$C_{22}H_{20}O_4S$

Crystal data

$C_{22}H_{20}O_4S$	Mo $K\alpha$ radiation
$M_r = 380.44$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 27
$P2_1/c$	reflections
$a = 9.3028\,(10)$ Å	$\theta = 5.15\text{--}12.53°$
$b = 8.5506\,(6)$ Å	$\mu = 0.196$ mm^{-1}
$c = 23.960\,(2)$ Å	$T = 293\,(2)$ K
$\beta = 97.636\,(8)°$	Rectangular block
$V = 1889.0\,(3)$ Å^3	$0.84 \times 0.68 \times 0.54$ mm
$Z = 4$	Colourless
$D_x = 1.338$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens $P4$ diffractometer	$\theta_{max} = 27.49°$
$\theta/2\theta$ scans	$h = -1 \rightarrow 12$
Absorption correction: none	$k = -1 \rightarrow 11$
5750 measured reflections	$l = -31 \rightarrow 31$
4303 independent reflections	3 standard reflections
3450 reflections with	every 97 reflections
$\quad I > 2\sigma(I)$	intensity decay: <3%
$R_{int} = 0.017$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = -0.007$
$R[F^2 > 2\sigma(F^2)] = 0.040$	$\Delta\rho_{max} = 0.217$ e Å^{-3}
$wR(F^2) = 0.116$	$\Delta\rho_{min} = -0.238$ e Å^{-3}
$S = 1.048$	Extinction correction: none
4303 reflections	Scattering factors from
324 parameters	*International Tables for*
All H atoms refined	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2$	
$\quad + 0.183P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Selected torsion angles (°)*

C12—C7—C8—C15	4.0 (2)	C12—C13—C14—C15	−57.7 (2)
C8—C7—C12—C13	−19.1 (2)	C7—C8—C15—C14	−15.7 (2)
C7—C12—C13—C14	46.0 (2)	C13—C14—C15—C8	41.5 (2)

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
C6—H6$\cdots$O11^i	1.01 (2)	2.58 (2)	3.470 (2)	147 (2)
C19—H19$\cdots$O11^{ii}	0.98 (2)	2.45 (2)	3.378 (2)	159 (2)

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

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometrical calculations *PARST* (Nardelli, 1983b).

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Diethyl 1-(3,4-Dichlorophenyl)-5-oxo-3-(2-thienyl)-2,2-pyrrolidinedicarboxylate

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Abstract

In the title molecule, $C_{20}H_{19}Cl_2NO_5S$, the pyrrolidine ring is in an envelope conformation. The dichlorophenyl and thiophene rings are planar. Of the two ethoxycarbonyl side chains, one is nearly planar but the other is distorted from planarity. The structure is stabilized by weak C—H$\cdots$O hydrogen bonds and van der Waals interactions.

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Comment

γ-Lactams are not only important constituents of novel antibiotics (Nozaki *et al.*, 1987), but are also key intermediates in the synthesis of five-membered heterocycles (Laskin & Lechevalier, 1984). The crystal structure determination of the title compound, (I), a γ-lactam with *gem*-diethyl ester substituents was carried out in order to elucidate the molecular conformation.

(I)
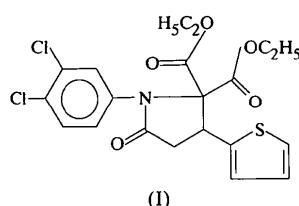
A displacement ellipsoid plot of (I) with the atom-numbering scheme is shown in Fig. 1. The pyrrolidine ring is in an envelope conformation, with asymmetry parameter ΔC_s(C9) = 0.024 (2) (Nardelli, 1983a). The deviation of the C9 atom from the mean plane defined by atoms N, C7, C8 and C10 is 0.514 (3) Å. The planes of the dichlorophenyl and thiophene rings define a dihedral angle of 107.3 (1)°. The thiophene ring shows no sign of the disorder typical of related structures (Sivakumar *et al.*, 1995a,b; Ray *et al.*, 1997) and exhibits normal C—C bond lengths in the thiophene ring.
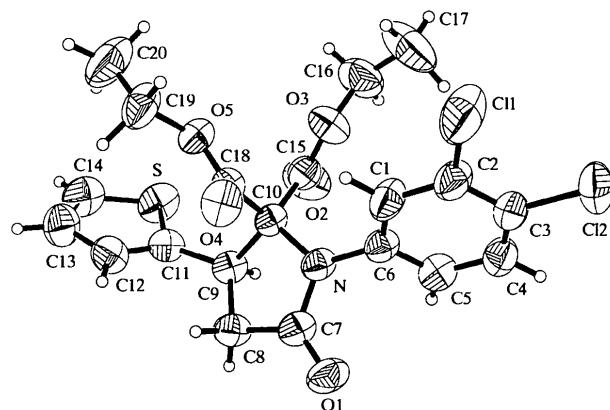

Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

The mean plane through the pyrrolidine ring makes dihedral angles of 58.2 (1) and 59.9 (1)° with the thiophene and phenyl rings, respectively. One ethoxycarbonyl side chain [C18(=O4)—O5—C19—C20] is nearly planar, but the other is distorted from planarity [C15—O3—C16—C17 −153.9 (5)°] due to steric interactions. Slight disorder of the C17 atom cannot be

excluded given its large U_{11} value [0.211 (8) Å^2]. The two ethoxycarbonyl planes make angles of 110.1 (1) and 111.8 (2)° with the phenyl-ring plane, and angles of 32.4 (2) and 76.8 (2)° with the thiophene-ring plane. The crystal structure is stabilized by weak C—H $\cdots$ O hydrogen bonds (Table 2) and van der Waals interactions.

Experimental

The title compound was synthesized directly from arylaminomalonates and arylacryloyl chloride in the presence of base (Ray *et al.*, 1994). Single crystals were grown by slow evaporation from a 2-propanol solution of the compound.

Crystal data

$C_{20}H_{19}Cl_2NO_5S$	Mo $K\alpha$ radiation
$M_r = 456.32$	$\lambda = 0.71073$ Å
Triclinic	Cell parameters from 28
$P\bar{1}$	reflections
$a = 10.441$ (1) Å	$\theta = 5.39$–$12.32°$
$b = 10.772$ (1) Å	$\mu = 0.433$ mm^{-1}
$c = 11.079$ (1) Å	$T = 293$ (2) K
$\alpha = 87.95$ (1)°	Rectangular slab
$\beta = 75.16$ (1)°	$0.42 \times 0.32 \times 0.28$ mm
$\gamma = 62.90$ (1)°	Colourless
$V = 1067.5$ (2) Å^3	
$Z = 2$	
$D_x = 1.420$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens *P4* diffractometer	$R_{int} = 0.036$
$\theta/2\theta$ scans	$\theta_{max} = 27.50°$
Absorption correction:	$h = -1 \rightarrow 13$
empirical ψ scans	$k = -12 \rightarrow 13$
(Siemens, 1994)	$l = -14 \rightarrow 14$
$T_{min} = 0.814$, $T_{max} = 0.877$	3 standard reflections
5718 measured reflections	every 97 reflections
4910 independent reflections	intensity decay: <3%
3078 reflections with	
$I > 2\sigma(I)$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.053$	$\Delta\rho_{max} = 0.31$ e Å^{-3}
$wR(F^2) = 0.141$	$\Delta\rho_{min} = -0.36$ e Å^{-3}
$S = 1.015$	Extinction correction:
4909 reflections	*SHELXL93*
339 parameters	Extinction coefficient:
All H atoms refined	0.009 (2)
$w = 1/[\sigma^2(F_o^2) + (0.0435P)^2$	Scattering factors from
$+ 0.5622P]$	*International Tables for*
where $P = (F_o^2 + 2F_c^2)/3$	*Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S—C14	1.696 (4)	C7—C8	1.503 (4)
S—C11	1.721 (3)	C8—C9	1.523 (4)
O1—C7	1.214 (3)	C9—C11	1.496 (4)
O2—C15	1.189 (3)	C9—C10	1.574 (3)

