

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-(4-Chlorophenyl)-N-(1,3-thiazol-2-yl)-acetamide

 Hoong-Kun Fun,^{a*} ‡ Ching Kheng Quah,^{a§} Prakash S. Nayak,^b B. Narayana^b and B. K. Sarojini^c

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of Studies in Chemistry, Mangalore University, Mangalagangothri 574 199, India, and ^cDepartment of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalore 574 153, India
Correspondence e-mail: hkfun@usm.my

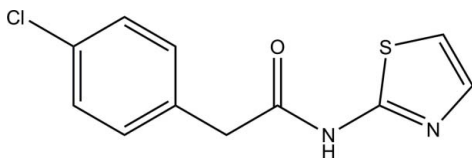
Received 31 July 2012; accepted 3 August 2012

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.116; data-to-parameter ratio = 23.0.

In the title compound, $\text{C}_{11}\text{H}_9\text{ClN}_2\text{OS}$, the thiazole ring is nearly planar (r.m.s. deviation = 0.003 Å) and forms a dihedral angle of 64.18 (7)° with the benzene ring. In the crystal, inversion dimers linked by pairs of $\text{N}-\text{H}\cdots\text{N}_t$ ($t = \text{thiazole}$) hydrogen bonds generate $R_2^2(8)$ loops.

Related literature

For general background to the title compound and for related structures, see: Fun *et al.* (2011a,b, 2012a,b). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{11}\text{H}_9\text{ClN}_2\text{OS}$
 $M_r = 252.71$
Monoclinic, $P2_1/c$
 $a = 13.9169$ (13) Å
 $b = 5.5188$ (5) Å
 $c = 15.1836$ (14) Å
 $\beta = 100.311$ (2)°

$V = 1147.34$ (18) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.49$ mm⁻¹
 $T = 296$ K
0.35 × 0.29 × 0.18 mm

Data collection

Bruker SMART APEXII DUO
CCD diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
 $T_{\min} = 0.847$, $T_{\max} = 0.918$

11463 measured reflections
3421 independent reflections
2769 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.116$
 $S = 1.02$
3421 reflections
149 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.29$ e Å⁻³
 $\Delta\rho_{\min} = -0.29$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H1N2}\cdots\text{N1}^i$	0.866 (18)	2.096 (18)	2.9606 (16)	176.2 (17)

Symmetry code: (i) $-x + 1, -y + 1, -z + 2$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

The authors would like to thank Universiti Sains Malaysia (USM) for the Research University Grant (No. 1001/PFIZIK/811160). BN also thanks the UGC, New Delhi, and the Government of India for the purchase of chemicals through the SAP-DRS-Phase 1 programme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6927).

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‡ Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5525-2009.

supporting information

Acta Cryst. (2012). E68, o2679 [doi:10.1107/S1600536812034629]

2-(4-Chlorophenyl)-N-(1,3-thiazol-2-yl)acetamide

Hoong-Kun Fun, Ching Kheng Quah, Prakash S. Nayak, B. Narayana and B. K. Sarojini

S1. Comment

In continuation of our work on synthesis of amides (Fun *et al.*, 2011a, 2011b, 2012a, 2012b), we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the thiazol-2-yl ring (S1/N1/C1-C3) is nearly planar (r.m.s. deviation = 0.003 Å) and it forms a dihedral angle of 64.18 (7)° with the benzene ring (C6-C11). Bond lengths and angles are within normal ranges and are comparable to related structures (Fun *et al.*, 2011a, 2011b, 2012a, 2012b).

In the crystal structure, Fig. 2, molecules are linked into an inversion dimer by pairs of N2–H1N2···N1 hydrogen bonds (Table 1), generating R₂²(8) ring motifs (Bernstein *et al.*, 1995).

S2. Experimental

4-Chlorophenylacetic acid (0.170 g, 1 mmol), 2-aminothiazole (0.1 g, 1 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)-carbodiimide hydrochloride (1.0 g, 0.01 mol) were dissolved in dichloromethane (20 ml). The mixture was stirred in presence of triethylamine at 273 K for about 3 h. The contents were poured into 100 ml of ice-cold aqueous hydrochloric acid with stirring. The resulting solution was extracted thrice with dichloromethane. The organic layer was washed with saturated NaHCO₃ solution and brine solution, dried and concentrated under reduced pressure to give the title compound (I). Orange blocks were grown from an acetone and toluene (1:1) solvent mixture by the slow evaporation method (*m.p.*: 441K).

