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Hydrogen-bonded supramolecular structures of three related 4-(5-nitro-2-furyl)-1,4-dihydropyridines

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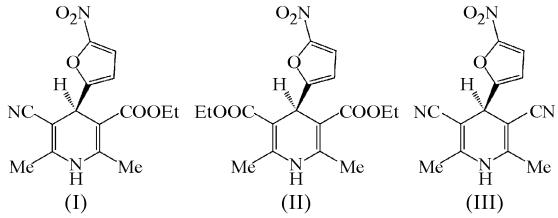
In ethyl 5-cyano-2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3-carboxylate, $C_{15}H_{15}N_3O_5$, the molecules are linked into chains by a single N—H···O hydrogen bond. The molecules in diethyl 2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3,5-dicarboxylate, $C_{17}H_{20}N_2O_7$, are linked by a combination of one N—H···O hydrogen bond and two C—H···O hydrogen bonds into sheets built from equal numbers of $R_2^2(17)$ and $R_4^4(18)$ rings. In 2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3,5-dicarbonitrile, $C_{13}H_{10}N_4O_3$, the molecules are linked by a combination of a three-centre N—H···$(O)_2$ hydrogen bond and two independent two-centre C—H···O hydrogen bonds into complex sheets containing four types of ring.

Comment

1,4-Dihydropyridine (1,4-DHP) derivatives, which are analogues of NADH coenzymes, are an important class of drugs, acting as potent blockers of calcium channels with application in the treatment of various cardiovascular diseases (Bou *et al.*, 1983; Godfraind *et al.*, 1986; Wagner *et al.*, 1988). In addition, 1,4-DHP compounds such as nifedipine, nisoldipine and nicardipine exhibit potential trypanocidal activity, inhibiting culture growth and oxygen uptake in *Trypanosoma cruzi* epimastigotes, the parasite causing Chagas' disease (Núñez-Vergara *et al.*, 1997, 1998). The drug action can be associated with the reduction of the nitro groups in these compounds. The presence of ester groups at the 3- and 5-positions in the 1,4-dihydropyridine ring is of crucial importance for the pharmaceutical effects. It has been suggested that these groups form hydrogen bonds with the receptor site (Goldmann & Stoltefuss, 1991). Previous studies of the title compounds, namely ethyl 5-cyano-2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3-carboxylate, (I), diethyl 2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3,5-dicarboxylate, (II), and



2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3,5-dicarbonitrile, (III), have involved their NMR spectra (DaSilva *et al.*, 2005) and electroreduction of the nitro groups (Argüello *et al.*, 2005). The NMR study revealed the non-equivalence of the methylene H atoms in the ethoxycarbonyl groups, and we now report the molecular and supramolecular structures of three representative examples, *viz.* (I)–(III).

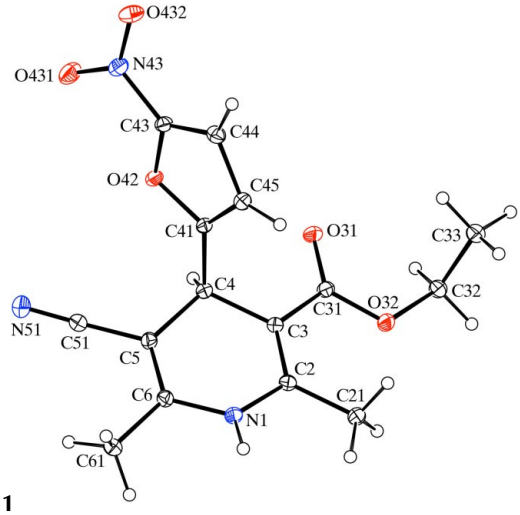


Figure 1
The *R* enantiomer of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

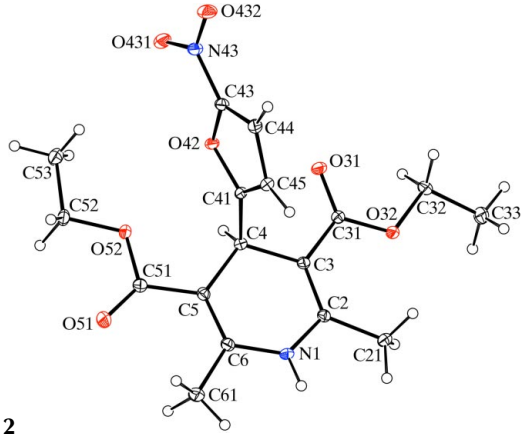


Figure 2
The molecule of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

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In each of compounds (I)–(III) (Figs. 1–3), the 1,4-dihydropyrimidine ring adopt a flat-boat conformation, as generally observed when this ring system carries an aryl or heteroaryl substituent at position 4 (Fossheim *et al.*, 1982; Lokaj *et al.*, 1991; Kožíšek *et al.*, 1993), although an example containing a planar ring has recently been reported (Mahendra *et al.*, 2003). In each compound, the distortion of the ring from planarity is modest, with total puckering amplitudes (Cremer & Pople, 1975) of only 0.190 (2), 0.105 (2) and 0.089 (2) Å for (I)–(III), respectively. In (I), atom C4 is a stereogenic centre and the selected reference molecule has the *R* configuration at this centre. However, the centrosymmetric space group accommodates equal numbers of *R* and *S* molecules.

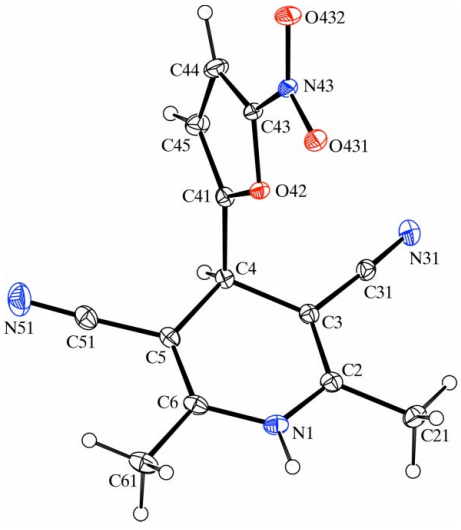

Figure 3
The molecule of (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

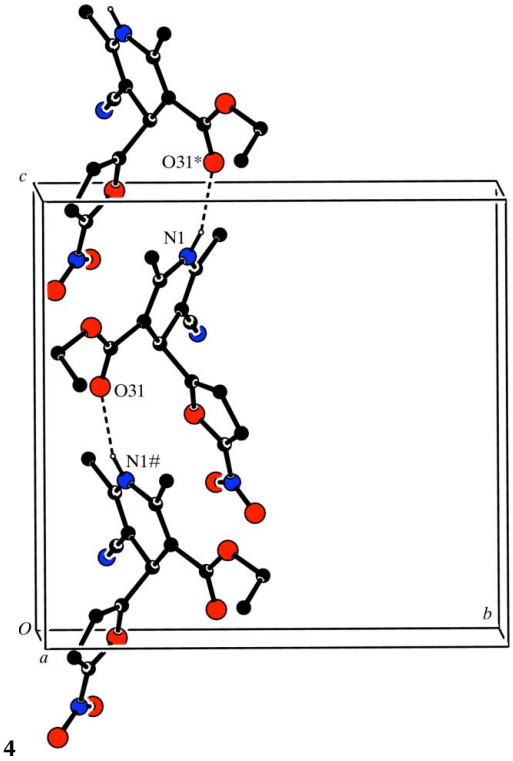

Figure 4
Part of the crystal structure of (I), showing the formation of a $C(6)$ chain along [001]. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ and $(x, \frac{1}{2}-y, -\frac{1}{2}+z)$, respectively.

