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

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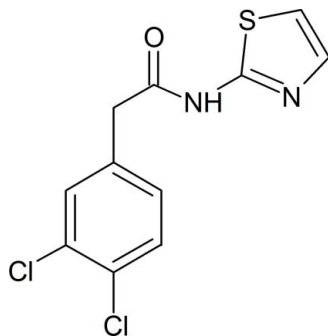
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In the title compound, $C_{11}H_8Cl_2N_2OS$, the mean plane of the dichlorophenyl ring is twisted by 61.8 (1)° from that of the thiazole ring. In the crystal, molecules are linked *via* pairs of N—H···N hydrogen bonds with an $R_2^2(8)$ graph-set motif, forming inversion dimers which stack along the *a*-axis direction.

Related literature

For the structural similarity of *N*-substituted 2-arylacetamides to the lateral chain of natural benzylpenicillin, see: Mijin & Marinkovic (2006); Mijin *et al.* (2008). For the coordination abilities of amides, see: Wu *et al.* (2008, 2010). For related structures, see: Fun *et al.* (2012*a,b,c,d,e*); Butcher *et al.* (2013*a,b*). For standard bond lengths, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{11}H_8Cl_2N_2OS$	a = 4.6185 (3) Å
M_r = 287.15	b = 7.7741 (7) Å
Triclinic, $P\bar{1}$	c = 17.1188 (12) Å

α = 100.278 (7)°	Mo $K\alpha$ radiation
β = 94.250 (6)°	μ = 0.72 mm^{-1}
γ = 105.001 (7)°	T = 123 K
V = 579.47 (8) Å^3	0.55 × 0.19 × 0.12 mm
Z = 2	

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer	9162 measured reflections
Absorption correction: analytical (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)	5597 independent reflections
T_{min} = 0.776, T_{max} = 0.929	3860 reflections with $I > 2\sigma(I)$
	R_{int} = 0.151

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	154 parameters
$wR(F^2)$ = 0.118	H-atom parameters constrained
S = 1.01	$\Delta\rho_{max}$ = 0.64 e Å^{-3}
5597 reflections	$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^i	0.88	2.03	2.8946 (16)	169

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

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

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supplementary materials

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

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Comment

N-Substituted 2-arylacetamides are very interesting compounds because of their structural similarity to the lateral chain of natural benzylpenicillin (Mijin *et al.*, 2006, 2008). Amides are also used as ligands due to their excellent coordination abilities (Wu *et al.*, 2008, 2010). Crystal structures of some acetamide derivatives viz., (2,2-diphenyl-N-(1,3-thiazol-2-yl)acetamide, 2-(4-chlorophenyl)-N-(1,3-thiazol-2-yl)acetamide, 2-(naphthalen-1-yl)-N-(1,3-thiazol-2-yl)acetamide, N-(1,3-thiazol-2-yl)-2-(2,4,6-trimethyl phenyl)acetamide, 2-(2-fluorophenyl)-N-(1,3-thiazol-2-yl)acetamide (Fun *et al.*, 2012*a,b,c,d,e*), 2-(2,6-dichlorophenyl)-N-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro- 1H-pyrazol-4-yl)acetamide, 2-(2,4-Dichlorophenyl)-N-(1,5-dimethyl-3-oxo- 2-phenyl-2,3-dihydro-1H-pyrazol-4-yl)acetamide (Butcher *et al.*, 2013*a,b*) have been reported. In view of the importance of amides, we report herein the crystal structure of the title compound, $C_{11}H_8Cl_2N_2OS$, (I).

In (I), the mean plane of the dichlorophenyl ring is twisted by 61.8 (1)° from that of the thiazol ring (Fig. 1). Bond lengths are in normal ranges (Allen *et al.*, 1987). In the crystal, the molecules are linked via pairs of N—H···N hydrogen bonds in an $R_2^2(8)$ graph-set motif forming inversion dimers which stack along the *a* axis (Fig. 2).

