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Sulfachloropyridazine

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In the title compound, 4-amino-*N*-(6-chloro-3-pyridazinyl)-benzenesulfonamide, $C_{10}H_9ClN_4O_2S$, the pyridazine ring and the benzene ring form a distorted V configuration, with a dihedral angle of 82.86 (6)°. The crystal packing is stabilized by π–π stacking interactions between the pyridazine rings and intermolecular N—H···O, C—H···O and C—H···N hydrogen bonds, linking the molecules into a two-dimensional network.

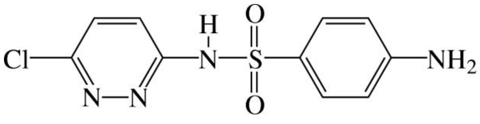
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.100
Data-to-parameter ratio = 15.5

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

Comment

Sulfachloropyridazine [4-amino-*N*-(6-chloro-3-pyridazinyl)-benzenesulfonamide], (I), is a synthetic antibacterial drug of the sulfanilamides. A number of sufanilamide drugs have been crystallographically characterized in the past few decades (Acharya *et al.*, 1982; Adsmond & Grant, 2001; Basak *et al.*, 1983; Caria & Mohamed, 1992; Deo *et al.*, 1980; Joshi *et al.*, 1983). However, as far as we are aware, up to now, no crystal structure of sulfanilamide derivatives containing chloropyridazine has been published.



(I)

The bond lengths and angles of (I) are normal and agree with the corresponding values found in sulfamerazine and sulfamethazine (Acharya *et al.*, 1982; Adsmond & Grant, 2001; Basak *et al.*, 1983; Caira & Mohamed, 1992; Deo *et al.*, 1980). Since the S—C bond [1.734 (2) Å] is a normal single bond and shorter than those found in the metal–sulfadiazinate complexes [1.746 (4) or 1.768 (5) Å; Garcia-Raso *et al.*, 1997; Yuan *et al.*, 2001], it appears that there is no extension of the benzene ring electron delocalization including the S atom. The S—O bond lengths are very similar [1.4339 (15) and 1.4327 (16) Å] and comparable to those found in free sulfadiazine (Joshi *et al.*, 1983). The heterocyclic ring geometry in (I) is comparable to that found for free pyridazine (Blake & Rankin, 1991). The pyridazine and benzene rings form a distorted V configuration, with a dihedral angle of 82.86 (6)°.

The C4—H4···O1 hydrogen bond, involving benzene C and H atoms and a sulfonamide O atom, forms a six-membered pseudo-ring within the molecule. There exist intermolecular N—H···O, C—H···O and C—H···N hydrogen bonds (Table 2) involving the amine group, the sulfonamide NH group, and benzene C and H atoms, which act as donors, and the two sulfonamide O and two pyridazinyl N atoms, which act

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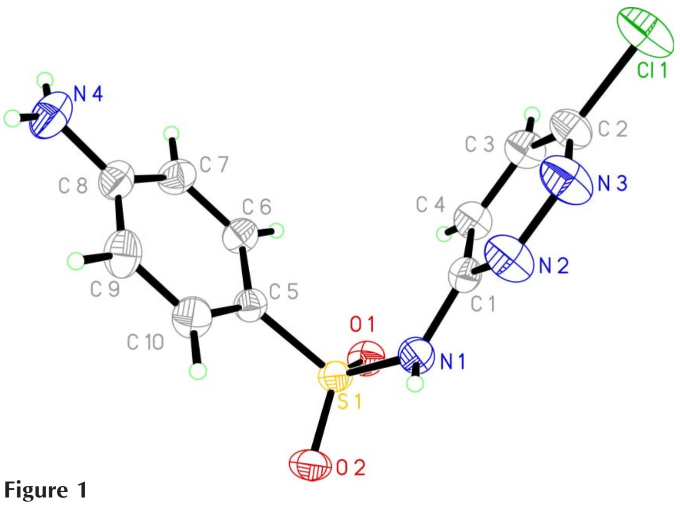

Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

as acceptors. A two-dimensional supramolecular structure is formed through these hydrogen-bonding and π–π stacking interactions between the pyridazine rings; the interplanar and centroid–centroid separations are 3.619 and 4.537 (2) Å, respectively.

Experimental

Sulfachloropyridazine (0.5 mmol, purchased from Fluka) and $AgNO_3$ (0.5 mmol) were placed in an autoclave. After addition of EtOH (13 ml), pyridine (0.5 ml) and CH_2Cl_2 (3 ml), the autoclave was heated at 353 K for 7 d to give unexpected orange–yellow block-shaped crystals of (I), with a yield of 50%. IR (KBr, cm^{-1}): 3395 (*s*), 3317 (*s*), 3138 (*w*), 1596 (*s*), 1575 (*s*), 1440 (*s*), 1241 (*s*), 944 (*s*), 908 (*m*), 825 (*m*), 749 (*w*), 680 (*s*).

