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N-(Phenylsulfonyl)-L-asparagine

Muhammad Nadeem Arshad,[a] Hafiz Mubashar-ur-Rehman,[a] Islam Ullah Khan,[a]* Muhammad Shafiq[a] and Kong Mun Lo[b]*

[a]Materials Chemistry Laboratory, Department of Chemistry, GC University, Lahore 54000, Pakistan, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: iukhan.gcu@gmail.com, kmlo@um.edu.my

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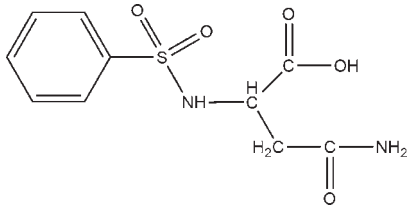
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.076; data-to-parameter ratio = 15.5.

In the title compound, $C_{10}H_{12}N_2O_5S$, one of the sulfonyl O atoms is hydrogen bonded to the amido N atom of an adjacent molecule. There is also a weak hydrogen-bonding interaction between the other sulfonyl O atom and the secondary amino N atom. In addition, the amido O atom is also hydrogen bonded to a carboxyl O atom. These hydrogen-bonding interactions give rise to a layer structure parallel to the *bc* plane.

Related literature

For related compounds, see: Koroniak *et al.* (2003); Arshad *et al.* (2008, 2009).



Experimental

Crystal data

$C_{10}H_{12}N_2O_5S$
M_r = 272.28
Monoclinic, $P2_1$
a = 10.5479 (6) Å
b = 5.1587 (3) Å
c = 11.0157 (7) Å
β = 92.011 (3)°
V = 599.03 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 296 K
0.25 × 0.21 × 0.13 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.932, T_{max} = 0.964
6832 measured reflections
2732 independent reflections
2518 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.076
S = 1.06
2732 reflections
176 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1187 Friedel pairs
Flack parameter: −0.01 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1^{i}	0.77 (2)	2.31 (2)	3.076 (2)	168.6 (19)
N2—H2A···O2^{ii}	0.95 (3)	2.06 (3)	2.998 (3)	172 (3)
O4—H4A···O6^{iii}	0.82	1.82	2.5804 (18)	155

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2295).

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supporting information

Acta Cryst. (2009). E**65**, o3229 [doi:10.1107/S1600536809050247]

N-(Phenylsulfonyl)-L-asparagine

Muhammad Nadeem Arshad, Hafiz Mubashar-ur-Rehman, Islam Ullah Khan, Muhammad Shafiq and Kong Mun Lo

S1. Comment

Sulfonamides compounds have long been studied due to their biological activity. *N*-acylsufonamide derivatives of asparagine have synthesized and characterized (Koroniak *et al.*, 2003). The title compound is also a sulfonamide derivative and synthesized in continuation of our studies for the synthesis of acyclic and cyclic sulfonamides (Arshad *et al.*, 2008), (Arshad *et al.*, 2009). In this sulfonamide derivative of L-asparagine (Fig. 1), the molecule is twisted due to the tetrahedral geometries at S1, C7 and C9. One of the sulfonyl oxygen O2 is hydrogen-bonded to the amido nitrogen atom N2 of an adjacent molecule. The amido oxygen O6 also forms hydrogen bond with the carboxylate oxygen O4 of an adjacent molecule. There is a weak hydrogen bonding interaction between the other sulfonyl oxygen O1 and the secondary amino nitrogen N1 (Fig. 2). No significant intramolecular hydrogen bonding interaction is found in the molecule.

S2. Experimental

Asparagine (0.175 g, 1.32 mmol) was dissolved in distilled water (10 ml) in a round bottom flask (25 ml). The pH of the solution was adjusted at 8–9 using 1*M*, Na_2CO_3 solution. Benzenesulfonyl chloride (0.169 ml, 0.234 g, 1.32 mmol) was suspended to the above solution and stirred at room temperature until all the benzenesulfonyl chloride was consumed. The completion of the reaction was achieved when the suspension turned to a clear solution. Upon completion of the reaction, the pH was adjusted 1–2, using 1 N HCl solution. The precipitate obtained was filtered, washed with distilled water, dried and recrystalized in methanol to yield white crystals.

