Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Diethyl 2,6-dimethyl-4-[5-(4-nitrophenyl)-2-furyl]-1,4-dihydropyridine-3,5-dicarboxylate

**Viktor Vrábel,[a]* Marek Fronc,[b] Ingrid Svoboda,[b] Štefan Marchalín[c] and Barbora Baumlová[c]**

[a]Department of Analytical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic, [b]Department of Physical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic, and [c]Department of Organic Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic

Correspondence e-mail: vrabel@cvt.stuba.sk

**Key indicators**

Single-crystal X-ray study
*T* = 300 K
Mean σ(C–C) = 0.008 Å
*R* factor = 0.064
*wR* factor = 0.186
Data-to-parameter ratio = 7.8

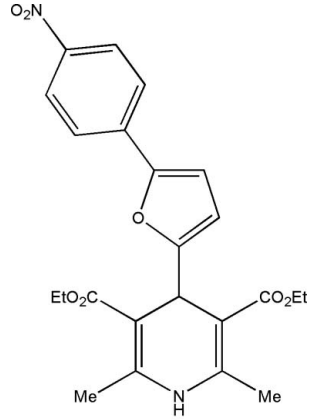
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the crystal structure of the title compound, $C_{23}H_{24}N_2O_7$, molecules are linked by N—H···O hydrogen bonds into infinite chains. The substituted 1,4-dihydropyridine ring has a shallow boat conformation. The 4-nitrophenyl and 2-furyl rings are twisted in the same direction and are approximately perpendicular to the 1,4-dihydropyridine (DHP) ring. The carbonyl groups of the ester groups, at positions 3 and 5 in the 1,4-DHP ring, have different (*cis*/*trans*) configurations with respect to the double bonds in the 1,4-DHP ring

Received 23 June 2005
Accepted 3 October 2005
Online 8 October 2005

## Comment

A wide range of chemical substances influence the flow of $Ca^{2+}$ ions through the channels found in cell membranes. While some compounds, the calcium agonists, activate this flow, other compounds, the calcium antagonists, selectively inhibit the flow of $Ca^{2+}$ ions through the $Ca^{2+}$-conducting channels (Nayler, 1988). 1,4-Dihydropyridine (1,4-DHP) derivatives constitute a major class of calcium agonists or antagonists and have been a target of structure–activity relationship studies (Langs & Triggle, 1985; Langs *et al.*, 1987; Rose, 1989, 1990). Nifedipine is the prototype of this group, and both nifedipine and its structural analogues are used as antihypertensive and anti-anginal drugs. These compounds inhibit the normal excitation–contraction coupling in muscle tissue by blocking the flow of $Ca^{2+}$ ions through plasma membrane channels into the muscle cell (Janis & Triggle, 1983; Triggle *et al.*, 1989). Our interest is in the structure and calcium antagonistic behaviour of condensed derivatives of 1,4-DHP. The crystal structures of some of these derivatives have already been reported (Vrábel *et al.*, 2001, 2003, 2003*a*,*b*). The title compound, (I), has been prepared as another 1,4-DHP derivative with potential biological activity.




(I)

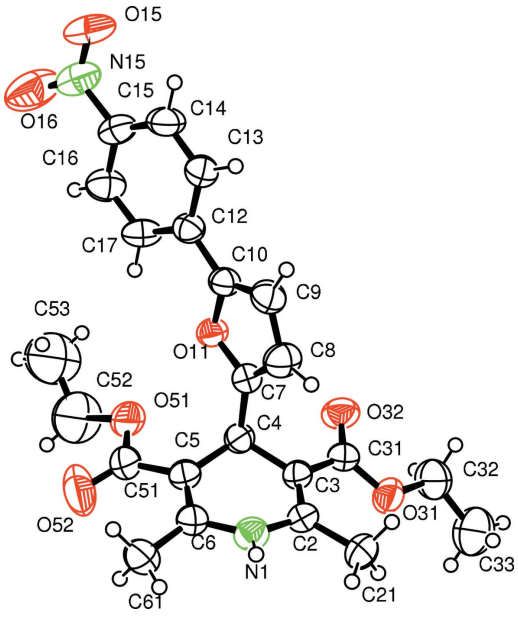



**Figure 1**
Molecular structure of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Atom H4 is behind atom C4.

The molecules of (I) are linked by N—H···O hydrogen bonds. The imine atom N1 (Fig. 1) acts as a donor, *via* H1, to carbonyl atom O32 of a neighbouring molecule (Table 2). The molecules are hydrogen bonded into infinite one-dimensional zigzag chains, which run parallel to the *a* axis. The 1,4-dihydropyridine ring adopts a shallow boat conformation, with atoms C4 and N1 deviating by 0.365 (5) and 0.175 (4) Å, respectively, from the base of the boat. The planar 2-furyl ring is approximately perpendicular to the 1,4-DHP ring. The 4-nitrophenyl and 2-furyl rings, which are twisted in the same direction, are nearly coplanar, with an angle of 10.5 (2)° between the least-squares planes. The ester group at position 3, which has antiperiplanar (*ap*) conformation, is significantly rotated out of the plane defined by atoms C2, C3, C5 and C6, as indicated by the torsion angle C2—C3—C31—O32, while the ester group at position 5, which has synperiplanar (*sp*) conformation, is slightly rotated out of the same plane, as indicated by the torsion angle C6—C5—C51—O52.

