Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Prakash S. Nayak,[a] B. Narayana,[a] H. S. Yathirajan,[b] Jerry P. Jasinski[c]* and Ray J. Butcher[d]

[a]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, [b]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, [c]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, and [d]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA
Correspondence e-mail: jjasinski@keene.edu

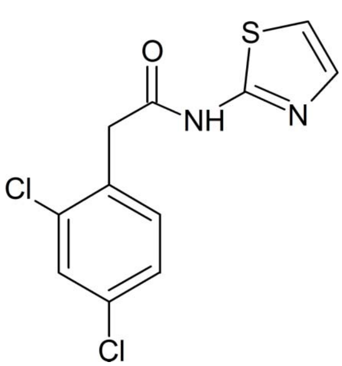
Received 24 March 2013; accepted 27 March 2013

Key indicators: single-crystal X-ray study; *T* = 123 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.046; *wR* factor = 0.112; data-to-parameter ratio = 39.0.

In the title compound, $C_{11}H_8Cl_2N_2OS$, the mean plane of the dichlorophenyl ring is twisted by 72.4 (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 and weak C—H···O interactions, forming inversion dimers which stack along the *c*-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

$C_{11}H_8Cl_2N_2OS$
M_r = 287.15
Triclinic, $P\bar{1}$
a = 5.3262 (2) Å
b = 10.5083 (4) Å
c = 10.8096 (4) Å
α = 83.900 (3)°
β = 86.301 (3)°
γ = 87.279 (4)°
V = 599.83 (4) Å^3
Z = 2
Mo *K*α radiation
μ = 0.70 mm^{-1}
T = 123 K
0.35 × 0.25 × 0.12 mm

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)
T_{min} = 0.873, T_{max} = 1.000
10769 measured reflections
6006 independent reflections
4329 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.112
S = 1.07
6006 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.65 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O1^{i}	0.95	2.50	3.3253 (15)	145
N1—H1*A*···N2^{ii}	0.88	2.04	2.9052 (15)	168

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

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*.

BN thanks the UGC for financial assistance through the BSR one-time grant for the purchase of chemicals. PSN thanks Mangalore University for research facilities and DST–PURSE financial assistance. RJB acknowledges the NSF–MRI program (grant No. CHE-0619278) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5302).

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.
Butcher, R. J., Mahan, A., Nayak, P. S., Narayana, B. & Yathirajan, H. S. (2013*a*). *Acta Cryst.* E**69**, o46–o47.
Butcher, R. J., Mahan, A., Nayak, P. S., Narayana, B. & Yathirajan, H. S. (2013*b*). *Acta Cryst.* E**69**, o39.
Fun, H.-K., Ooi, C. W., Nayak, P. S., Narayana, B. & Sarojini, B. K. (2012*a*). *Acta Cryst.* E**68**, o1312–o1313.
Fun, H.-K., Quah, C. K., Nayak, P. S., Narayana, B. & Sarojini, B. K. (2012*b*). *Acta Cryst.* E**68**, o2679.
Fun, H.-K., Quah, C. K., Nayak, P. S., Narayana, B. & Sarojini, B. K. (2012*c*). *Acta Cryst.* E**68**, o2464.
Fun, H.-K., Quah, C. K., Nayak, P. S., Narayana, B. & Sarojini, B. K. (2012*d*). *Acta Cryst.* E**68**, o2461.
Fun, H.-K., Quah, C. K., Nayak, P. S., Narayana, B. & Sarojini, B. K. (2012*e*). *Acta Cryst.* E**68**, o2565.
Mijin, D. & Marinkovic, A. (2006). *Synth. Commun.* **36**, 193–198.