S3. Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.93 to 0.97 Å) and were treated as riding on their parent carbon atoms, with *U*(H) set to 1.2–1.5 times *U*~eq~(C). The hydroxy H was refined with a restraint of 0.82 (1) Å. The Flack parameter refined to -0.01 (6); there were 1187 measured Friedel pairs.

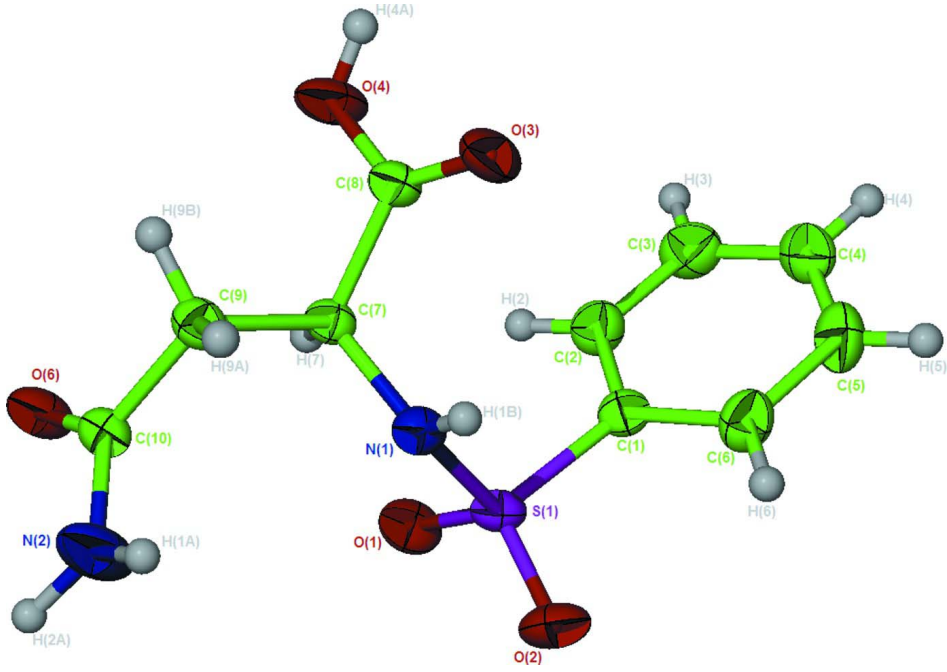

Figure 1

The molecular structure of *N*-(benzenesulfonyl)-L-asparagine showing 70% probability displacement ellipsoids and the atom numbering scheme. Hydrogen atoms are drawn as spheres of arbitrary radius.

