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Key indicators

Single-crystal X-ray study
 T = 113 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 Disorder in main residue
 R factor = 0.038
 wR factor = 0.102
 Data-to-parameter ratio = 8.0

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

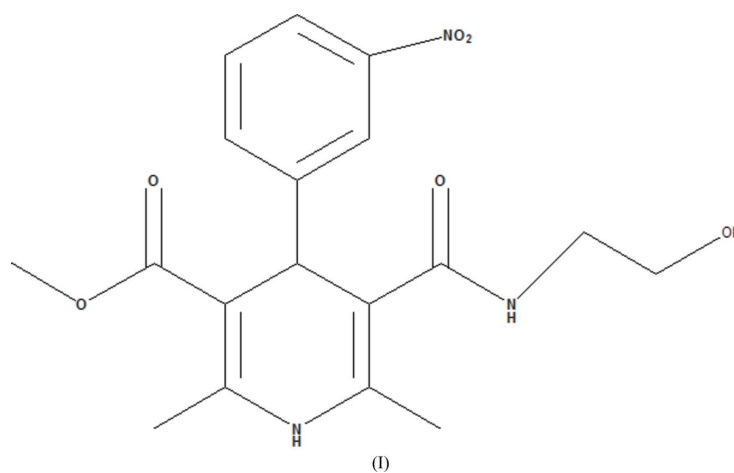
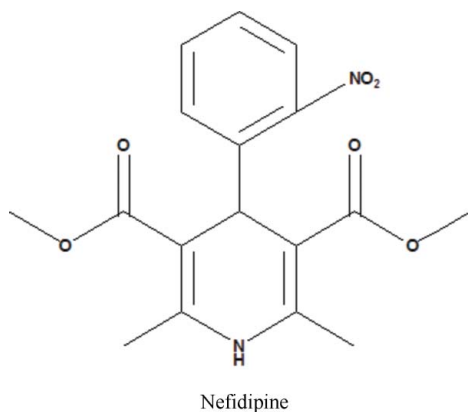
Methyl 5-[N-(2-hydroxyethyl)carbamoyl]-2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3-carboxylate

The title compound, $\text{C}_{18}\text{H}_{21}\text{N}_3\text{O}_6$, is a nefidipine analog. The crystal packing is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

4-Aryl-1,4-dihydropyridine-3,5-dicarboxylic diesters of the nefidipine type have become almost indispensable for the treatment of cardiovascular diseases since they first appeared on the market in 1975 (Yiu & Knaus, 1999; Goldmann & Stoltefuss, 1991).



The structure of (I) is shown in Fig. 1. The dihydropyridine ring displays an envelope conformation, with C3 displaced from the mean plane formed by the other atoms in the same ring by 0.165 (1) Å. The dihedral angle between the benzene ring and the N1/C1/C2/C4/C5 plane is 96.77°. Three of the four substituents on the dihydropyridine ring are almost coplanar

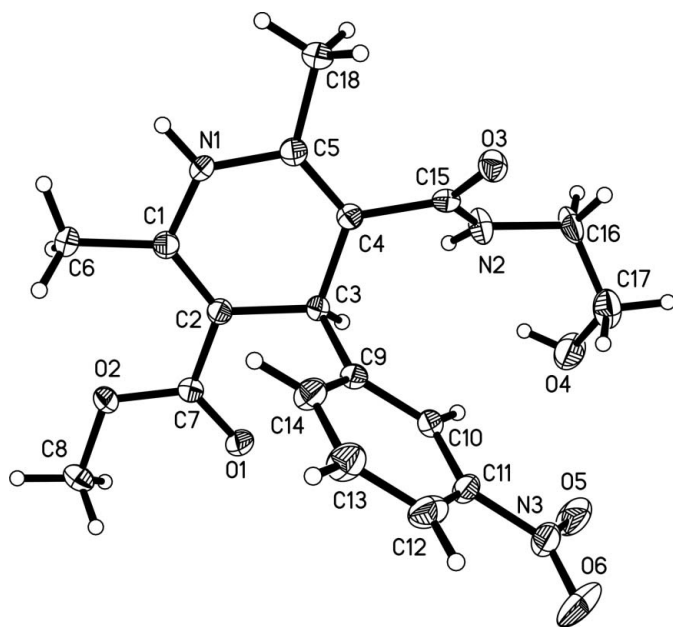


Figure 1

A view of the title compound, (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Only one disorder component is shown.

