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2-(2-Iodobenzenesulfonamido)acetic acid

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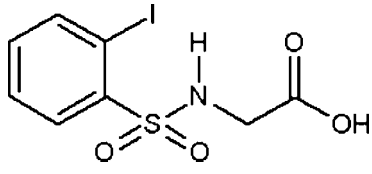
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.030; wR factor = 0.086; data-to-parameter ratio = 19.6.

The title compound, $C_8H_8INO_4S$, is a halogenated sulfonamide, a medicinally important class of organic compounds. In the crystal structure, intermolecular O—H···O hydrogen bonds involving the carboxylic acid groups form characteristic centrosymmetric dimers. These dimers are further linked through centrosymmetric dimeric N—H···O interactions involving the amido H atom and a sulfonyl O atom. This leads to the formation of a ribbon-like polymer structure propagating in the b direction.

Related literature

For background on sulfonamides, or sulfa drugs, see: Pandya *et al.* (2003). For the structure of the non-halogenated analogue, see: Arshad *et al.* (2008*b*). For the synthesis of the title compound, see: Deng & Mani (2006). For details of related structures: see Arshad *et al.* (2008*a*,*c*). For background on related thiazine heterocycles, see: Arshad *et al.* (2008*d*). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_8INO_4S$
M_r = 341.11
Triclinic, $P\overline{1}$
a = 5.5877 (2) Å
b = 8.0145 (2) Å
c = 12.3584 (4) Å
α = 80.923 (2)°
β = 83.398 (2)°
γ = 88.038 (2)°
V = 542.81 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.14 mm^{-1}
T = 296 K
0.22 × 0.10 × 0.06 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.691, T_{max} = 0.834
11492 measured reflections
2691 independent reflections
2297 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.086
S = 1.02
2691 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.43 e Å^{-3}
$\Delta\rho_{min}$ = −1.09 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.86	2.47	3.142 (3)	135
O2—H2O···O1^{ii}	0.82	1.86	2.676 (4)	176

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2093).

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supplementary materials

Acta Cryst. (2009). E**65**, o549 [**doi:10.1107/S1600536809005248**]

2-(2-Iodobenzenesulfonamido)acetic acid

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Comment

Sulfonamides, commonly known as sulfa drugs, belong to a class of organic compounds which have widespread applications in Medicinal Chemistry for curing bacterial infections (Pandya *et al.*, 2003). As a continuation of our studies on the synthesis of thiazine related compounds (Arshad *et al.*, 2008*a*,*b*,*c*,*d*) we present here the structure of the title compound.

The molecular structure of the title compound is illustrated in Fig. 1. We have previously reported on the crystal structures of the non-halogenated analogue of the title compound, 2-(phenylsulfonamido)acetic acid, (Arshad *et al.*, 2008*b*), and the K^+ and Na^+ salts of 2-iodobenzenesulfonates (Arshad *et al.*, 2008*a*,*c*). The bond lengths and angles in the title compound are similar to those reported there and are within normal ranges (Allen *et al.*, 1987).

In the crystal structure intermolecular O—H···O hydrogen bonds involving the carboxylic acid groups form characteristic centrosymmetric dimers. These dimers are further linked through centrosymmetric intermolecular N—H···O interactions involving the amido H atoms and a sulfonyl O atom (Fig. 2). This leads to the formation of a ribbon-like polymer structure propagating along the *b* axis. This arrangement is very similar to that observed in the non-halogenated analogue mentioned above.

Experimental

The title compound was synthesized following the literature method (Deng & Mani, 2006). Glycine methyl ester hydrochloride (207 mg, 1.653 mmol) was dissolved in distilled water (5 ml). The pH of the solution was maintained at 8–9 using 1*M*, Na_2CO_3 solution. 2-Iodo benzene sulfonyl chloride (500 mg, 1.653 mmol) was then added to the solution, which was stirred at room temperature until all the 2-iodo benzene sulfonyl chloride had been consumed. During the reaction the pH was again strictly maintained at 8–9 using 1*M*, Na_2CO_3. On completion of the reaction the pH was adjusted 1–2, using 1N HCl under vigorous stirring. The precipitate obtained was filtered off, washed with distilled water and dried. Crystals of the title compound were obtained by recrystallisation from methanol.