The supramolecular structures of compounds (I)–(III) are all different and each is based on a different selection of hydrogen bonds. It is of interest to note the changes in the supramolecular structures which are associated with the changes in the substituents at positions 3 and 5 of the dihydropyridine ring.

In compound (I), the molecules are linked into simple chains by a single hydrogen bond (Table 1). Atom N1 in the molecule at (x, y, z) acts as hydrogen-bond donor to carbonyl atom O31 in the molecule at $(x, \frac{1}{2}-y, \frac{1}{2}+z)$, thereby producing a $C(6)$ (Bernstein *et al.*, 1995) chain running parallel to the [001] direction and generated by the *c*-glide plane at $y = \frac{1}{4}$ (Fig. 4). Two such chains, running antiparallel to one another, pass through each unit cell, but there are no direction-specific interactions between adjacent chains.

The formation of the sheet structure in compound (II) can readily be analysed in terms of two one-dimensional substructures, one involving both N—H···O and C—H···O hydrogen bonds, and the other only a C—H···O hydrogen bond (Table 2). In the first substructure, atoms N1 and C45 in the molecule at (x, y, z) act as hydrogen-bond donors to atoms O31 and O431, respectively, in the molecule at $(x, \frac{1}{2}-y, \frac{1}{2}+z)$, so forming a chain of edge-fused $R_2^2(17)$ rings running parallel to the [001] direction and generated by the *c*-glide plane at $y = \frac{1}{4}$ (Fig. 5). The second substructure is much simpler: atom C44 in the molecule at (x, y, z) acts as hydrogen-bond donor to ester atom O32 in the molecule at $(1+x, y, z)$, so generating by translation a simple $C(8)$ chain running parallel to the [100] direction. The combination of these two one-dimensional motifs then generates an (010) sheet consisting of alternating columns, all parallel to [001], of $R_2^2(17)$ and $R_4^4(18)$ rings (Fig. 6). Two sheets of this type, related to one another by inversion, pass through each unit cell. The only direction-

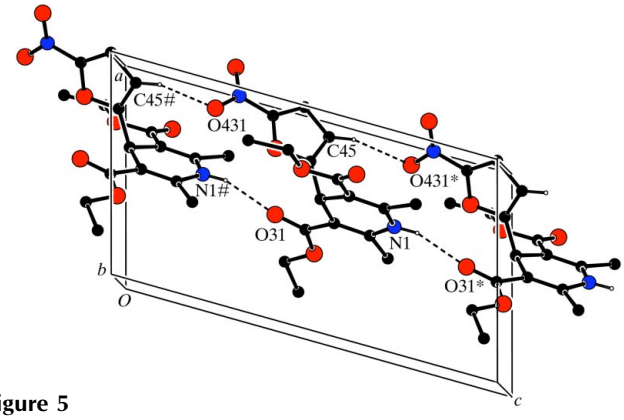

Figure 5
Part of the crystal structure of (II), showing the formation of a chain of edge-fused $R_2^2(17)$ rings along [001]. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ and $(x, \frac{1}{2}-y, -\frac{1}{2}+z)$, respectively.

specific interaction of possible significance is a C—H···π(furan) hydrogen bond (Table 2). Atom C52 in the molecule at (x, y, z), which lies in the sheet generated by the glide planes at $y = \frac{1}{4}$, acts as hydrogen-bond donor to the furyl ring of the molecule at ($2 - x$, $1 - y$, $1 - z$), which forms part of the sheet generated by the glide plane at $y = \frac{3}{4}$. Propagation of this interaction then links each (010) sheet to the two adjacent sheets.

The supramolecular structure of compound (III) consists of hydrogen-bonded sheets containing four types of ring. However, as for (II), the formation of the sheet in (III) is readily analysed in terms of simpler zero- and one-dimensional substructures. The basic building block in the supramolecular structure of (III) can be regarded as a cyclic centrosymmetric dimer. Atom N1 in the molecule at (x, y, z) acts as hydrogen-bond donor to both O42 and O431 in the molecule at ($1 - x$, $1 - y$, $1 - z$), forming an effectively planar three-centre N—H···$(O)_2$ system (Table 3). The resulting dimer centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) contains an $R_2^2(14)$ ring generated by the shorter component of the three-centre hydrogen bond and two $R_1^2(5)$ rings generated by both components (Fig. 7). Two independent C—H···O hydrogen bonds then link these dimers into sheets, and it is convenient to consider the action of each hydrogen bond in turn. Atom C4 in the molecule at (x, y, z), part of the dimer centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$), acts as hydrogen-bond donor to atom O431 in the molecule at (x, $1 + y$, z), part of the dimer centred at ($\frac{1}{2}$, $\frac{3}{2}$, $\frac{1}{2}$). Propagation of this hydrogen bond by translation and inversion then generates a chain of edge-fused rings along ($\frac{1}{2}$, y, $\frac{1}{2}$), with $R_2^2(14)$ rings centred at ($\frac{1}{2}$, $n + \frac{1}{2}$, $\frac{1}{2}$) (n = zero or integer) and $R_4^4(14)$ rings centred at ($\frac{1}{2}$, n, $\frac{1}{2}$) (n = zero or integer) (Fig. 8). Finally, these chains are linked by the second C—H···O hydrogen bond. Atom C44 in the molecule at (x, y, z), which lies in the chain of rings along ($\frac{1}{2}$, y, $\frac{1}{2}$), acts as hydrogen-bond donor to atom O432 in the molecule at ($-x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$), which itself lies in the chain of rings along ($-\frac{1}{2}$, y, 1). Propagation by the space group of this hydrogen bond then links the [010] chains of rings into a (102) sheet (Fig. 8). There are no direction-specific interactions between adjacent sheets.

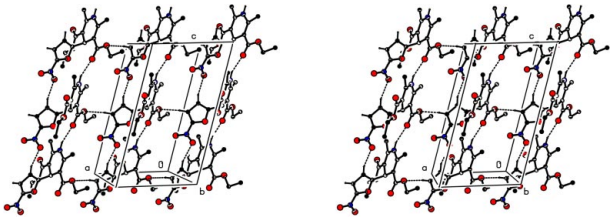

Figure 6
Stereoview of part of the crystal structure of (II), showing the formation of an (010) sheet built from $R_2^2(17)$ and $R_4^4(18)$ rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

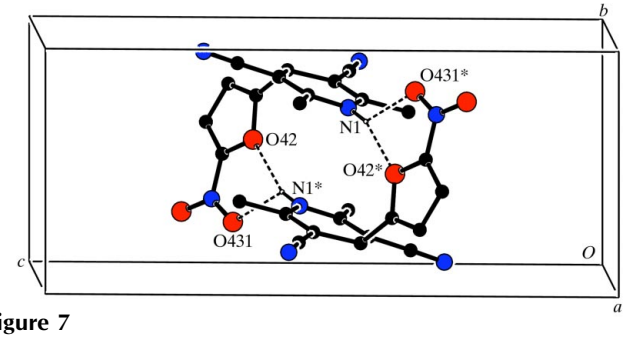

Figure 7
Part of the crystal structure of (III), showing the formation of a cyclic centrosymmetric dimer. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position ($1 - x$, $1 - y$, $1 - z$).

