Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-[2-(3-Ethyl-4-methyl-2-oxo-3-pyrrolidine-1-carboxamido)ethyl]benzenesulfonamide

Xiang Li

Chemistry and Chemical Engineering Department, Pingdingshan Institute of Technology, Pingdingshan 467000, People's Republic of China

Correspondence e-mail: lixiang_acta@yahoo.com.cn

Received 21 May 2006
Accepted 7 June 2006

In the title molecular structure, $C_{16}H_{21}N_3O_4S$, the dihedral angle between the essentially planar 2,5-dihydro-1*H*-pyrrole and benzene rings is 20.8 (2)°. In the crystal structure, molecules are linked by N—H···O hydrogen bonds [H···O = 2.07 (3)–2.57 (3) Å] to form a two-dimensional network.

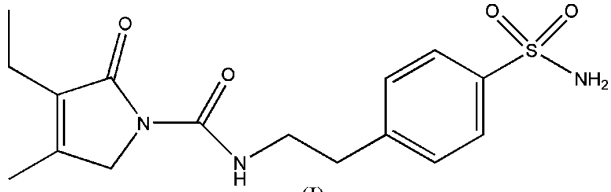
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.051
wR factor = 0.126
Data-to-parameter ratio = 13.5

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

Comment

The title compound is an intermediate of glimepiride (Hoe490), which is an oral antidiabetic drug (Kirchheiner *et al.*, 2005). The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles (Table 1) are in agreement with values reported for glimepiride (Iwata *et al.*, 1997). The N—S bond length is 1.603 (2) Å, in good agreement with the mean value of 1.600 Å reported by Allen *et al.* (1987). The deviation of atom C5 from the C1–C5/C7/C8/O1/O2/N1/N2 plane is 0.056 (3) Å. Atoms C10 and S1 are approximately in the plane of the benzene ring (C11–C16), the largest deviation being 0.018 (2) Å for atom S1. The dihedral angle between the essentially planar 2,5-dihydro-1*H*-pyrrole and benzene rings is 20.8 (2)°. In the crystal structure, molecules are linked by N—H···O hydrogen bonds to form a two-dimensional network (Fig. 2 and Table 2).



(I)

Experimental

The title compound was prepared from 3-ethyl-4-methyl-2-oxo-3-pyrrolidine (0.01 mol) and 2-phenylethyl isocyanate (0.01 mol),

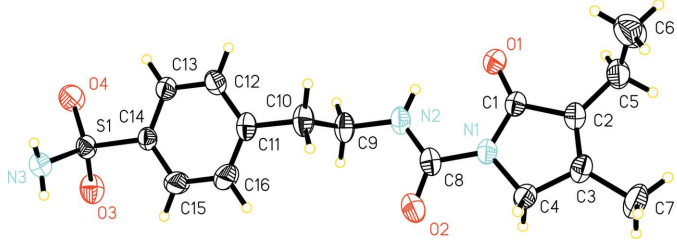


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 40% probability level. H atoms are shown as small spheres.

which were introduced with cooling and agitation into chlorosulfonic acid (0.01 mol). The mixture was poured on to ice, whereupon the sulfochloride was separated and treated with concentrated ammonia (20 ml). The sulfonamide was suction-filtered and recrystallized from ethanol (Weyer *et al.*, 1980) (yield 86%, 3.02 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a methanol solution of the title compound at room temperature for two weeks.

Crystal data

$C_{16}H_{21}N_3O_4S$
M_r = 351.42
Monoclinic, $P2_1/n$
a = 8.2325 (11) Å
b = 14.2854 (18) Å
c = 14.8231 (19) Å
β = 93.474 (2)°
V = 1740.1 (4) Å^3
Z = 4
D_x = 1.341 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 298 (2) K
Block, colorless
0.51 × 0.41 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.900, T_{max} = 0.930
8711 measured reflections
3058 independent reflections
2675 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.126
S = 1.10
3058 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.8277P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0173 (16)

Table 1
Selected geometric parameters (Å, °).

S1—O3	1.4261 (19)	N1—C1	1.383 (3)
S1—O4	1.4263 (18)	N1—C8	1.399 (3)
S1—N3	1.603 (2)	N1—C4	1.457 (3)
S1—C14	1.767 (2)	N2—C8	1.332 (3)
O1—C1	1.227 (3)	N2—C9	1.457 (3)
O2—C8	1.224 (3)		
O3—S1—O4	119.68 (12)	C1—N1—C4	110.75 (18)
N3—S1—C14	107.96 (11)	C8—N2—C9	121.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*A*···O1	0.86	2.08	2.742 (3)	134
N3—H3*A*···O1^{i}	0.80 (3)	2.57 (3)	3.193 (3)	135 (3)
N3—H3*B*···O2^{ii}	0.79 (3)	2.07 (3)	2.851 (3)	168 (3)

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

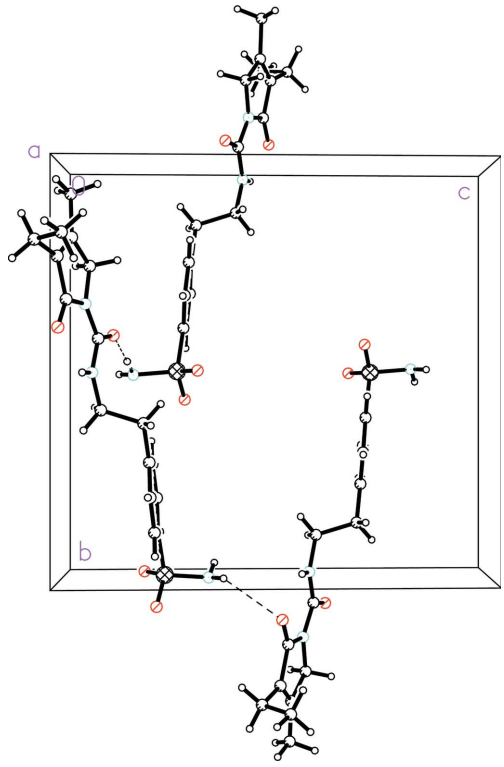


Figure 2
Partial packing plot of (I) (Spek, 2003), showing hydrogen bonds as dashed lines.

The positions and isotropic displacement parameters of the amino H atoms (H3*A* and H3*B*) were refined independently. All other H atoms were placed in calculated positions (C—H = 0.93–0.97 Å and N—H = 0.86 Å) and were constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C,N), or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Iwata, M., Nagase, H., Endo, T. & Ueda, H. (1997). *Acta Cryst.* C**53**, 329–331.
Kirchheiner, J., Roots, I., Goldammer, M., Rosenkranz, B. & Brockmoller, J. (2005). *Clin. Pharmacokinet.* **44**, 1209–1225.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Weyer, R., Geisen, K., Hitzel, V., Geisen, K. & Regitz, G. (1980). US Patent 4 3797 85 (CA: 95,168964c).