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(2*SR*,5*SR*,6*SR*)-Ethyl 5-[(4-chlorophenyl)sulfanyl]-2,6-bis(4-fluorophenyl)-4-hydroxy-1,2,5,6-tetrahydropyridine-3-carboxylate

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Key indicators

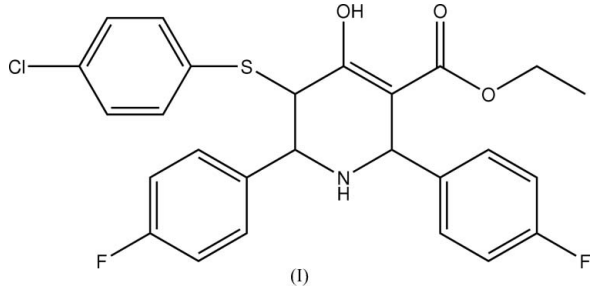
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.141
Data-to-parameter ratio = 13.7

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

In the title compound, $C_{26}H_{22}ClF_2NO_3S$, a polysubstituted piperidine enol, the tetrahydropyridine ring adopts a half-chair conformation. The crystal structure is stabilized by van der Waals and weak C—H···π interactions. An intramolecular O—H···S interaction generates an *S*(5) graph-set motif.

Comment

The piperidine ring is a distinct structural feature of a variety of alkaloid natural products and drug candidates. Watson *et al.* (2000) observed that during the past decade thousands of piperidine compounds were mentioned in clinical and preclinical studies. Piperidinones, though relatively less prominent, have also been regarded as precursors of a host of biologically active compounds and natural alkaloids, prior to their conversion to piperidines. Recently, we have determined the crystal structures of some piperidinone derivatives with varying substituents on the phenyl rings of the piperidone, namely 4-chlorophenyl (Suresh *et al.*, 2007*a*), *p*-tolyl (Suresh *et al.*, 2007*b*) and 4-fluorophenyl (Suresh *et al.*, 2007*c*). As an extension of our work, the structure of the title compound, (I), is reported here.



(I)

The piperidine enol ring (Fig. 1) adopts a half-chair conformation, as indicated by the puckering parameters Q = 0.517 (3)Å, θ = 125.4 (3) and φ = 157.9 (5)° (Cremer & Pople, 1975). The half-chair conformation is also evident from the torsion angles in the piperidine ring (Table 1). The chlorophenylsulfanyl group is *cis* to the fluorophenyl group at C6, making a dihedral angle of 19.3 (2)°. The ethoxycarbonyl group at C3 is in a nearly perpendicular orientation to the fluorophenyl ring at C2, forming a dihedral angle of 85.5 (2)°. The bond distances (Table 1) show the conjugation of S1 with the phenyl ring and of O3 with the C7═O2 carbonyl group. The piperidine ring has three chiral C atoms, *viz*. C2, C5 and C6. Of the eight possible stereoisomers, those present in this structure are *S*,*S*,*S* and its enantiomer *R*,*R*,*R*.

An intramolecular hydrogen bond found between hydroxyl and phenylsulfanyl groups (O1—H1*A*···S1; Table 2) generates a graph-set motif of *S*(5) (Etter, 1990; Bernstein *et al.*, 1995). The crystal structure is stabilized by van der Waals interactions, and by three weak C—H···π interactions (Table 2).

Experimental

To a solution of ammonium acetate (0.142 g, 2 mmol) in ethanol (20 ml), was added a mixture of ethyl 4-[(4-chlorophenyl)sulfanyl]-3-oxobutanoate (0.5 g, 2mmol) and freshly distilled *p*-fluorobenzaldehyde (0.39 ml, 4 mmol). The resulting mixture was warmed on a water bath for 5 min and then set aside at room temperature. The precipitate was filtered off and recrystallized from ethanol (yield 0.26 g, 52%; m.p. 402–403 K).

Crystal data

$C_{26}H_{22}ClF_2NO_3S$
M_r = 501.96
Monoclinic, $P2_1/c$
a = 10.1341 (9) Å
b = 13.8290 (13) Å
c = 17.9627 (15) Å
β = 103.729 (17)°
V = 2445.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 (2) K
0.20 × 0.16 × 0.11 mm

Data collection

Nonius MACH-3 four-circle diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.945, T_{max} = 0.969
4955 measured reflections
4290 independent reflections
2252 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.141
S = 1.01
4290 reflections
313 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C5—S1	1.843 (3)	C8—O3	1.436 (4)
C7—O2	1.222 (4)	C51—S1	1.786 (3)
C7—O3	1.352 (4)		
N1—C2—C3—C4	−15.9 (4)	C4—C5—C6—N1	40.7 (3)
C21—C2—C3—C4	112.5 (3)	C4—C5—C6—C61	160.3 (3)
C21—C2—C3—C7	−72.7 (4)	C3—C2—N1—C6	54.3 (4)
C2—C3—C4—C5	−6.0 (5)	C61—C6—N1—C2	169.3 (3)
C3—C4—C5—C6	−7.4 (4)	C5—C6—N1—C2	−67.5 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···S1	0.82	2.55	3.075 (3)	123
C22—H22···*Cg*1	0.93	2.77	3.598 (4)	149
C25—H25···*Cg*2^{i}	0.93	2.88	3.792 (5)	167
C55—H55···*Cg*2^{ii}	0.93	2.96	3.771 (4)	146

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$. *Cg*1 and *Cg*2 are the C51–C56 and C61–C66 ring centroids.

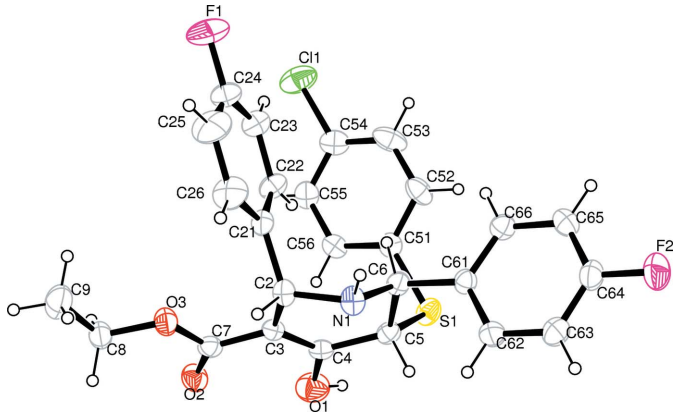


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

The N-bound H atom was located in a difference Fourier map and its parameters were refined. O- and C-bound H atoms were placed at calculated positions, with C—H = 0.93–0.98 Å and O—H = 0.82 Å, and allowed to ride on their carrier atoms, with U_{iso}(H) = 1.2U_{eq}(C,N) for CH_2 and CH groups, and 1.5U_{eq}(C,O) for CH_3 and OH groups.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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