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6-(3-Chlorophenyl)imidazo[2,1-*b*][1,3,4]-thiadiazoleA.S. Praveen,^a Jerry P. Jasinski,^{b*} Shannon T. Krauss,^b
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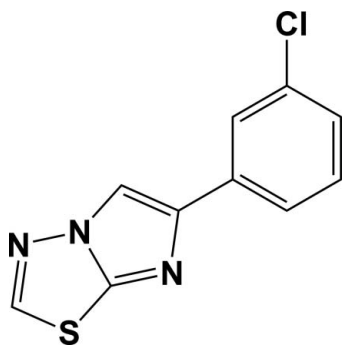
Received 25 November 2012; accepted 4 December 2012

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 13.5.

In the title compound, $\text{C}_{10}\text{H}_8\text{ClN}_3\text{S}$, the dihedral angle between the mean planes of the benzene and imidazo[2,1-*b*][1,3,4]thiadiazole rings is $6.0(9)^\circ$. In the crystal, molecules are assembled by the formation of centrosymmetric dimers by π -stacking of the thiadiazole and benzene rings of neighboring molecules [centroid-centroid distance = $3.6938(11)$ Å] along [010].

Related literature

For related imidazothiadiazole derivatives and their pharmacological potential, see: Palagiano *et al.* (1995). For related structures, see: Banu *et al.* (2011*a,b*); Fun *et al.* (2011*a,b*). For standard bond lengths, see Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{10}\text{H}_8\text{ClN}_3\text{S}$	$V = 941.77(5)$ Å ³
$M_r = 235.70$	$Z = 4$
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation
$a = 5.43804(19)$ Å	$\mu = 5.37$ mm ⁻¹
$b = 12.4222(4)$ Å	$T = 173$ K
$c = 14.1684(4)$ Å	$0.26 \times 0.12 \times 0.06$ mm
$\beta = 100.269(3)^\circ$	

Data collection

Agilent Xcalibur (Eos, Gemini) diffractometer	5495 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> and <i>CrysAlis RED</i> ; Agilent, 2012)	1846 independent reflections
$T_{\min} = 0.691$, $T_{\max} = 1.000$	1650 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$	137 parameters
$wR(F^2) = 0.100$	H-atom parameters constrained
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.36$ e Å ⁻³
1846 reflections	$\Delta\rho_{\text{min}} = -0.23$ e Å ⁻³

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2414).

References

- Agilent (2012). *CrysAlis PRO* and *CrysAlis RED* Agilent Technologies, Yarnton, England.
- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Banu, A., Begum, N. S., Lamani, R. S. & Khazi, I. M. (2011*a*). *Acta Cryst. E67*, o779.
- Banu, A., Ziaulla, M., Begum, N. S., Lamani, R. S. & Khazi, I. M. (2011*b*). *Acta Cryst. E67*, o617–o618.
- Fun, H.-K., Hemamalini, M., Prasad, D. J., Nagaraja, G. K. & Anitha, V. V. (2011*b*). *Acta Cryst. E67*, o207.
- Fun, H.-K., Yeap, C. S., Prasad, D. J., Castelino, P. A. & Anitha, V. V. (2011*a*). *Acta Cryst. E67*, o255.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Palagiano, F., Arenare, L., Laraschi, E., de Caprariis, P., Abignente, E., Amico, M. D., Filippelli, W. & Rossi, F. (1995). *Eur. J. Med. Chem.* **30**, 901–910.
- Sheldrick, G. M. (2008). *Acta Cryst. A64*, 112–122.

supplementary materials

Acta Cryst. (2013). E69, o66 [doi:10.1107/S1600536812049793]