S3. Refinement

Atom H1N2 was located in a difference Fourier map and refined freely [N–H = 0.868 (19) Å]. The remaining H atoms were positioned geometrically and refined using a riding model with C–H = 0.93 or 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

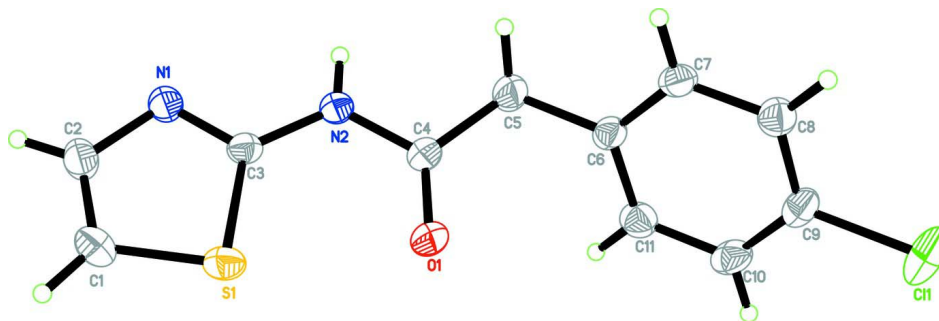


Figure 1

The molecular structure of the title compound showing 30% probability displacement ellipsoids for non-H atoms.

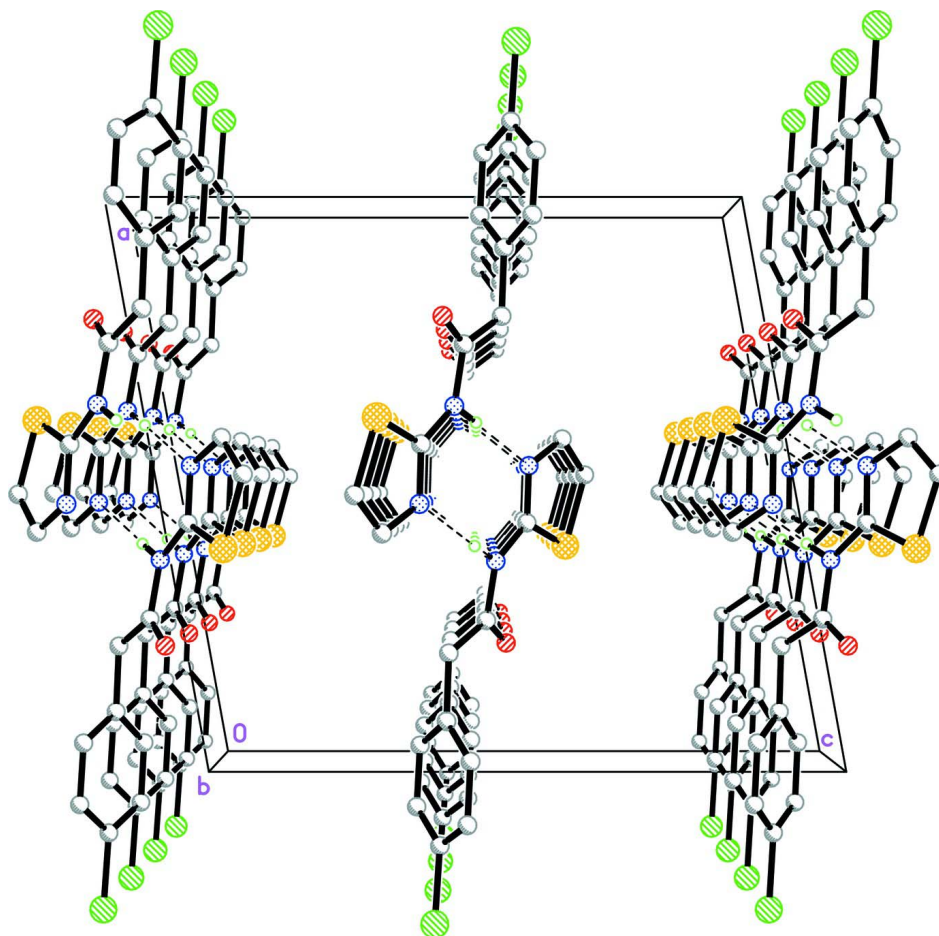


Figure 2

The crystal structure of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

2-(4-Chlorophenyl)-*N*-(1,3-thiazol-2-yl)acetamide

Crystal data

$C_{11}H_9ClN_2OS$

$M_r = 252.71$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 13.9169$ (13) Å

$b = 5.5188$ (5) Å

$c = 15.1836$ (14) Å

$\beta = 100.311$ (2)°

$V = 1147.34$ (18) Å³

$Z = 4$

$F(000) = 520$

$D_x = 1.463$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4547 reflections

$\theta = 2.7\text{--}30.3^\circ$

$\mu = 0.49$ mm⁻¹

$T = 296$ K

Block, orange

$0.35 \times 0.29 \times 0.18$ mm

Data collection

Bruker SMART APEXII DUO CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.847$, $T_{\max} = 0.918$

11463 measured reflections
 3421 independent reflections
 2769 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 30.3^\circ$, $\theta_{\text{min}} = 1.5^\circ$
 $h = -19 \rightarrow 19$
 $k = -7 \rightarrow 7$
 $l = -21 \rightarrow 21$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.116$
 $S = 1.02$
 3421 reflections
 149 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.2113P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.29 \text{ e } \text{Å}^{-3}$

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.