Refinement

The O, N and C-bound H atoms were included in calculated positions and treated as riding: O—H = 0.82 Å, N—H = 0.86 Å, C—H = 0.93–0.97 Å, with U_{iso}(H) = 1.5U_{eq}(parent O atom) and = 1.2U_{eq}(parent C and N atom).

Figures

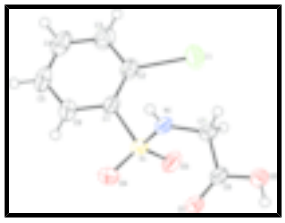

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. The thermal ellipsoids are drawn at the 50% probability level.

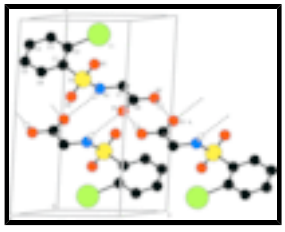

Fig. 2. Crystal packing of the title compound, viewed along the axis, showing the intermolecular hydrogen bonds as dashed lines.

2-(2-Iodobenzenesulfonamido)acetic acid

Crystal data

$C_8H_8INO_4S$	$Z = 2$
$M_r = 341.11$	$F_{000} = 328$
Triclinic, $P\overline{1}$	$D_x = 2.087$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.5877$ (2) Å	Cell parameters from 6313 reflections
$b = 8.0145$ (2) Å	$\theta = 2.6$–28.3º
$c = 12.3584$ (4) Å	$\mu = 3.14$ mm^{-1}
$\alpha = 80.923$ (2)º	$T = 296$ K
$\beta = 83.398$ (2)º	Needle-like, light brown
$\gamma = 88.038$ (2)º	0.22 × 0.10 × 0.06 mm
$V = 542.81$ (3) Å^3	

Data collection

Bruker Kappa APEXII CCD diffractometer	2691 independent reflections
Radiation source: fine-focus sealed tube	2297 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.028$
$T = 296$ K	$\theta_{max} = 28.3$º
φ and ω scans	$\theta_{min} = 2.6$º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -7 \rightarrow 7$
$T_{min} = 0.691$, $T_{max} = 0.834$	$k = -10 \rightarrow 10$
11492 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.6546P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
2691 reflections	$\Delta\rho_{max} = 1.43$ e Å^{-3}
137 parameters	$\Delta\rho_{min} = -1.09$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.10590 (5)	0.34414 (3)	0.90179 (2)	0.0537 (1)
S1	0.59293 (13)	0.42594 (9)	0.68657 (7)	0.0327 (2)
O1	0.5522 (4)	0.7909 (3)	0.5250 (2)	0.0405 (7)
O2	0.2240 (4)	0.9320 (3)	0.5837 (3)	0.0506 (8)
O3	0.6165 (5)	0.5362 (3)	0.7649 (2)	0.0447 (8)
O4	0.7874 (4)	0.4121 (3)	0.6022 (2)	0.0448 (8)
N1	0.3478 (5)	0.4818 (3)	0.6322 (2)	0.0348 (8)
C1	0.5418 (5)	0.2165 (4)	0.7568 (3)	0.0319 (8)
C2	0.3582 (6)	0.1730 (4)	0.8409 (3)	0.0376 (9)
C3	0.3352 (8)	0.0057 (4)	0.8907 (3)	0.0496 (11)
C4	0.4944 (8)	−0.1169 (4)	0.8574 (3)	0.0520 (13)
C5	0.6787 (8)	−0.0735 (4)	0.7758 (4)	0.0514 (11)
C6	0.7032 (7)	0.0926 (4)	0.7254 (3)	0.0434 (10)
C7	0.2290 (6)	0.6417 (4)	0.6433 (3)	0.0371 (9)
C8	0.3550 (6)	0.7946 (4)	0.5770 (3)	0.0346 (9)
H1N	0.28640	0.41450	0.59550	0.0420*
H2O	0.29800	1.01380	0.54930	0.0760*
H3	0.21160	−0.02410	0.94690	0.0600*
H4	0.47630	−0.22890	0.89050	0.0630*

