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N^2-(2-Nitrophenylsulfonyl)-N^5-*n*-propylglutamine

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.068
Data-to-parameter ratio = 13.8

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

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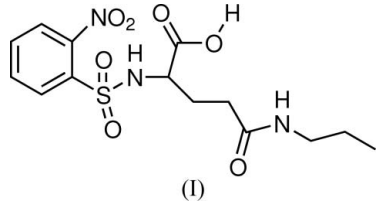
The title compound, $C_{14}H_{19}N_3O_7S$, is a potent new herbicide. X-ray analysis reveals that the nitro group is twisted away from the benzene ring and the glutamine residue adopts a folded conformation. The crystal packing is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds.

Comment

Based on the reported 2.8 Å high-resolution crystal structure of yeast AHAS (acetohydroxyacid synthase) complex (Pang *et al.*, 2003), we obtained 300 molecules with low binding energy toward AHAS from the MDL/ACD three-dimensional database, by searching with the program *DOCK 4.0* (Wang *et al.*, 2005). These structures provide information for further design of new targeted AHAS herbicidal molecules. According to the structural information and bioactivity data of 5-*N*-substituted-2-(substituted benzenesulfonyl)glutamine, a series of these derivatives has been designed and synthesized. The X-ray crystal structure determination of the title compound, (I), was undertaken to investigate the relationship between structure and herbicidal activity.



(I)

The bond lengths and angles in (I) show normal values (Table 1). The O3—S1—O4 angle [120.39 (10)°] deviates significantly from the ideal tetrahedral value. The O—N—C—C torsion angles indicate that the nitro group is twisted away from the plane of the benzene ring (Fig. 1). In the glutamine residue, the ψ^1 (N2—C7—C8—O5), ψ^2 (N2—C7—C8—O6), χ^1 (N2—C7—C9—C10) and χ^2 (C7—C9—C10—C11) torsion angles are 11.2 (3), −169.67 (16), 66.3 (2) and −62.7 (2)°, respectively; the two planar groups in the residue, C7/C8/O5/O6 and C10/C11/O7/N3, form a dihedral angle of 32.0 (1)°.

The molecular structure is stabilized by intramolecular N—H···O hydrogen bonds (Table 2). The crystal structure involves intermolecular O—H···O and N—H···O hydrogen bonds, which link the molecules into a layer parallel to the *ab* plane (Fig. 2).

Experimental

Compound (I) was prepared according to the procedure of Srikanth *et al.* (2002). Colourless single crystals suitable for X-ray diffraction

analysis were obtained by recrystallization from ethanol and water (4:1 *v*/*v*).

Crystal data

$C_{14}H_{19}N_3O_7S$
M_r = 373.38
Monoclinic, $P2_1$
a = 6.9215 (13) Å
b = 7.7539 (16) Å
c = 15.797 (3) Å
β = 91.858 (3)°
V = 847.3 (3) Å^3
Z = 2
D_x = 1.463 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2760 reflections
θ = 2.9–26.2°
μ = 0.23 mm^{-1}
T = 294 (2) K
Prism, colourless
0.26 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.935, T_{max} = 0.959
4767 measured reflections
3293 independent reflections
2977 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 26.4°
$h = -8 \rightarrow 5$
$k = -9 \rightarrow 9$
$l = -16 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.069
S = 1.06
3293 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 0.1123P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), 1439 Friedel Pairs
Flack parameter: 0.00 (6)

Table 1
Selected geometric parameters (Å, °).

S1—N2	1.6157 (16)	N1—C1	1.470 (3)
S1—C6	1.793 (2)	N2—C7	1.469 (2)
O5—C8	1.198 (2)	N3—C11	1.326 (2)
O6—C8	1.305 (2)	N3—C12	1.459 (3)
O7—C11	1.245 (2)		
O5—C8—O6	125.90 (18)	O7—C11—C10	121.98 (17)
O6—C8—C7	111.43 (18)	N3—C12—C13	112.45 (18)
O1—N1—C1—C2	−45.2 (3)	C12—N3—C11—O7	−4.5 (3)
O2—N1—C1—C2	132.6 (2)	C9—C10—C11—O7	127.2 (2)
O1—N1—C1—C6	135.4 (2)	C9—C10—C11—N3	−54.1 (2)
O2—N1—C1—C6	−46.8 (3)	N3—C12—C13—C14	54.2 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O7^{i}	0.82 (3)	1.73 (3)	2.549 (2)	172 (3)
N2—H2A···O5	0.81 (2)	2.19 (2)	2.639 (2)	115 (2)
N2—H2A···O2	0.81 (2)	2.35 (2)	2.952 (2)	132 (2)
N3—H3A···O5^{ii}	0.83 (2)	2.52 (2)	3.190 (2)	139 (2)

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

The amine and hydroxy H atoms were located in a difference map and refined isotropically (see Table 2 for distances). The C-bound H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

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); software used to prepare material for publication: *SHELXTL*.

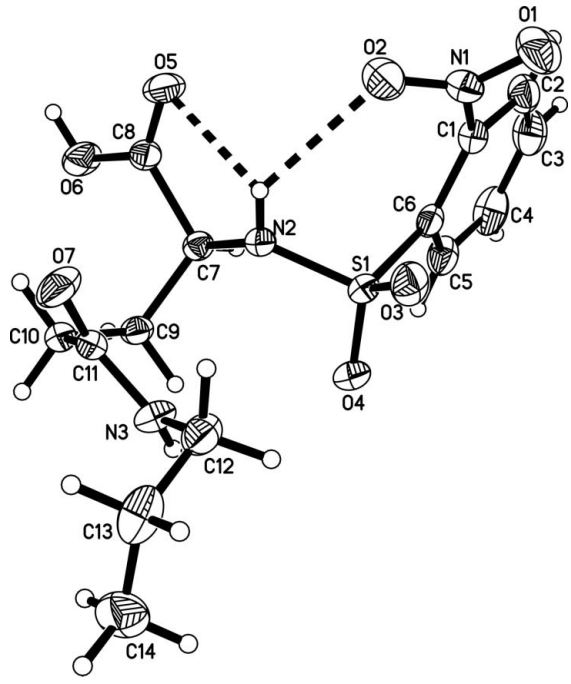


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate intramolecular hydrogen bonds.

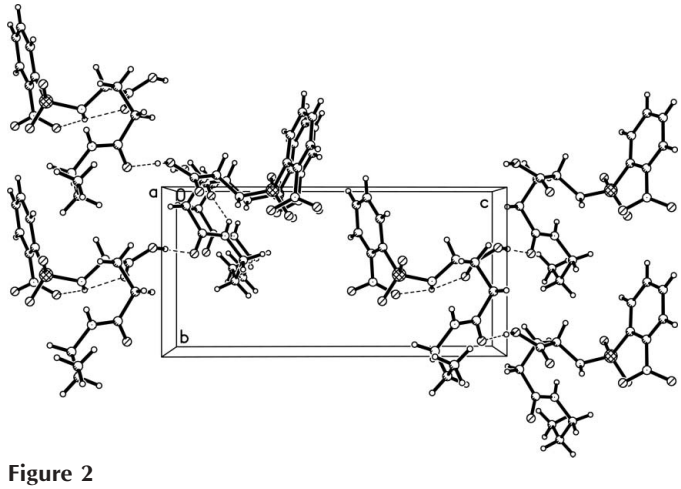


Figure 2
The crystal packing of (I), viewed along the *c* axis. Dashed lines indicate hydrogen bonds.

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