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2-[(5-Amino-1,3,4-thiadiazol-2-yl)sulfanyl]-*N*-(2,4,5-trichlorophenyl)acetamide

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Keywords: crystal structure; imidazothiadiazole; hydrogen bonding.

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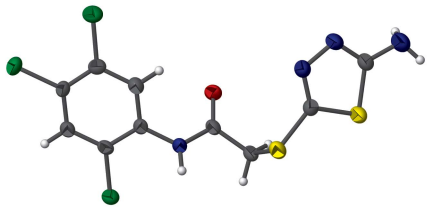
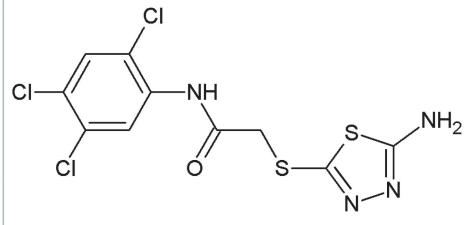
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{10}H_7Cl_3N_4OS_2$, the dihedral angle between the trichlorobenzene and thiadiazole rings is 29.26 (17)°. In the crystal, molecules are connected by N—H···O and C—H···O hydrogen bonds, forming chains propagating along [001]. The chains are linked *via* N—H···N hydrogen bonds to form slabs parallel to (100).

3D view



Chemical scheme



Structure description

As part of our research on the syntheses and crystal structure analyses of thiadiazole derivatives, we report herein on the synthesis and crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The dihedral angle between the trichlorobenzene ring (C5–C10) and the thiadiazol moiety (C1/C2/N1/N2/S1) is 29.26 (17)°.

In the crystal, molecules are connected by N—H···O and C—H···O hydrogen bonds, forming chains propagating along the *c*-axis direction (Table 1 and Fig. 2). The chains are linked *via* N—H···N hydrogen bonds to form slabs parallel to the *bc* plane (Table 1 and Fig. 2).

Synthesis and crystallization

An equimolar ratio of compound 2-((5-amino-1,3,4-thiadiazol-2-yl)thio)-*N*-(trichlorophenyl)acetamide (0.005 mol) and ethyl chloroacetate (0.005 mol) in glacial acetic acid

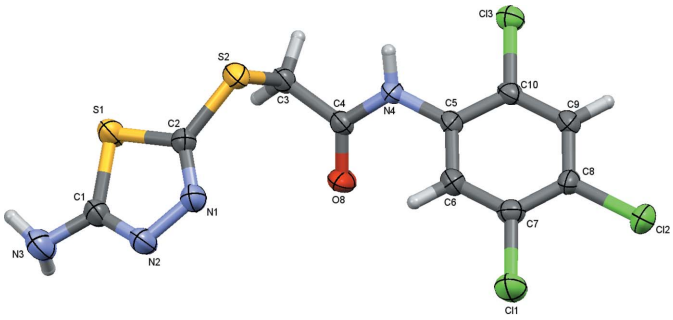


Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

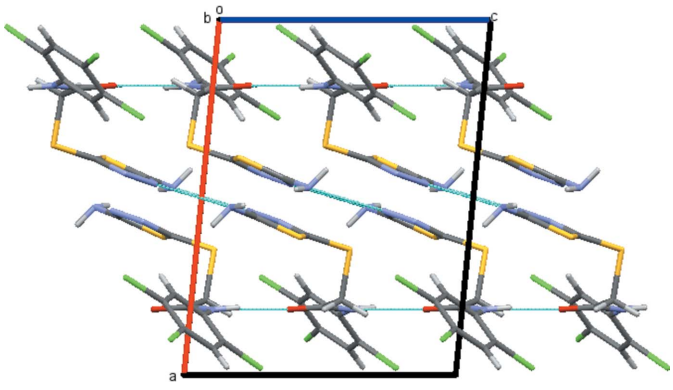


Figure 2
A viewed along the *b* axis of the crystal packing of the title compound. Hydrogen bonds are drawn as dashed lines (see Table 1).