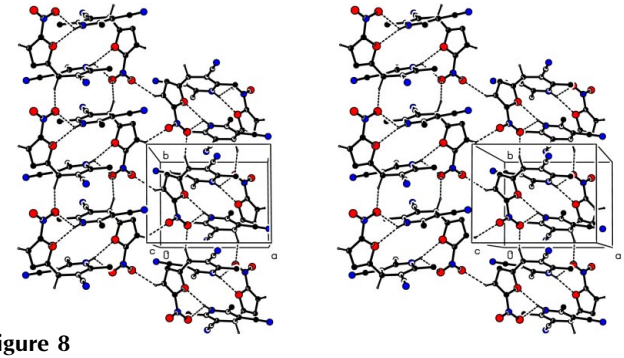

Figure 8
Stereoview of part of the crystal structure of (III), showing the formation of a (102) sheet built from $R_1^2(5)$, $R_2^2(14)$, $R_3^3(14)$ and $R_4^4(14)$ rings. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

Experimental

Samples of compounds (I)–(III) were prepared according to published procedures (Hafiz *et al.*, 1999; DaSilva *et al.*, 2005; Argüello *et al.*, 2005). Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of solutions in ethanol. Attempts to cut small fragments from the rather large blocks of compound (III) led to shattering of the crystals.

Compound (I)

Crystal data

$C_{15}H_{15}N_3O_5$
$M_r = 317.30$
Monoclinic, $P2_1/c$
$a = 8.0214$ (3) Å
$b = 13.7477$ (4) Å
$c = 13.2847$ (4) Å
$\beta = 95.3019$ (17)°
$V = 1458.71$ (8) Å^3
$Z = 4$
$D_x = 1.445$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3361 reflections
$\theta = 3.0$–$27.6°$
$\mu = 0.11$ mm^{-1}
$T = 120$ (2) K
Block, brown
0.14 × 0.12 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.979$, $T_{max} = 0.991$
17958 measured reflections
3361 independent reflections
2614 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 27.6°$
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 17$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.132$
$S = 1.06$
3361 reflections
211 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.6243P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O31^{i}	0.88	2.12	2.953 (2)	157

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

Compound (II)

Crystal data

$C_{17}H_{20}N_2O_7$
M_r = 364.35
Monoclinic, $P2_1/c$
a = 8.0511 (2) Å
b = 15.173 (4) Å
c = 14.470 (4) Å
β = 105.760 (2)°
V = 1701.2 (7) Å^3
Z = 4
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3898 reflections
θ = 2.9–27.5°
μ = 0.11 mm^{-1}
T = 120 (2) K
Plate, yellow
0.26 × 0.22 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.969, T_{max} = 0.993
17840 measured reflections
3898 independent reflections
3073 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.5°
$h = -10 \rightarrow 7$
$k = -19 \rightarrow 19$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.06
3898 reflections
239 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.8476P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

Cg is the centroid of the C41/O42/C43/C44/C45 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O31^{i}	0.86	2.18	2.986 (2)	157
C44—H44···O32^{ii}	0.95	2.38	3.330 (2)	174
C45—H45···O431^{i}	0.95	2.45	3.369 (2)	163
C52—H52A···Cg^{iv}	0.99	2.68	3.473 (2)	134

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

Compound (III)

Crystal data

$C_{13}H_{10}N_4O_3$
M_r = 270.25
Monoclinic, $P2_1/c$
a = 9.5651 (3) Å
b = 7.5735 (2) Å
c = 17.6385 (5) Å
β = 96.2570 (13)°
V = 1270.14 (6) Å^3
Z = 4
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2907 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Block, colourless
0.90 × 0.34 × 0.22 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.906, T_{max} = 0.977
16132 measured reflections
2907 independent reflections
2333 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°
$h = -12 \rightarrow 12$
$k = -9 \rightarrow 9$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.05
2907 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.4785P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O42^{i}	0.88	2.35	3.2019 (15)	162
N1—H1···O431^{i}	0.88	2.32	2.9390 (16)	128
C4—H4···O431^{ii}	1.00	2.47	3.3262 (16)	143
C44—H44···O432^{iii}	0.95	2.32	3.0446 (18)	132

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

For each of compounds (I), (II) and (III), the space group $P2_1/c$ was uniquely assigned from the systematic absences. All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aromatic), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (aliphatic CH) and N—H = 0.88 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(methyl C).

For all three compounds, data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, England; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1887). Services for accessing these data are described at the back of the journal.

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supporting information

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Hydrogen-bonded supramolecular structures of three related 4-(5-nitro-2-furyl)-1,4-dihydropyridines

Antonio Quesada, Jacqueline Argüello, Juan A. Squella, James L. Wardell, John N. Low and Christopher Glidewell

Computing details

For all compounds, data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

(I) ethyl 5-cyano-2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3-carboxylate