Experimental

3,4-Dichlorophenylacetic acid (0.240 g, 1 mmol) and 2-aminothiazole (0.1 g, 1 mmol), 1-ethyl-3-(3-dimethylamino-propyl)-carbodiimide hydrochloride (1.0 g, 0.01 mol) and 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, which was extracted thrice with dichloromethane (Fig. 3). The organic layer was washed with saturated $NaHCO_3$ solution and brine solution, dried and concentrated under reduced pressure to give the title compound (I). Single crystals were grown from methanol and acetone mixture (1:1) by the slow evaporation method (M.P.: 459–461K).

Refinement

All of the H atoms were placed in their calculated positions and then refined using the riding model with Atom—H lengths of 0.95Å (CH), 0.99Å (CH₂) or 0.88Å (NH). Isotropic displacement parameters for these atoms were set to 1.18-1.22 (CH, CH₂) times U_{eq} of the parent atom.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (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); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).
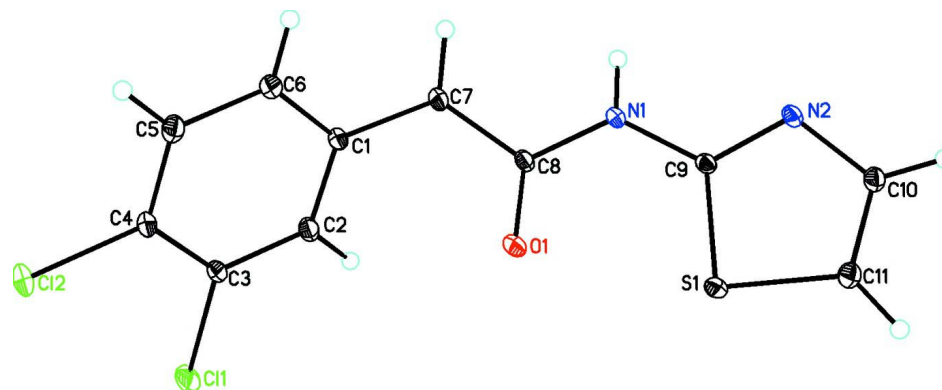
Figure 1

Molecular structure of the title compound showing the atom labeling scheme and 30% probability displacement ellipsoids.
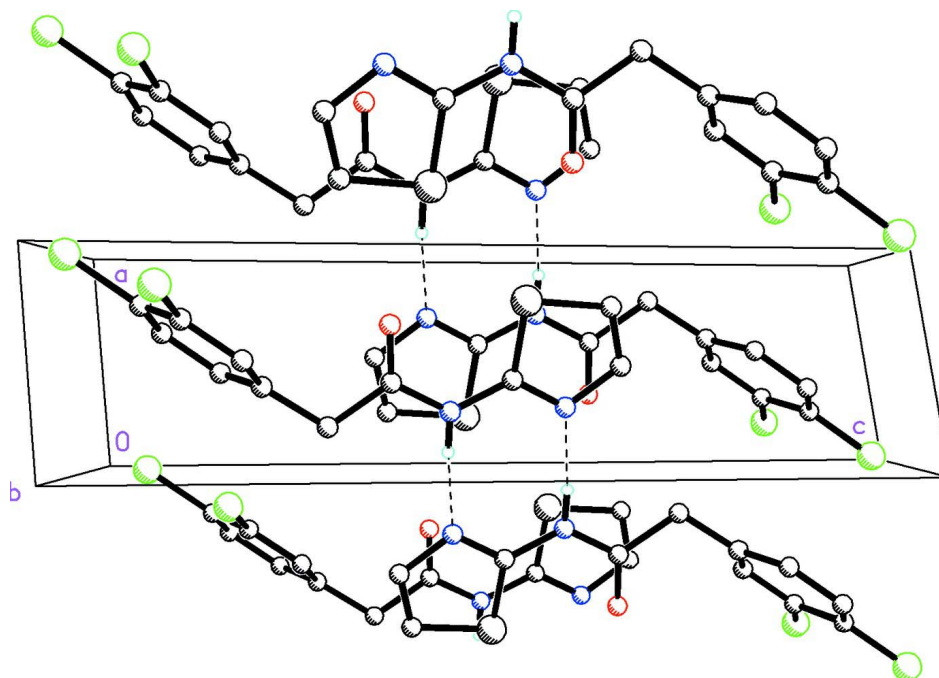
Figure 2