H5	0.78740	−0.15580	0.75430	0.0620*
H6	0.82870	0.12170	0.67000	0.0520*
H7A	0.21220	0.65480	0.72050	0.0440*
H7B	0.06810	0.63890	0.62140	0.0440*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0578 (2)	0.0421 (2)	0.0529 (2)	0.0132 (1)	0.0143 (1)	0.0003 (1)
S1	0.0304 (3)	0.0232 (3)	0.0442 (4)	−0.0049 (3)	−0.0027 (3)	−0.0042 (3)
O1	0.0375 (12)	0.0252 (10)	0.0551 (14)	−0.0047 (9)	0.0058 (10)	−0.0016 (9)
O2	0.0413 (13)	0.0247 (11)	0.0772 (18)	0.0014 (9)	0.0106 (12)	0.0057 (11)
O3	0.0498 (14)	0.0282 (11)	0.0602 (16)	−0.0014 (10)	−0.0147 (12)	−0.0130 (10)
O4	0.0337 (11)	0.0411 (13)	0.0565 (15)	−0.0076 (10)	0.0046 (10)	−0.0034 (11)
N1	0.0353 (13)	0.0217 (11)	0.0466 (15)	−0.0039 (9)	−0.0051 (11)	−0.0016 (10)
C1	0.0348 (15)	0.0227 (12)	0.0391 (16)	0.0011 (11)	−0.0065 (12)	−0.0059 (11)
C2	0.0442 (17)	0.0279 (14)	0.0394 (17)	0.0028 (12)	−0.0029 (13)	−0.0035 (12)
C3	0.063 (2)	0.0326 (17)	0.048 (2)	−0.0015 (15)	0.0038 (17)	0.0022 (14)
C4	0.075 (3)	0.0260 (16)	0.054 (2)	0.0038 (16)	−0.009 (2)	−0.0024 (14)
C5	0.062 (2)	0.0287 (16)	0.065 (2)	0.0135 (15)	−0.0084 (19)	−0.0137 (16)
C6	0.0454 (18)	0.0322 (16)	0.052 (2)	0.0049 (13)	0.0002 (15)	−0.0102 (14)
C7	0.0333 (15)	0.0247 (13)	0.0495 (18)	−0.0027 (11)	0.0011 (13)	0.0018 (12)
C8	0.0343 (15)	0.0229 (13)	0.0449 (17)	−0.0033 (11)	−0.0040 (13)	−0.0002 (12)

Geometric parameters (Å, °)

I1—C2	2.094 (3)	C2—C3	1.388 (5)
S1—O3	1.429 (3)	C3—C4	1.382 (5)
S1—O4	1.431 (2)	C4—C5	1.369 (6)
S1—N1	1.613 (3)	C5—C6	1.382 (5)
S1—C1	1.780 (3)	C7—C8	1.509 (5)
O1—C8	1.212 (4)	C3—H3	0.9300
O2—C8	1.310 (4)	C4—H4	0.9300
O2—H2O	0.8200	C5—H5	0.9300
N1—C7	1.442 (4)	C6—H6	0.9300
N1—H1N	0.8600	C7—H7A	0.9700
C1—C6	1.389 (5)	C7—H7B	0.9700
C1—C2	1.385 (5)		
O3—S1—O4	118.92 (15)	C1—C6—C5	120.5 (4)
O3—S1—N1	106.70 (15)	N1—C7—C8	115.3 (3)
O3—S1—C1	109.54 (16)	O1—C8—O2	124.4 (3)
O4—S1—N1	110.20 (14)	O1—C8—C7	124.5 (3)
O4—S1—C1	105.65 (15)	O2—C8—C7	111.1 (3)
N1—S1—C1	105.02 (13)	C2—C3—H3	120.00
C8—O2—H2O	109.00	C4—C3—H3	120.00
S1—N1—C7	121.8 (2)	C3—C4—H4	120.00
C7—N1—H1N	119.00	C5—C4—H4	120.00
S1—N1—H1N	119.00	C4—C5—H5	120.00