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 $F^2 > 2\sigma(F^2)$ 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 (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	-0.20822 (3)	0.54220 (12)	0.82496 (4)	0.08193 (19)
S1	0.39710 (3)	1.03306 (7)	1.11917 (3)	0.04855 (13)
O1	0.23806 (7)	0.8565 (2)	1.00294 (8)	0.0547 (3)
N1	0.53067 (8)	0.7454 (2)	1.08411 (8)	0.0444 (3)
N2	0.37558 (8)	0.6335 (2)	1.00962 (8)	0.0410 (2)
C1	0.51611 (12)	1.0855 (3)	1.16882 (11)	0.0530 (4)
H1A	0.5365	1.2115	1.2085	0.064*
C2	0.57568 (11)	0.9199 (3)	1.14263 (11)	0.0503 (3)
H2A	0.6429	0.9223	1.1626	0.060*
C3	0.43688 (9)	0.7846 (2)	1.06618 (8)	0.0370 (3)
C4	0.27847 (9)	0.6768 (2)	0.98005 (9)	0.0409 (3)
C5	0.22953 (11)	0.4817 (3)	0.91743 (12)	0.0508 (4)
H5A	0.2547	0.4902	0.8619	0.061*
H5B	0.2473	0.3248	0.9442	0.061*
C6	0.11993 (10)	0.4988 (2)	0.89603 (9)	0.0396 (3)
C7	0.06207 (10)	0.3160 (2)	0.92138 (9)	0.0425 (3)
H7A	0.0916	0.1835	0.9531	0.051*
C8	-0.03890 (11)	0.3270 (3)	0.90033 (10)	0.0458 (3)
H8A	-0.0773	0.2037	0.9174	0.055*
C9	-0.08118 (10)	0.5259 (3)	0.85331 (9)	0.0457 (3)

C10	-0.02621 (11)	0.7100 (3)	0.82739 (9)	0.0501 (3)
H10A	-0.0562	0.8422	0.7958	0.060*
C11	0.07454 (11)	0.6961 (3)	0.84901 (9)	0.0465 (3)
H11A	0.1124	0.8204	0.8319	0.056*
H1N2	0.4009 (14)	0.518 (3)	0.9827 (12)	0.052 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0398 (2)	0.1231 (5)	0.0797 (3)	0.0207 (2)	0.0020 (2)	0.0027 (3)
S1	0.0515 (2)	0.03917 (19)	0.0564 (2)	0.00832 (14)	0.01343 (16)	-0.00790 (14)
O1	0.0402 (5)	0.0473 (6)	0.0736 (7)	0.0117 (4)	0.0022 (5)	-0.0138 (5)
N1	0.0366 (5)	0.0441 (6)	0.0521 (6)	0.0018 (5)	0.0068 (5)	-0.0104 (5)
N2	0.0334 (5)	0.0361 (5)	0.0530 (6)	0.0044 (4)	0.0067 (5)	-0.0074 (5)
C1	0.0612 (9)	0.0455 (8)	0.0523 (8)	-0.0065 (7)	0.0099 (7)	-0.0129 (6)
C2	0.0433 (7)	0.0531 (8)	0.0529 (8)	-0.0051 (6)	0.0044 (6)	-0.0103 (6)
C3	0.0374 (6)	0.0328 (6)	0.0418 (6)	0.0036 (5)	0.0098 (5)	-0.0006 (5)
C4	0.0347 (6)	0.0398 (6)	0.0480 (7)	0.0047 (5)	0.0069 (5)	-0.0009 (5)
C5	0.0387 (7)	0.0504 (8)	0.0615 (9)	0.0064 (6)	0.0041 (6)	-0.0147 (7)
C6	0.0377 (6)	0.0405 (6)	0.0388 (6)	0.0062 (5)	0.0016 (5)	-0.0073 (5)
C7	0.0460 (7)	0.0368 (6)	0.0422 (6)	0.0087 (5)	0.0012 (5)	-0.0007 (5)
C8	0.0445 (7)	0.0466 (7)	0.0465 (7)	0.0006 (6)	0.0085 (6)	-0.0025 (6)
C9	0.0369 (6)	0.0582 (8)	0.0395 (6)	0.0115 (6)	0.0003 (5)	-0.0069 (6)
C10	0.0560 (8)	0.0472 (8)	0.0421 (7)	0.0150 (6)	-0.0051 (6)	0.0032 (6)
C11	0.0524 (8)	0.0415 (7)	0.0437 (7)	-0.0003 (6)	0.0033 (6)	0.0019 (5)

