Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*S*)-N^5-Ethyl-N^2-(2-nitrophenylsulfonyl)glutamine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.070
Data-to-parameter ratio = 10.9

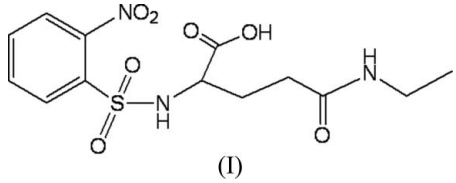
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 March 2007
Accepted 16 April 2007

The title compound, $C_{13}H_{17}N_3O_7S$, is a potential AHAS (acetohydroxyacid synthase) inhibitor. In the crystal structure, the nitro group is twisted away from the plane of the aromatic ring and the glutamine residue adopts a folded conformation. The crystal packing is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds.

Comment

Based on the AHAS (acetohydroxyacid synthase, EC 2.2.1.6) crystal structure (McCourt *et al.*, 2006), we have succeeded in identifying a few novel AHAS inhibitors (Wang, *et al.*, 2007). Among the 296 possible inhibitors from that virtual screening, we have also synthesized some new compounds with altered structure and validated their *in vivo* and *in vitro* biological activity (Wang *et al.*, 2006). These results indicated that it was possible to design new lead herbicidal AHAS inhibitors from a computer-aided design strategy. We previously reported the crystal structure of N^2-(2-nitrophenylsulfonyl)-N^5-*n*-propyl-glutamine, (II) (Xiao *et al.*, 2005). In order to further investigate the structure–activity relationship of this series of compounds, we have obtained and determined the crystal structure of another compound, (I), in this series (Fig. 1).



(I)

The X-ray crystallographic analysis reveals that all the bond lengths and angles in (I) show normal values (Allen *et al.*,

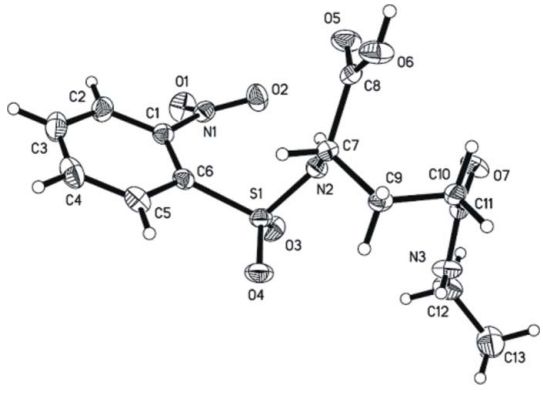


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level (arbitrary spheres for the H atoms).

1987). The chiral atom C7 has an *S* configuration. The C9—C10—C11—O7 and C9—C10—C11—N3 torsion angles of 115.8 (2) and −65.7 (2)°, respectively, are significantly different from the corresponding values of 127.2 (2) and −54.1 (2)° in (II).

The crystal structure of (I) is stabilized by N—H···O and O—H···O hydrogen bonds (Table 1 and Fig. 2).

Experimental

The title compound was synthesized according to the method of Srikanth *et al.* (2002). Colourless single crystals of (I) were obtained by recrystallization from ethanol and water (19:1 *v*/*v*).

Crystal data

$C_{13}H_{17}N_3O_7S$
M_r = 359.36
Monoclinic, $P2_1$
a = 6.7756 (10) Å
b = 7.4223 (11) Å
c = 15.848 (2) Å
β = 91.901 (2)°
V = 796.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 (2) K
0.22 × 0.16 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.928, T_{max} = 0.966
4510 measured reflections
2469 independent reflections
2350 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.070
S = 1.06
2469 reflections
227 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 722 Friedel Pairs
Flack parameter: −0.08 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O5	0.81 (2)	2.25 (2)	2.641 (2)	110.7 (19)
N2—H2···O2	0.81 (2)	2.29 (2)	2.918 (2)	135 (2)
N3—H3···O5^{i}	0.73 (3)	2.35 (3)	3.053 (3)	164 (3)
O6—H6···O7^{ii}	0.79 (3)	1.77 (3)	2.546 (2)	173 (3)

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

The N- and O-bound H atoms were located in difference maps and their positions were freely refined with U_{iso}(H) = 1.2U_{eq}(carrier). The C-bound H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *SMART* (Bruker, 1999); 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); software used to prepare material for publication: *SHELXTL*.

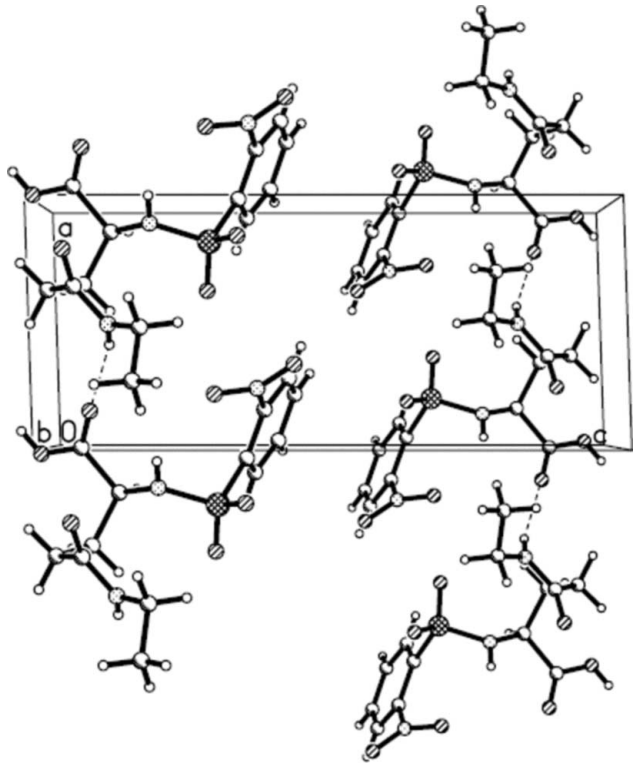


Figure 2
The packing in (I), with hydrogen bonds shown as dashed lines.

This project was supported by the National Natural Science Foundation of China (No. 20602021) and the Basic Research Development Program of China (973 Program) (grant No. 2003CB114406).

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