Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 5-ethoxymethylene-2-methyl-6-oxo-4-phenyl-1,4,5,6-tetrahydropyridine-3-carboxylate

Héctor Novoa de Armas,[a]* Oswald M. Peeters,[a] Norbert M. Blaton,[a] Camiel J. De Ranter,[a] Margarita Suárez Navarro,[b] Yamila Verdecia Reyes,[b] Estael Ochoa Rodríguez[b] and Esperanza Salfrán[b]

[a]Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium, and [b]Laboratorio de Síntesis Orgánica, Facultad de Química, Universidad de La Habana, Apartado 10400 La Habana, Cuba

Correspondence e-mail: hector.novoa@pharm.kuleuven.ac.be

Received 13 January 2003
Accepted 20 January 2003
Online 31 January 2003

In the title compound, $C_{18}H_{21}NO_4$, the molecules form dimers by means of a pair of N—H···O hydrogen bonds. The 2(1*H*)-pyridone ring displays a screw–boat conformation.

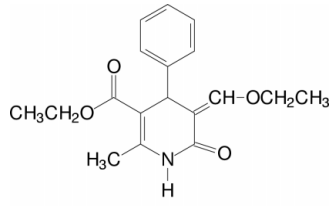
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.067
wR factor = 0.219
Data-to-parameter ratio = 14.0

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

Comment

A wide variety of compounds containing the 2(1*H*)-pyridone ring are found in nature, and in some cases they display useful biological properties (Overman *et al.*, 1980). 2(1*H*)-Pyridones are very close in structure to the 1,4-dihidropyridines, which have been used as effective drugs in the treatment of cardiovascular diseases. In a previous study, we have reported a general procedure (Verdecia *et al.*, 1996) for the synthesis of novel *o*-chloroformyl-substituted ethyl 1,4-dihydropyridine-5-carboxylates from the corresponding 3,4-dihydropyridones (Goldmann & Stoltefuss, 1991). The reaction proceeds through a mechanism involving several steps and one of the intermediates formed in the reaction is the title compound, (I).



(I)

The 2(1*H*)-pyridone ring of (I) has a screw–boat conformation, with puckering parameters (Cremer & Pople, 1975) Q = 0.280 (2) Å, θ = 109.5 (4)° and φ = 339.1 (5)°, with the axis through C2—C3, for N1—C1—C2—C3—C4—C5—C6. The mean Csp^2—Csp^2 bond length within this ring is 1.434 (1) Å. A centrosymmetric pair of N—H···O hydrogen bonds holds the molecules together in a dimeric association (Table 2).

Experimental

The title compound was prepared by stirring a solution of 5-ethoxycarbonyl-6-methyl-4-phenyl-3,4-dihydro-2(1*H*)-pyridone (10 mmol) in anhydrous *N,N*-dimethylformamide (40 mmol), and phosphorus oxychloride (40 mmol) in dry chloroform (50 ml). After stirring at room temperature for 18 h, 20 ml of anhydrous ethanol was added. After 0.5 h, a white solid had precipitated and was filtered off. Further purification was accomplished by recrystallization from methanol (yield 68%; m.p. 746–747 K). IR (KBr, cm^{-1}): 3202 (NH), 1697 (C═O), 1674 (C═O), 1637 (C═C); ^{1}H NMR (DMSO-d_6, p.p.m.): 9.68 (*s*, 1H, NH), 7.36 (*s*, 1H, ═CH), 7.31–7.11 (*m*, 5H, Ph), 4.89 (*s*, 1H, H-4), 4.13 (*q*, 2H, CH_2), 3.96 (*q*, 2H, CH_2), 2.29 (*s*, 3H, CH_3), 1.22 (*t*, 3H, CH_3), 1.06 (*t*, 3H, CH_3); ^{13}C NMR (DMSO-d_6,

p.p.m.): 171.4 (C2), 169.7 (COO), 161.2 (=CH), 152.2 (C6), 149.5 (C1′), 133.4 (C2′, C6′), 131.9 (C3′, C5′), 131.4 (C4′), 114.5 (C3), 109.2 (C5), 75.3 (CH_2), 64.5 (CH_2), 43.4 (C4), 23.5 (CH_3), 20.5 (CH_3), 19.3 (CH_3); MS: *m*/*z* (intensity%): 315 (M^+, 64), 286 (60), 270 (31), 238 (65), 210 (100), 182 (44). Crystals suitable for X-ray analysis were obtained by slow evaporation from methanol.

