

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

5-Bromo-4-(3,4-dimethoxyphenyl)thiazol-2-amine

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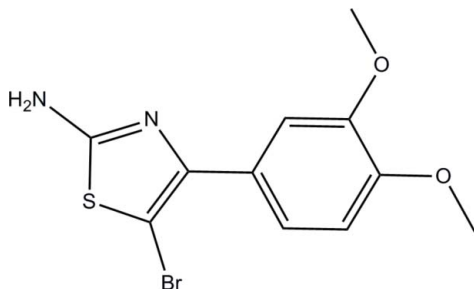
Received 28 April 2012; accepted 30 April 2012

 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.024; wR factor = 0.071; data-to-parameter ratio = 12.9.

In the title compound, $\text{C}_{11}\text{H}_{11}\text{BrN}_2\text{O}_2\text{S}$, the thiazole ring makes a dihedral angle of $53.16(11)^\circ$ with the adjacent benzene ring. The two methoxy groups are slightly twisted from the attached benzene ring with $\text{C}-\text{O}-\text{C}-\text{C}$ torsion angles of $-9.2(3)$ and $-5.5(3)^\circ$. In the crystal, molecules are linked by a pair of $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds into an inversion dimer with an $R_2^2(8)$ ring motif. The dimers are further connected by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into a tape along $[\bar{1}10]$.

Related literature

For applications of the thiazole ring system, see: Hargrave *et al.* (1983); Patt *et al.* (1992); Haviv *et al.* (1988); Jaen *et al.* (1990); Tsuji & Ishikawa (1994); Bell *et al.* (1995). For applications of aminothiazoles, see: Fink *et al.* (1999); Van Muijlwijk-Koezen *et al.* (2001); Metzger (1984). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For the preparation, see: Das *et al.* (2006). For stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



‡ Thomson Reuters ResearcherID: A-3561-2009.

Experimental

Crystal data

$\text{C}_{11}\text{H}_{11}\text{BrN}_2\text{O}_2\text{S}$
 $M_r = 315.19$
 Triclinic, $P\bar{1}$
 $a = 7.4873(2)$ Å
 $b = 8.0359(2)$ Å
 $c = 10.6428(3)$ Å
 $\alpha = 86.571(2)^\circ$
 $\beta = 77.633(2)^\circ$
 $\gamma = 85.330(2)^\circ$
 $V = 622.82(3)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 3.46$ mm⁻¹
 $T = 100$ K
 $0.45 \times 0.20 \times 0.09$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.305$, $T_{\max} = 0.737$
 10861 measured reflections
 2121 independent reflections
 1888 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.071$
 $S = 1.12$
 2121 reflections
 164 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 1.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.73$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2N2}\cdots\text{O1}^i$	0.78 (3)	2.40 (3)	2.992 (3)	134 (3)
$\text{N2}-\text{H2N2}\cdots\text{O2}^i$	0.78 (3)	2.37 (3)	3.112 (3)	161 (3)
$\text{N2}-\text{H1N2}\cdots\text{N1}^{ii}$	0.81 (3)	2.20 (3)	2.998 (3)	168 (3)

 Symmetry codes: (i) $x - 1, y + 1, z$; (ii) $-x, -y + 1, -z + 1$.

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).

HAG thanks the Deanship of Scientific Research and Research Center, College of Pharmacy, King Saud University. HKF and TSC thank Universiti Sains Malaysia (USM) for the Research University Grant (1001/PFIZIK/811160). TSC also thanks the Malaysian Government and USM for the award of a research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5132).

