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3-Cinnamyl 5-(2-methoxyethyl) 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate

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Key indicators

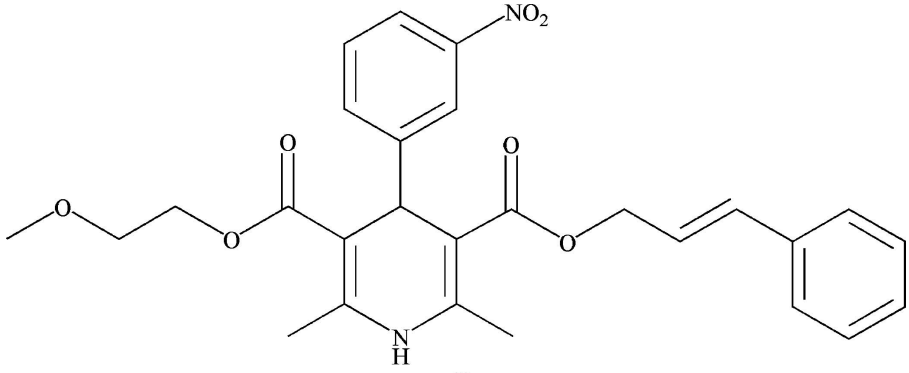
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.152
Data-to-parameter ratio = 8.7

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

The title compound, $C_{27}H_{28}N_2O_7$, has been synthesized by the reaction of 2-methoxyethyl 2-(3-nitrobenzylidene)acetoacetate with cinnamyl 3-aminocrotonate and crystallized from methanol. The substituted 1,4-dihydropyridine ring adopts a flattened boat conformation. The 3-nitrophenyl ring is approximately perpendicular to the mean plane of the 1,4-dihydropyridine ring.

Comment

Since nifedipine was found to be a highly effective calcium antagonist in 1975, 1,4-dihydropyridine derivatives have been widely investigated for their pharmacological activities, especially calcium antagonist activity. Many compounds similar in structure to nifedipine have already been used as therapeutic agents for the treatment of cerebral circulatory disorder, hypertension, and so on (Goldmann *et al.*, 1991). The title compound, (I), is a new 1,4-dihydropyridine calcium antagonist. It has a slow-onset long-lasting hypotensive effect in hypertensive patients and has been claimed to antagonize both the *L*-type and *N*-type voltage-dependent calcium-channels (VDCC) (Mealy *et al.*, 1996). Inhibition of the *N*-type VDCC may contribute positively to the clinical profile, as the *N*-type VDCC is extensively involved in peripheral sympathetic neurotransmission.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The 1,4-dihydropyridne ring has a flattened boat conformation. The 3-nitrophenyl ring and the 1,4-dihydropyridine ring are almost perpendicular to each other, the dihedral angle between their mean planes being 85.2 (2)°. The C19—C20—C21—C22 torsion angle of 179.9 (4)° indicates an *E* configuration of the planar cinnamyl unit. The crystal structure is stabilized by intermolecular N—H···O hydrogen bonding (Table 1). The crystal packing is illustrated in Fig. 2.

Experimental

The synthesis of (I) was carried out by heating a mixture of 2-methoxyethyl 2-(3-nitrobenzylidene)acetoacetate (2.93 g, 0.01 mol) and cinnamyl 3-aminocrotonate (2.17 g, 0.01 mol) at 383–393 K, and stirring for 3 h; ethanol (10 ml) and active carbon (0.05 g) were then added to the mixture, refluxed for 20 min and filtered, the filtrate was cooled in an ice bath and filtered again to obtain a yellow crude product. Recrystallization from ethanol gave 4.18 g of (I) (yield 85%, m.p. 375–377 K) (Wu *et al.*, 2002). Single crystals of (I) were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{27}H_{28}N_2O_7$
M_r = 492.51
Orthorhombic, *Fdd*2
a = 15.1280 (8) Å
b = 59.932 (3) Å
c = 10.9468 (5) Å
V = 9924.9 (9) Å^3
Z = 16
D_x = 1.318 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 (2) K
Block, yellow
0.42 × 0.34 × 0.28 mm

Data collection

Bruker AXS SMART 1000 CCD diffractometer
ω scans
Absorption correction: none
20624 measured reflections
2851 independent reflections
2094 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.152
S = 1.09
2851 reflections
328 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.099P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O5^{i}	0.88	2.04	2.925 (4)	179

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

Methyl H atoms were placed in calculated positions, with C—H = 0.98 Å, and torsion angles were refined, with U_{iso}(H)= 1.5U_{eq}(C). Other H atoms were positioned geometrically, with C—H = 0.99 (methylene), 0.95 (methine) and 0.95 Å (aromatic), and N—H = 0.88 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(carrier). In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; 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: *SHELXTL* (Bruker, 1997).

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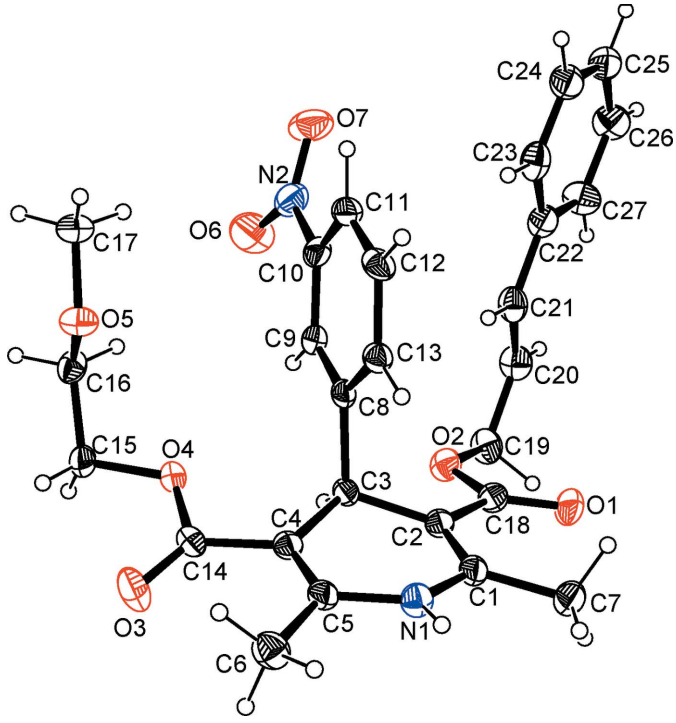

Figure 1
The molecular structure of (I) shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

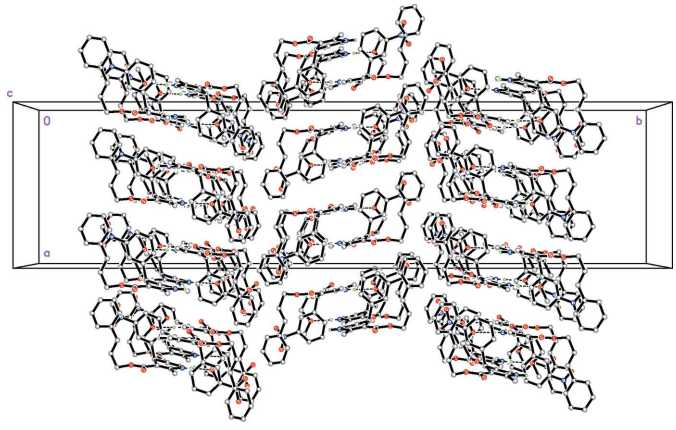

Figure 2
The packing of (I). All C-bound H atoms have been omitted for clarity.

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