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

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Keywords: crystal structure; 1,3,4-thiadiazole; intermolecular hydrogen bonds.

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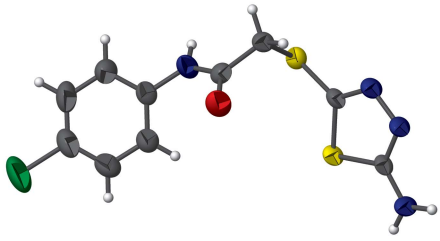
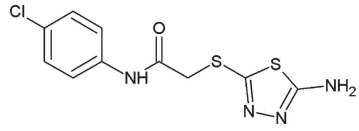
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{10}H_9ClN_4OS_2$, the dihedral angle between the planes of the chlorophenyl and thiadiazole groups is 32.93 (16)°. The molecules are connected through intermolecular N—H···N and N—H···O hydrogen bonds. An N—H···N hydrogen bond forms $R_2^2(8)$ ring motifs.

3D view



Chemical scheme



Structure description

As part of our research on synthesis and crystal structure determination of imidazo-[2,1-*b*][1,3,4]thiadiazole derivatives, we report here the crystal and molecular structure of 2-[(5-amino-1,3,4-thiadiazol-2-yl)sulfanyl]-*N*-(4-chlorophenyl)acetamide (Fig. 1).

The dihedral angle between the chlorophenyl (C5–C10) and thiadiazol (C1/C2/N1/N2/S1) rings is 32.93 (16)°. In the crystal (Fig. 2), the molecules are connected through N—H···N and N—H···O hydrogen bonds (Table 1), the N3—H3*A*···N2 hydrogen bonds forming $R_2^2(8)$ ring motifs. Overall, these interactions generate a two-dimensional network parallel to (100).