Crystal data

$C_{18}H_{21}NO_4$	D_x = 1.230 Mg m^{-3}
M_r = 315.36	Cu Kα radiation
Monoclinic, *C*2/*c*	Cell parameters from 40 reflections
a = 22.652 (1) Å	θ = 10.8–27.9°
b = 10.5508 (3) Å	μ = 0.71 mm^{-1}
c = 15.7162 (7) Å	*T* = 293 K
β = 114.921 (4)°	Prism, colourless
V = 3406.4 (3) Å^3	0.60 × 0.40 × 0.40 mm
Z = 8	

Data collection

Siemens *P*4 four-circle diffractometer	R_{int} = 0.030
$\omega/2\theta$ scans	θ_{max} = 69.1°
Absorption correction: ψ scan (North *et al.*, 1968)	*h* = −1 → 27
T_{min} = 0.565, T_{max} = 0.753	*k* = −1 → 12
7611 measured reflections	*l* = −19 → 17
2962 independent reflections	3 standard reflections every 100 reflections
2690 reflections with $F^2 > 2\sigma(F^2)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1525P)^2 + 3.0912P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.067	$(\Delta/\sigma)_{max}$ = 0.110
$wR(F^2)$ = 0.219	$\Delta\rho_{max}$ = 0.62 e Å^{-3}
S = 0.98	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
2962 reflections	Extinction correction: *SHELXL*97
212 parameters	Extinction coefficient: 0.0018 (3)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O21—C2	1.234 (3)	O53—C51	1.333 (3)
O32—C31	1.342 (3)	O53—C54	1.453 (3)
O32—C33	1.445 (4)	N1—C2	1.368 (3)
O52—C51	1.204 (3)	N1—C6	1.394 (3)
C31—O32—C33	114.7 (2)	N1—C6—C61	111.3 (2)
C51—O53—C54	116.3 (2)	O32—C31—C3	121.9 (2)
C2—N1—C6	125.4 (2)	O32—C33—C34	109.8 (3)
O21—C2—N1	119.6 (2)	O52—C51—O53	122.0 (2)
O21—C2—C3	125.0 (2)	O52—C51—C5	122.3 (2)
N1—C2—C3	115.4 (2)	O53—C51—C5	115.8 (2)
N1—C6—C5	120.2 (2)	O53—C54—C55	107.7 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O21^{i}	0.86	2.03	2.885 (3)	171
C31—H31···O21	0.93	2.46	2.785 (3)	100
C61—H61*B*···O53	0.96	2.20	2.803 (3)	119

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

The value of the max shift/s.u. parameter corresponds to the torsion angle variation of atom H34 attached to C34 of a terminal methyl group. H atoms were positioned geometrically and included in the refinement, but were constrained to ride on their parent atoms. The isotropic displacement parameters of the H atoms were fixed at $1.3U_{eq}$ of their parent atoms.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PLATON* (Spek, 1990), *PARST* (Nardelli, 1995) and *PARSTCIF* (Nardelli, 1991).

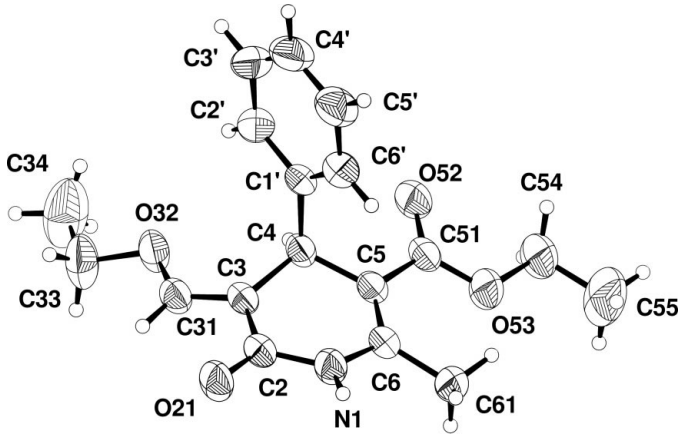


Figure 1
Plot showing the atomic numbering scheme in (I). Displacement ellipsoids for non-H atoms are shown at the 50% probability level.

HNdA thanks the K. U. Leuven (Belgium) for support through IRO Scholarships. MS is grateful to the Alma Mater projects, University of Havana (Cuba).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Bergerhoff, G. (1996). *DIAMOND*. Gerhard-Domagk-Str. 1, 53121 Bonn, Germany.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **95**, 1354–1358.
Goldmann, S. & Stoltefuss, J. (1991). *Angew. Chem. Int. Ed Engl.* **30**, 1559–1578.
Nardelli, M. (1991). *PARSTCIF*. University of Parma, Italy.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Overman, L., Tsuboi, S., Roos, J. & Taylor, G. (1980). *J. Am. Chem. Soc.* **102**, 747–754.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1996). *XSCANS*. Version 2.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Verdecia, Y., Suárez, M., Morales, A., Rodriguez, E., Ochoa, E., González, L., Martín, N., Quinteiro, M., Seaone, C. & Soto, J. L. (1996). *J. Chem. Soc. Perkin Trans.* pp. 947–952.