Crystal data

$C_{10}H_9ClN_4O_2S$
$M_r = 284.72$
Monoclinic, $P2_1/n$
$a = 5.549$ (2) Å
$b = 17.097$ (7) Å
$c = 12.606$ (5) Å
$\beta = 92.647$ (10)°
$V = 1194.6$ (8) Å^3
$Z = 4$
$D_x = 1.583$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5140 reflections
$\theta = 3.2$–27.5°
$\mu = 0.49$ mm^{-1}
$T = 193$ (2) K
Block, orange–yellow
0.39 × 0.29 × 0.20 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{min} = 0.831$, $T_{max} = 0.908$
13 214 measured reflections
2731 independent reflections
2481 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$
$h = -7 \rightarrow 6$
$k = -22 \rightarrow 22$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.100$
$S = 1.10$
2731 reflections
176 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.8089P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

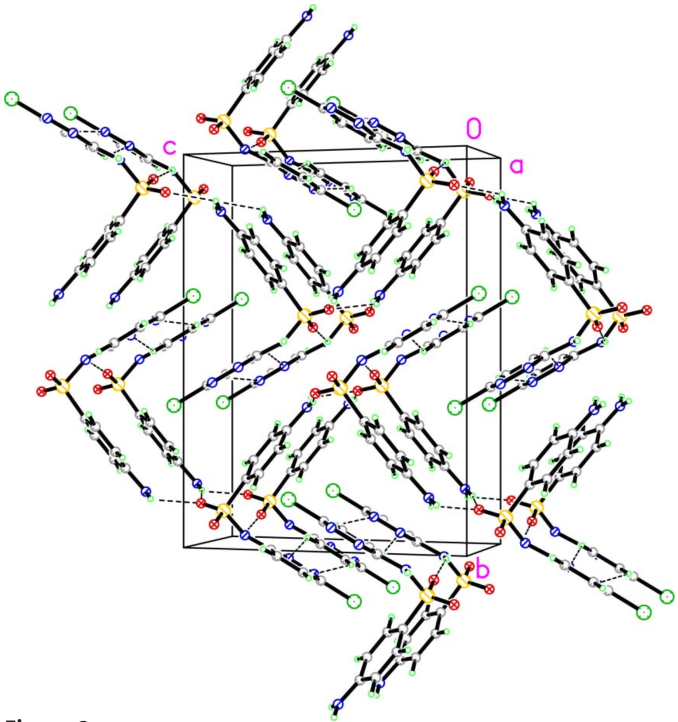

Figure 2
A packing diagram of (I). Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

Cl1—C2	1.729 (2)	N1—C1	1.394 (3)
S1—O1	1.4327 (16)	N2—N3	1.347 (3)
S1—O2	1.4339 (15)	N3—C2	1.306 (3)
S1—N1	1.6467 (18)	N4—C8	1.356 (3)
S1—C5	1.734 (2)		
O1—S1—O2	119.40 (9)	N2—C1—C4	123.32 (19)
O1—S1—N1	108.22 (10)	N1—C1—C4	124.01 (18)
O2—S1—N1	102.70 (9)	N3—C2—C3	124.4 (2)
O1—S1—C5	109.00 (9)	N3—C2—Cl1	115.28 (16)
O2—S1—C5	109.99 (10)	C3—C2—Cl1	120.32 (16)
N1—S1—C5	106.70 (9)	C4—C3—C2	117.13 (19)
C1—N1—S1	126.60 (14)	C6—C5—C10	120.24 (18)
N2—C1—N1	112.58 (17)	C7—C6—C5	119.92 (19)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O2^{i}	0.86 (3)	2.54 (3)	3.204 (3)	135 (3)
N4—H4*A*···O2^{i}	0.86 (3)	2.54 (3)	3.204 (3)	135 (3)
N1—H1···O1^{ii}	0.78 (3)	2.55 (3)	3.290 (3)	158 (2)
C4—H4···N2^{iii}	0.95	2.48	3.237 (3)	137
C4—H4···O1	0.95	2.42	3.053 (3)	124
C3—H3···N3^{iii}	0.95	2.61	3.248 (3)	125

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $x + 1, y, z$; (iii) $x - 1, y, z$.

H atoms on C atoms were positioned geometrically and were treated as riding, with C—H distances of 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms bound to N1 and N4 were located in a difference map and refined iostropically [N—H = 0.78 (3), 0.84 (3) and 0.86 (3) Å].

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku & Rigaku/

MSC, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXTL*.

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