Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Diethyl 1-(*p*-fluorophenyl)-5-oxo-3-(2-thienyl)pyrrolidine-2,2-dicarboxylate

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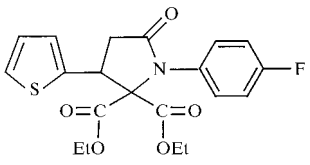
Received 19 September 2001
Accepted 9 October 2001

In the title compound, $C_{20}H_{20}FNO_5S$, the pyrrolidine ring adopts an envelope conformation. The fluorophenyl and thiophene rings are individually planar. The molecular and crystal structures are stabilized by intra- and intermolecular C—H···O interactions.

Comment

N-Phenyl-γ-lactams have been observed to exhibit Gram-positive and Gram-negative antibacterial activities (Ray *et al.*, 1995). Though the bioactivity of the γ-lactam derivatives is controlled by the substituents attached to the γ-lactam ring and includes the ability of several proteins to inhibit the crosslinking of the bacterial wall (Baldwin *et al.*, 1984), the introduction of a thiophene ring enhances the bioactivities of the γ-lactam systems so that they are comparable in bioactivity with ampicilines (Kar *et al.*, 1998). The title compound, (I), one of the thieno-γ-lactam derivatives having fluoro and *gem*-diethyl ester substituents, was synthesized in order to obtain a novel γ-lactam analogue with potential as a biological surrogate. The crystal structure of (I) was undertaken in order to elucidate its molecular conformation (Fig. 1).



(I)

The bond lengths and angles in (I) show normal values (Table 1) and agree with those of related structures studied previously (Sivakumar *et al.*, 1995*a*,*b*; Ray *et al.*, 1997). The pyrrolidine ring adopts an envelope conformation, with atom C9 deviating by −0.196 (3) Å from the C10/N1/C7/C8 plane and with puckering parameters (Cremer & Pople, 1975) Q_2 = 0.319 (3) Å and φ_2 = 114.9 (5)°. The fluorophenyl and thiophene rings are planar to within ±0.007 (4) and ±0.001 (4) Å, respectively, with a dihedral angle of 67.1 (2)° between them. These fluorophenyl and thiophene rings form dihedral angles of 81.7 (2) and 46.8 (2)° with the pyrrolidine ring, corresponding to an equatorial and a bisectional configuration, respectively.

One of the two ethyl carboxylate groups shows disorder in the C15 and C16 atoms, and both the major and minor configurations are in a crisscross pattern and deviate from the C10/C14/O4/O5 plane by 0.46 (2) and 0.73 (3) Å for C15*A* and

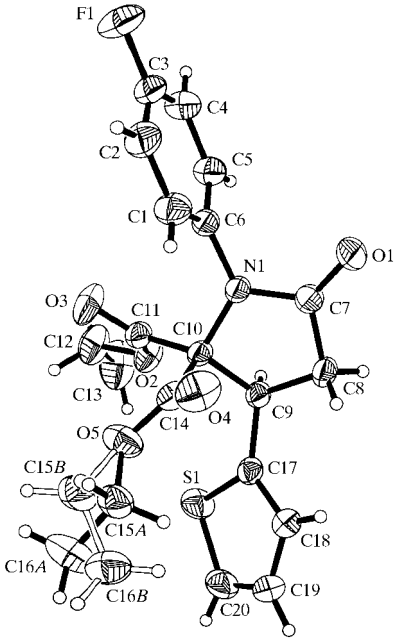


Figure 1
The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

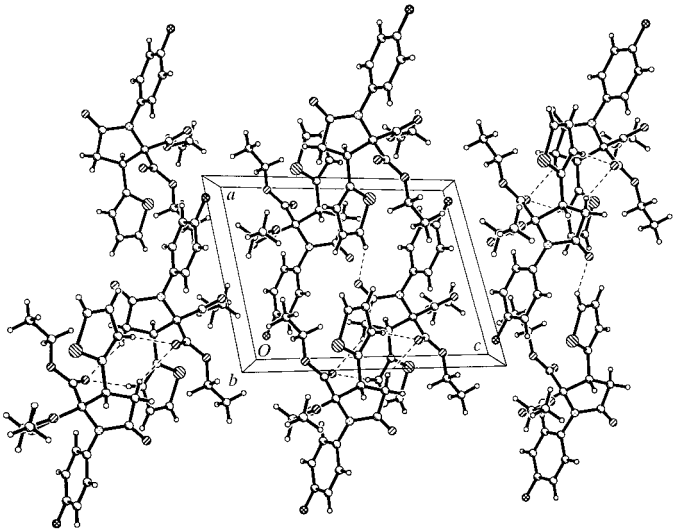


Figure 2
Packing diagram of the title compound viewed down the *b* axis. Disordered components are not shown as they are not involved in any hydrogen-bonding or weak interactions.