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supporting information

Acta Cryst. (2012). E68, o1631–o1632 [doi:10.1107/S1600536812019320]

5-Bromo-4-(3,4-dimethoxyphenyl)thiazol-2-amine**Hazem A. Ghabbour, Tze Shyang Chia and Hoong-Kun Fun****S1. Comment**

The thiazole ring system is a useful structural motif found in numerous biologically active molecules. This structure has found applications in drug development for the treatment of allergies (Hargrave *et al.*, 1983), hypertension (Patt *et al.*, 1992), inflammation (Haviv *et al.*, 1988), schizophrenia (Jaen *et al.*, 1990), bacterial (Tsuji & Ishikawa, 1994) and HIV infections (Bell *et al.*, 1995). Aminothiazoles are known to be ligands of estrogen receptors (Fink *et al.*, 1999) as well as a novel class of adenosine receptor antagonists (Van Muijlwijk-Koezen *et al.*, 2001). Other analogues are used as fungicides, inhibiting *in vivo* growth of *Xanthomonas*, as an ingredient of herbicides or as schistosomicidal and anthelmintic drugs (Metzger, 1984).

In the title compound (Fig. 1), the thiazole ring (S1/N1/C7–C9) makes a dihedral angle of 53.16 (11)° with the adjacent benzene ring (C1–C6). The two methoxy groups (O1/C10 & O2/C11) are slightly twisted from the C1–C6 ring with torsion angles C10—O1—C3—C2 = -9.2 (3) and C11—O2—C4—C5 = -5.5 (3)°.

In the crystal packing (Fig. 2 & 3), the molecules are linked by intermolecular N2—H1N2···N1 hydrogen bonds into dimers with $R_2^2(8)$ ring motifs (Bernstein *et al.*, 1995). The dimers are further connected by intermolecular N2—H2N2···O1 and N2—H2N2···O2 hydrogen bonds (Table 1) into infinite tapes along $[\bar{1}10]$.

S2. Experimental

The title compound was prepared from the reaction of 4-(3,4-dimethoxyphenyl)thiazol-2-amine (236 mg, 1 mmol) with bromine (161 mg, 1.1 mmol) in glacial acetic acid and heated at 80 °C for 1.5 h. Single crystals of the title compound suitable for X-ray structure determination were recrystallized from ethanol by the slow evaporation of the solvent at room temperature after several days (Das *et al.*, 2006).

S3. Refinement

Atom H1N2 and H2N2 were located in a difference Fourier map and refined freely [N—H = 0.80 (3) and 0.78 (3) Å]. The remaining H atoms were positioned geometrically (C—H = 0.95 and 0.98 Å) and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl groups. Three outliers (-2 6 4), (5 -3 8) and (5 -2 9) were omitted.