(20 ml) was heated under reflux for 17 h. The reaction mixture was poured into ice-cold water. The precipitated solid was filtered, dried and recrystallized from ethanol.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank DST–PURSE, Mangalore University, Mangaluru, for providing the single-crystal X-ray diffraction facility.

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···O8^{i}	0.86	2.09	2.927 (3)	163
C3—H3D···O8^{i}	0.97	2.43	3.166 (3)	133
N3—H3A···N1^{ii}	0.86	2.51	3.304 (4)	154

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_7Cl_3N_4OS_2$
M_r	369.67
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	273
a, b, c (Å)	12.4679 (12), 11.9467 (11), 9.5278 (8)
β (°)	95.701 (7)
V (Å^3)	1412.1 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.94
Crystal size (mm)	0.32 × 0.23 × 0.1
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.771, 0.910
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9999, 3200, 2292
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.128, 1.09
No. of reflections	3200
No. of parameters	182
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.41

Computer programs: *CrystalClear* (Rigaku, 2011), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

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Crystal data

$C_{10}H_7Cl_3N_4OS_2$
M_r = 369.67
Monoclinic, $P2_1/c$
a = 12.4679 (12) Å
b = 11.9467 (11) Å
c = 9.5278 (8) Å
β = 95.701 (7)°
V = 1412.1 (2) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.739 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 3200 reflections
θ = 3.1–27.5°
μ = 0.94 mm^{-1}
T = 273 K
Block, colourless
0.32 × 0.23 × 0.1 mm

Data collection

Rigaku Saturn724+ diffractometer
profile data from ω–scans
Absorption correction: multi-scan (NUMABS; Rigaku, 1999)
T_{min} = 0.771, T_{max} = 0.910
9999 measured reflections
3200 independent reflections
2292 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −16→16
k = −15→15
l = −9→12
3200 standard reflections