Mijin, D. Z., Prascevic, M. & Petrovic, S. D. (2008). *J. Serb. Chem. Soc.* **73**, 945–950.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wu, W.-N., Cheng, F.-X., Yan, L. & Tang, N. (2008). *J. Coord. Chem.* **61**, 2207–2215.
Wu, W.-N., Wang, Y., Zhang, A.-Y., Zhao, R.-Q. & Wang, Q.-F. (2010). *Acta Cryst.* E**66**, m288.

supplementary materials

Acta Cryst. (2013). E**69**, o656–o657 [doi:10.1107/S1600536813008532]

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

Prakash S. Nayak, B. Narayana, H. S. Yathirajan, Jerry P. Jasinski and Ray J. Butcher

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 72.4 (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 a R2,2(8)graph-set motif and weak C—H···O intermolecular interactions forming inversion dimers which stack along the *c* axis (Fig. 2).

Experimental

2,4-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.: 493–495 K).

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 (CH_2) or 0.88Å (NH). Isotropic displacement parameters for these atoms were set to 1.18-1.23 (CH, CH_2, NH) 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).

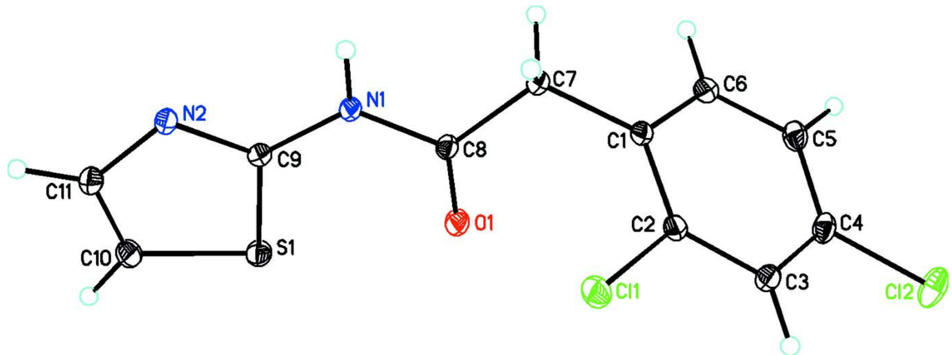


Figure 1

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

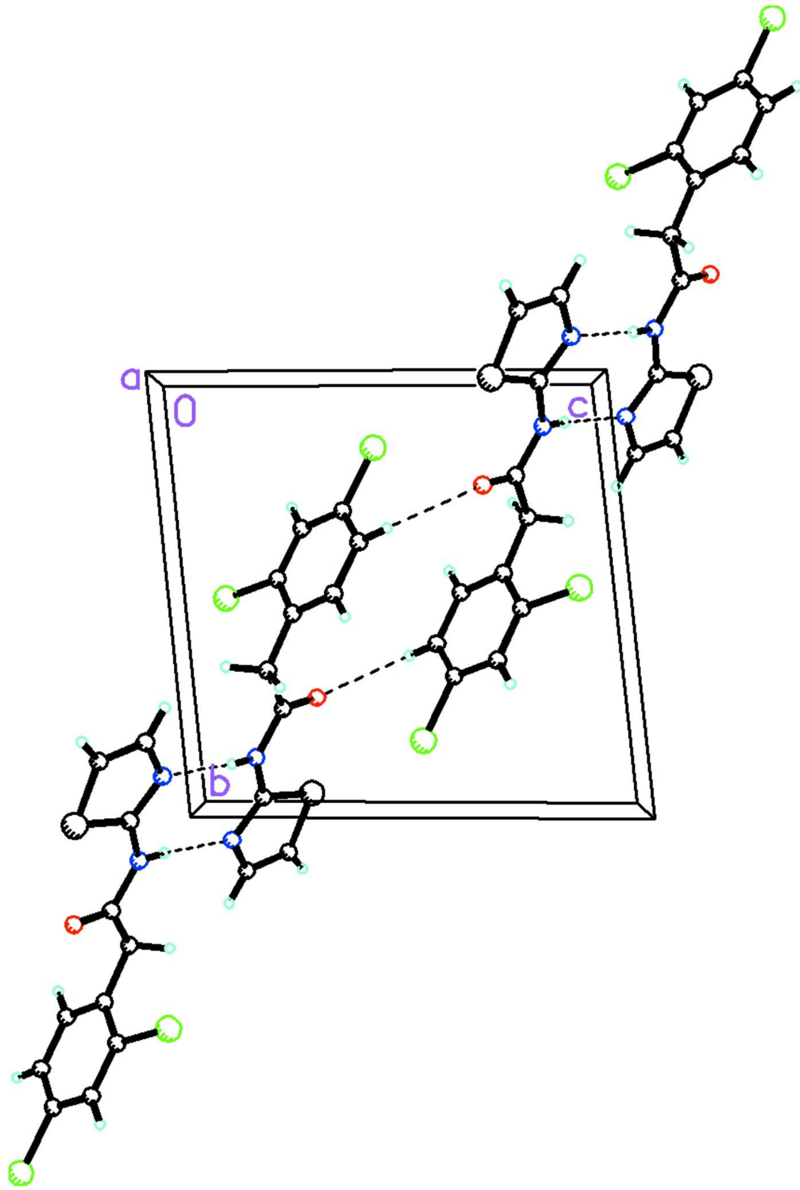


Figure 2

Packing diagram of the title compound viewed along the *a* axis. Dashed lines indicate N—H···N intermolecular hydrogen bonds in an R2,2(8) graph-set motif and weak C—H···O intemolecular interactions forming inversion dimers which stack along the *c* axis.

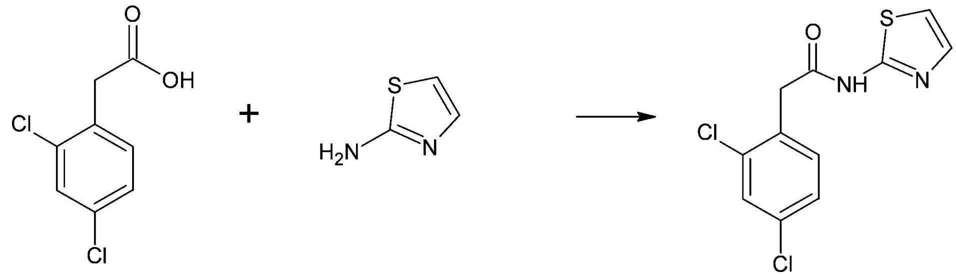


Figure 3

Reaction scheme.

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

Crystal data

$C_{11}H_8Cl_2N_2OS$
$M_r = 287.15$
Triclinic, $P\bar{1}$
Hall symbol: -P 1
$a = 5.3262$ (2) Å
$b = 10.5083$ (4) Å
$c = 10.8096$ (4) Å
$\alpha = 83.900$ (3)°
$\beta = 86.301$ (3)°
$\gamma = 87.279$ (4)°
$V = 599.83$ (4) Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.590$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4451 reflections
$\theta = 3.8$–37.4°
$\mu = 0.70$ mm^{-1}
$T = 123$ K
Prism, colorless
0.35 × 0.25 × 0.12 mm