6-(3-Chlorophenyl)imidazo[2,1-*b*][1,3,4]thiadiazole

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Comment

Many imidazothiadiazole derivatives have been reported to possess diverse medicinal properties such as anthelmintic, antimicrobial, anti-inflammatory, antipyretic, analgesic properties and many other activities of therapeutic significance (Palagiano *et al.*, 1995). The crystal structures of some imidazothiadiazole molecules, viz., 6-(4-bromophenyl)-2-(4-fluorobenzyl)imidazo[2,1-*b*][1,3,4]thiadiazole, 3-{{6-(4-Chlorophenyl)imidazo[2,1-*b*][1,3,4]thiadiazol-2-yl}methyl}-1,2-benzoxazole (Banu *et al.*, 2011*a,b*), 2-isobutyl-6-(4-methoxyphenyl)imidazo[2,1-*b*][1,3,4]thiadiazole and 6-(4-chlorophenyl)-2-isobutylimidazo[2,1-*b*][1,3,4]thiadiazole (Fun *et al.*, 2011*a,b*) have been reported.

In the title compound, C₁₀H₈N₃SCl, the dihedral angle between the mean planes of the benzene and imidazo[2,1*b*][1,3,4]thiadiazole rings is 6.0 (9)° (Fig. 1). Bond lengths are in normal ranges (Allen *et al.*, 1987). In the crystal, molecules are assembled by the formation of centrosymmetric dimers by π -stacking of thiadiazole and benzene rings of neighboring molecules (centroid-centroid distance = 3.6938 (11) Å) along [010] (Fig. 2). Additional weak π -stacking interactions involving nearby benzene rings (centroid-centroid distance = 3.7739 (11) Å) are also observed.

Experimental

A solution of 1,3,4-thiadiazol-2-amine (500 mg, 4.9 mmol) and 2-bromo-1-(3-chlorophenyl)ethanone (1.1 g, 4.9 mmol) in DMF (10 mL) was placed in a microwave pyrex tube which was introduced into a Biotage Initiator-microwave reactor fitted with a rotational system. Microwave irradiation was performed for 10 minutes at 373 K, then the mixture was cooled to ambient temperature. The reaction mass was poured into ice, the precipitated solid was filtered and dried. The single crystal was grown from a solution in ethyl acetate by slow evaporation of the solvent (yield: 97%; (m.p.: 437–440 K).

Refinement

All H atoms were placed in their calculated positions and then refined using the riding model with C—H lengths of 0.93 Å. Isotropic displacement parameters for hydrogen atoms were set to 1.18–1.21 (CH) times U_{eq} of the parent atom.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

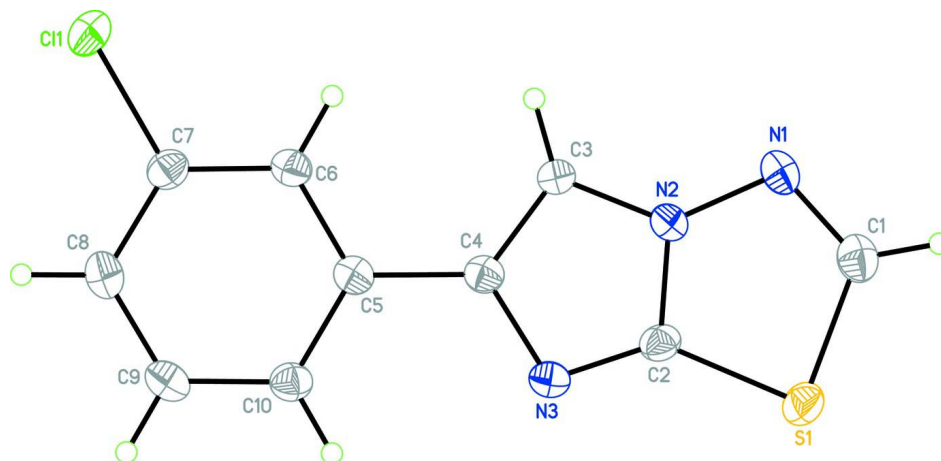


Figure 1

Molecular structure of the title compound showing the atom labeling scheme and 50% probability displacement ellipsoids.