Crystal data

$C_{15}H_{15}N_3O_5$
$M_r = 317.30$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.0214\ (3)$ Å
$b = 13.7477\ (4)$ Å
$c = 13.2847\ (4)$ Å
$\beta = 95.3019\ (17)°$
$V = 1458.71\ (8)$ Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.445$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3361 reflections
$\theta = 3.0$–$27.6°$
$\mu = 0.11$ mm^{-1}
$T = 120$ K
Block, brown
0.14 × 0.12 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR91 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.979$, $T_{max} = 0.991$
17958 measured reflections
3361 independent reflections
2614 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.0°$
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 17$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.132$
$S = 1.06$
3361 reflections
211 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.6243P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O31	0.77237 (15)	0.12586 (9)	0.57702 (9)	0.0223 (3)
O32	0.94984 (15)	0.10372 (9)	0.71630 (9)	0.0226 (3)
O42	0.48921 (14)	0.33505 (8)	0.50276 (9)	0.0194 (3)
O431	0.29866 (16)	0.38423 (10)	0.34165 (10)	0.0328 (3)
O432	0.50117 (17)	0.46435 (10)	0.28064 (9)	0.0313 (3)
N1	0.69121 (17)	0.31836 (10)	0.86343 (11)	0.0198 (3)
N43	0.44161 (19)	0.41652 (10)	0.34731 (11)	0.0234 (3)
N51	0.16274 (19)	0.34964 (13)	0.66906 (13)	0.0321 (4)
C2	0.7913 (2)	0.25597 (11)	0.81411 (12)	0.0178 (3)
C3	0.7368 (2)	0.22212 (12)	0.72070 (12)	0.0179 (3)
C4	0.5763 (2)	0.25943 (12)	0.66380 (12)	0.0179 (3)
C5	0.4669 (2)	0.30909 (12)	0.73598 (13)	0.0190 (4)
C6	0.5276 (2)	0.33856 (12)	0.82929 (13)	0.0187 (3)
C21	0.9568 (2)	0.23557 (13)	0.87286 (13)	0.0225 (4)
C31	0.8207 (2)	0.14805 (12)	0.66446 (12)	0.0189 (4)
C32	1.0373 (2)	0.02987 (13)	0.66327 (13)	0.0224 (4)
C33	1.1581 (2)	0.07439 (14)	0.59697 (14)	0.0258 (4)
C41	0.6142 (2)	0.32718 (12)	0.58037 (12)	0.0185 (3)
C43	0.5491 (2)	0.39835 (12)	0.43664 (12)	0.0193 (4)
C44	0.7033 (2)	0.43184 (12)	0.46804 (13)	0.0218 (4)
C45	0.7457 (2)	0.38513 (12)	0.56259 (13)	0.0210 (4)
C51	0.2976 (2)	0.33115 (13)	0.70004 (13)	0.0226 (4)
C61	0.4281 (2)	0.39324 (13)	0.90073 (13)	0.0243 (4)
H1	0.7341	0.3466	0.9194	0.024*
H4	0.5133	0.2024	0.6329	0.022*
H21A	1.0477	0.2575	0.8339	0.034*
H21B	0.9678	0.1655	0.8855	0.034*
H21C	0.9628	0.2704	0.9375	0.034*
H32A	0.9546	−0.0099	0.6214	0.027*
H32B	1.0987	−0.0137	0.7132	0.027*
H33A	1.2409	0.1131	0.6384	0.039*
H33B	1.0972	0.1164	0.5465	0.039*
H33C	1.2152	0.0228	0.5626	0.039*
H44	0.7691	0.4767	0.4344	0.026*
H45	0.8466	0.3930	0.6052	0.025*
H61A	0.3085	0.3819	0.8828	0.037*
H61B	0.4520	0.4629	0.8962	0.037*
H61C	0.4589	0.3707	0.9699	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O31	0.0246 (6)	0.0267 (6)	0.0151 (6)	0.0016 (5)	−0.0007 (5)	−0.0020 (5)
O32	0.0223 (6)	0.0269 (6)	0.0182 (6)	0.0065 (5)	−0.0007 (5)	−0.0029 (5)
O42	0.0194 (6)	0.0228 (6)	0.0154 (6)	0.0000 (5)	−0.0012 (5)	0.0017 (5)
O431	0.0273 (7)	0.0398 (8)	0.0294 (7)	−0.0043 (6)	−0.0071 (6)	0.0056 (6)
O432	0.0416 (8)	0.0309 (7)	0.0209 (7)	−0.0007 (6)	0.0010 (6)	0.0090 (5)
N1	0.0197 (7)	0.0233 (7)	0.0160 (7)	0.0001 (6)	−0.0004 (5)	−0.0037 (6)
N43	0.0277 (8)	0.0218 (7)	0.0200 (8)	0.0034 (6)	−0.0021 (6)	−0.0003 (6)
N51	0.0217 (8)	0.0426 (10)	0.0318 (9)	0.0042 (7)	0.0016 (7)	0.0039 (7)
C2	0.0189 (8)	0.0180 (8)	0.0168 (8)	−0.0009 (6)	0.0028 (6)	0.0012 (6)
C3	0.0170 (8)	0.0217 (8)	0.0152 (8)	0.0006 (6)	0.0019 (6)	0.0010 (6)
C4	0.0177 (8)	0.0204 (8)	0.0153 (8)	0.0007 (6)	0.0002 (6)	−0.0002 (6)
C5	0.0162 (8)	0.0211 (8)	0.0199 (8)	0.0007 (6)	0.0022 (6)	0.0029 (7)
C6	0.0180 (8)	0.0192 (8)	0.0191 (8)	0.0008 (6)	0.0028 (6)	0.0017 (6)
C21	0.0190 (8)	0.0297 (9)	0.0182 (8)	−0.0003 (7)	−0.0015 (7)	−0.0035 (7)
C31	0.0187 (8)	0.0216 (8)	0.0162 (8)	−0.0009 (6)	0.0002 (6)	0.0018 (6)
C32	0.0247 (9)	0.0231 (8)	0.0195 (9)	0.0056 (7)	0.0022 (7)	−0.0032 (7)
C33	0.0261 (9)	0.0293 (9)	0.0223 (9)	0.0052 (7)	0.0046 (7)	−0.0001 (7)
C41	0.0175 (8)	0.0240 (8)	0.0136 (8)	0.0027 (6)	−0.0009 (6)	−0.0017 (6)
C43	0.0229 (8)	0.0200 (8)	0.0150 (8)	0.0031 (6)	0.0014 (7)	0.0021 (6)
C44	0.0238 (8)	0.0209 (8)	0.0214 (9)	−0.0001 (7)	0.0056 (7)	0.0011 (7)
C45	0.0193 (8)	0.0242 (8)	0.0191 (9)	0.0000 (7)	0.0004 (7)	−0.0004 (7)
C51	0.0212 (9)	0.0271 (9)	0.0201 (9)	0.0011 (7)	0.0044 (7)	0.0019 (7)
C61	0.0252 (9)	0.0285 (9)	0.0199 (9)	0.0046 (7)	0.0050 (7)	−0.0013 (7)

Geometric parameters (Å, º)

N1—C6	1.377 (2)	C4—C5	1.520 (2)
N1—C2	1.381 (2)	C4—H4	1.00
N1—H1	0.88	C41—C45	1.359 (2)
C2—C3	1.359 (2)	C41—O42	1.3745 (19)
C2—C21	1.503 (2)	O42—C43	1.356 (2)
C21—H21A	0.98	C43—C44	1.349 (2)
C21—H21B	0.98	C43—N43	1.423 (2)
C21—H21C	0.98	N43—O431	1.225 (2)
C3—C31	1.463 (2)	N43—O432	1.2341 (19)
C3—C4	1.520 (2)	C44—C45	1.424 (2)
C31—O31	1.228 (2)	C44—H44	0.95
C31—O32	1.3369 (19)	C45—H45	0.95
C32—O32	1.453 (2)	C5—C6	1.351 (2)
C32—C33	1.499 (3)	C5—C51	1.430 (2)
C32—H32A	0.99	C51—N51	1.149 (2)
C32—H32B	0.99	C6—C61	1.497 (2)
C33—H33A	0.98	C61—H61A	0.98
C33—H33B	0.98	C61—H61B	0.98
C33—H33C	0.98	C61—H61C	0.98

C4—C41	1.500 (2)

C6—N1—C2	123.19 (14)	C5—C4—C3	110.45 (13)
C6—N1—H1	118.4	C41—C4—H4	108.2
C2—N1—H1	118.4	C5—C4—H4	108.2
C3—C2—N1	119.58 (15)	C3—C4—H4	108.2
C3—C2—C21	127.19 (16)	C45—C41—O42	110.32 (14)
N1—C2—C21	113.21 (14)	C45—C41—C4	134.88 (15)
C2—C21—H21A	109.5	O42—C41—C4	114.80 (14)
C2—C21—H21B	109.5	C43—O42—C41	105.01 (12)
H21A—C21—H21B	109.5	C44—C43—O42	112.85 (14)
C2—C21—H21C	109.5	C44—C43—N43	131.71 (16)
H21A—C21—H21C	109.5	O42—C43—N43	115.43 (14)
H21B—C21—H21C	109.5	O431—N43—O432	124.82 (15)
C2—C3—C31	125.46 (15)	O431—N43—C43	118.61 (15)
C2—C3—C4	121.65 (15)	O432—N43—C43	116.56 (15)
C31—C3—C4	112.90 (13)	C43—C44—C45	104.84 (15)
O31—C31—O32	122.38 (15)	C43—C44—H44	127.6
O31—C31—C3	122.41 (15)	C45—C44—H44	127.6
O32—C31—C3	115.17 (14)	C41—C45—C44	106.98 (15)
O32—C32—C33	111.52 (14)	C41—C45—H45	126.5
O32—C32—H32A	109.3	C44—C45—H45	126.5
C33—C32—H32A	109.3	C6—C5—C51	119.55 (16)
O32—C32—H32B	109.3	C6—C5—C4	122.23 (14)
C33—C32—H32B	109.3	C51—C5—C4	118.06 (15)
H32A—C32—H32B	108.0	N51—C51—C5	178.4 (2)
C31—O32—C32	117.04 (13)	C5—C6—N1	119.56 (15)
C32—C33—H33A	109.5	C5—C6—C61	124.33 (15)
C32—C33—H33B	109.5	N1—C6—C61	116.11 (14)
H33A—C33—H33B	109.5	C6—C61—H61A	109.5
C32—C33—H33C	109.5	C6—C61—H61B	109.5
H33A—C33—H33C	109.5	H61A—C61—H61B	109.5
H33B—C33—H33C	109.5	C6—C61—H61C	109.5
C41—C4—C5	110.81 (13)	H61A—C61—H61C	109.5
C41—C4—C3	110.89 (13)	H61B—C61—H61C	109.5