Data collection

Agilent Xcalibur (Ruby, Gemini) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO* and *CrysAlis RED*; Agilent, 2012)
$T_{min} = 0.873$, $T_{max} = 1.000$
10769 measured reflections
6006 independent reflections
4329 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 37.5°$, $\theta_{min} = 3.8°$
$h = -8 \rightarrow 8$
$k = -17 \rightarrow 17$
$l = -18 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.112$
$S = 1.07$
6006 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.65$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ 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	0.79895 (7)	0.48741 (3)	0.89686 (3)	0.02461 (8)
Cl2	0.55890 (9)	0.83944 (4)	0.52220 (4)	0.03766 (11)
S1	1.04354 (7)	0.01947 (3)	0.74851 (3)	0.02147 (8)
O1	0.79783 (18)	0.25361 (10)	0.71093 (9)	0.0215 (2)
N1	0.6082 (2)	0.11595 (11)	0.86027 (10)	0.0186 (2)
H1A	0.4759	0.1039	0.9126	0.022*
N2	0.7798 (2)	−0.08345 (11)	0.93865 (10)	0.0192 (2)
C1	0.4325 (2)	0.45348 (12)	0.74111 (11)	0.0171 (2)
C2	0.6152 (2)	0.53376 (12)	0.77059 (11)	0.0174 (2)
C3	0.6586 (3)	0.65234 (13)	0.70372 (12)	0.0206 (3)
H3A	0.7876	0.7045	0.7242	0.025*
C4	0.5064 (3)	0.69139 (13)	0.60622 (12)	0.0217 (3)
C5	0.3151 (3)	0.61785 (14)	0.57565 (12)	0.0222 (3)
H5A	0.2096	0.6478	0.5102	0.027*
C6	0.2813 (3)	0.49878 (13)	0.64318 (12)	0.0199 (2)
H6A	0.1522	0.4469	0.6223	0.024*
C7	0.3992 (2)	0.32251 (13)	0.80963 (12)	0.0191 (2)
H7A	0.2454	0.2866	0.7829	0.023*
H7B	0.3745	0.3306	0.9001	0.023*
C8	0.6212 (2)	0.23051 (12)	0.78742 (11)	0.0166 (2)
C9	0.7900 (2)	0.01826 (12)	0.85656 (11)	0.0170 (2)
C10	1.1457 (3)	−0.12670 (14)	0.81987 (13)	0.0237 (3)
H10A	1.2938	−0.1734	0.7945	0.028*
C11	0.9844 (3)	−0.16548 (13)	0.91734 (13)	0.0216 (3)
H11A	1.0106	−0.2441	0.9678	0.026*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02368 (17)	0.02474 (17)	0.02565 (16)	0.00123 (13)	−0.00852 (12)	−0.00047 (12)
Cl2	0.0557 (3)	0.02104 (18)	0.03370 (19)	−0.00763 (18)	−0.00098 (18)	0.01074 (15)
S1	0.02054 (16)	0.02107 (16)	0.02141 (15)	0.00019 (13)	0.00441 (12)	0.00039 (12)
O1	0.0212 (5)	0.0200 (5)	0.0211 (4)	0.0003 (4)	0.0032 (4)	0.0045 (4)
N1	0.0157 (5)	0.0160 (5)	0.0220 (5)	0.0006 (4)	0.0036 (4)	0.0045 (4)
N2	0.0183 (5)	0.0156 (5)	0.0224 (5)	0.0004 (4)	0.0010 (4)	0.0023 (4)
C1	0.0153 (5)	0.0155 (5)	0.0193 (5)	0.0004 (4)	0.0007 (4)	0.0018 (4)
C2	0.0164 (5)	0.0166 (5)	0.0189 (5)	0.0014 (4)	−0.0016 (4)	−0.0002 (4)

C3	0.0206 (6)	0.0169 (6)	0.0238 (6)	−0.0017 (5)	0.0011 (5)	−0.0008 (5)
C4	0.0277 (7)	0.0154 (6)	0.0201 (5)	0.0007 (5)	0.0041 (5)	0.0024 (4)
C5	0.0255 (7)	0.0208 (6)	0.0191 (5)	0.0030 (5)	−0.0022 (5)	0.0023 (5)
C6	0.0180 (6)	0.0197 (6)	0.0218 (5)	−0.0002 (5)	−0.0026 (4)	0.0002 (5)
C7	0.0169 (6)	0.0158 (5)	0.0231 (6)	−0.0005 (5)	0.0009 (4)	0.0036 (4)
C8	0.0178 (6)	0.0148 (5)	0.0170 (5)	−0.0013 (4)	−0.0026 (4)	0.0012 (4)
C9	0.0155 (5)	0.0165 (5)	0.0185 (5)	−0.0015 (4)	0.0003 (4)	0.0002 (4)
C10	0.0206 (6)	0.0205 (6)	0.0291 (6)	0.0041 (5)	0.0020 (5)	−0.0027 (5)
C11	0.0218 (6)	0.0166 (6)	0.0256 (6)	0.0008 (5)	−0.0007 (5)	0.0002 (5)

Geometric parameters (Å, º)