O3—C15	1.313 (3)	C10—C18	1.525 (3)
O3—C16	1.461 (4)	C10—C15	1.535 (4)
O4—C18	1.194 (3)	C11—C12	1.358 (4)
O5—C18	1.320 (3)	C12—C13	1.412 (4)
O5—C19	1.467 (4)	C13—C14	1.346 (5)
N—C7	1.377 (4)	C16—C17	1.470 (7)
N—C6	1.434 (3)	C19—C20	1.489 (7)
N—C10	1.470 (3)		
C14—S—C11	92.3 (2)	N—C10—C15	110.2 (2)
C15—O3—C16	118.0 (3)	C18—C10—C15	112.1 (2)
C18—O5—C19	116.0 (2)	N—C10—C9	101.2 (2)
C7—N—C10	113.0 (2)	C18—C10—C9	109.9 (2)
C6—N—C10	125.6 (2)	C15—C10—C9	111.3 (2)
C5—C6—N	119.2 (2)	C12—C11—C9	129.3 (2)
O1—C7—N	124.4 (3)	C12—C11—S	110.3 (2)
N—C7—C8	108.2 (2)	C9—C11—S	120.4 (2)
C7—C8—C9	104.4 (2)	C11—C12—C13	112.8 (3)
C11—C9—C8	115.4 (2)	C14—C13—C12	113.1 (4)
C8—C9—C10	103.0 (2)	C13—C14—S	111.5 (3)
N—C10—C18	111.7 (2)		
C10—N—C7—C8	4.0 (3)	C8—C9—C10—N	30.8 (3)
N—C7—C8—C9	17.0 (3)	N—C10—C15—O2	98.5 (3)
C7—C8—C9—C10	−29.4 (3)	N—C10—C18—O4	−17.0 (4)
C7—N—C10—C9	−22.2 (3)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C4—H4⋯O1^i	1.00 (3)	2.57 (3)	3.366 (3)	137 (3)
C12—H12⋯O4^{ii}	0.91 (4)	2.52 (4)	3.351 (5)	153 (3)
C14—H14⋯O1^{iii}	0.92 (5)	2.58 (5)	3.433 (6)	154 (4)

Symmetry codes: (i) $2-x, -y, 1-z$; (ii) $1-x, -y, 2-z$; (iii) $x-1, y, z$.

The title structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Programs used for data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994); for structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); for structure refinement: *SHELXL93* (Sheldrick, 1993); for geometrical calculations: *PARST* (Nardelli, 1983b).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1338). Services for accessing these data are described at the back of the journal.

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2-Acetyl-5,8-dihydronaphthalen-1-ol

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Abstract

The heavy-atom skeleton of the title molecule, $C_{12}H_{12}O_2$, is planar to within ±0.023 (2) Å and an O—H⋯O intramolecular hydrogen bond contributes to this planarity.

Comment

Dihydronaphthalene derivatives are widely used as intermediates in the synthesis of several polycyclic phenols which are useful antifibrillatory agents, disinfectants and water softeners (Hauck *et al.*, 1977). Furthermore, hydroxy–ketone derivatives of naphthalene are useful in synthesizing the sub-units of daunomycinone and adiramycin, which are important anticancer drugs (Crouse *et al.*, 1981).

The title molecule, (I), as a whole, is planar within ±0.023 (2) Å. The planarity is stabilized by an O—H⋯O intramolecular hydrogen bond involving atoms O1 and O2 [O1⋯O2 2.546 (2), H1O2⋯O1 1.65 (2) Å and O2—H1O2⋯O1 154 (2)°]. In the dihydrobenzene ring, the C_{sp^2}—C_{sp^3} distances C5—C6 [1.481 (2) Å] and C9—C10 [1.491 (2) Å] are longer than the C6—C7 [1.465 (2) Å] and C8—C9 [1.461 (3) Å] distances because of the steric interactions caused by the planarity of the dihydrobenzene ring. The C5—C6—C7 [115.1 (2)°] and C8—C9—C10 [115.5 (2)°] angles are also widened