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.128
S = 1.09
3200 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.4363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.27431 (8)	0.33324 (7)	0.75005 (10)	0.0522 (3)
Cl2	0.10023 (8)	0.19165 (6)	0.56097 (9)	0.0479 (3)
Cl3	0.01952 (7)	0.54537 (6)	0.22229 (8)	0.0393 (2)
S1	0.38818 (8)	1.08239 (7)	0.62462 (9)	0.0427 (3)
S2	0.35323 (7)	0.87823 (7)	0.43499 (8)	0.0381 (2)
O8	0.1844 (2)	0.73427 (18)	0.6362 (2)	0.0415 (6)
N1	0.4321 (3)	0.8833 (2)	0.7082 (3)	0.0447 (7)
N2	0.4623 (3)	0.9512 (2)	0.8237 (3)	0.0505 (8)
N3	0.4728 (3)	1.1403 (3)	0.8894 (3)	0.0576 (9)
H3A	0.4773	1.2027	0.8453	0.069*
H3B	0.4237	1.1454	0.9466	0.069*
N4	0.1841 (2)	0.6632 (2)	0.4152 (3)	0.0332 (6)
H4	0.1894	0.6797	0.3283	0.040*
C1	0.4452 (3)	1.0570 (3)	0.7939 (3)	0.0390 (8)
C2	0.3929 (3)	0.9389 (3)	0.5991 (3)	0.0333 (7)
C3	0.2102 (3)	0.8607 (2)	0.4479 (3)	0.0331 (7)
H3C	0.1845	0.9190	0.5071	0.040*
H3D	0.1710	0.8665	0.3550	0.040*
C4	0.1910 (2)	0.7478 (2)	0.5102 (3)	0.0291 (7)
C5	0.1687 (2)	0.5499 (2)	0.4494 (3)	0.0285 (7)
C6	0.2261 (3)	0.4997 (2)	0.5656 (3)	0.0336 (7)
H6	0.2784	0.5405	0.6202	0.040*
C7	0.2061 (3)	0.3899 (3)	0.6005 (3)	0.0336 (7)
C8	0.1297 (3)	0.3284 (2)	0.5181 (3)	0.0316 (7)
C9	0.0733 (3)	0.3762 (2)	0.4015 (3)	0.0315 (7)
H9	0.0224	0.3346	0.3457	0.038*
C10	0.0930 (2)	0.4854 (2)	0.3683 (3)	0.0295 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0694 (7)	0.0412 (5)	0.0413 (5)	0.0011 (4)	−0.0173 (4)	0.0066 (4)
Cl2	0.0716 (7)	0.0291 (4)	0.0417 (5)	−0.0121 (4)	−0.0004 (4)	0.0057 (3)
Cl3	0.0487 (5)	0.0355 (4)	0.0316 (4)	−0.0010 (4)	−0.0065 (4)	0.0017 (3)
S1	0.0567 (6)	0.0308 (4)	0.0373 (5)	0.0017 (4)	−0.0116 (4)	−0.0020 (4)
S2	0.0461 (5)	0.0388 (4)	0.0295 (5)	−0.0096 (4)	0.0043 (4)	−0.0046 (4)
O8	0.0680 (17)	0.0346 (12)	0.0224 (12)	−0.0034 (11)	0.0067 (11)	−0.0042 (9)
N1	0.065 (2)	0.0304 (14)	0.0357 (16)	−0.0071 (14)	−0.0080 (14)	0.0003 (12)
N2	0.078 (2)	0.0364 (16)	0.0329 (16)	−0.0065 (15)	−0.0137 (15)	−0.0010 (13)
N3	0.086 (3)	0.0422 (17)	0.0415 (19)	−0.0042 (17)	−0.0088 (17)	−0.0072 (15)
N4	0.0512 (18)	0.0266 (12)	0.0218 (13)	−0.0073 (12)	0.0028 (12)	−0.0026 (10)
C1	0.046 (2)	0.0341 (16)	0.0348 (18)	−0.0052 (15)	−0.0067 (15)	−0.0024 (14)
C2	0.0362 (18)	0.0303 (15)	0.0324 (17)	−0.0055 (13)	−0.0007 (14)	0.0014 (13)
C3	0.0391 (19)	0.0272 (15)	0.0310 (17)	0.0001 (14)	−0.0073 (14)	−0.0026 (13)
C4	0.0306 (17)	0.0295 (15)	0.0266 (17)	−0.0016 (13)	−0.0012 (13)	−0.0043 (13)

C5	0.0368 (18)	0.0255 (14)	0.0236 (15)	−0.0027 (13)	0.0054 (12)	−0.0039 (12)
C6	0.0387 (19)	0.0317 (16)	0.0298 (17)	−0.0047 (14)	0.0003 (14)	−0.0033 (13)
C7	0.0398 (19)	0.0328 (16)	0.0279 (16)	0.0030 (14)	0.0011 (14)	0.0016 (13)
C8	0.045 (2)	0.0233 (14)	0.0271 (16)	−0.0053 (13)	0.0061 (14)	−0.0008 (12)
C9	0.0396 (18)	0.0297 (15)	0.0254 (16)	−0.0053 (14)	0.0032 (13)	−0.0040 (13)
C10	0.0347 (18)	0.0319 (15)	0.0216 (15)	0.0006 (13)	0.0011 (13)	−0.0039 (12)

Geometric parameters (Å, º)