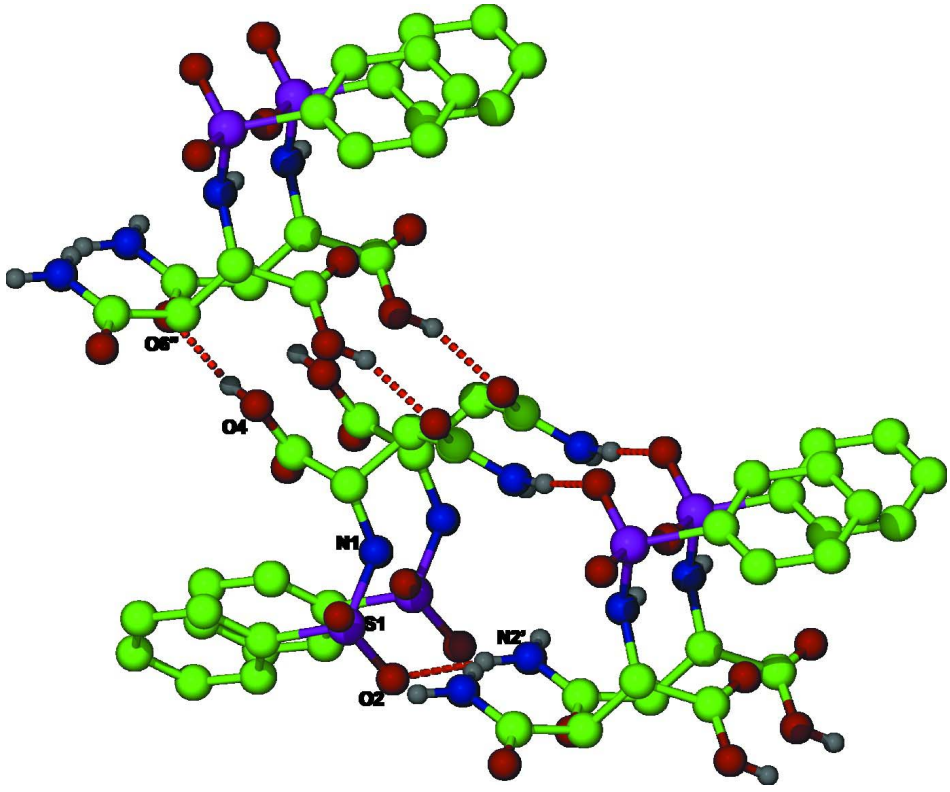

Figure 2

Crystal packing as viewed down the crystallographic b-axis showing the hydrogen bonding interactions. Symmetry code: ii = -x+1,y+1/2,-z.

N-(Phenylsulfonyl)-L-asparagine

Crystal data

$C_{10}H_{12}N_2O_5S$
$M_r = 272.28$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 10.5479\ (6)$ Å
$b = 5.1587\ (3)$ Å
$c = 11.0157\ (7)$ Å
$\beta = 92.011\ (3)°$
$V = 599.03\ (6)$ Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.510$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3825 reflections
$\theta = 2.6–27.4°$
$\mu = 0.29$ mm^{-1}
$T = 296$ K
Block, colorless
0.25 × 0.21 × 0.13 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.932$, $T_{max} = 0.964$
6832 measured reflections
2732 independent reflections
2518 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.6°$
$h = -13\rightarrow13$
$k = -6\rightarrow6$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.076$
$S = 1.06$
2732 reflections
176 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.0133P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Absolute structure: Flack (1983), 1187 Friedel pairs
Absolute structure parameter: −0.01 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.91282 (16)	0.8736 (3)	0.28950 (15)	0.0323 (4)
C2	0.93349 (19)	0.7209 (4)	0.18909 (17)	0.0411 (4)
H2	0.8795	0.5838	0.1688	0.049*
C3	1.03713 (19)	0.7776 (5)	0.11920 (19)	0.0503 (5)
H3	1.0534	0.6766	0.0516	0.060*
C4	1.11535 (19)	0.9813 (5)	0.1493 (2)	0.0499 (5)
H4	1.1837	1.0187	0.1012	0.060*
C5	1.09413 (19)	1.1314 (5)	0.2501 (2)	0.0517 (5)
H5	1.1483	1.2683	0.2700	0.062*
C6	0.99250 (18)	1.0783 (4)	0.32109 (19)	0.0439 (4)
H6	0.9775	1.1783	0.3893	0.053*
C7	0.59899 (15)	0.9058 (3)	0.19546 (14)	0.0279 (3)
H7	0.6111	0.7183	0.1873	0.034*
C8	0.64948 (16)	1.0412 (3)	0.08415 (14)	0.0311 (3)
C9	0.45593 (16)	0.9649 (3)	0.20037 (16)	0.0333 (4)
H9A	0.4444	1.1370	0.2336	0.040*
H9B	0.4187	0.9630	0.1186	0.040*
C10	0.38860 (15)	0.7715 (3)	0.27662 (15)	0.0315 (4)
H1A	0.379 (3)	1.000 (7)	0.406 (3)	0.073 (9)*
H2A	0.312 (3)	0.722 (7)	0.433 (3)	0.090 (10)*
N1	0.66143 (14)	0.9939 (3)	0.30846 (13)	0.0300 (3)
H1B	0.6757 (17)	1.141 (5)	0.3130 (17)	0.027 (5)*
N2	0.3616 (2)	0.8376 (5)	0.38858 (16)	0.0575 (5)