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C16*A*, and by −0.05 (2) and 1.31 (2) Å for C15*B* and C16*B*, respectively. In the other ethyl carboxylate group, atom O3 deviates by −0.118 (4) Å from the C10/C11/O2/C12/C13 plane, which makes an angle of 50.3 (3)° with the pyrrolidine-ring plane.

The molecular structure of (I) is maintained by an intramolecular interaction between C9 and O2 [2.718 (3) Å]. In the crystal, the molecules form four C—H···O interactions (Table 2) which, together with van der Waals interactions, stabilize the crystal structure (Fig. 2).

Experimental

The title compound was synthesized (overall yield 80%) from *p*-fluoroaniline (Aldrich) through the formation of arylaminomalonate (bromoethylmalonate) and condensation with 3-(2-thienyl)acrloyl chloride in the presence of triethylamine.

Crystal data

$C_{20}H_{20}FNO_5S$
M_r = 405.43
Triclinic, $P\overline{1}$
a = 9.6560 (2) Å
b = 10.1195 (1) Å
c = 12.3126 (2) Å
α = 110.704 (1)°
β = 92.659 (1)°
γ = 115.856 (1)°
V = 983.58 (3) Å^3
Z = 2
D_x = 1.369 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3864 reflections
θ = 1.8–28.6°
μ = 0.21 mm^{-1}
T = 293 (2) K
Slab, colourless
0.42 × 0.32 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
T_{min} = 0.919, T_{max} = 0.968
5604 measured reflections
3391 independent reflections
2358 reflections with $I > 2\sigma(I)$
R_{int} = 0.045
θ_{max} = 25.0°
$h = -9 \rightarrow 11$
$k = -11 \rightarrow 12$
$l = -11 \rightarrow 14$

Table 1
Selected geometric parameters (Å, °).

S1—C20	1.704 (3)	N1—C6	1.442 (3)
S1—C17	1.724 (2)	N1—C10	1.471 (3)
F1—C3	1.361 (3)	C7—C8	1.503 (4)
O1—C7	1.213 (3)	C8—C9	1.533 (3)
O2—C11	1.315 (3)	C9—C17	1.497 (3)
O2—C12	1.465 (3)	C9—C10	1.573 (3)
O3—C11	1.194 (3)	C10—C11	1.531 (3)
O4—C14	1.201 (3)	C17—C18	1.369 (4)
O5—C14	1.318 (3)	C18—C19	1.419 (4)
N1—C7	1.372 (3)	C19—C20	1.337 (5)
C20—S1—C17	92.45 (14)	C8—C9—C10	102.68 (18)
C7—N1—C6	121.00 (19)	N1—C10—C9	101.06 (18)
N1—C7—C8	108.0 (2)	C9—C17—S1	121.57 (18)
C12—O2—C11—C10	175.9 (2)		

Table 2
C—H···O interactions (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O4^{i}	0.97	2.54	3.415 (4)	150
C9—H9A···O2	0.98	2.36	2.718 (3)	100
C12—H12B···O3^{ii}	0.97	2.59	3.443 (5)	147
C18—H18A···O4^{i}	0.93	2.57	3.496 (5)	171
C20—H20B···O1^{iii}	0.93	2.47	3.327 (4)	154

Symmetry codes: (i) $-x, -y, 1-z$; (ii) $-1-x, -y, -z$; (iii) $1+x, 1+y, z$.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.161
S = 0.95
3391 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.096P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

After checking their presence in the difference map, all H atoms were fixed geometrically and allowed to ride on their parent atoms (C—H = 0.93–0.98 Å). Due to the large fraction of weak data at higher angles, 2θ was limited to a maximum of 50°.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610961, and AU wishes to thank Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1510). Services for accessing these data are described at the back of the journal.

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