Cl1—C7	1.724 (3)	N4—H4	0.8600
Cl2—C8	1.733 (3)	N4—C4	1.354 (4)
Cl3—C10	1.742 (3)	N4—C5	1.410 (4)
S1—C1	1.723 (3)	C3—H3C	0.9700
S1—C2	1.733 (3)	C3—H3D	0.9700
S2—C2	1.750 (3)	C3—C4	1.503 (4)
S2—C3	1.812 (3)	C5—C6	1.393 (4)
O8—C4	1.222 (4)	C5—C10	1.392 (4)
N1—N2	1.388 (4)	C6—H6	0.9300
N1—C2	1.287 (4)	C6—C7	1.383 (4)
N2—C1	1.308 (4)	C7—C8	1.383 (4)
N3—H3A	0.8607	C8—C9	1.378 (4)
N3—H3B	0.8611	C9—H9	0.9300
N3—C1	1.369 (4)	C9—C10	1.371 (4)
C1—S1—C2	86.57 (15)	O8—C4—N4	123.5 (3)
C2—S2—C3	100.39 (15)	O8—C4—C3	122.4 (3)
C2—N1—N2	113.0 (3)	N4—C4—C3	114.1 (3)
C1—N2—N1	111.5 (3)	C6—C5—N4	121.7 (3)
H3A—N3—H3B	109.4	C10—C5—N4	120.2 (3)
C1—N3—H3A	109.3	C10—C5—C6	118.0 (3)
C1—N3—H3B	109.0	C5—C6—H6	119.7
C4—N4—H4	117.9	C7—C6—C5	120.6 (3)
C4—N4—C5	124.3 (3)	C7—C6—H6	119.7
C5—N4—H4	117.9	C6—C7—Cl1	119.0 (2)
N2—C1—S1	114.6 (2)	C6—C7—C8	119.9 (3)
N2—C1—N3	122.2 (3)	C8—C7—Cl1	121.1 (2)
N3—C1—S1	123.2 (3)	C7—C8—Cl2	121.1 (2)
S1—C2—S2	121.61 (18)	C9—C8—Cl2	118.5 (2)
N1—C2—S1	114.4 (2)	C9—C8—C7	120.4 (3)
N1—C2—S2	124.0 (3)	C8—C9—H9	120.3
S2—C3—H3C	109.9	C10—C9—C8	119.4 (3)
S2—C3—H3D	109.9	C10—C9—H9	120.3
H3C—C3—H3D	108.3	C5—C10—Cl3	119.3 (2)
C4—C3—S2	109.1 (2)	C9—C10—Cl3	118.9 (2)
C4—C3—H3C	109.9	C9—C10—C5	121.8 (3)
C4—C3—H3D	109.9		
Cl1—C7—C8—Cl2	−0.5 (4)	C2—N1—N2—C1	−1.0 (5)

Cl1—C7—C8—C9	177.8 (2)	C3—S2—C2—S1	−87.5 (2)
Cl2—C8—C9—C10	177.9 (2)	C3—S2—C2—N1	95.8 (3)
S2—C3—C4—O8	95.0 (3)	C4—N4—C5—C6	−44.6 (5)
S2—C3—C4—N4	−83.7 (3)	C4—N4—C5—C10	134.0 (3)
N1—N2—C1—S1	1.5 (4)	C5—N4—C4—O8	−0.2 (5)
N1—N2—C1—N3	−177.3 (3)	C5—N4—C4—C3	178.6 (3)
N2—N1—C2—S1	0.0 (4)	C5—C6—C7—Cl1	−176.9 (2)
N2—N1—C2—S2	177.0 (3)	C5—C6—C7—C8	1.1 (5)
N4—C5—C6—C7	177.1 (3)	C6—C5—C10—Cl3	179.9 (2)
N4—C5—C10—Cl3	1.3 (4)	C6—C5—C10—C9	0.9 (5)
N4—C5—C10—C9	−177.7 (3)	C6—C7—C8—Cl2	−178.4 (2)
C1—S1—C2—S2	−176.4 (2)	C6—C7—C8—C9	−0.2 (5)
C1—S1—C2—N1	0.6 (3)	C7—C8—C9—C10	−0.4 (5)
C2—S1—C1—N2	−1.2 (3)	C8—C9—C10—Cl3	−179.0 (2)
C2—S1—C1—N3	177.5 (3)	C8—C9—C10—C5	0.0 (5)
C2—S2—C3—C4	−90.1 (2)	C10—C5—C6—C7	−1.5 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···O8^{i}	0.86	2.09	2.927 (3)	163
C3—H3*D*···O8^{i}	0.97	2.43	3.166 (3)	133
N3—H3*A*···N1^{ii}	0.86	2.51	3.304 (4)	154

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2.