## Experimental

Full details of the synthetic procedure have been published by Čupka *et al.* (1987). Yellow needle-like single crystals were prepared by recrystallization from an ethanol solution.

*Crystal data*

$C_{23}H_{24}N_2O_7$
$M_r$ = 440.44
Orthorhombic, $Pca2_1$
$a$ = 12.940 (2) Å
$b$ = 8.143 (1) Å
$c$ = 20.114 (2) Å
$V$ = 2119.4 (5) Å$^3$
$Z$ = 4
$D_x$ = 1.380 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2029 reflections
$\theta$ = 4.5–19.1°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 300 (2) K
Short needle, yellow
0.50 × 0.25 × 0.15 mm

*Data collection*

Oxford Diffraction Xcalibur CCD diffractometer
$\omega$ and $\varphi$ scans
Absorption correction: none
13748 measured reflections
2222 independent reflections
1615 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.059
$\theta_{max}$ = 26.4°
$h = -16 \rightarrow 11$
$k = -10 \rightarrow 10$
$l = -25 \rightarrow 25$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.186
$S$ = 1.00
2222 reflections
286 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1339P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.42 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.50 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C2—C3 | 1.331 (7) | C8—C9 | 1.401 (8) |
| C2—N1 | 1.351 (7) | C10—C12 | 1.444 (7) |
| C4—C7 | 1.486 (7) | C15—N15 | 1.444 (7) |
| C5—C6 | 1.356 (7) | C32—C33 | 1.484 (5) |
| C7—C8 | 1.333 (7) | C52—C53 | 1.447 (5) |
| | | | |
| C3—C2—N1 | 118.6 (5) | O52—C51—O51 | 120.5 (6) |
| C2—C3—C4 | 119.7 (4) | C2—N1—C6 | 123.7 (5) |
| C7—C4—C3 | 109.1 (4) | O15—N15—O16 | 122.6 (6) |
| O32—C31—O31 | 122.5 (5) | C7—O11—C10 | 108.0 (4) |
| | | | |
| C2—C3—C4—C7 | 96.4 (5) | C4—C3—C31—O31 | −167.6 (4) |
| C7—C4—C5—C6 | −96.2 (5) | C6—C5—C51—O52 | −4.1 (12) |
| C9—C10—C12—C13 | 10.0 (9) | C4—C5—C51—O52 | 176.0 (8) |
| C2—C3—C31—O32 | −166.8 (5) | C6—C5—C51—O51 | −179.3 (5) |
| C4—C3—C31—O32 | 9.5 (7) | C16—C15—N15—O15 | −175.3 (6) |
| C2—C3—C31—O31 | 16.1 (7) | C16—C15—N15—O16 | 8.2 (9) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O32$^{i}$ | 0.86 | 2.31 | 3.027 (6) | 141 |

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

All H-atom positions were calculated geometrically and the H atoms were treated as riding atoms (N—H = 0.86 Å; $CH_3$, $CH_2$, CH and aromatic C—H bonds set equal to 0.96, 0.97, 0.98 and 0.93 Å, respectively), with $U_{iso}$(H) values set to 1.2$U_{eq}$ of the parent atom. The C52—C53 and C32—C33 bonds always refined to about 1.3 Å. Therefore, the distances between atoms C32 and C33, and C52 and C53, were restrained to 1.500 (5) Å. The large $U_{eq}$(max)/$U_{eq}$(min) ratio for C and H atoms is caused by large displacements of terminal $CH_3$ groups in the ester chains compared with the C and H atoms that are fixed in the relatively rigid part of the molecule, and may indicate unresolved disorder in the ethyl groups. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows*

(Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Grant Agency of the Slovak Republic (grant Nos. 1/2456/05 and 1/2449/05).

## References

Čupka, P., Bella, J. & Martvoň, A. (1987). *Collect. Czech. Chem. Commun.* **52**, 742–751.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Janis, R. A. & Triggle, D. J. (1983). *J. Med. Chem.* **26**, 775–785.
Langs, D. A., Strong, P. D. & Triggle, D. J. (1987). *Acta Cryst.* C**43**, 707–711.
Langs, D. A. & Triggle, D. J. (1985). *Mol. Pharmacol.* **27**, 257–274.
Nayler, W. G. (1988). *Calcium Antagonists*. New York: Academic Press.
Oxford Diffraction (2002). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Köln, Germany.
Rose, U. (1989). *Arzneim.-Forsch. (Drug Res.)*, **39**, 1393–1398.
Rose, U. (1990). *Arch. Pharm. (Weinheim)*, **323**, 281–286.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Triggle, D. J., Langs, D. A. & Janis, R. A. (1989). *Med. Res. Rev.* **9**, 123–180.
Vrábel, V., Kožíšek, J. & Marchalín, Š. (2003*a*). *Acta Cryst.* E**59**, o376–o377.
Vrábel, V., Kožíšek, J. & Marchalín, Š. (2003*b*). *Acta Cryst.* E**59**, o1964–o1966.
Vrábel, V., Lehotay, J., Oktavec, D. & Marchalín, Š. (2001). *Acta Cryst.* C**57**, 1073–1074.
Vrábel, V., Oktavec, D. & Marchalín, Š. (2003). *Acta Cryst.* E**59**, o306–o307.