Geometric parameters (Å, °)

C11—C9	1.7455 (15)	C5—C6	1.5045 (19)
S1—C1	1.7170 (17)	C5—H5A	0.9700
S1—C3	1.7300 (13)	C5—H5B	0.9700
O1—C4	1.2211 (16)	C6—C7	1.388 (2)
N1—C3	1.3029 (17)	C6—C11	1.3904 (19)
N1—C2	1.3820 (18)	C7—C8	1.386 (2)
N2—C4	1.3666 (16)	C7—H7A	0.9300
N2—C3	1.3778 (17)	C8—C9	1.382 (2)
N2—H1N2	0.868 (19)	C8—H8A	0.9300
C1—C2	1.341 (2)	C9—C10	1.371 (2)
C1—H1A	0.9300	C10—C11	1.384 (2)
C2—H2A	0.9300	C10—H10A	0.9300
C4—C5	1.515 (2)	C11—H11A	0.9300
C1—S1—C3	88.50 (7)	C4—C5—H5B	108.6
C3—N1—C2	109.91 (12)	H5A—C5—H5B	107.6
C4—N2—C3	124.38 (11)	C7—C6—C11	118.63 (13)
C4—N2—H1N2	115.8 (12)	C7—C6—C5	120.71 (13)
C3—N2—H1N2	118.8 (12)	C11—C6—C5	120.65 (14)
C2—C1—S1	110.73 (12)	C8—C7—C6	121.25 (13)

C2—C1—H1A	124.6	C8—C7—H7A	119.4
S1—C1—H1A	124.6	C6—C7—H7A	119.4
C1—C2—N1	115.61 (14)	C9—C8—C7	118.34 (14)
C1—C2—H2A	122.2	C9—C8—H8A	120.8
N1—C2—H2A	122.2	C7—C8—H8A	120.8
N1—C3—N2	121.01 (11)	C10—C9—C8	121.92 (13)
N1—C3—S1	115.24 (10)	C10—C9—C11	118.92 (11)
N2—C3—S1	123.75 (10)	C8—C9—C11	119.15 (13)
O1—C4—N2	121.81 (13)	C9—C10—C11	118.98 (13)
O1—C4—C5	125.28 (12)	C9—C10—H10A	120.5
N2—C4—C5	112.91 (11)	C11—C10—H10A	120.5
C6—C5—C4	114.56 (11)	C10—C11—C6	120.88 (14)
C6—C5—H5A	108.6	C10—C11—H11A	119.6
C4—C5—H5A	108.6	C6—C11—H11A	119.6
C6—C5—H5B	108.6		
C3—S1—C1—C2	0.48 (13)	C4—C5—C6—C7	-116.45 (15)
S1—C1—C2—N1	-0.8 (2)	C4—C5—C6—C11	64.72 (19)
C3—N1—C2—C1	0.7 (2)	C11—C6—C7—C8	0.3 (2)
C2—N1—C3—N2	-179.52 (13)	C5—C6—C7—C8	-178.55 (13)
C2—N1—C3—S1	-0.32 (16)	C6—C7—C8—C9	-0.1 (2)
C4—N2—C3—N1	-172.65 (13)	C7—C8—C9—C10	0.0 (2)
C4—N2—C3—S1	8.23 (19)	C7—C8—C9—C11	179.30 (11)
C1—S1—C3—N1	-0.08 (12)	C8—C9—C10—C11	0.0 (2)
C1—S1—C3—N2	179.09 (12)	C11—C9—C10—C11	-179.33 (11)
C3—N2—C4—O1	-0.6 (2)	C9—C10—C11—C6	0.2 (2)
C3—N2—C4—C5	179.13 (13)	C7—C6—C11—C10	-0.3 (2)
O1—C4—C5—C6	-10.4 (2)	C5—C6—C11—C10	178.52 (13)
N2—C4—C5—C6	169.89 (13)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H1N2 \cdots N1 ⁱ	0.866 (18)	2.096 (18)	2.9606 (16)	176.2 (17)

Symmetry code: (i) $-x+1, -y+1, -z+2$.