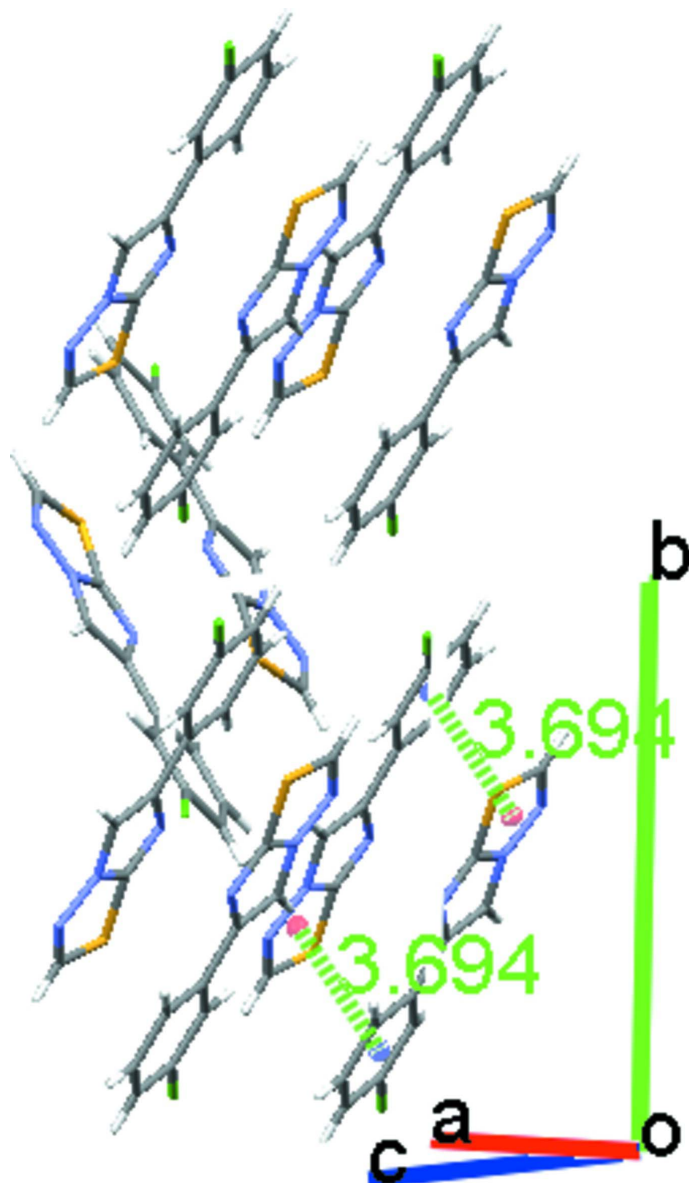


Figure 2

Packing diagram of the title compound viewed along the a axis. Dashed lines indicate π -stacking of thiazole and benzene rings of neighboring molecules (centroid–centroid distance = 3.6938 (11) Å) into centrosymmetric dimers along [010]. Hydrogen atoms have been removed for clarity.

6-(3-Chlorophenyl)imidazo[2,1- b][1,3,4]thiadiazole

Crystal data

$C_{10}H_6ClN_3S$

$M_r = 235.70$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1/n$

$a = 5.43804$ (19) Å

$b = 12.4222$ (4) Å

$c = 14.1684$ (4) Å

$\beta = 100.269$ (3)°

$V = 941.77$ (5) Å³

$Z = 4$

$F(000) = 480$

$D_x = 1.676$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 2788 reflections

$\theta = 3.2\text{--}72.4^\circ$
 $\mu = 5.37\text{ mm}^{-1}$
 $T = 173\text{ K}$

Chunk, colorless
 $0.26 \times 0.12 \times 0.06\text{ mm}$

Data collection

Agilent Xcalibur (Eos, Gemini)
 diffractometer
 Radiation source: Enhance (Cu) X-ray Source
 Graphite monochromator
 Detector resolution: 16.0416 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO and CrysAlis RED; Agilent,
 2012)

$T_{\min} = 0.691$, $T_{\max} = 1.000$
 5495 measured reflections
 1846 independent reflections
 1650 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\max} = 72.5^\circ$, $\theta_{\min} = 4.8^\circ$
 $h = -6 \rightarrow 6$
 $k = -12 \rightarrow 15$
 $l = -17 \rightarrow 13$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.100$
 $S = 1.06$
 1846 reflections
 137 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.2711P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.36\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23\text{ e \AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0028 (5)