Synthesis and crystallization

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

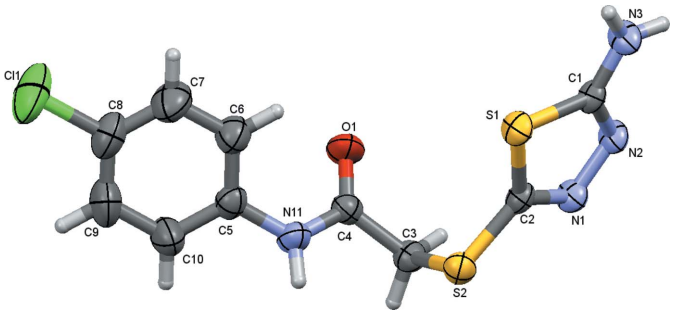


Figure 1
A view of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

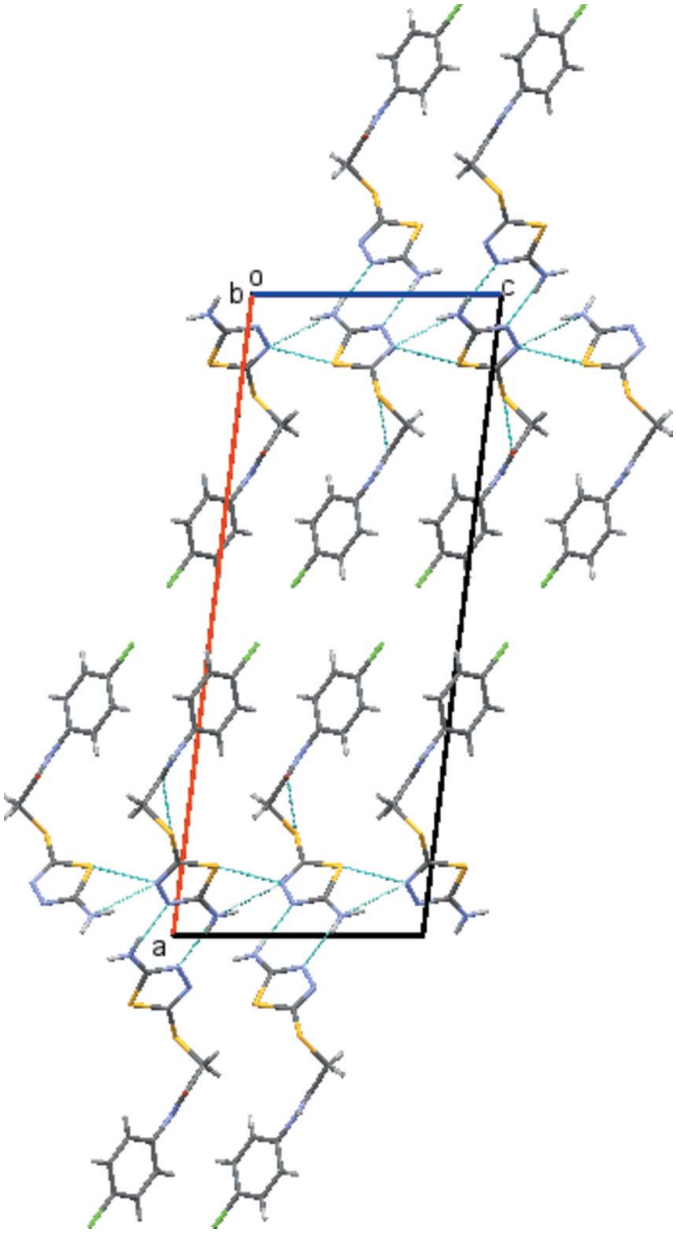


Figure 2
A view along the *b* axis of the crystal packing of the title compound. Hydrogen bonds are drawn as dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2^{i}	0.86	2.12	2.978 (3)	172
N3—H3B···N1^{ii}	0.86	2.17	2.973 (3)	156
N3—H3B···N2^{ii}	0.86	2.58	3.319 (3)	144
N11—H11···O1^{iii}	0.86	2.09	2.926 (3)	164

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_9ClN_4OS_2$
M_r	300.78
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	26.379 (5), 4.7704 (8), 10.1866 (18)
β (°)	96.996 (7)
V (Å^3)	1272.3 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.62
Crystal size (mm)	0.32 × 0.23 × 0.12
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (*NUMABS*; Rigaku 1999)
T_{min}, T_{max}	0.843, 0.928
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10559, 2881, 2019
R_{int}	0.066
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.127, 1.06
No. of reflections	2881
No. of parameters	163
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.34

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

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161139 [https://doi.org/10.1107/S2414314616011391]

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Crystal data

$C_{10}H_9ClN_4OS_2$
M_r = 300.78
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 26.379 (5) Å
b = 4.7704 (8) Å
c = 10.1866 (18) Å
β = 96.996 (7)°
V = 1272.3 (4) Å^3
Z = 4
$F(000)$ = 616
D_x = 1.570 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 2881 reflections
θ = 3.1–27.5°
μ = 0.62 mm^{-1}
T = 293 K
Block, brown
0.32 × 0.23 × 0.12 mm