C6—N1—C2—C3	9.2 (2)	C4—C41—O42—C43	−179.70 (14)
C6—N1—C2—C21	−172.45 (15)	C41—O42—C43—C44	−0.68 (18)
N1—C2—C3—C31	−173.06 (15)	C41—O42—C43—N43	178.48 (14)
C21—C2—C3—C31	8.8 (3)	C44—C43—N43—O431	−172.54 (18)
N1—C2—C3—C4	6.7 (2)	O42—C43—N43—O431	8.5 (2)
C21—C2—C3—C4	−171.46 (15)	C44—C43—N43—O432	7.5 (3)
C2—C3—C31—O31	−174.16 (17)	O42—C43—N43—O432	−171.46 (14)
C4—C3—C31—O31	6.1 (2)	O42—C43—C44—C45	0.31 (19)
C2—C3—C31—O32	8.2 (2)	N43—C43—C44—C45	−178.67 (17)
C4—C3—C31—O32	−171.54 (14)	O42—C41—C45—C44	−0.62 (19)
O31—C31—O32—C32	2.6 (2)	C4—C41—C45—C44	180.00 (18)
C3—C31—O32—C32	−179.77 (14)	C43—C44—C45—C41	0.19 (19)

C33—C32—O32—C31	80.06 (18)	C41—C4—C5—C6	−106.28 (18)
C2—C3—C4—C41	104.92 (18)	C3—C4—C5—C6	17.0 (2)
C31—C3—C4—C41	−75.33 (17)	C41—C4—C5—C51	69.09 (19)
C2—C3—C4—C5	−18.3 (2)	C3—C4—C5—C51	−167.61 (14)
C31—C3—C4—C5	161.42 (14)	C51—C5—C6—N1	−179.22 (15)
C5—C4—C41—C45	98.5 (2)	C4—C5—C6—N1	−3.9 (2)
C3—C4—C41—C45	−24.5 (3)	C51—C5—C6—C61	0.7 (3)
C5—C4—C41—O42	−80.86 (17)	C4—C5—C6—C61	176.00 (16)
C3—C4—C41—O42	156.10 (14)	C2—N1—C6—C5	−10.6 (2)
C45—C41—O42—C43	0.79 (18)	C2—N1—C6—C61	169.48 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O31^{i}	0.88	2.12	2.953 (2)	157

Symmetry code: (i) *x*, −*y*+1/2, *z*+1/2.

(II) diethyl 2,6-dimethyl-4-(5-nitro-2-furyl)-1,4-dihydropyridine-3,5-dicarboxylate

Crystal data

$C_{17}H_{20}N_2O_7$
M_r = 364.35
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 8.0511 (2) Å
b = 15.173 (4) Å
c = 14.470 (4) Å
β = 105.760 (2)°
V = 1701.2 (7) Å^3
Z = 4
$F(000)$ = 768
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3898 reflections
θ = 2.9–27.5°
μ = 0.11 mm^{-1}
T = 120 K
Plate, yellow
0.26 × 0.22 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR91 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.969, T_{max} = 0.993
17840 measured reflections
3898 independent reflections
3073 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.5°, θ_{min} = 2.9°
h = −10→7
k = −19→19
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.06
3898 reflections
239 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.8476P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O31	0.51174 (15)	0.19208 (8)	0.39397 (8)	0.0203 (3)
O32	0.38877 (15)	0.11796 (7)	0.49219 (8)	0.0174 (3)
O42	0.80303 (15)	0.31933 (7)	0.39983 (8)	0.0165 (3)
O431	0.90638 (17)	0.34441 (8)	0.24520 (9)	0.0254 (3)
O432	1.11994 (18)	0.25194 (9)	0.29460 (10)	0.0297 (3)
O51	0.77729 (19)	0.55027 (8)	0.61859 (9)	0.0316 (3)
O52	0.73600 (15)	0.48699 (7)	0.47419 (8)	0.0194 (3)
N1	0.60132 (19)	0.30689 (9)	0.70679 (10)	0.0186 (3)
N43	0.99410 (19)	0.29324 (10)	0.30501 (10)	0.0206 (3)
C2	0.5338 (2)	0.24179 (10)	0.64101 (11)	0.0155 (3)
C3	0.5438 (2)	0.25016 (10)	0.54904 (11)	0.0147 (3)
C4	0.6314 (2)	0.32908 (10)	0.51643 (11)	0.0152 (3)
C5	0.6769 (2)	0.40033 (10)	0.59366 (11)	0.0156 (3)
C6	0.6669 (2)	0.38535 (11)	0.68400 (12)	0.0176 (4)
C21	0.4572 (2)	0.16731 (11)	0.68472 (12)	0.0211 (4)
C31	0.4822 (2)	0.18551 (10)	0.47192 (11)	0.0149 (3)
C32	0.3187 (2)	0.05729 (11)	0.41336 (12)	0.0206 (4)
C33	0.2033 (2)	−0.00630 (12)	0.44639 (13)	0.0255 (4)
C41	0.7893 (2)	0.29747 (10)	0.48935 (11)	0.0154 (3)
C43	0.9483 (2)	0.27909 (11)	0.39198 (12)	0.0175 (3)
C44	1.0287 (2)	0.23332 (11)	0.47184 (12)	0.0192 (4)
C45	0.9246 (2)	0.24588 (10)	0.53550 (12)	0.0177 (3)
C51	0.7346 (2)	0.48642 (11)	0.56723 (12)	0.0185 (3)
C52	0.8006 (2)	0.56725 (11)	0.44191 (12)	0.0205 (4)
C53	0.7937 (3)	0.55517 (12)	0.33795 (13)	0.0270 (4)
C61	0.7228 (3)	0.44752 (12)	0.76791 (12)	0.0257 (4)
H1	0.6024	0.2981	0.7657	0.022*
H12A	0.3331	0.1636	0.6537	0.032*
H12B	0.4761	0.1781	0.7536	0.032*
H12C	0.5127	0.1118	0.6754	0.032*
H13A	0.4133	0.0252	0.3963	0.025*
H13B	0.2520	0.0900	0.3561	0.025*
H14	0.5495	0.3549	0.4580	0.018*
H33A	0.2712	−0.0389	0.5025	0.038*
H33B	0.1526	−0.0478	0.3945	0.038*
H33C	0.1111	0.0263	0.4637	0.038*
H44	1.1322	0.2000	0.4828	0.023*
H45	0.9454	0.2226	0.5986	0.021*
H52A	0.9208	0.5784	0.4801	0.025*
H52B	0.7286	0.6181	0.4497	0.025*
H52C	0.8662	0.5050	0.3312	0.040*
H52D	0.8362	0.6086	0.3139	0.040*
H52E	0.6743	0.5440	0.3009	0.040*
H61A	0.8443	0.4633	0.7773	0.039*
H61B	0.7086	0.4188	0.8260	0.039*