Packing diagram of the title compound viewed along the *b* axis. Dashed lines indicate N—H···N intermolecular hydrogen bonds in an $R_2^2(8)$ graph-set motif forming inversion dimers which stack along the *a* axis. H atoms not involved in hydrogen bonding have been removed for clarity.
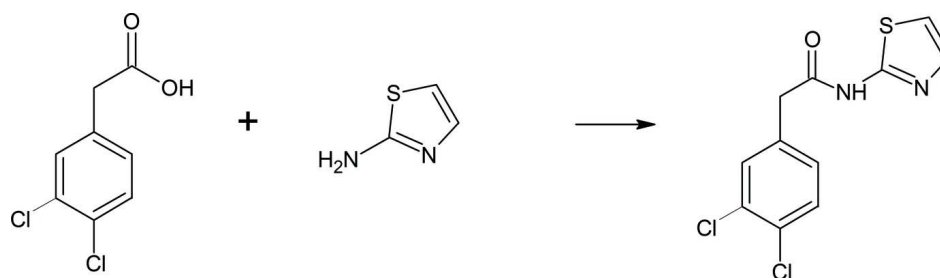
Figure 3

The preparation of the title compound.

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

Crystal data

$C_{11}H_8Cl_2N_2OS$	$Z = 2$
$M_r = 287.15$	$F(000) = 292$
Triclinic, $P\overline{1}$	$D_x = 1.646$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 4.6185$ (3) Å	Cell parameters from 4182 reflections
$b = 7.7741$ (7) Å	$\theta = 3.3–37.5°$
$c = 17.1188$ (12) Å	$\mu = 0.72$ mm^{-1}
$\alpha = 100.278$ (7)°	$T = 123$ K
$\beta = 94.250$ (6)°	Prism, colorless
$\gamma = 105.001$ (7)°	$0.55 \times 0.19 \times 0.12$ mm
$V = 579.47$ (8) Å^3	

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer	$T_{min} = 0.776$, $T_{max} = 0.929$
Radiation source: Enhance (Mo) X-ray Source	9162 measured reflections
Graphite monochromator	5597 independent reflections
Detector resolution: 10.5081 pixels mm^{-1}	3860 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.151$
Absorption correction: analytical	$\theta_{max} = 37.6°$, $\theta_{min} = 3.3°$
(*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)	$h = -7 \rightarrow 7$
	$k = -12 \rightarrow 13$
	$l = -25 \rightarrow 28$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	
$wR(F^2) = 0.118$	H-atom parameters constrained
$S = 1.01$	$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2]$
5597 reflections	where $P = (F_o^2 + 2F_c^2)/3$
154 parameters	$(\Delta/\sigma)_{max} < 0.001$
0 restraints	$\Delta\rho_{max} = 0.64$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.44$ 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.

