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Methyl 5-[*N*-(2-methoxycarbonyl-1-methylvinyl)-carbamoyl]-2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3-carboxylate monohydrate

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Key indicators

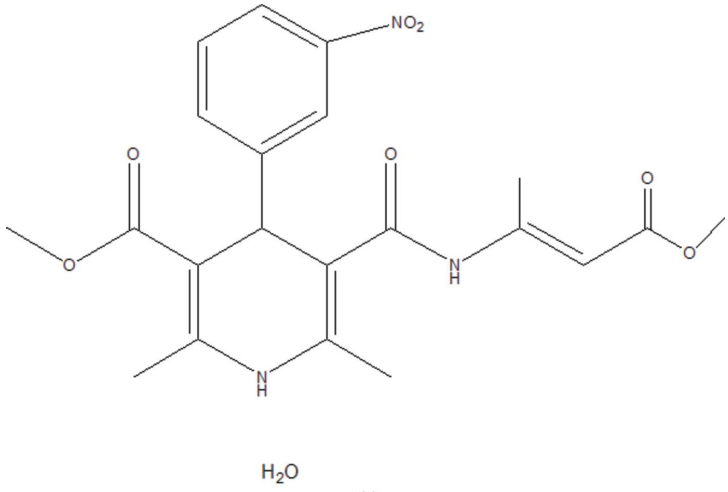
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.138
Data-to-parameter ratio = 12.5

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

The title compound, $C_{21}H_{25}N_3O_7$, is a nefidipine analog. The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds, which link the molecules into chains running parallel to the *c* axis.

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 title compound, (I), is a nefidipine analog.



(I)

Fig. 1 shows the structure of (I). Bond lengths and angles are unexceptional. The dihydropyridine ring has a boat conformation. This compares well with the structures of 3-(benzotriazol-1-yl) 5-ethyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate ethyl acetate hemisolvate (Sun *et al.*, 2006) and nefidipine (Hofmann & Cimiraglia, 1990). Atoms C3 and N1 are displaced from the mean plane through C1/C2/C4/C5 by 0.413 (1) and 0.180 (1) Å, respectively. The dihedral angle between the benzene ring and the C1/C2/C4/C5 plane is 88.71 (1)°.

The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds (Table 1), which link the molecules into chains running parallel to the *c* axis.

Experimental

The title compound was prepared by dissolving 2,6-dimethyl-4-(*p*-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylic acid monomethyl

ester (332 mg, 1 mmol) in CH_2Cl_2 (25 ml) and triethylamine (1 ml). $SOCl_2$ (119 mg, 1 mmol) in CH_2Cl_2 (2 ml) was added dropwise to this solution at 275–277 K. The reaction mixture was stirred at 275–277 K for a further 1 h. Methyl 3-aminobut-2-enoate (330 mg, 2 mmol) was added to this solution. The reaction mixture was stirred at 275–277 K for a further 8 h. Water (20 ml) was added and two layers formed. The organic layer was collected and the solvent was removed by vacuum evaporation at 293 K. The target compound was purified by chromatography on a silica-gel column (eluting with ethyl acetate and petroleum, 1:6) at room temperature. The product was obtained in 20% yield. Suitable crystals were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{21}H_{25}N_3O_8$
M_r = 447.44
Triclinic, $P\overline{1}$
a = 7.872 (5) Å
b = 11.800 (7) Å
c = 12.277 (7) Å
α = 90.238 (10)°
β = 91.757 (10)°
γ = 108.391 (9)°
V = 1081.5 (10) Å^3
Z = 2
D_x = 1.374 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
Block, yellow
0.26 × 0.24 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.973, T_{max} = 0.985
5562 measured reflections
3802 independent reflections
2355 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.138
S = 1.04
3802 reflections
304 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.1639P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8A···O3′^{i}	0.85	2.09	2.916 (10)	162
O8—H8A···O3^{i}	0.85	2.11	2.967 (12)	178

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

All methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C); the torsion angles were refined to fit the electron density. Other H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, N—H = 0.86 Å, O—H = 0.85 Å and U_{iso}(H) = 1.2U_{eq}(C,N,O). One O atom of the nitro group was disordered over two sites, O3 and O3′, with refined occupancy factors of 0.54 (4) and 0.46 (4), respectively.

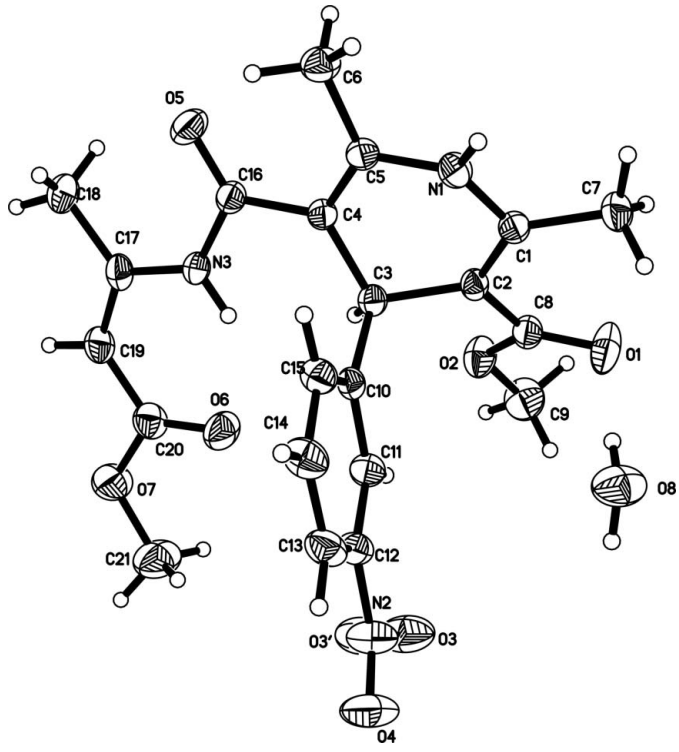


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius. Both disorder components are shown.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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