H61C	0.6518	0.5009	0.7550	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O31	0.0246 (7)	0.0232 (6)	0.0153 (6)	−0.0031 (5)	0.0091 (5)	−0.0019 (5)
O32	0.0207 (6)	0.0170 (6)	0.0160 (6)	−0.0041 (5)	0.0074 (5)	−0.0027 (4)
O431	0.0309 (7)	0.0283 (7)	0.0186 (6)	0.0012 (6)	0.0094 (5)	0.0033 (5)
O432	0.0319 (8)	0.0331 (7)	0.0309 (7)	0.0066 (6)	0.0200 (6)	−0.0011 (6)
O51	0.0504 (9)	0.0212 (7)	0.0264 (7)	−0.0119 (6)	0.0161 (6)	−0.0065 (5)
O52	0.0263 (7)	0.0151 (6)	0.0183 (6)	−0.0046 (5)	0.0087 (5)	−0.0003 (4)
N1	0.0259 (8)	0.0193 (7)	0.0128 (6)	−0.0024 (6)	0.0088 (6)	−0.0022 (5)
N43	0.0238 (8)	0.0219 (7)	0.0192 (7)	−0.0033 (6)	0.0110 (6)	−0.0038 (6)
C2	0.0155 (8)	0.0145 (7)	0.0174 (8)	0.0010 (6)	0.0059 (6)	−0.0007 (6)
C3	0.0137 (8)	0.0149 (7)	0.0165 (8)	0.0005 (6)	0.0060 (6)	−0.0007 (6)
C4	0.0165 (8)	0.0156 (7)	0.0141 (7)	−0.0001 (6)	0.0054 (6)	0.0000 (6)
C5	0.0154 (8)	0.0145 (7)	0.0173 (8)	0.0003 (6)	0.0050 (6)	−0.0011 (6)
C6	0.0185 (9)	0.0164 (8)	0.0190 (8)	−0.0001 (6)	0.0070 (7)	−0.0024 (6)
C21	0.0273 (10)	0.0214 (8)	0.0169 (8)	−0.0044 (7)	0.0097 (7)	−0.0001 (7)
C31	0.0132 (8)	0.0160 (8)	0.0156 (8)	0.0014 (6)	0.0041 (6)	0.0010 (6)
C32	0.0250 (9)	0.0191 (8)	0.0184 (8)	−0.0033 (7)	0.0069 (7)	−0.0051 (7)
C33	0.0284 (10)	0.0224 (9)	0.0256 (9)	−0.0086 (7)	0.0071 (8)	−0.0041 (7)
C41	0.0191 (8)	0.0153 (7)	0.0132 (7)	−0.0039 (6)	0.0068 (6)	−0.0012 (6)
O42	0.0189 (6)	0.0177 (6)	0.0148 (6)	0.0020 (5)	0.0079 (5)	0.0005 (4)
C43	0.0186 (8)	0.0179 (8)	0.0181 (8)	−0.0012 (6)	0.0086 (6)	−0.0033 (6)
C44	0.0195 (9)	0.0182 (8)	0.0215 (8)	0.0001 (7)	0.0081 (7)	0.0000 (7)
C45	0.0206 (9)	0.0175 (8)	0.0165 (8)	−0.0009 (6)	0.0075 (7)	0.0007 (6)
C51	0.0187 (8)	0.0179 (8)	0.0198 (8)	0.0004 (7)	0.0069 (6)	−0.0004 (6)
C52	0.0231 (9)	0.0146 (8)	0.0250 (9)	−0.0023 (7)	0.0085 (7)	0.0034 (7)
C53	0.0343 (11)	0.0246 (9)	0.0233 (9)	−0.0046 (8)	0.0100 (8)	0.0032 (7)
C61	0.0349 (11)	0.0242 (9)	0.0204 (9)	−0.0069 (8)	0.0115 (8)	−0.0067 (7)

Geometric parameters (Å, º)

N1—C2	1.377 (2)	O42—C43	1.351 (2)
N1—C6	1.378 (2)	C43—C44	1.353 (2)
N1—H1	0.8599	C43—N43	1.420 (2)
C2—C3	1.361 (2)	N43—O431	1.2323 (19)
C2—C21	1.506 (2)	N43—O432	1.2348 (19)
C21—H12A	0.98	C44—C45	1.416 (2)
C21—H12B	0.98	C44—H44	0.95
C21—H12C	0.98	C45—H45	0.95
C3—C31	1.467 (2)	C5—C6	1.351 (2)
C3—C4	1.528 (2)	C5—C51	1.471 (2)
C31—O31	1.2185 (19)	C6—C61	1.507 (2)
C31—O32	1.3499 (19)	C51—O51	1.213 (2)
O32—C32	1.4551 (19)	C51—O52	1.349 (2)
C32—C33	1.504 (2)	O52—C52	1.4507 (19)

C32—H13A	0.99	C52—C53	1.501 (2)
C32—H13B	0.99	C52—H52A	0.99
C33—H33A	0.98	C52—H52B	0.99
C33—H33B	0.98	C53—H52C	0.98
C33—H33C	0.98	C53—H52D	0.98
C4—C41	1.507 (2)	C53—H52E	0.98
C4—C5	1.526 (2)	C61—H61A	0.98
C4—H14	1.00	C61—H61B	0.98
C41—C45	1.360 (2)	C61—H61C	0.98
C41—O42	1.3703 (19)		

C2—N1—C6	124.01 (14)	O42—C43—C44	112.59 (14)
C2—N1—H1	118.0	O42—C43—N43	116.42 (14)
C6—N1—H1	118.0	C44—C43—N43	130.96 (16)
C3—C2—N1	119.46 (14)	O431—N43—O432	124.52 (14)
C3—C2—C21	128.35 (15)	O431—N43—C43	118.71 (14)
N1—C2—C21	112.19 (14)	O432—N43—C43	116.76 (14)
C2—C21—H12A	109.5	C43—C44—C45	104.85 (15)
C2—C21—H12B	109.5	C43—C44—H44	127.6
H12A—C21—H12B	109.5	C45—C44—H44	127.6
C2—C21—H12C	109.5	C41—C45—C44	107.12 (15)
H12A—C21—H12C	109.5	C41—C45—H45	126.4
H12B—C21—H12C	109.5	C44—C45—H45	126.4
C2—C3—C31	125.71 (14)	C6—C5—C51	120.47 (15)
C2—C3—C4	122.02 (14)	C6—C5—C4	121.60 (14)
C31—C3—C4	112.22 (13)	C51—C5—C4	117.93 (14)
O31—C31—O32	121.58 (14)	C5—C6—N1	120.29 (15)
O31—C31—C3	122.59 (15)	C5—C6—C61	126.26 (15)
O32—C31—C3	115.83 (13)	N1—C6—C61	113.46 (14)
C31—O32—C32	115.49 (12)	O51—C51—O52	121.91 (15)
O32—C32—C33	107.34 (13)	O51—C51—C5	127.37 (16)
O32—C32—H13A	110.2	O52—C51—C5	110.72 (14)
C33—C32—H13A	110.2	C51—O52—C52	115.31 (13)
O32—C32—H13B	110.2	O52—C52—C53	107.53 (14)
C33—C32—H13B	110.2	O52—C52—H52A	110.2
H13A—C32—H13B	108.5	C53—C52—H52A	110.2
C32—C33—H33A	109.5	O52—C52—H52B	110.2
C32—C33—H33B	109.5	C53—C52—H52B	110.2
H33A—C33—H33B	109.5	H52A—C52—H52B	108.5
C32—C33—H33C	109.5	C52—C53—H52C	109.5
H33A—C33—H33C	109.5	C52—C53—H52D	109.5
H33B—C33—H33C	109.5	H52C—C53—H52D	109.5
C41—C4—C5	111.36 (13)	C52—C53—H52E	109.5
C41—C4—C3	108.88 (13)	H52C—C53—H52E	109.5
C5—C4—C3	111.59 (13)	H52D—C53—H52E	109.5
C41—C4—H14	108.3	C6—C61—H61A	109.5
C5—C4—H14	108.3	C6—C61—H61B	109.5
C3—C4—H14	108.3	H61A—C61—H61B	109.5

