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3-Ethyl-2-phenylsulfonyl-1,2,3,3a,10,10a,11,11a-octahydro-5*H*-pyrrolo[3,4:2′,3′]pyrrolo[1,5-*b*]-isoquinolin-5-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.001 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.131
Data-to-parameter ratio = 44.8

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

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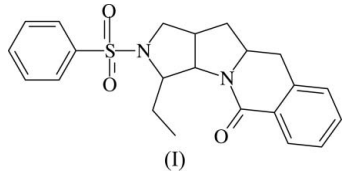
In the title molecule, $C_{22}H_{24}N_2O_3S$, the fused pyridinone ring adopts a half-chair conformation while the pyrrolidine rings adopt envelope and twist conformations. C—H···O interactions link the molecules into a three-dimensional network.

Comment

Isoquinolin-1-ones are potent 5-HT3 receptor antagonists (Clark *et al.*, 1993). Pyrrolopyrrole compounds are used as anti-inflammatory and analgesic agents (Muchowski *et al.*, 1989) and some of them act as potent and selective orphanin FQ/nociceptin (N/OFQ) receptor (NOP) agonists (Kolczewski *et al.*, 2003). We report here the crystal structure of the title compound, (I) (Fig. 1).



(I)

Bond distances and angles in the phenylsulfonyl-pyrrolopyrrole fragment of (I) (Fig. 1) are comparable to those observed in other similar structures (Senthil Kumar *et al.*, 2006*a*,*b*,*c*). The N1/C1–C4 pyrrolidine ring adopts a twist conformation. In the other pyrrolidine ring, one conformer (with N2) adopts an envelope conformation with atom C5 at the flap position, while the other conformer (with N2*A*) is in a twist conformation. The Cremer & Pople (1975) puckering parameters q_2 and φ_2 are, respectively, 0.2497 (9) Å and 93.2 (2)° for the N1/C1–C4 ring, 0.3562 (16) Å and 285.9 (5)° for the N2/C3/C2/C5/C6 ring, and 0.3433 (14) Å and 267.9 (6)° for the N2*A*/C3/C2/C5/C6 ring. Both conformers of the pyridinone ring adopt half-chair conformations, with a smallest displacement asymmetry parameter (Nardelli, 1999) ΔC_2(C6–C7) of 8.8 (3) (N2) or 6.6 (4)° (N2*A*). The dihedral angle between the benzene (C8/C9/C11–C14) and phenyl (C15–C20) rings is 34.42 (3)°.

In the crystal structure, C3—H3···O1^{ii} intermolecular hydrogen bonds link the molecules into a chain along the *c* axis. Adjacent chains are cross-linked *via* C1—H1*A*···O2^{i}, C14—H14···O3^{iii} and C22—H22*A*···O3^{iv} interactions, forming a three-dimensional framework (Fig. 2); see Table 1 for symmetry codes.

Experimental

A mixture of 2-(*N*-allyl-*N*-phenylsulfonylamino)butanal (1 mmol) and isoquinoline-3-carboxylic acid (1 mmol) in toluene (20 ml) was

refluxed until the starting materials had disappeared, as indicated by thin-layer chromatography. The solvent was evaporated *in vacuo* and the residue was column chromatographed with a hexane–ethyl acetate mixture (8:2 *v*/*v*) to yield the title compound. Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{22}H_{24}N_2O_3S$
M_r = 396.49
Monoclinic, $P2_1/c$
a = 10.7021 (2) Å
b = 11.8115 (2) Å
c = 15.7264 (3) Å
β = 106.660 (1)°
V = 1904.49 (6) Å^3
Z = 4
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 100.0 (1) K
Block, yellow
0.58 × 0.55 × 0.45 mm

Data collection

Bruker SMART APEX2 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.890, T_{max} = 0.917
40101 measured reflections
11737 independent reflections
9292 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 40.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.131
S = 1.07
11737 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.5945P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.94 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1*A*···O2^{i}	0.99	2.56	3.2113 (11)	124
C3—H3···O1^{ii}	1.00	2.46	3.2798 (10)	139
C4—H4···O3	1.00	2.49	3.0481 (11)	115
C14—H14···O3^{iii}	0.95	2.52	3.3214 (12)	141
C16—H16···O1	0.95	2.56	2.9229 (13)	103
C22—H22*A*···O3^{iv}	0.98	2.56	3.4371 (13)	149

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x, -y+1, -z+1$.

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95–1.00 Å and U_{iso}(H) = 1.2 or 1.5 (methyl) times U_{eq}(C). One of the N atoms is disordered over two distinct sites, N2 and N2*A*. The occupancy factors for these positions were initially refined and later fixed at 0.5. The C—N bond lengths involving the disordered atoms were restrained to be equal.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

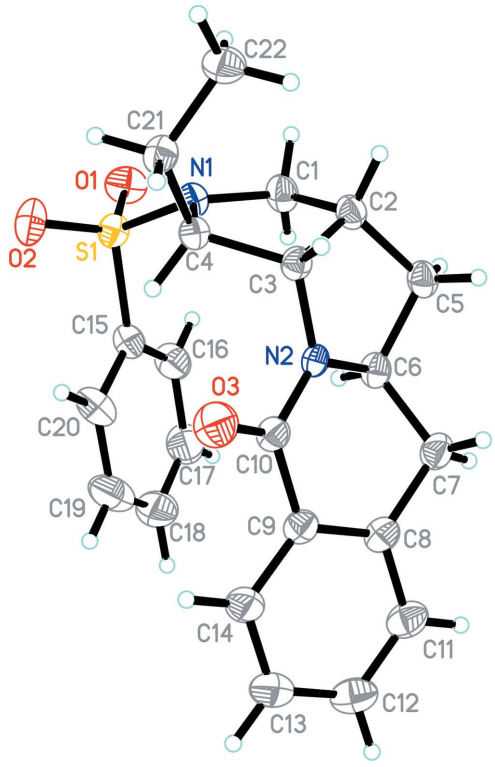


Figure 1
The molecular structure of (I), showing 70% probability displacement ellipsoids and the atom-numbering scheme. Only one component of the disordered N atom is shown.

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