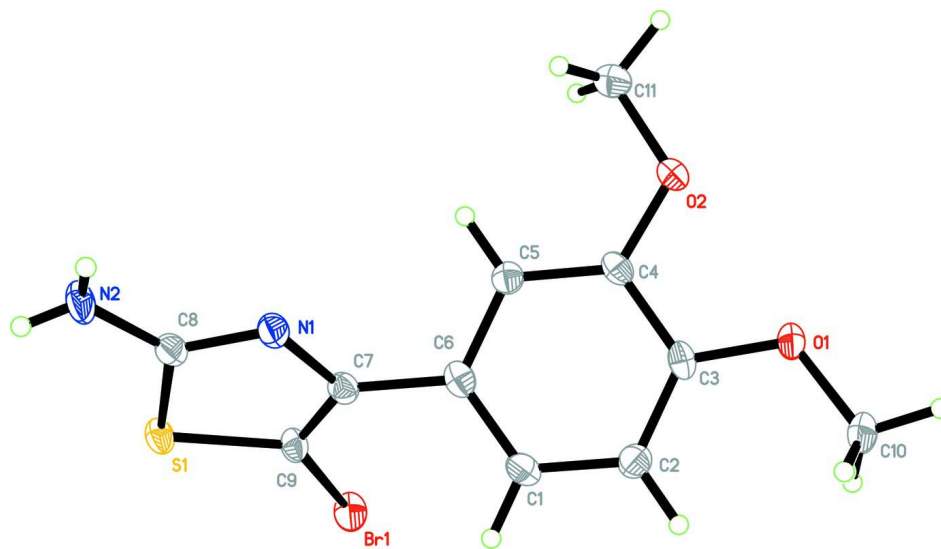
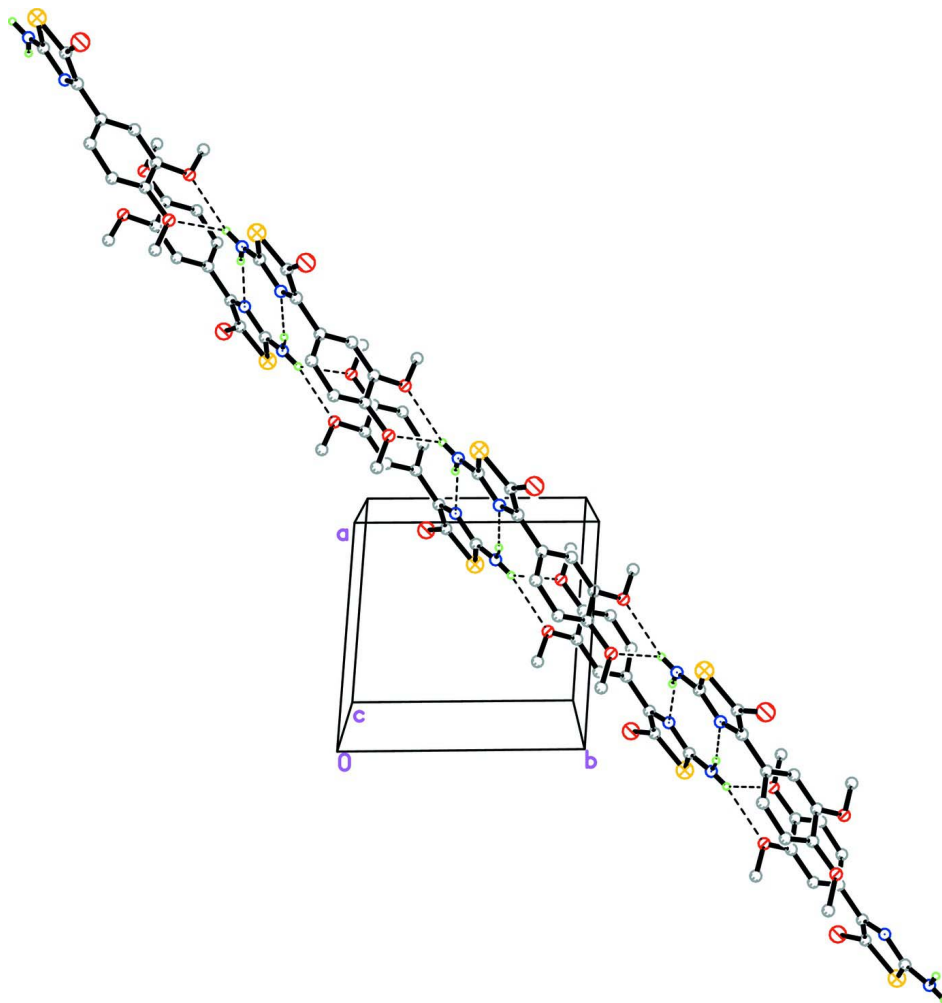
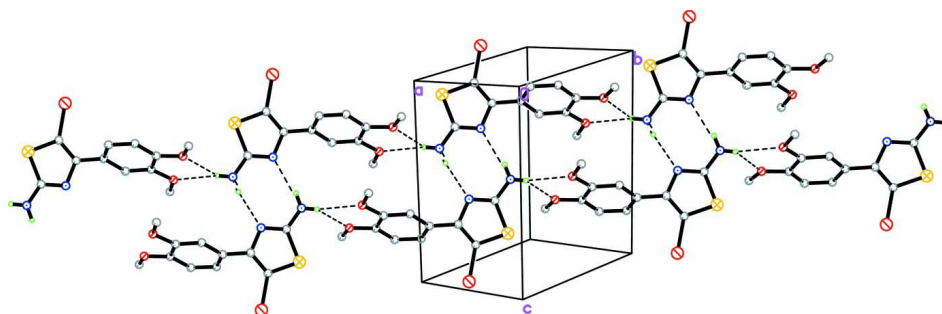


Figure 1

The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids.

