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2-(2-Chlorophenylamino)-4-methylsulfanyl-1,3,5-triazine

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.096
Data-to-parameter ratio = 14.0

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

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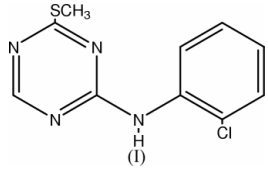
The crystal structure of the title compound, $C_{10}H_9ClN_4S$, is reported. There is π-conjugation between triazine and the bridging N4 atom, where N atoms are sp^2 hybridized. The plane of the triazine ring forms a dihedral angle of 54.35 (4)° with the phenyl plane. There exists an intermolecular N—H···N hydrogen bond to form a dimer with an N···N separation of 2.991 (2) Å.

Comment

Heterocycles are of very important consideration in a study on new pharmaceuticals and agrochemicals. In recent years, many new chemicals have been synthesized that have structures containing heterocyclic rings, such as triazine. In our study on this type of compounds, we have found that substituted triazine-2-amine exhibited good biological activity. Adenosine antagonistic effect on human A_1 receptor has been reported (Kuefner-Mueal *et al.*, 1999). Nitrification inhibitory effects have been studied (Koizumi *et al.*, 1994) and pest control, fungicidal activity and herbicidal activity effects have also been observed (Schapter *et al.*, 1999; Kubuyama *et al.*, 1998; Riebel *et al.*, 1998). Since knowledge of the stereochemistry is useful in the rational design of pharmaceuticals and agrochemicals, we herein report the synthesis and X-ray crystal structure of the title complex, (I).



(I)

In the title compound, the N4—C4 bond length is remarkably shorter than a single C—N bond (1.47 Å; Sasada, 1984) and close to the C═N double-bond distance (1.28 Å; Wang *et al.*, 1998), which is indicative of significant double-bond character. The N4 atom and the triazine ring form a π_7^8 configuration in which the N4 atom is sp^2 hybridized. The C1—S1 bond length is slightly longer than that of C2—S1, which may be due to the fact that an sp^2 C atom has a smaller covalent radius than an sp^3 C atom. The lengths of N—C of triazine ring are in the range of 1.319 (2)-1.362 (2) Å, in which the N3—C4 bond length is slightly longer than that of N3—C3. Atoms C1–C4/N1–N4/S1 are nearly coplanar, with an average deviation of 0.016 (1) Å, and form a 54.35 (4)° dihedral angle with the phenyl plane. Molecules of (I) are linked into dimers by intermolecular N—H···N hydrogen bonds (see Table 2).

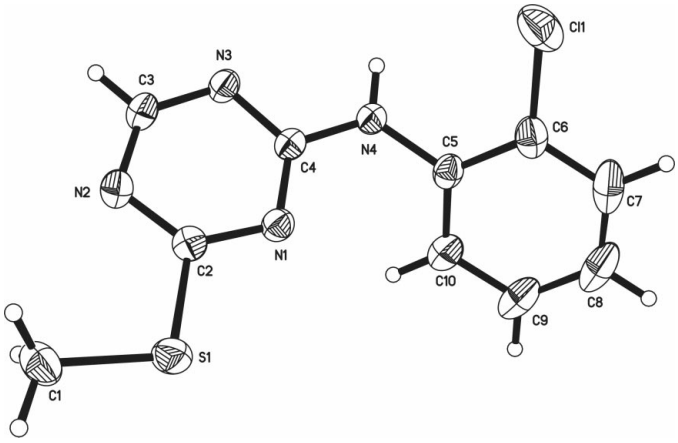


Figure 1
The molecular structure of the title compound with 30% probability ellipsoids.