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.81248 (12)	1.09847 (6)	0.14647 (3)	0.03477 (13)
Cl2	0.96349 (10)	0.77677 (6)	0.02962 (2)	0.02982 (11)
S1	0.76516 (8)	0.85914 (5)	0.54589 (2)	0.01625 (8)
O1	0.6604 (2)	0.80798 (16)	0.38585 (6)	0.0197 (2)
N1	0.2761 (3)	0.64533 (17)	0.44080 (7)	0.0139 (2)
H1A	0.0927	0.5703	0.4317	0.017*
N2	0.2939 (3)	0.63193 (17)	0.57692 (7)	0.0150 (2)
C1	0.3891 (3)	0.6615 (2)	0.22801 (8)	0.0152 (2)
C2	0.5038 (3)	0.8399 (2)	0.21780 (8)	0.0182 (3)
H2A	0.4619	0.9377	0.2526	0.022*
C3	0.6792 (3)	0.8744 (2)	0.15679 (8)	0.0193 (3)
C4	0.7428 (3)	0.7331 (2)	0.10493 (8)	0.0189 (3)
C5	0.6263 (4)	0.5551 (2)	0.11406 (9)	0.0204 (3)
H5A	0.6675	0.4575	0.0790	0.024*
C6	0.4486 (3)	0.5206 (2)	0.17508 (8)	0.0185 (3)
H6A	0.3667	0.3986	0.1805	0.022*
C7	0.2098 (3)	0.6289 (2)	0.29693 (8)	0.0172 (3)
H7A	0.1172	0.4966	0.2912	0.021*
H7B	0.0447	0.6883	0.2951	0.021*
C8	0.4065 (3)	0.70281 (19)	0.37754 (8)	0.0143 (2)
C9	0.4195 (3)	0.69989 (19)	0.51855 (8)	0.0129 (2)
C10	0.4805 (3)	0.7104 (2)	0.64816 (8)	0.0171 (3)
H10A	0.4282	0.6798	0.6976	0.020*
C11	0.7421 (3)	0.8335 (2)	0.64325 (8)	0.0181 (3)
H11B	0.8918	0.8969	0.6873	0.022*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0566 (3)	0.01772 (18)	0.0251 (2)	−0.00311 (18)	0.01475 (18)	0.00759 (15)
Cl2	0.0326 (2)	0.0360 (2)	0.01865 (17)	0.00221 (18)	0.01304 (15)	0.00638 (15)
S1	0.01359 (15)	0.01624 (16)	0.01658 (15)	−0.00031 (12)	0.00308 (11)	0.00343 (12)
O1	0.0146 (5)	0.0231 (5)	0.0182 (5)	−0.0024 (4)	0.0032 (4)	0.0067 (4)
N1	0.0127 (5)	0.0152 (5)	0.0132 (5)	0.0014 (4)	0.0032 (4)	0.0044 (4)
N2	0.0146 (5)	0.0162 (5)	0.0143 (5)	0.0024 (4)	0.0044 (4)	0.0048 (4)
C1	0.0147 (6)	0.0172 (6)	0.0129 (5)	0.0017 (5)	0.0015 (4)	0.0047 (5)
C2	0.0230 (7)	0.0170 (6)	0.0151 (6)	0.0047 (5)	0.0059 (5)	0.0044 (5)

C3	0.0245 (7)	0.0157 (6)	0.0146 (6)	−0.0014 (5)	0.0039 (5)	0.0047 (5)
C4	0.0198 (7)	0.0228 (7)	0.0121 (5)	0.0016 (5)	0.0038 (5)	0.0041 (5)
C5	0.0251 (7)	0.0205 (7)	0.0153 (6)	0.0067 (6)	0.0049 (5)	0.0017 (5)
C6	0.0218 (7)	0.0165 (6)	0.0159 (6)	0.0024 (5)	0.0027 (5)	0.0039 (5)
C7	0.0148 (6)	0.0210 (7)	0.0148 (6)	0.0016 (5)	0.0037 (4)	0.0055 (5)
C8	0.0143 (6)	0.0152 (6)	0.0138 (5)	0.0029 (5)	0.0044 (4)	0.0047 (4)
C9	0.0120 (5)	0.0125 (5)	0.0145 (5)	0.0030 (4)	0.0040 (4)	0.0032 (4)
C10	0.0190 (6)	0.0182 (6)	0.0140 (5)	0.0049 (5)	0.0030 (5)	0.0032 (5)
C11	0.0193 (7)	0.0190 (7)	0.0150 (6)	0.0049 (5)	0.0023 (5)	0.0018 (5)

Geometric parameters (Å, º)