**Figure 2**

The crystal packing of the title compound. The dashed lines represent the hydrogen bonds. For clarity sake, hydrogen atoms not involved in hydrogen bonding have been omitted.

**Figure 3**

The crystal packing of the title compound viewed along $[1\ 1\ 0]$.

5-Bromo-4-(3,4-dimethoxyphenyl)thiazol-2-amine

Crystal data

C₁₁H₁₁BrN₂O₂S
M_r = 315.19
 Triclinic, *P* $\bar{1}$
 Hall symbol: -P 1
a = 7.4873 (2) Å
b = 8.0359 (2) Å
c = 10.6428 (3) Å
 α = 86.571 (2)°
 β = 77.633 (2)°
 γ = 85.330 (2)°
V = 622.82 (3) Å³

Z = 2
F(000) = 316
D_x = 1.681 Mg m⁻³
 Mo *K* α radiation, λ = 0.71073 Å
 Cell parameters from 7495 reflections
 θ = 2.6–35.5°
 μ = 3.46 mm⁻¹
T = 100 K
 Plate, brown
 0.45 × 0.20 × 0.09 mm

Data collection

Bruker SMART APEXII CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2009)
T_{min} = 0.305, *T_{max}* = 0.737

10861 measured reflections
 2121 independent reflections
 1888 reflections with *I* > 2 σ (*I*)
R_{int} = 0.030
 θ_{\max} = 25.0°, θ_{\min} = 2.0°
h = -8→8
k = -9→9
l = -12→12

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2 σ (*F*²)] = 0.024
wR(*F*²) = 0.071
S = 1.12
 2121 reflections
 164 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
 $w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.1359P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 1.17 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.73 \text{ e \AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 > σ (*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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i> */ <i>U_{eq}</i>
Br1	-0.16418 (3)	0.27703 (3)	1.04837 (2)	0.02360 (12)