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.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.35900 (10)	0.10200 (4)	0.67741 (4)	0.02949 (18)
S1	0.39725 (10)	0.62878 (4)	0.24567 (3)	0.02652 (18)
N1	0.0469 (3)	0.61595 (14)	0.34925 (13)	0.0253 (4)
N2	0.2006 (3)	0.52794 (13)	0.36757 (12)	0.0199 (4)
N3	0.5314 (3)	0.43233 (13)	0.34504 (12)	0.0222 (4)
C1	0.1320 (4)	0.67447 (17)	0.28675 (15)	0.0261 (4)
H1	0.0545	0.7386	0.2645	0.031*
C2	0.3979 (4)	0.51895 (15)	0.32099 (13)	0.0208 (4)
C3	0.2043 (4)	0.44025 (16)	0.42652 (14)	0.0217 (4)
H3	0.0929	0.4235	0.4671	0.026*
C4	0.4101 (4)	0.38271 (15)	0.41207 (13)	0.0190 (4)
C5	0.5067 (3)	0.28278 (15)	0.46084 (13)	0.0192 (4)

C6	0.3963 (3)	0.24196 (15)	0.53512 (14)	0.0205 (4)
H6	0.2605	0.2769	0.5531	0.025*
C7	0.4911 (4)	0.14891 (16)	0.58176 (13)	0.0213 (4)
C8	0.6921 (4)	0.09440 (16)	0.55644 (16)	0.0258 (4)
H8	0.7526	0.0317	0.5881	0.031*
C9	0.8008 (4)	0.13562 (16)	0.48276 (15)	0.0254 (4)
H9	0.9360	0.1001	0.4650	0.030*
C10	0.7107 (4)	0.22930 (15)	0.43499 (14)	0.0219 (4)
H10	0.7861	0.2563	0.3860	0.026*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0325 (3)	0.0305 (3)	0.0269 (3)	0.00059 (19)	0.0091 (2)	0.00925 (19)
S1	0.0346 (3)	0.0247 (3)	0.0222 (3)	0.00174 (19)	0.0104 (2)	0.00440 (18)
N1	0.0239 (9)	0.0224 (8)	0.0297 (9)	0.0063 (6)	0.0050 (7)	0.0021 (7)
N2	0.0198 (8)	0.0193 (8)	0.0212 (8)	0.0029 (6)	0.0052 (6)	-0.0002 (6)
N3	0.0228 (8)	0.0234 (8)	0.0221 (8)	0.0031 (7)	0.0087 (7)	0.0004 (6)
C1	0.0292 (11)	0.0235 (10)	0.0246 (10)	0.0033 (8)	0.0019 (8)	0.0003 (8)
C2	0.0243 (10)	0.0224 (9)	0.0167 (9)	-0.0007 (7)	0.0069 (7)	-0.0006 (7)
C3	0.0237 (10)	0.0215 (9)	0.0220 (9)	0.0030 (7)	0.0096 (8)	0.0032 (7)
C4	0.0195 (10)	0.0208 (9)	0.0166 (9)	0.0000 (7)	0.0027 (7)	-0.0023 (7)
C5	0.0190 (9)	0.0200 (9)	0.0181 (9)	-0.0005 (7)	0.0021 (7)	-0.0034 (7)
C6	0.0194 (9)	0.0220 (9)	0.0203 (9)	0.0013 (7)	0.0045 (7)	-0.0021 (7)
C7	0.0212 (10)	0.0228 (9)	0.0197 (9)	-0.0031 (8)	0.0035 (8)	0.0002 (7)
C8	0.0256 (10)	0.0220 (10)	0.0289 (11)	0.0044 (8)	0.0023 (9)	0.0022 (8)
C9	0.0220 (10)	0.0258 (10)	0.0292 (10)	0.0043 (8)	0.0069 (8)	-0.0028 (8)
C10	0.0229 (10)	0.0221 (9)	0.0214 (9)	-0.0003 (8)	0.0063 (8)	-0.0023 (7)

