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3,5-Diacetyl-2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine

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Key indicators

Single-crystal X-ray study
T = 301 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.149
Data-to-parameter ratio = 14.6

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

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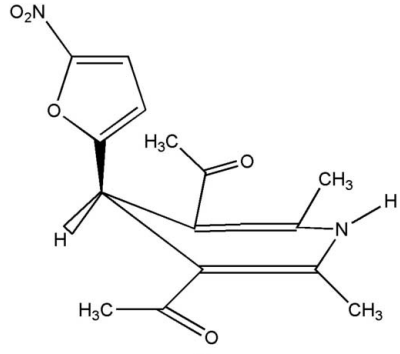
In the molecule of the title compound, $C_{15}H_{16}N_2O_5$, the 5-nitro substituent on the furan ring is rotated from coplanarity with the ring by only 2.1 (2)°. The central 1,4-dihydropyridine (1,4-DHP) ring adopts a flattened boat conformation with the 4-furyl group in a pseudo-axial orientation. Both acetyl substituents of the 1,4-DHP ring at positions 3 and 5 have a synperiplanar conformation. In the crystal structure, intramolecular C—H···O and intermolecular N—H···O and C—H···O hydrogen bonds lead to the formation of infinite sheets of molecules; they seem to be effective in the stabilization of the structure.

Comment

1,4-Dihydropyridine (1,4-DHP) derivatives are an important class of drugs, acting as potent blockers of calcium channels with application in the treatment of various cardiovascular diseases (Triggle *et al.*, 1980; Godfraind *et al.*, 1986; Goldmann & Stoltefuss, 1991). In recent years, active compounds have been prepared by the introduction of the 1,4-DHP unit in condensed systems and the replacement of the ester group with various carbonyl-containing groups, such as amides, nitriles and the acetyl group (Loev *et al.*, 1974; Rose, 1990; Rose & Dräger, 1992). The title compound, (I), has been prepared as a further potentially active 1,4-DHP derivative.



(I)

The structure of (I) is illustrated in Fig. 1. The 1,4-DHP ring adopts a shallow boat conformation, with atoms C4 and N1 deviating by 0.321 (1) and 0.108 (1) Å, respectively, from the base of the boat. The planar furan ring is approximately perpendicular to the DHP ring; the dihedral angle between the plane of the five-membered ring and the plane of the base of the boat (C2/C3/C5/C6) is 82.6 (1)° (Nardelli, 1995). The maximum deviation of these latter four atoms from their mean plane is 0.002 (2) Å. Both acetyl groups in positions 3 and 5 are twisted in the same direction and are synperiplanar (*sp*, *sp*)

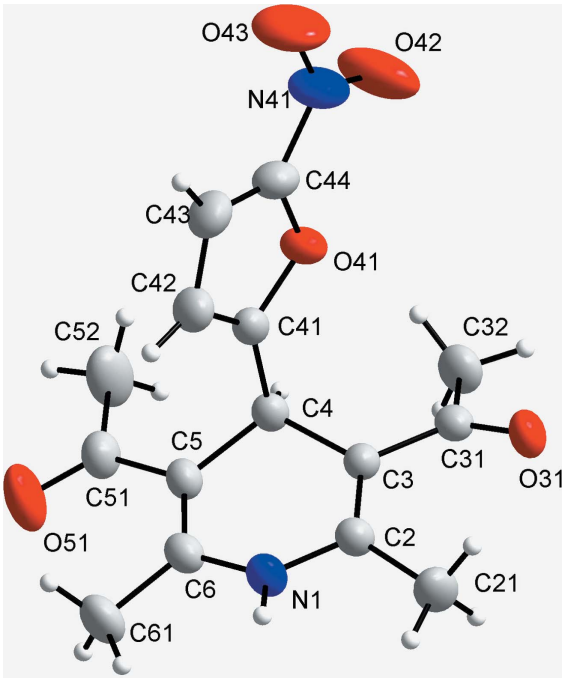


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

to the ring double bonds. One acetyl group is coplanar with the 1,4-DHP, with a C6—C5—C51—O51 torsion angle of 3.8 (3)°, while the second is significantly oriented out of the plane, with a C2—C3—C31—O31 torsion angle of −23.5 (2)°. Fossheim (1986) previously commented that the ester conformation observed in the crystal structure was probably a result of intermolecular hydrogen bonding and crystal-packing interactions. The carbonyl C31═O31 bond length of 1.230 (2) Å is somewhat longer than typical carbonyl bonds, possibly due to the involvement of atom O31 in an intermolecular C—H···O hydrogen bond. The nitro substituent lies above the C4—H bond in a synperiplanar orientation and not over the centre of the boat.

In the crystal structure, intramolecular C—H···O and intermolecular N—H···O and C—H···O hydrogen bonds (Table 1) lead to the formation of infinite sheets of molecules; they seem to be effective in the stabilization of the structure.

Experimental

The title compound was prepared by the condensation reaction of 3-(5-nitrofur-2-ylidene)-1,3-dicarbonyl with 2-amino-2-penten-4-one in dry ethanol (Ilavský & Milata, 1996). Yellow single crystals were obtained by recrystallization from an ethanol solution.

Crystal data

$C_{15}H_{16}N_2O_5$
M_r = 304.30
Monoclinic, $P2_1/c$
a = 8.7056 (7) Å
b = 12.2393 (9) Å
c = 13.8290 (10) Å
β = 93.608 (7)°
V = 1470.6 (2) Å^3
Z = 4
D_x = 1.374 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 301 (2) K
Block, yellow
0.50 × 0.30 × 0.30 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
ω and φ scans
Absorption correction: none
8961 measured reflections
2981 independent reflections
2378 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.149
S = 1.10
2981 reflections
204 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2 + 0.2265P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.006 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O31^{i}	0.86	2.14	2.9562 (17)	158
N1—H1···O43^{ii}	0.86	2.92	3.219 (3)	103
C42—H42···O51^{iii}	0.93	2.42	3.257 (2)	151
C43—H43···O31^{iv}	0.93	2.41	3.324 (2)	170
C21—H21*C*···O31	0.96	2.31	2.817 (2)	113
C32—H32*B*···O41	0.96	2.60	3.314 (2)	131
C61—H61*B*···O51	0.96	2.46	2.792 (3)	100

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

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93, 0.98 and 0.96 Å for aromatic, methine and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,N), where x = 1.5 for methyl and x = 1.2 for all other H atoms.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2002); software used to prepare material for publication: *SHELXL97*.

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