S1	-0.30599 (8)	0.51061 (8)	0.84026 (6)	0.01865 (17)
O1	0.6319 (2)	-0.1031 (2)	0.74403 (16)	0.0203 (4)
O2	0.4088 (2)	-0.1521 (2)	0.60126 (16)	0.0213 (4)
N1	-0.0368 (3)	0.4074 (3)	0.6600 (2)	0.0187 (5)
N2	-0.2487 (4)	0.5970 (3)	0.5852 (2)	0.0237 (5)
C1	0.2811 (3)	0.2379 (3)	0.8495 (2)	0.0195 (6)
H1A	0.2539	0.3246	0.9090	0.023*
C2	0.4415 (3)	0.1345 (3)	0.8433 (2)	0.0193 (6)
H2A	0.5241	0.1524	0.8968	0.023*
C3	0.4794 (3)	0.0060 (3)	0.7588 (2)	0.0168 (5)
C4	0.3576 (3)	-0.0193 (3)	0.6795 (2)	0.0162 (5)
C5	0.2018 (3)	0.0867 (3)	0.6835 (2)	0.0166 (5)
H5A	0.1218	0.0719	0.6274	0.020*
C6	0.1613 (3)	0.2164 (3)	0.7706 (2)	0.0169 (5)
C7	-0.0059 (3)	0.3294 (3)	0.7743 (2)	0.0167 (5)
C8	-0.1867 (3)	0.5072 (3)	0.6792 (2)	0.0180 (6)
C9	-0.1366 (3)	0.3686 (3)	0.8796 (2)	0.0174 (5)
C10	0.7724 (3)	-0.0657 (3)	0.8075 (2)	0.0233 (6)
H10A	0.8799	-0.1439	0.7821	0.035*
H10B	0.7274	-0.0762	0.9010	0.035*
H10C	0.8065	0.0488	0.7831	0.035*
C11	0.2828 (4)	-0.1928 (4)	0.5250 (3)	0.0260 (6)
H11A	0.3359	-0.2877	0.4723	0.039*
H11B	0.2589	-0.0961	0.4689	0.039*
H11C	0.1675	-0.2225	0.5819	0.039*
H2N2	-0.324 (4)	0.669 (4)	0.602 (3)	0.028 (9)*
H1N2	-0.182 (4)	0.606 (4)	0.515 (3)	0.033 (9)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02354 (18)	0.02661 (19)	0.01783 (16)	0.00291 (11)	-0.00083 (11)	0.00253 (12)
S1	0.0173 (3)	0.0182 (4)	0.0182 (3)	0.0037 (3)	-0.0003 (3)	-0.0012 (3)
O1	0.0161 (9)	0.0188 (10)	0.0266 (9)	0.0041 (7)	-0.0071 (7)	-0.0039 (8)
O2	0.0207 (9)	0.0200 (10)	0.0238 (9)	0.0045 (8)	-0.0060 (8)	-0.0086 (8)
N1	0.0190 (11)	0.0185 (12)	0.0178 (10)	0.0028 (9)	-0.0033 (9)	-0.0020 (9)
N2	0.0244 (14)	0.0241 (15)	0.0187 (12)	0.0125 (11)	-0.0005 (10)	-0.0019 (11)
C1	0.0203 (14)	0.0167 (14)	0.0208 (12)	-0.0003 (11)	-0.0019 (10)	-0.0048 (11)
C2	0.0187 (13)	0.0199 (15)	0.0202 (13)	-0.0014 (11)	-0.0057 (10)	-0.0008 (11)
C3	0.0142 (13)	0.0132 (13)	0.0209 (12)	0.0013 (10)	-0.0003 (10)	0.0021 (10)
C4	0.0178 (13)	0.0135 (14)	0.0152 (11)	-0.0001 (10)	0.0006 (10)	-0.0003 (10)
C5	0.0172 (13)	0.0173 (14)	0.0149 (11)	-0.0009 (10)	-0.0029 (10)	0.0007 (10)
C6	0.0188 (13)	0.0137 (13)	0.0162 (11)	-0.0003 (10)	-0.0001 (10)	0.0030 (10)
C7	0.0175 (13)	0.0136 (13)	0.0190 (12)	-0.0011 (10)	-0.0039 (10)	-0.0008 (11)
C8	0.0193 (14)	0.0170 (14)	0.0171 (12)	0.0013 (11)	-0.0030 (10)	-0.0024 (11)
C9	0.0192 (13)	0.0136 (14)	0.0183 (12)	0.0021 (10)	-0.0035 (10)	0.0017 (11)
C10	0.0140 (13)	0.0262 (16)	0.0296 (14)	0.0026 (11)	-0.0050 (11)	-0.0034 (12)
C11	0.0263 (15)	0.0265 (16)	0.0270 (14)	0.0004 (12)	-0.0080 (12)	-0.0097 (12)

Geometric parameters (Å, °)