C45—C41—O42	110.13 (14)	C6—C61—H61C	109.5
C45—C41—C4	132.47 (15)	H61A—C61—H61C	109.5
O42—C41—C4	117.33 (13)	H61B—C61—H61C	109.5
C43—O42—C41	105.30 (12)		

C6—N1—C2—C3	5.9 (2)	C44—C43—N43—O431	−173.70 (17)
C6—N1—C2—C21	−174.76 (15)	O42—C43—N43—O432	−175.20 (14)
N1—C2—C3—C31	178.28 (15)	C44—C43—N43—O432	7.0 (3)
C21—C2—C3—C31	−1.0 (3)	O42—C43—C44—C45	0.03 (19)
N1—C2—C3—C4	1.3 (2)	N43—C43—C44—C45	177.90 (17)
C21—C2—C3—C4	−177.98 (15)	O42—C41—C45—C44	−0.73 (18)
C2—C3—C31—O31	−171.65 (16)	C4—C41—C45—C44	175.86 (16)
C4—C3—C31—O31	5.6 (2)	C43—C44—C45—C41	0.43 (18)
C2—C3—C31—O32	8.8 (2)	C41—C4—C5—C6	−110.93 (17)
C4—C3—C31—O32	−173.91 (13)	C3—C4—C5—C6	11.0 (2)
O31—C31—O32—C32	−3.4 (2)	C41—C4—C5—C51	68.32 (18)
C3—C31—O32—C32	176.18 (13)	C3—C4—C5—C51	−169.78 (14)
C31—O32—C32—C33	−173.60 (14)	C51—C5—C6—N1	175.41 (15)
C2—C3—C4—C41	114.40 (16)	C4—C5—C6—N1	−5.4 (2)
C31—C3—C4—C41	−62.98 (17)	C51—C5—C6—C61	−4.9 (3)
C2—C3—C4—C5	−8.9 (2)	C4—C5—C6—C61	174.35 (16)
C31—C3—C4—C5	173.69 (13)	C2—N1—C6—C5	−3.8 (3)
C5—C4—C41—C45	73.2 (2)	C2—N1—C6—C61	176.46 (15)
C3—C4—C41—C45	−50.3 (2)	C6—C5—C51—O51	−0.1 (3)
C5—C4—C41—O42	−110.41 (15)	C4—C5—C51—O51	−179.33 (17)
C3—C4—C41—O42	126.12 (14)	C6—C5—C51—O52	179.62 (15)
C45—C41—O42—C43	0.74 (17)	C4—C5—C51—O52	0.4 (2)
C4—C41—O42—C43	−176.44 (13)	O51—C51—O52—C52	3.2 (2)
C41—O42—C43—C44	−0.46 (18)	C5—C51—O52—C52	−176.55 (13)
C41—O42—C43—N43	−178.67 (13)	C51—O52—C52—C53	179.52 (14)
O42—C43—N43—O431	4.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O31^{i}	0.86	2.18	2.986 (2)	157
C44—H44···O32^{ii}	0.95	2.38	3.330 (2)	174
C45—H45···O431^{i}	0.95	2.45	3.369 (2)	163

Symmetry codes: (i) x, $-y+1/2$, $z+1/2$; (ii) $x+1$, y, z.

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

Crystal data

$C_{13}H_{10}N_4O_3$	c = 17.6385 (5) Å
M_r = 270.25	β = 96.2570 (13)°
Monoclinic, $P2_1/c$	V = 1270.14 (6) Å^3
Hall symbol: -P 2ybc	Z = 4
a = 9.5651 (3) Å	F(000) = 560
b = 7.5735 (2) Å	D_x = 1.413 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2907 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 K
Block, colourless
0.90 × 0.34 × 0.22 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR91 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.906, T_{max} = 0.977
16132 measured reflections
2907 independent reflections
2333 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 2.9°
h = −12→12
k = −9→9
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.05
2907 reflections
183 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.4785P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O42	0.27258 (9)	0.53845 (11)	0.61731 (5)	0.0164 (2)
O431	0.30100 (10)	0.20533 (12)	0.65330 (6)	0.0221 (2)
O432	0.15430 (11)	0.22748 (14)	0.73896 (6)	0.0278 (3)
N1	0.50332 (13)	0.70103 (16)	0.45769 (7)	0.0234 (3)
N31	0.55647 (13)	0.93396 (17)	0.71152 (7)	0.0286 (3)
N43	0.21791 (11)	0.28884 (15)	0.68804 (6)	0.0188 (3)
N51	0.00913 (15)	0.8255 (2)	0.42460 (9)	0.0423 (4)
C2	0.55496 (15)	0.75829 (17)	0.52956 (8)	0.0201 (3)
C3	0.46578 (14)	0.82043 (17)	0.57746 (7)	0.0180 (3)
C4	0.30672 (13)	0.82641 (17)	0.55835 (7)	0.0169 (3)
C5	0.26985 (14)	0.77735 (17)	0.47480 (8)	0.0194 (3)
C6	0.36409 (15)	0.71607 (17)	0.42962 (8)	0.0213 (3)
C21	0.71118 (15)	0.74498 (19)	0.54819 (9)	0.0251 (3)
C31	0.51887 (14)	0.88193 (18)	0.65156 (8)	0.0204 (3)
C41	0.23281 (14)	0.71315 (17)	0.61106 (7)	0.0173 (3)
C43	0.19267 (13)	0.46889 (18)	0.66885 (7)	0.0178 (3)
C44	0.10486 (15)	0.5872 (2)	0.69481 (8)	0.0256 (3)
C51	0.12583 (16)	0.80078 (19)	0.44537 (8)	0.0259 (3)
C45	0.13092 (16)	0.74698 (19)	0.65650 (9)	0.0248 (3)
C61	0.33009 (18)	0.6621 (2)	0.34784 (8)	0.0298 (4)
H1	0.5620	0.6528	0.4286	0.028*

