabilized by intermolecular hydrogen bonds between the NH group and one O atom of the carbonyl group. The six-membered ring adopts a twist conformation and the lactone ring a planar conformation.

Comment

Much effort has been devoted to the synthesis of 1,4-dihydropyridines (1,4-DHPs) because their calcium antagonist effect is useful in the treatment of cardiovascular diseases (Bossert & Vater, 1989). The presence of an aryl group on C-4 and ester groups on C-3 and C-5 of the 1,4-DHP ring has proved to be a basic requirement for pharmacological activity (Goldmann & Stoltefuss, 1991).

There are no unusual intermolecular or intramolecular distances or angles in the title compound, (I).



† Alternative name: methyl 1,4,5,6-tetrahydro-2-methyl-6-oxo-4-phenylpyridine-3-carboxylate.

There is an intramolecular N—H···O hydrogen bond with O1···H1(−*x*, 1−*y*, 1−*z*) = 2.05 (4) Å. The six-membered ring adopts a twist conformation with asymmetry parameters (Nardelli, 1983*a*) $\Delta C_2(\text{C10—N1}) = 0.005$ (2) and $\Delta C_2(\text{C11}) = 0.073$ (2).

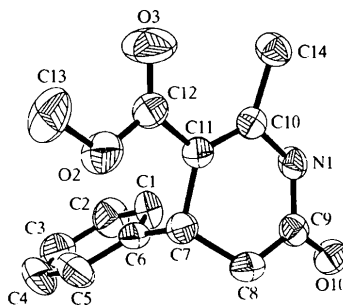


Fig. 1. A view of (I) showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared in a two-step procedure from readily available 3,4-dihydro-2(1*H*)-pyridones by reaction with the Vilsmeier–Haack reagent (Verdecia *et al.*, 1995). Crystals suitable for X-ray analysis were obtained by slow evaporation from ethanol.

Crystal data

C₁₄H₁₅NO₃
M_r = 245.28
 Orthorhombic
Pbca
a = 8.983 (2) Å
b = 21.125 (4) Å
c = 13.196 (3) Å
V = 2504.1 (9) Å³
Z = 8
D_x = 1.301 Mg m^{−3}
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 3–25°
 μ = 0.0918 mm^{−1}
T = 293 (2) K
 Prismatic
 0.15 × 0.11 × 0.05 mm
 Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω –2 θ scans
 Absorption correction: none
 2870 measured reflections
 2200 independent reflections
 960 reflections with $I > 2\sigma(I)$

*R*_{int} = 0.041
 θ_{max} = 25.0°
h = −1 → 10
k = −1 → 25
l = −15 → 1
 2 standard reflections every 100 reflections
 intensity decay: <5%

Refinement

Refinement on *F*²
R(*F*) = 0.050
wR(*F*²) = 0.10
S = 1.165

(Δ/σ)_{max} = 0.059
 $\Delta\rho_{\text{max}}$ = 0.203 e Å^{−3}
 $\Delta\rho_{\text{min}}$ = −0.158 e Å^{−3}
 Extinction correction: none