Cl1—C3	1.7362 (15)	C2—C3	1.3897 (18)
Cl2—C4	1.7286 (13)	C2—H2A	0.9500
S1—C9	1.7203 (14)	C3—C4	1.392 (2)
S1—C11	1.7212 (14)	C4—C5	1.390 (2)
O1—C8	1.2251 (16)	C5—C6	1.397 (2)
N1—C8	1.3658 (15)	C5—H5A	0.9500
N1—C9	1.3829 (18)	C6—H6A	0.9500
N1—H1A	0.8800	C7—C8	1.523 (2)
N2—C9	1.3153 (16)	C7—H7A	0.9900
N2—C10	1.382 (2)	C7—H7B	0.9900
C1—C6	1.391 (2)	C10—C11	1.352 (2)
C1—C2	1.398 (2)	C10—H10A	0.9500
C1—C7	1.5116 (17)	C11—H11B	0.9500
C9—S1—C11	88.74 (7)	C1—C6—H6A	119.4
C8—N1—C9	122.90 (11)	C5—C6—H6A	119.4
C8—N1—H1A	118.5	C1—C7—C8	111.93 (11)
C9—N1—H1A	118.5	C1—C7—H7A	109.2
C9—N2—C10	109.29 (12)	C8—C7—H7A	109.2
C6—C1—C2	118.72 (12)	C1—C7—H7B	109.2
C6—C1—C7	122.39 (13)	C8—C7—H7B	109.2
C2—C1—C7	118.88 (14)	H7A—C7—H7B	107.9
C3—C2—C1	120.10 (15)	O1—C8—N1	122.09 (13)
C3—C2—H2A	119.9	O1—C8—C7	123.10 (11)
C1—C2—H2A	119.9	N1—C8—C7	114.80 (11)
C2—C3—C4	120.91 (13)	N2—C9—N1	120.89 (12)
C2—C3—Cl1	118.03 (13)	N2—C9—S1	115.75 (10)
C4—C3—Cl1	121.05 (10)	N1—C9—S1	123.35 (9)
C5—C4—C3	119.33 (12)	C11—C10—N2	115.89 (12)
C5—C4—Cl2	119.89 (13)	C11—C10—H10A	122.1
C3—C4—Cl2	120.78 (11)	N2—C10—H10A	122.1
C4—C5—C6	119.68 (15)	C10—C11—S1	110.32 (11)
C4—C5—H5A	120.2	C10—C11—H11B	124.8
C6—C5—H5A	120.2	S1—C11—H11B	124.8
C1—C6—C5	121.23 (13)		
C6—C1—C2—C3	−1.4 (2)	C2—C1—C7—C8	−67.93 (18)
C7—C1—C2—C3	177.54 (14)	C9—N1—C8—O1	−0.4 (2)

C1—C2—C3—C4	0.2 (2)	C9—N1—C8—C7	−179.28 (13)
C1—C2—C3—Cl1	179.42 (11)	C1—C7—C8—O1	14.2 (2)
C2—C3—C4—C5	0.6 (2)	C1—C7—C8—N1	−166.90 (13)
Cl1—C3—C4—C5	−178.58 (12)	C10—N2—C9—N1	−179.19 (13)
C2—C3—C4—Cl2	−179.47 (12)	C10—N2—C9—S1	0.25 (17)
Cl1—C3—C4—Cl2	1.3 (2)	C8—N1—C9—N2	−175.45 (14)
C3—C4—C5—C6	−0.2 (2)	C8—N1—C9—S1	5.2 (2)
Cl2—C4—C5—C6	179.89 (12)	C11—S1—C9—N2	0.07 (12)
C2—C1—C6—C5	1.8 (2)	C11—S1—C9—N1	179.50 (13)
C7—C1—C6—C5	−177.07 (14)	C9—N2—C10—C11	−0.6 (2)
C4—C5—C6—C1	−1.0 (2)	N2—C10—C11—S1	0.62 (19)
C6—C1—C7—C8	110.97 (16)	C9—S1—C11—C10	−0.38 (12)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2[i]	0.88	2.03	2.8946 (16)	169

Symmetry code: (i) −x, −y+1, −z+1.