H4	0.2758	0.9511	0.5650	0.020*
H21A	0.7380	0.7908	0.5997	0.038*
H21B	0.7578	0.8143	0.5113	0.038*
H21C	0.7400	0.6211	0.5458	0.038*
H44	0.0396	0.5683	0.7309	0.031*
H45	0.0853	0.8570	0.6618	0.030*
H61A	0.2278	0.6613	0.3348	0.045*
H61B	0.3676	0.5437	0.3404	0.045*
H61C	0.3727	0.7461	0.3149	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O42	0.0169 (5)	0.0165 (5)	0.0165 (5)	0.0010 (3)	0.0048 (3)	0.0001 (3)
O431	0.0215 (5)	0.0197 (5)	0.0255 (5)	0.0011 (4)	0.0050 (4)	−0.0010 (4)
O432	0.0273 (6)	0.0307 (6)	0.0271 (5)	−0.0071 (4)	0.0100 (4)	0.0067 (4)
N1	0.0264 (6)	0.0230 (6)	0.0221 (6)	0.0015 (5)	0.0086 (5)	−0.0023 (5)
N31	0.0259 (7)	0.0333 (7)	0.0254 (7)	0.0000 (5)	−0.0028 (5)	−0.0001 (5)
N51	0.0326 (8)	0.0529 (9)	0.0385 (8)	0.0007 (7)	−0.0099 (6)	−0.0074 (7)
C2	0.0232 (7)	0.0148 (6)	0.0229 (7)	−0.0008 (5)	0.0044 (6)	0.0043 (5)
C3	0.0201 (7)	0.0155 (6)	0.0183 (6)	−0.0013 (5)	0.0011 (5)	0.0021 (5)
C4	0.0186 (6)	0.0154 (6)	0.0164 (6)	0.0010 (5)	0.0007 (5)	−0.0011 (5)
C5	0.0233 (7)	0.0177 (6)	0.0167 (6)	−0.0021 (5)	0.0000 (5)	0.0011 (5)
C6	0.0302 (8)	0.0156 (6)	0.0184 (7)	−0.0045 (5)	0.0044 (6)	0.0021 (5)
C21	0.0217 (7)	0.0224 (7)	0.0322 (8)	0.0019 (5)	0.0071 (6)	0.0052 (6)
C31	0.0180 (7)	0.0199 (7)	0.0229 (7)	0.0006 (5)	0.0011 (5)	0.0037 (5)
C41	0.0185 (6)	0.0168 (6)	0.0165 (6)	0.0027 (5)	0.0008 (5)	−0.0010 (5)
C43	0.0150 (6)	0.0219 (7)	0.0168 (6)	−0.0023 (5)	0.0036 (5)	0.0013 (5)
N43	0.0154 (5)	0.0226 (6)	0.0185 (6)	−0.0040 (4)	0.0026 (5)	0.0003 (5)
C44	0.0222 (7)	0.0297 (8)	0.0267 (8)	0.0027 (6)	0.0109 (6)	0.0012 (6)
C51	0.0301 (8)	0.0265 (8)	0.0201 (7)	−0.0028 (6)	−0.0014 (6)	−0.0026 (6)
C45	0.0254 (7)	0.0234 (7)	0.0270 (7)	0.0070 (6)	0.0088 (6)	−0.0004 (6)
C61	0.0441 (10)	0.0277 (8)	0.0182 (7)	−0.0066 (7)	0.0064 (6)	−0.0022 (6)

Geometric parameters (Å, º)

N1—C6	1.3741 (19)	O42—C43	1.3563 (15)
N1—C2	1.3794 (18)	C43—C44	1.3424 (19)
N1—H1	0.88	C43—N43	1.4193 (18)
C2—C3	1.3491 (19)	N43—O432	1.2297 (15)
C2—C21	1.498 (2)	N43—O431	1.2302 (14)
C21—H21A	0.98	C44—C45	1.421 (2)
C21—H21B	0.98	C44—H44	0.95
C21—H21C	0.98	C5—C6	1.349 (2)
C3—C31	1.4278 (19)	C5—C51	1.429 (2)
C3—C4	1.5227 (18)	C51—N51	1.152 (2)
C31—N31	1.1490 (18)	C45—H45	0.95
C4—C41	1.4978 (18)	C6—C61	1.500 (2)

C4—C5	1.5234 (18)	C61—H61A	0.98
C4—H4	1.00	C61—H61B	0.98
C41—C45	1.352 (2)	C61—H61C	0.98
C41—O42	1.3778 (16)		

C6—N1—C2	122.76 (12)	C44—C43—O42	112.86 (12)
C6—N1—H1	118.6	C44—C43—N43	131.04 (12)
C2—N1—H1	118.6	O42—C43—N43	116.06 (11)
C3—C2—N1	119.85 (13)	O432—N43—O431	124.58 (12)
C3—C2—C21	124.74 (13)	O432—N43—C43	116.90 (11)
N1—C2—C21	115.42 (12)	O431—N43—C43	118.52 (11)
C2—C21—H21A	109.5	C43—C44—C45	104.92 (12)
C2—C21—H21B	109.5	C43—C44—H44	127.5
H21A—C21—H21B	109.5	C45—C44—H44	127.5
C2—C21—H21C	109.5	C6—C5—C51	120.52 (13)
H21A—C21—H21C	109.5	C6—C5—C4	123.80 (12)
H21B—C21—H21C	109.5	C51—C5—C4	115.68 (12)
C2—C3—C31	120.07 (12)	N51—C51—C5	176.57 (16)
C2—C3—C4	123.81 (12)	C41—C45—C44	107.20 (12)
C31—C3—C4	116.11 (11)	C41—C45—H45	126.4
N31—C31—C3	177.33 (15)	C44—C45—H45	126.4
C41—C4—C3	111.92 (10)	C5—C6—N1	119.96 (12)
C41—C4—C5	112.68 (11)	C5—C6—C61	124.96 (13)
C3—C4—C5	109.10 (11)	N1—C6—C61	115.08 (13)
C41—C4—H4	107.6	C6—C61—H61A	109.5
C3—C4—H4	107.6	C6—C61—H61B	109.5
C5—C4—H4	107.6	H61A—C61—H61B	109.5
C45—C41—O42	110.18 (12)	C6—C61—H61C	109.5
C45—C41—C4	132.76 (12)	H61A—C61—H61C	109.5
O42—C41—C4	117.05 (11)	H61B—C61—H61C	109.5
C43—O42—C41	104.82 (10)		

C6—N1—C2—C3	5.0 (2)	O42—C43—N43—O432	174.51 (11)
C6—N1—C2—C21	−175.16 (12)	C44—C43—N43—O431	177.01 (14)
N1—C2—C3—C31	−179.21 (12)	O42—C43—N43—O431	−5.36 (17)
C21—C2—C3—C31	0.9 (2)	O42—C43—C44—C45	−0.04 (17)
N1—C2—C3—C4	2.2 (2)	N43—C43—C44—C45	177.65 (14)
C21—C2—C3—C4	−177.65 (12)	C41—C4—C5—C6	−116.84 (14)
C2—C3—C4—C41	117.40 (14)	C3—C4—C5—C6	8.12 (18)
C31—C3—C4—C41	−61.23 (15)	C41—C4—C5—C51	63.42 (15)
C2—C3—C4—C5	−8.01 (17)	C3—C4—C5—C51	−171.62 (11)
C31—C3—C4—C5	173.37 (11)	O42—C41—C45—C44	0.85 (16)
C3—C4—C41—C45	124.76 (16)	C4—C41—C45—C44	−178.71 (14)
C5—C4—C41—C45	−111.83 (17)	C43—C44—C45—C41	−0.49 (17)
C3—C4—C41—O42	−54.78 (15)	C51—C5—C6—N1	177.31 (12)
C5—C4—C41—O42	68.63 (14)	C4—C5—C6—N1	−2.4 (2)
C45—C41—O42—C43	−0.85 (14)	C51—C5—C6—C61	−2.0 (2)
C4—C41—O42—C43	178.79 (11)	C4—C5—C6—C61	178.24 (13)

C41—O42—C43—C44	0.54 (15)	C2—N1—C6—C5	−4.9 (2)
C41—O42—C43—N43	−177.52 (11)	C2—N1—C6—C61	174.54 (12)
C44—C43—N43—O432	−3.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O42^{i}	0.88	2.35	3.2019 (15)	162
N1—H1···O431^{i}	0.88	2.32	2.9390 (16)	128
C4—H4···O431^{ii}	1.00	2.47	3.3262 (16)	143
C44—H44···O432^{iii}	0.95	2.32	3.0446 (18)	132

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*, *y*+1, *z*; (iii) −*x*, *y*+1/2, −*z*+3/2.