Geometric parameters (\AA , $^\circ$)

C11—C7	1.7432 (19)	C4—C5	1.471 (3)
S1—C2	1.7318 (19)	C5—C6	1.397 (3)
S1—C1	1.744 (2)	C5—C10	1.397 (3)
N1—C1	1.293 (3)	C6—C7	1.385 (3)
N1—N2	1.373 (2)	C6—H6	0.9300
N2—C2	1.361 (3)	C7—C8	1.386 (3)
N2—C3	1.371 (2)	C8—C9	1.386 (3)
N3—C2	1.309 (2)	C8—H8	0.9300
N3—C4	1.393 (2)	C9—C10	1.391 (3)
C1—H1	0.9300	C9—H9	0.9300
C3—C4	1.373 (3)	C10—H10	0.9300
C3—H3	0.9300		
C2—S1—C1	87.81 (9)	C6—C5—C10	119.54 (18)
C1—N1—N2	107.27 (17)	C6—C5—C4	119.66 (17)
C2—N2—C3	107.63 (16)	C10—C5—C4	120.79 (18)
C2—N2—N1	118.68 (16)	C7—C6—C5	119.29 (17)
C3—N2—N1	133.69 (17)	C7—C6—H6	120.4
C2—N3—C4	103.49 (15)	C5—C6—H6	120.4

N1—C1—S1	117.63 (16)	C6—C7—C8	121.87 (18)
N1—C1—H1	121.2	C6—C7—C11	118.69 (15)
S1—C1—H1	121.2	C8—C7—C11	119.41 (16)
N3—C2—N2	112.95 (17)	C9—C8—C7	118.44 (19)
N3—C2—S1	138.46 (15)	C9—C8—H8	120.8
N2—C2—S1	108.59 (14)	C7—C8—H8	120.8
N2—C3—C4	104.29 (16)	C8—C9—C10	121.00 (19)
N2—C3—H3	127.9	C8—C9—H9	119.5
C4—C3—H3	127.9	C10—C9—H9	119.5
C3—C4—N3	111.64 (16)	C9—C10—C5	119.86 (18)
C3—C4—C5	126.96 (18)	C9—C10—H10	120.1
N3—C4—C5	121.37 (17)	C5—C10—H10	120.1
C1—N1—N2—C2	0.2 (2)	C2—N3—C4—C3	0.4 (2)
C1—N1—N2—C3	179.7 (2)	C2—N3—C4—C5	-177.68 (17)
N2—N1—C1—S1	0.3 (2)	C3—C4—C5—C6	-4.9 (3)
C2—S1—C1—N1	-0.45 (17)	N3—C4—C5—C6	172.90 (17)
C4—N3—C2—N2	-0.1 (2)	C3—C4—C5—C10	176.33 (19)
C4—N3—C2—S1	179.75 (18)	N3—C4—C5—C10	-5.9 (3)
C3—N2—C2—N3	-0.2 (2)	C10—C5—C6—C7	-0.1 (3)
N1—N2—C2—N3	179.44 (17)	C4—C5—C6—C7	-178.88 (17)
C3—N2—C2—S1	179.87 (13)	C5—C6—C7—C8	-0.4 (3)
N1—N2—C2—S1	-0.5 (2)	C5—C6—C7—C11	177.50 (14)
C1—S1—C2—N3	-179.4 (2)	C6—C7—C8—C9	0.5 (3)
C1—S1—C2—N2	0.47 (14)	C11—C7—C8—C9	-177.37 (15)
C2—N2—C3—C4	0.5 (2)	C7—C8—C9—C10	-0.1 (3)
N1—N2—C3—C4	-179.12 (19)	C8—C9—C10—C5	-0.4 (3)
N2—C3—C4—N3	-0.6 (2)	C6—C5—C10—C9	0.5 (3)
N2—C3—C4—C5	177.43 (17)	C4—C5—C10—C9	179.27 (18)