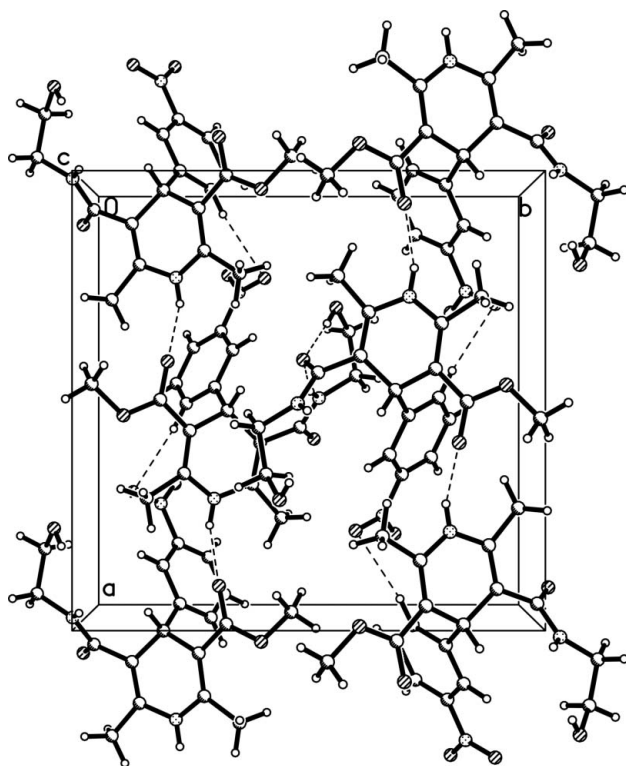


Figure 2

A packing diagram of (I). Dashed lines indicate hydrogen bonds. Only one disorder component is shown.

with it, with atoms N1, C3 and C7 displaced from the mean C1/C2/C4/C5/C6/C15/C18 plane by 0.113 (1), 0.134 (1) and 0.129 (1) Å, respectively. This compares well with values in the structures of similar compounds reported previously (Liu *et*

al., 2006; Jiang & Sun, 2006; Sun *et al.*, 2006). One methyl group and the hydroxyethyl group were found to be disordered.

Three intermolecular N—H···O hydrogen bonds link the molecules into zigzag chains along *a* axis.

Experimental

2,6-Dimethyl-4-(*m*-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylic acid monomethyl ester (332 mg, 1 mmol) and dicyclohexylcarbodiimide (206 mg, 1 mmol) were dissolved in 28 ml CH₂Cl₂, and 2-aminoethanol (305 mg, 5 mmol) was added dropwise at 278 K. The reaction mixture was stirred at 276–279 K for a further 10 h after which the CH₂Cl₂ was removed by vacuum evaporation at 293 K. Compound (I) was purified by chromatography on a silica gel column (eluted by ethyl acetate–petroleum 1:4) at room temperature (yield 350 mg, 93% yield). Suitable crystals were obtained by slow evaporation of an ethyl acetate–methanol (1:1) solution.

Crystal data

C ₁₈ H ₂₁ N ₃ O ₆	Z = 4
<i>M_r</i> = 375.38	<i>D_x</i> = 1.407 Mg m ⁻³
Orthorhombic, <i>Pna</i> 2 ₁	Mo Kα radiation
<i>a</i> = 14.1028 (15) Å	<i>μ</i> = 0.11 mm ⁻¹
<i>b</i> = 14.4948 (16) Å	<i>T</i> = 113 (2) K
<i>c</i> = 8.6701 (10) Å	Block, yellow
<i>V</i> = 1772.3 (3) Å ³	0.26 × 0.20 × 0.16 mm

Data collection

Rigaku Saturn diffractometer	15964 measured reflections
<i>ω</i> scans	2244 independent reflections
Absorption correction: multi-scan	2066 reflections with <i>I</i> > 2σ(<i>I</i>)
Jacobson (1998)	<i>R_{int}</i> = 0.039
<i>T_{min}</i> = 0.970, <i>T_{max}</i> = 0.983	<i>θ_{max}</i> = 27.9°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.102$	(Δ/σ) _{max} = 0.002
<i>S</i> = 1.06	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
2244 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
279 parameters	Extinction correction: SHELXL97
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.034 (4)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O4—H4A···O3 ⁱ	0.84	2.05	2.892 (3)	176
O4'—H4'A···O3 ⁱ	0.84	1.92	2.76 (2)	176
N1—H1···O1 ⁱⁱ	0.87 (3)	2.10 (3)	2.920 (2)	157 (2)
N2—H2···O3 ⁱ	0.87 (3)	2.00 (3)	2.836 (3)	161 (3)

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

H atoms were treated by a mixture of independent and restrained refinement. The restrained H atoms were positioned geometrically and refined using a riding model with C—H 0.97 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C). The disordered methyl group has two positions with a 1:1 ratio. The disordered hydroxyethyl group was refined on two alternative sites with equal occupancies. The C—O, O—H and C—C single bonds were restrained to 1.40–1.43 (1), 0.84 (1) and 1.47–

1.52 (1) Å, respectively. The bond angles were also restrained by restraining the 1–3 atom distances. In the absence of significant anomalous scattering effects, Friedel pairs have been merged.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Bruker, 1997b); software used to prepare material for publication: *CrystalStructure*.

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