Data collection

Rigaku Saturn724+ diffractometer
profile data from ω–scans
Absorption correction: multi-scan (NUMABS; Rigaku 1999)
T_{min} = 0.843, T_{max} = 0.928
10559 measured reflections
2881 independent reflections
2019 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −34→34
k = −6→5
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.127
S = 1.06
2881 reflections
163 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\Sigma^2(F_o^2) + (0.049P)^2 + 0.4848P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.45400 (4)	0.6782 (3)	0.30886 (13)	0.1062 (6)
S1	0.10780 (3)	0.59735 (16)	0.36162 (7)	0.0389 (2)
S2	0.15876 (3)	0.17391 (16)	0.55874 (7)	0.0409 (2)
O1	0.24709 (8)	0.7463 (4)	0.6213 (2)	0.0543 (8)
N1	0.08143 (8)	0.5253 (5)	0.5913 (2)	0.0386 (8)
N2	0.04937 (8)	0.7301 (5)	0.5344 (2)	0.0372 (7)
N3	0.03064 (8)	0.9734 (6)	0.3363 (2)	0.0422 (8)
N11	0.27991 (9)	0.3229 (5)	0.5821 (3)	0.0476 (9)
C1	0.05795 (10)	0.7883 (6)	0.4124 (2)	0.0331 (8)
C2	0.11406 (10)	0.4409 (6)	0.5160 (2)	0.0348 (9)
C3	0.20296 (10)	0.3442 (6)	0.6846 (3)	0.0394 (9)
C4	0.24508 (10)	0.4915 (6)	0.6270 (3)	0.0382 (9)
C5	0.32186 (11)	0.4125 (6)	0.5165 (3)	0.0435 (10)
C6	0.31545 (12)	0.6126 (7)	0.4193 (3)	0.0554 (11)
C7	0.35637 (14)	0.6942 (9)	0.3561 (4)	0.0704 (14)
C8	0.40275 (13)	0.5755 (9)	0.3893 (4)	0.0638 (14)
C9	0.40974 (13)	0.3734 (9)	0.4832 (4)	0.0719 (15)
C10	0.36869 (13)	0.2904 (8)	0.5480 (4)	0.0654 (16)
H3A	0.00600	1.06190	0.36610	0.0510*
H3B	0.03760	1.00480	0.25730	0.0510*
H3C	0.18440	0.47870	0.73200	0.0470*
H3D	0.21750	0.20480	0.74750	0.0470*
H6	0.28350	0.69330	0.39610	0.0670*
H7	0.35210	0.83100	0.29060	0.0840*
H9	0.44160	0.29080	0.50410	0.0860*
H10	0.37310	0.15200	0.61270	0.0790*
H11	0.27660	0.14560	0.59390	0.0570*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0674 (7)	0.1549 (13)	0.1040 (9)	−0.0259 (7)	0.0419 (6)	0.0066 (8)
S1	0.0432 (4)	0.0475 (5)	0.0289 (3)	0.0042 (3)	0.0162 (3)	−0.0013 (3)
S2	0.0409 (4)	0.0356 (4)	0.0466 (4)	−0.0003 (3)	0.0076 (3)	−0.0044 (3)
O1	0.0574 (13)	0.0294 (12)	0.0784 (17)	0.0021 (10)	0.0176 (12)	−0.0010 (11)
N1	0.0427 (13)	0.0462 (15)	0.0290 (11)	0.0023 (11)	0.0127 (10)	0.0006 (11)
N2	0.0389 (12)	0.0488 (15)	0.0259 (11)	0.0054 (10)	0.0127 (9)	0.0016 (10)
N3	0.0479 (14)	0.0551 (17)	0.0260 (11)	0.0120 (12)	0.0140 (10)	0.0046 (11)
N11	0.0482 (15)	0.0326 (14)	0.0654 (17)	−0.0006 (11)	0.0206 (12)	0.0012 (13)
C1	0.0339 (13)	0.0420 (17)	0.0251 (12)	−0.0035 (12)	0.0105 (10)	−0.0049 (12)
C2	0.0353 (13)	0.0394 (17)	0.0315 (14)	−0.0064 (12)	0.0109 (11)	−0.0019 (12)
C3	0.0434 (15)	0.0429 (18)	0.0328 (14)	0.0029 (13)	0.0086 (12)	0.0016 (13)
C4	0.0378 (14)	0.0358 (17)	0.0399 (15)	0.0025 (12)	0.0000 (12)	−0.0009 (13)
C5	0.0424 (16)	0.0399 (18)	0.0497 (18)	−0.0041 (13)	0.0116 (13)	−0.0058 (15)
C6	0.0480 (18)	0.065 (2)	0.054 (2)	−0.0009 (16)	0.0099 (15)	0.0080 (18)

C7	0.066 (2)	0.087 (3)	0.060 (2)	−0.006 (2)	0.0153 (18)	0.020 (2)
C8	0.051 (2)	0.082 (3)	0.061 (2)	−0.0152 (19)	0.0178 (17)	−0.006 (2)
C9	0.0408 (18)	0.091 (3)	0.086 (3)	0.0049 (19)	0.0159 (18)	−0.003 (2)
C10	0.050 (2)	0.072 (3)	0.077 (3)	0.0151 (18)	0.0187 (18)	0.019 (2)

Geometric parameters (Å, º)