Br1—C9	1.876 (2)	C2—C3	1.382 (3)
S1—C9	1.738 (3)	C2—H2A	0.9500
S1—C8	1.755 (2)	C3—C4	1.402 (3)
O1—C3	1.369 (3)	C4—C5	1.382 (4)
O1—C10	1.426 (3)	C5—C6	1.407 (3)
O2—C4	1.372 (3)	C5—H5A	0.9500
O2—C11	1.437 (3)	C6—C7	1.480 (4)
N1—C8	1.312 (3)	C7—C9	1.355 (3)
N1—C7	1.390 (3)	C10—H10A	0.9800
N2—C8	1.340 (4)	C10—H10B	0.9800
N2—H2N2	0.78 (3)	C10—H10C	0.9800
N2—H1N2	0.80 (3)	C11—H11A	0.9800
C1—C6	1.380 (3)	C11—H11B	0.9800
C1—C2	1.395 (4)	C11—H11C	0.9800
C1—H1A	0.9500		
C9—S1—C8	88.28 (12)	C1—C6—C7	121.1 (2)
C3—O1—C10	116.65 (19)	C5—C6—C7	119.8 (2)
C4—O2—C11	117.3 (2)	C9—C7—N1	114.3 (2)
C8—N1—C7	111.7 (2)	C9—C7—C6	126.9 (2)
C8—N2—H2N2	120 (2)	N1—C7—C6	118.7 (2)
C8—N2—H1N2	119 (2)	N1—C8—N2	123.9 (2)
H2N2—N2—H1N2	116 (3)	N1—C8—S1	114.24 (19)
C6—C1—C2	121.1 (2)	N2—C8—S1	121.9 (2)
C6—C1—H1A	119.4	C7—C9—S1	111.44 (19)
C2—C1—H1A	119.4	C7—C9—Br1	128.9 (2)
C3—C2—C1	119.6 (2)	S1—C9—Br1	119.41 (13)
C3—C2—H2A	120.2	O1—C10—H10A	109.5
C1—C2—H2A	120.2	O1—C10—H10B	109.5
O1—C3—C2	124.9 (2)	H10A—C10—H10B	109.5
O1—C3—C4	115.1 (2)	O1—C10—H10C	109.5
C2—C3—C4	120.0 (2)	H10A—C10—H10C	109.5
O2—C4—C5	125.4 (2)	H10B—C10—H10C	109.5
O2—C4—C3	114.6 (2)	O2—C11—H11A	109.5
C5—C4—C3	120.1 (2)	O2—C11—H11B	109.5
C4—C5—C6	120.1 (2)	H11A—C11—H11B	109.5
C4—C5—H5A	119.9	O2—C11—H11C	109.5
C6—C5—H5A	119.9	H11A—C11—H11C	109.5
C1—C6—C5	119.1 (2)	H11B—C11—H11C	109.5
C6—C1—C2—C3	-1.4 (4)	C8—N1—C7—C9	1.5 (3)
C10—O1—C3—C2	-9.2 (3)	C8—N1—C7—C6	-178.0 (2)
C10—O1—C3—C4	170.4 (2)	C1—C6—C7—C9	-52.8 (4)
C1—C2—C3—O1	179.9 (2)	C5—C6—C7—C9	128.5 (3)
C1—C2—C3—C4	0.3 (4)	C1—C6—C7—N1	126.6 (2)
C11—O2—C4—C5	-5.5 (3)	C5—C6—C7—N1	-52.1 (3)

C11—O2—C4—C3	175.2 (2)	C7—N1—C8—N2	-179.6 (2)
O1—C3—C4—O2	1.3 (3)	C7—N1—C8—S1	-1.3 (3)
C2—C3—C4—O2	-179.1 (2)	C9—S1—C8—N1	0.6 (2)
O1—C3—C4—C5	-178.0 (2)	C9—S1—C8—N2	179.0 (2)
C2—C3—C4—C5	1.6 (4)	N1—C7—C9—S1	-1.0 (3)
O2—C4—C5—C6	178.4 (2)	C6—C7—C9—S1	178.36 (19)
C3—C4—C5—C6	-2.4 (3)	N1—C7—C9—Br1	172.86 (17)
C2—C1—C6—C5	0.6 (4)	C6—C7—C9—Br1	-7.7 (4)
C2—C1—C6—C7	-178.1 (2)	C8—S1—C9—C7	0.28 (19)
C4—C5—C6—C1	1.3 (3)	C8—S1—C9—Br1	-174.28 (14)
C4—C5—C6—C7	-180.0 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2N2 \cdots O1 ⁱ	0.78 (3)	2.40 (3)	2.992 (3)	134 (3)
N2—H2N2 \cdots O2 ⁱ	0.78 (3)	2.37 (3)	3.112 (3)	161 (3)
N2—H1N2 \cdots N1 ⁱⁱ	0.81 (3)	2.20 (3)	2.998 (3)	168 (3)

Symmetry codes: (i) $x-1, y+1, z$; (ii) $-x, -y+1, -z+1$.