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3-Methyl 5-isopropyl 2-methoxyiminomethyl-6-methyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate

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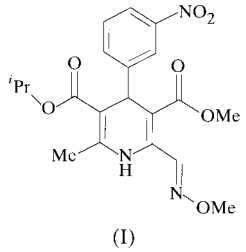
The crystal structure of the title compound, $C_{20}H_{23}N_3O_7$, consists of relatively isolated molecules. The substituted 1,4-dihydropyridine ring adopts a flattened boat conformation. Both ester groups, at positions 3 and 5, have *cis,cis* geometry. The phenyl ring is nearly planar and is approximately perpendicular to the 1,4-dihydropyridine ring (dihedral angle 87.70°).

Comment

Many derivatives of 1,4-dihydropyridine (DHP) exhibit high affinity for calcium channel receptors and may act as agonists or antagonists, depending on the nature of the derivative, the physiological state of the channel and, in some cases, the side of the membrane containing the channel receptor to which the compound is added (Kokubun & Reuter, 1984). Amongst these derivatives is nifedipine [2,6-dimethyl-3,5-dicarbomethoxy-4-(2-nitrophenyl)-1,4-dihydropyridine], which is one of the most potent of the calcium antagonists and is a powerful



(I)

negative ionotropic and smooth-muscle relaxant species (Triggle *et al.*, 1980). We have studied the crystal structure of methyl isopropyl 2-methoxyiminomethyl-6-methyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate, (I), and present its structure here.

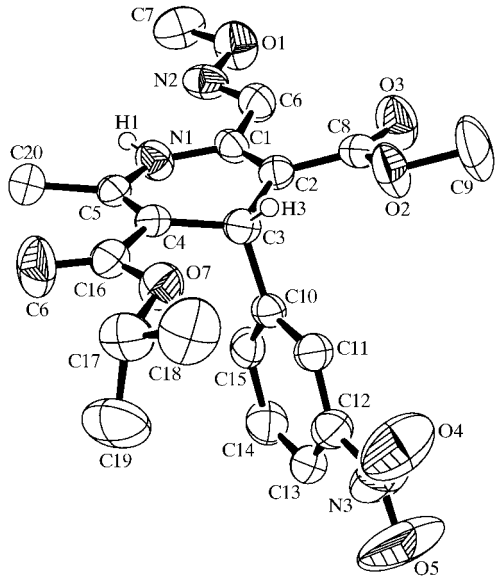


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The structure of (I) (Fig. 1) consists of discrete molecules. The 1,4-DHP ring exhibits a boat conformation. The N1 and C3 atoms lie 0.110 (3) and 0.228 (3) Å, respectively, from the least-squares plane defined by the remaining four atoms of the DHP ring. The orientation of the 3-nitrophenyl ring relative to the 1,4-DHP ring, characterized by the torsion angle C2—C3—C10—C15, is −63.8 (4)°. A similar orientation of the phenyl ring is preferred in all the investigated phenyl-substituted derivatives (Mehdi & Ravikumar, 1992). It is probable that in this way the steric strain, which is imposed by the phenyl substituent and the groups at the 3 and 5 positions, is minimized (Fossheim, 1985). The nitro group is twisted 5.0 (3)° from the plane of the phenyl-ring system. Langs & Triggle (1985) have observed that the majority of 1,4-DHP analogs have one of the ester groups at C3 and C5 of the 1,4-DHP ring in the *cis* conformation and the other in the *trans* conformation. A small number of very active antagonists are found to have *cis,cis* geometry. In the present compound, the carbonyl groups are twisted in the same *cis,cis* direction. One ester group is slightly oriented out of the 1,4-DHP plane and the second ester group is coplanar with the 1,4-DHP ring (Table 1).

Experimental

The title compound was prepared according to known synthetic methods (Hantzsch, 1882) and recrystallized from ethanol solution. Slow evaporation of an ethanol solution yielded yellow plate-like crystals.

Table 1
Selected geometric parameters (Å, °).

O1—N2	1.384 (3)	C1—C2	1.341 (3)
O3—C8	1.196 (3)	C2—C3	1.521 (4)
O6—C16	1.214 (3)	C3—C4	1.505 (4)
N1—C1	1.372 (4)	C3—C10	1.515 (4)
N1—C5	1.376 (3)	C4—C5	1.326 (4)
N3—C12	1.461 (4)		
N2—O1—C7	108.9 (2)	C4—C3—C2	111.7 (3)
C1—N1—C5	123.6 (3)	C15—C10—C3	120.7 (3)
O5—N3—O4	122.0 (4)	C13—C12—C11	122.5 (4)
C4—C3—C10	111.4 (3)	O6—C16—O7	122.1 (3)
C5—N1—C1—C2	10.7 (5)	C4—C3—C10—C15	60.9 (4)
C1—C2—C3—C4	−18.6 (4)	C2—C3—C10—C15	−63.8 (4)
C2—C3—C4—C5	17.8 (4)	O4—N3—C12—C11	5.2 (6)
C1—N1—C5—C4	−11.8 (4)	C5—C4—C16—O6	1.0 (5)
C1—C2—C8—O3	−13.8 (5)	C5—C4—C16—O7	−179.2 (3)
C1—C2—C8—O2	167.1 (3)		

Crystal data

$C_{20}H_{23}N_3O_7$
M_r = 417.41
Triclinic, $P\overline{1}$
a = 9.563 (4) Å
b = 9.703 (4) Å
c = 12.031 (6) Å
α = 93.99 (4)°
β = 105.09 (4)°
γ = 103.66 (4)°
V = 1037.1 (8) Å^3
Z = 2
D_x = 1.337 Mg m^{-3}
D_m = 1.34 (1) Mg m^{-3}
D_m measured by flotation in bromoform–hexane
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 8.2–21.4°
μ = 0.10 mm^{-1}
T = 293 (2) K
Plate, yellow
0.35 × 0.30 × 0.10 mm

Data collection

Syntex $P2_1$ diffractometer
$\theta/2\theta$ scans
3527 measured reflections
3527 independent reflections
1209 reflections with $I > 2\sigma(I)$
θ_{max} = 25.1°
$h = 0 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 13$
2 standard reflections
frequency: 100 min
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.052
$wR(F^2)$ = 0.068
S = 0.78
3527 reflections
304 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0112P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 8.8 (6) × 10^{-3}

Atoms H1, H3, H6, H11, H13, H14 and H15 were located in a difference Fourier map and were refined isotropically. The positions of the methyl H atoms were calculated geometrically and fixed (C—H = 0.96 Å), while their isotropic displacement parameters were fixed at 1.5U_{eq} of the parent atom.

Data collection: Syntex $P2_1$ software; cell refinement: Syntex $P2_1$ software; data reduction: *XP*21 (Pavelčík, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXS*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1077). Services for accessing these data are described at the back of the journal.

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