Cl1—C8	1.735 (4)	N3—H3A	0.8600
S1—C1	1.730 (3)	C5—C10	1.368 (5)
S1—C2	1.730 (2)	C5—C6	1.371 (4)
S2—C2	1.754 (3)	C6—C7	1.379 (5)
S2—C3	1.816 (3)	C7—C8	1.353 (5)
O1—C4	1.218 (3)	C8—C9	1.355 (6)
N1—N2	1.373 (3)	C9—C10	1.393 (5)
N1—C2	1.285 (3)	N11—H11	0.8600
N2—C1	1.320 (3)	C3—H3C	0.9700
N3—C1	1.328 (4)	C3—H3D	0.9700
N11—C4	1.343 (4)	C6—H6	0.9300
N11—C5	1.426 (4)	C7—H7	0.9300
C3—C4	1.494 (4)	C9—H9	0.9300
N3—H3B	0.8600	C10—H10	0.9300
C1—S1—C2	86.98 (12)	C6—C7—C8	120.0 (4)
C2—S2—C3	101.82 (13)	Cl1—C8—C9	119.2 (3)
N2—N1—C2	113.2 (2)	Cl1—C8—C7	119.8 (3)
N1—N2—C1	112.3 (2)	C7—C8—C9	121.0 (4)
C4—N11—C5	125.6 (2)	C8—C9—C10	119.4 (3)
S1—C1—N2	113.3 (2)	C5—C10—C9	120.0 (3)
S1—C1—N3	123.62 (17)	C4—N11—H11	117.00
N2—C1—N3	123.1 (2)	C5—N11—H11	117.00
S1—C2—S2	121.71 (14)	S2—C3—H3C	109.00
S1—C2—N1	114.2 (2)	S2—C3—H3D	109.00
S2—C2—N1	123.92 (18)	C4—C3—H3C	109.00
S2—C3—C4	112.2 (2)	C4—C3—H3D	109.00
H3A—N3—H3B	120.00	H3C—C3—H3D	108.00
C1—N3—H3B	120.00	C5—C6—H6	120.00
C1—N3—H3A	120.00	C7—C6—H6	120.00
N11—C4—C3	115.1 (2)	C6—C7—H7	120.00
O1—C4—C3	121.8 (3)	C8—C7—H7	120.00
O1—C4—N11	123.1 (3)	C8—C9—H9	120.00
N11—C5—C6	120.8 (3)	C10—C9—H9	120.00
N11—C5—C10	119.5 (3)	C5—C10—H10	120.00
C6—C5—C10	119.6 (3)	C9—C10—H10	120.00
C5—C6—C7	120.0 (3)		
C2—S1—C1—N2	0.4 (2)	C4—N11—C5—C6	−44.4 (5)
C2—S1—C1—N3	−179.1 (3)	C4—N11—C5—C10	137.8 (3)
C1—S1—C2—S2	176.98 (19)	S2—C3—C4—O1	107.9 (3)

C1—S1—C2—N1	1.0 (2)	S2—C3—C4—N11	−71.8 (3)
C3—S2—C2—S1	112.82 (18)	N11—C5—C6—C7	−179.3 (3)
C3—S2—C2—N1	−71.6 (3)	C10—C5—C6—C7	−1.5 (5)
C2—S2—C3—C4	−89.6 (2)	N11—C5—C10—C9	179.1 (3)
C2—N1—N2—C1	2.3 (3)	C6—C5—C10—C9	1.2 (5)
N2—N1—C2—S1	−2.1 (3)	C5—C6—C7—C8	0.5 (6)
N2—N1—C2—S2	−177.95 (19)	C6—C7—C8—Cl1	179.7 (3)
N1—N2—C1—S1	−1.5 (3)	C6—C7—C8—C9	0.9 (6)
N1—N2—C1—N3	177.9 (2)	Cl1—C8—C9—C10	180.0 (3)
C5—N11—C4—O1	−3.4 (5)	C7—C8—C9—C10	−1.1 (6)
C5—N11—C4—C3	176.3 (3)	C8—C9—C10—C5	0.1 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2^{i}	0.86	2.12	2.978 (3)	172
N3—H3*B*···N1^{ii}	0.86	2.17	2.973 (3)	156
N3—H3*B*···N2^{ii}	0.86	2.58	3.319 (3)	144
N11—H11···O1^{iii}	0.86	2.09	2.926 (3)	164

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