Cl1—C2	1.7445 (12)	C2—C3	1.3939 (17)
Cl2—C4	1.7412 (13)	C3—C4	1.3866 (19)
S1—C10	1.7243 (14)	C3—H3A	0.9500
S1—C9	1.7272 (13)	C4—C5	1.384 (2)
O1—C8	1.2274 (15)	C5—C6	1.3933 (18)
N1—C8	1.3687 (15)	C5—H5A	0.9500
N1—C9	1.3794 (17)	C6—H6A	0.9500
N1—H1A	0.8800	C7—C8	1.5148 (19)
N2—C9	1.3155 (15)	C7—H7A	0.9900
N2—C11	1.3799 (18)	C7—H7B	0.9900
C1—C2	1.3912 (19)	C10—C11	1.3563 (19)
C1—C6	1.4009 (17)	C10—H10A	0.9500
C1—C7	1.5046 (17)	C11—H11A	0.9500
C10—S1—C9	88.73 (6)	C5—C6—H6A	119.1
C8—N1—C9	123.92 (11)	C1—C6—H6A	119.1
C8—N1—H1A	118.0	C1—C7—C8	113.05 (10)
C9—N1—H1A	118.0	C1—C7—H7A	109.0
C9—N2—C11	109.73 (11)	C8—C7—H7A	109.0
C2—C1—C6	117.23 (11)	C1—C7—H7B	109.0
C2—C1—C7	121.89 (11)	C8—C7—H7B	109.0
C6—C1—C7	120.88 (12)	H7A—C7—H7B	107.8
C1—C2—C3	122.75 (12)	O1—C8—N1	121.81 (12)
C1—C2—Cl1	119.76 (9)	O1—C8—C7	124.30 (11)
C3—C2—Cl1	117.49 (10)	N1—C8—C7	113.88 (10)
C4—C3—C2	117.54 (13)	N2—C9—N1	120.68 (11)
C4—C3—H3A	121.2	N2—C9—S1	115.46 (10)
C2—C3—H3A	121.2	N1—C9—S1	123.86 (9)
C5—C4—C3	122.29 (12)	C11—C10—S1	110.33 (11)
C5—C4—Cl2	119.82 (10)	C11—C10—H10A	124.8
C3—C4—Cl2	117.89 (11)	S1—C10—H10A	124.8
C4—C5—C6	118.37 (12)	C10—C11—N2	115.75 (12)
C4—C5—H5A	120.8	C10—C11—H11A	122.1
C6—C5—H5A	120.8	N2—C11—H11A	122.1
C5—C6—C1	121.75 (13)		
C6—C1—C2—C3	−2.9 (2)	C6—C1—C7—C8	112.46 (14)
C7—C1—C2—C3	176.27 (13)	C9—N1—C8—O1	1.1 (2)

C6—C1—C2—Cl1	176.52 (10)	C9—N1—C8—C7	−179.52 (12)
C7—C1—C2—Cl1	−4.33 (19)	C1—C7—C8—O1	−8.1 (2)
C1—C2—C3—C4	1.7 (2)	C1—C7—C8—N1	172.59 (11)
Cl1—C2—C3—C4	−177.67 (11)	C11—N2—C9—N1	−178.62 (12)
C2—C3—C4—C5	0.9 (2)	C11—N2—C9—S1	0.80 (15)
C2—C3—C4—Cl2	179.95 (11)	C8—N1—C9—N2	172.32 (12)
C3—C4—C5—C6	−2.1 (2)	C8—N1—C9—S1	−7.0 (2)
Cl2—C4—C5—C6	178.79 (11)	C10—S1—C9—N2	−0.69 (11)
C4—C5—C6—C1	0.9 (2)	C10—S1—C9—N1	178.71 (13)
C2—C1—C6—C5	1.5 (2)	C9—S1—C10—C11	0.35 (12)
C7—C1—C6—C5	−177.65 (13)	S1—C10—C11—N2	0.02 (18)
C2—C1—C7—C8	−66.66 (17)	C9—N2—C11—C10	−0.52 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*A*···O1^{i}	0.95	2.50	3.3253 (15)	145
N1—H1*A*···N2^{ii}	0.88	2.04	2.9052 (15)	168

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