C2—C1—C6	119.5 (3)	C6—C5—H5	120.00
S1—C1—C6	116.4 (3)	C1—C6—H6	120.00
S1—C1—C2	124.1 (2)	C5—C6—H6	120.00
I1—C2—C1	124.6 (2)	N1—C7—H7A	108.00
I1—C2—C3	116.0 (3)	N1—C7—H7B	108.00
C1—C2—C3	119.5 (3)	C8—C7—H7A	108.00
C2—C3—C4	120.5 (4)	C8—C7—H7B	108.00
C3—C4—C5	120.1 (3)	H7A—C7—H7B	107.00
C4—C5—C6	120.0 (4)		
O3—S1—N1—C7	15.3 (3)	C6—C1—C2—I1	178.1 (3)
O4—S1—N1—C7	−115.1 (3)	C6—C1—C2—C3	−1.5 (5)
C1—S1—N1—C7	131.6 (3)	S1—C1—C6—C5	179.6 (3)
O3—S1—C1—C2	54.6 (3)	C2—C1—C6—C5	1.3 (5)
O3—S1—C1—C6	−123.6 (3)	I1—C2—C3—C4	−179.3 (3)
O4—S1—C1—C2	−176.2 (3)	C1—C2—C3—C4	0.3 (6)
O4—S1—C1—C6	5.6 (3)	C2—C3—C4—C5	0.9 (6)
N1—S1—C1—C2	−59.7 (3)	C3—C4—C5—C6	−1.1 (6)
N1—S1—C1—C6	122.1 (3)	C4—C5—C6—C1	−0.1 (6)
S1—N1—C7—C8	72.2 (4)	N1—C7—C8—O1	−6.5 (5)
S1—C1—C2—I1	0.0 (4)	N1—C7—C8—O2	174.0 (3)
S1—C1—C2—C3	−179.6 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1^{i}	0.86	2.47	3.142 (3)	135
O2—H2O···O1^{ii}	0.82	1.86	2.676 (4)	176

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

Fig. 1

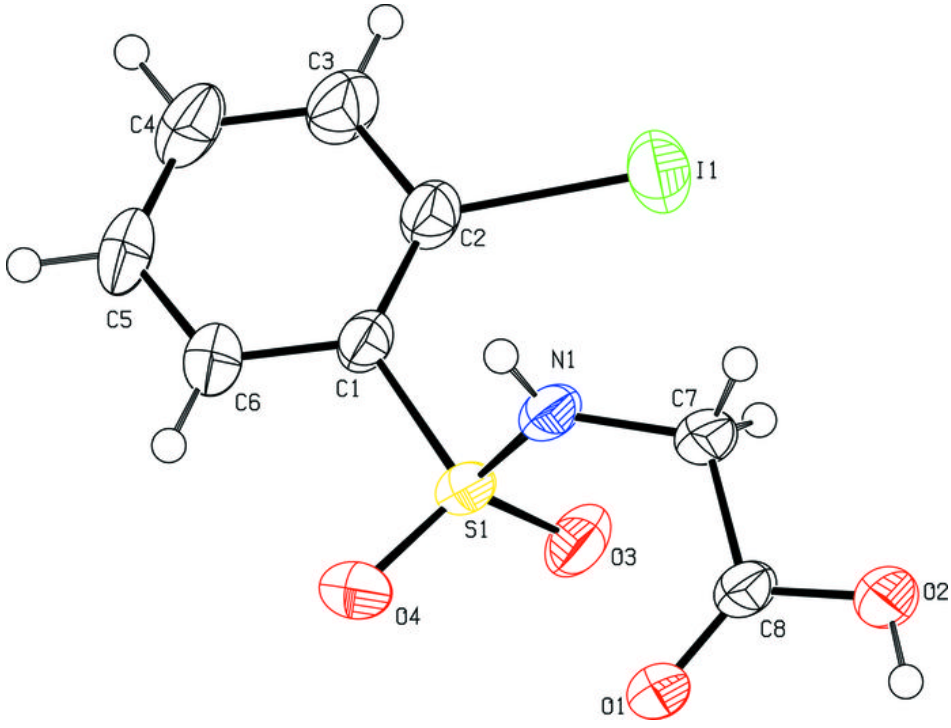

Fig. 2