O1	0.73791 (14)	0.5586 (3)	0.36312 (13)	0.0448 (3)
O2	0.79671 (14)	0.9371 (3)	0.49048 (11)	0.0458 (3)
O3	0.70376 (15)	1.2446 (3)	0.08654 (13)	0.0492 (4)
O4	0.61999 (16)	0.9065 (3)	−0.01409 (11)	0.0485 (4)
H4A	0.6403	0.9880	−0.0743	0.073*
O6	0.35901 (14)	0.5579 (3)	0.23573 (12)	0.0430 (3)
S1	0.77553 (4)	0.82468 (8)	0.37280 (3)	0.03153 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0338 (7)	0.0321 (10)	0.0308 (8)	0.0042 (6)	−0.0010 (6)	0.0009 (6)
C2	0.0425 (9)	0.0410 (10)	0.0399 (10)	0.0005 (8)	0.0027 (8)	−0.0079 (8)
C3	0.0454 (10)	0.0652 (15)	0.0407 (10)	0.0089 (10)	0.0084 (8)	−0.0096 (10)
C4	0.0310 (9)	0.0658 (14)	0.0532 (12)	0.0041 (9)	0.0049 (8)	0.0057 (10)
C5	0.0355 (10)	0.0530 (13)	0.0662 (14)	−0.0071 (9)	−0.0025 (9)	−0.0011 (11)
C6	0.0412 (9)	0.0438 (11)	0.0466 (11)	−0.0015 (8)	−0.0012 (8)	−0.0101 (9)
C7	0.0340 (8)	0.0237 (7)	0.0263 (8)	−0.0001 (6)	0.0039 (6)	0.0008 (6)
C8	0.0357 (8)	0.0312 (9)	0.0268 (8)	−0.0011 (7)	0.0059 (7)	0.0014 (6)
C9	0.0340 (8)	0.0315 (9)	0.0346 (9)	0.0006 (7)	0.0041 (7)	0.0053 (7)
C10	0.0326 (8)	0.0314 (10)	0.0306 (8)	0.0005 (6)	0.0049 (6)	0.0018 (6)
N1	0.0365 (7)	0.0248 (7)	0.0287 (7)	0.0013 (6)	0.0026 (6)	−0.0003 (6)
N2	0.0810 (13)	0.0565 (11)	0.0363 (9)	−0.0212 (12)	0.0216 (8)	−0.0110 (10)
O1	0.0570 (8)	0.0306 (7)	0.0472 (8)	0.0007 (6)	0.0059 (6)	0.0108 (6)
O2	0.0567 (8)	0.0564 (8)	0.0244 (6)	0.0107 (7)	0.0006 (6)	−0.0009 (6)
O3	0.0665 (9)	0.0457 (9)	0.0357 (7)	−0.0255 (7)	0.0061 (6)	0.0041 (6)
O4	0.0820 (10)	0.0388 (7)	0.0255 (6)	−0.0119 (7)	0.0105 (6)	−0.0018 (5)
O6	0.0593 (8)	0.0361 (7)	0.0345 (6)	−0.0074 (6)	0.0152 (6)	−0.0019 (5)
S1	0.0406 (2)	0.0300 (2)	0.02406 (18)	0.00257 (18)	0.00257 (14)	0.00307 (17)

Geometric parameters (Å, º)

C1—C2	1.382 (3)	C7—H7	0.9800
C1—C6	1.386 (3)	C8—O3	1.195 (2)
C1—S1	1.7596 (18)	C8—O4	1.314 (2)
C2—C3	1.390 (3)	C9—C10	1.499 (2)
C2—H2	0.9300	C9—H9A	0.9700
C3—C4	1.370 (3)	C9—H9B	0.9700
C3—H3	0.9300	C10—O6	1.226 (2)
C4—C5	1.378 (3)	C10—N2	1.320 (2)
C4—H4	0.9300	N1—S1	1.6289 (15)
C5—C6	1.377 (3)	N1—H1B	0.77 (2)
C5—H5	0.9300	N2—H1A	0.88 (4)
C6—H6	0.9300	N2—H2A	0.95 (3)
C7—N1	1.460 (2)	O1—S1	1.4319 (14)
C7—C8	1.523 (2)	O2—S1	1.4304 (14)
C7—C9	1.542 (2)	O4—H4A	0.8200