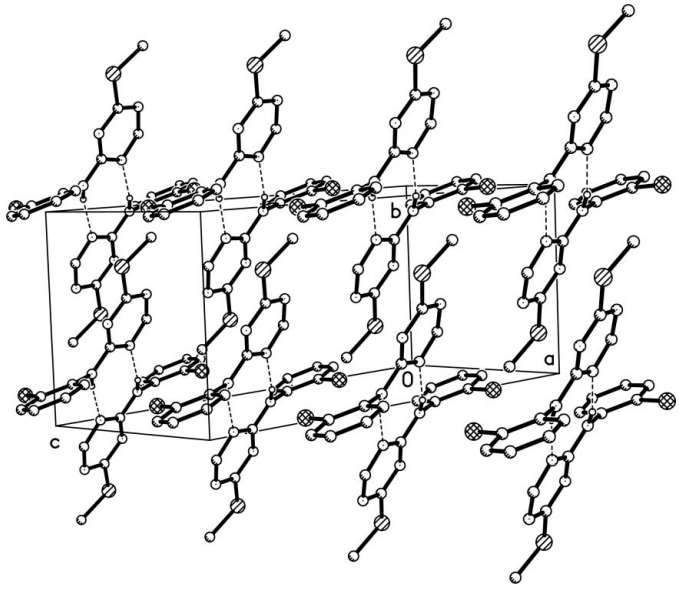


Figure 2
The packing of the title compound.

Experimental

To a solution of 0.77 g (5 mmol) *N*-(2-chlorophenyl)methanimidamide in 15 ml anhydrous toluene and 15 ml dimethylacetamide, 0.30 g (10 mmol) 80% NaH was added with stirring, then 0.73 g (5 mmol) N-cyanocarbonimidodithioic acid dimethyl ester dropwise. After stirring for 24 h, the mixture was poured into 50 ml ice water, then quenched carefully with 10% aqueous HCl. The solid was purified by silica-gel column chromatography to give the title compound, (I) (yield: 60%). FT–IR data (KBr pellet, cm^{-1}): 3398, 1598, 1543, 1497. Analysis calculated for (I): C 47.53, H 3.59, N 22.17%; Found: C 47.43, H 3.73, N 22.10%. Crystals of (I) were obtained as blocks by recrystallization from a petroleum ether/ethyl acetate mixture.

Crystal data

$C_{10}H_9ClN_4S$
$M_r = 252.72$
Monoclinic, $P2_1/c$
$a = 7.492$ (2) Å
$b = 7.797$ (2) Å
$c = 19.877$ (5) Å
$\beta = 94.962$ (5)°
$V = 1156.8$ (5) Å^3
$Z = 4$
$D_x = 1.451$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4658 reflections
$\theta = 2.1$–25.0°
$\mu = 0.49$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker SMART 1000 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
$T_{min} = 0.868$, $T_{max} = 0.909$
4658 measured reflections
2037 independent reflections
1730 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 8$
$l = -18 \rightarrow 23$

Refinement

Refinement on F^2
$R(F) = 0.031$
$wR(F^2) = 0.096$
$S = 1.04$
2037 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.2627P]$ $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.007$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*97

Table 1
Selected geometric parameters (Å, °).

S1—C2	1.7469 (17)	N2—C2	1.337 (2)
S1—C1	1.795 (2)	N3—C3	1.319 (2)
N1—C2	1.334 (2)	N3—C4	1.362 (2)
N1—C4	1.338 (2)	N4—C4	1.341 (2)
N2—C3	1.330 (2)	N4—C5	1.417 (2)
C2—S1—C1	102.28 (10)	N2—C2—S1	119.72 (13)
C2—N1—C4	114.23 (14)	N3—C3—N2	127.80 (15)
C3—N2—C2	112.60 (14)	N1—C4—N4	120.16 (14)
C3—N3—C4	113.85 (14)	N1—C4—N3	124.50 (15)
C4—N4—C5	126.43 (14)	N4—C4—N3	115.34 (14)
N1—C2—N2	126.99 (15)	C10—C5—N4	121.60 (15)
N1—C2—S1	113.28 (12)	C6—C5—N4	119.90 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4a···N3^{i}	0.86	2.15	2.991 (2)	168

Symmetry code: (i) $-x, 1-y, -z$.

All the H atoms were located geometrically and placed in calculated positions (C—H = 0.93 Å and N—H = 0.86 Å) and U_{iso}(H) was assigned as $1.2U_{eq}$(C/N).

Data collection: *SMART*1000 *Software* (Bruker, 1998); cell refinement: *SMART*1000 *Software*; data reduction: SAINT in *SMART*1000 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXL*97.

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