C2—C1—C6	121.60 (18)	O3—C8—C7	124.44 (15)
C2—C1—S1	119.45 (14)	O4—C8—C7	109.93 (14)
C6—C1—S1	118.76 (14)	C10—C9—C7	111.80 (14)
C1—C2—C3	118.21 (19)	C10—C9—H9A	109.3
C1—C2—H2	120.9	C7—C9—H9A	109.3
C3—C2—H2	120.9	C10—C9—H9B	109.3
C4—C3—C2	120.39 (19)	C7—C9—H9B	109.3
C4—C3—H3	119.8	H9A—C9—H9B	107.9
C2—C3—H3	119.8	O6—C10—N2	120.99 (18)
C3—C4—C5	120.86 (19)	O6—C10—C9	120.76 (16)
C3—C4—H4	119.6	N2—C10—C9	118.24 (18)
C5—C4—H4	119.6	C7—N1—S1	120.55 (12)
C6—C5—C4	119.82 (19)	C7—N1—H1B	116.2 (14)
C6—C5—H5	120.1	S1—N1—H1B	110.9 (14)
C4—C5—H5	120.1	C10—N2—H1A	113.9 (19)
C5—C6—C1	119.11 (18)	C10—N2—H2A	117.8 (19)
C5—C6—H6	120.4	H1A—N2—H2A	127 (3)
C1—C6—H6	120.4	C8—O4—H4A	109.5
N1—C7—C8	112.54 (13)	O2—S1—O1	119.35 (9)
N1—C7—C9	108.73 (13)	O2—S1—N1	105.46 (8)
C8—C7—C9	107.92 (13)	O1—S1—N1	106.44 (8)
N1—C7—H7	109.2	O2—S1—C1	108.01 (9)
C8—C7—H7	109.2	O1—S1—C1	109.25 (8)
C9—C7—H7	109.2	N1—S1—C1	107.77 (8)
O3—C8—O4	125.58 (16)		

C6—C1—C2—C3	−0.1 (3)	C7—C9—C10—O6	−79.6 (2)
S1—C1—C2—C3	174.80 (16)	C7—C9—C10—N2	100.7 (2)
C1—C2—C3—C4	−0.6 (3)	C8—C7—N1—S1	−99.21 (15)
C2—C3—C4—C5	0.9 (3)	C9—C7—N1—S1	141.28 (13)
C3—C4—C5—C6	−0.6 (3)	C7—N1—S1—O2	−168.94 (12)
C4—C5—C6—C1	−0.1 (3)	C7—N1—S1—O1	−41.22 (15)
C2—C1—C6—C5	0.5 (3)	C7—N1—S1—C1	75.87 (14)
S1—C1—C6—C5	−174.49 (16)	C2—C1—S1—O2	160.50 (14)
N1—C7—C8—O3	−21.2 (2)	C6—C1—S1—O2	−24.43 (17)
C9—C7—C8—O3	98.8 (2)	C2—C1—S1—O1	29.25 (17)
N1—C7—C8—O4	161.31 (14)	C6—C1—S1—O1	−155.68 (14)
C9—C7—C8—O4	−78.71 (17)	C2—C1—S1—N1	−86.01 (16)
N1—C7—C9—C10	−78.10 (18)	C6—C1—S1—N1	89.06 (15)
C8—C7—C9—C10	159.55 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O1^{i}	0.77 (2)	2.31 (2)	3.076 (2)	168.6 (19)

N2—H2*A*···O2^{ii}	0.95 (3)	2.06 (3)	2.998 (3)	172 (3)
O4—H4*A*···O6^{iii}	0.82	1.82	2.5804 (18)	155

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, *y*−1/2, −*z*+1; (iii) −*x*+1, *y*+1/2, −*z*.