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4-[(*E*)-2-Furylmethyleneamino]-3-phenyl-1*H*-1,2,4-triazole-5(4*H*)-thione

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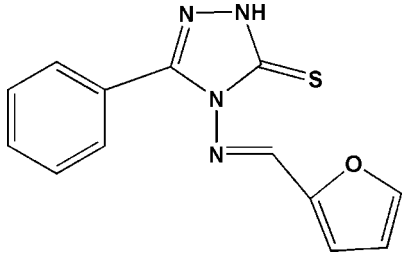
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 20.6.

In the title molecule, $C_{13}H_{10}N_4OS$, the triazole ring makes dihedral angles of 16.14 (9) and 58.51 (11)°, respectively, with the phenyl and furan rings. Intramolecular C—H···N hydrogen bonds generate S(5) and S(6) ring motifs. In the crystal structure, centrosymmetrically related molecules are linked *via* N—H···S hydrogen bonds to form dimeric pairs, which are interlinked *via* C—H···O and C—H···π interactions.

Related literature

For the biological activities of triazole derivatives, see: Clemons *et al.* (2004); Glerman *et al.* (1997); Holla *et al.* (2003); Johnston (2002); Kane *et al.* (1990); Kkgzel *et al.* (2004); Modzelewska & Kalabun (1999); Rollas *et al.* (1993); Shujuan *et al.* (2004); For bond-length data, see: Allen *et al.* (1987). For graph-set analysis of hydrogen bonding, see: Bernstein *et al.* (1995).



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Experimental

Crystal data

$C_{13}H_{10}N_4OS$
M_r = 270.31
Orthorhombic, *Pbcn*
a = 27.4006 (6) Å
b = 11.4940 (3) Å
c = 7.7886 (2) Å
V = 2452.96 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 100.0 (1) K
0.40 × 0.13 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.829, T_{max} = 0.974
40042 measured reflections
3627 independent reflections
2573 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.125
S = 1.05
3627 reflections
176 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

*Cg*1 is the centroid of the C3–C8 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N1···S1^{i}	0.85 (2)	2.42 (2)	3.265 (2)	169 (2)
C4—H4A···N2	0.93	2.55	2.859 (2)	100
C6—H6A···O1^{ii}	0.93	2.59	3.347 (2)	139
C8—H8A···N4	0.93	2.29	2.942 (2)	126
C5—H5A···Cg1^{iii}	0.93	2.92	3.522 (2)	123

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x-\frac{1}{2}, y+\frac{1}{2}, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); 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, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2631).

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supporting information

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4-[(*E*)-2-Furylmethyleneamino]-3-phenyl-1*H*-1,2,4-triazole-5(4*H*)-thione

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S1. Comment

1,2,4-Triazoles and their derivatives are found to be associated with various biological activities such as anticonvulsant (Kane *et al.*, 1990; Kkgzel *et al.*, 2004), antifungal (Rollas *et al.*, 1993), anticancer (Holla *et al.*, 2003), anti-inflammatory (Modzelewska & Kalabun, 1999) and antibacterial properties (Glerman *et al.*, 1997). Several compounds containing 1,2,4-triazole rings are well known as drugs. For example, fluconazole is used as an antimicrobial drug (Shujuan *et al.*, 2004), while vorozole, letrozole and anastrozole are non-steroidal drugs used for the threatment of cancer (Clemons *et al.*, 2004) and loreclezole is used as an anticonvulsant (Johnston, 2002) drug. In view of the above properties, we have synthesized the title compound and report here its crystal structure.

Bond lengths and angles in the title molecule (Fig. 1) are found to have normal values (Allen *et al.*, 1987). The furan ring is planar to within ±0.002 (2) Å and the triazole ring is also planar with a maximum deviation of 0.016 (2) Å for atom C1. The triazole and phenyl rings are twisted away from each other by an angle of 16.14 (9)°. The dihedral angle between the furan and triazole rings is 58.51 (11)°. Intramolecular C—H···N hydrogen bonds generate S(5) and S(6) ring motifs (Bernstein *et al.*, 1995).

The crystal structure is stabilized by intermolecular C—H···O and N—H···S hydrogen bonds together with C—H···π interactions involving the phenyl ring. The centrosymmetrically related molecules are linked by N—H···S hydrogen bonds to form a dimeric pair (Fig. 2) which are interlinked via C—H···O hydrogen bonds.

S2. Experimental

The title Schiff base compound was obtained by refluxing a mixture of 4-amino-5-methyl-2,4-dihydro-3*H*-1,2,4-triazole-3-thione (0.01 mol), furfural (0.01 mol) in ethanol (30 ml) and 2 drops of concentrated H_2SO_4 for 3 h. The solid product obtained was collected by filtration, washed with ethanol and dried. Single crystals suitable for X-ray analysis were obtained from acetone-N,N-dimethylformamide (DMF) (1:2) solution by slow evaporation (yield 63%; m.p. 451–453 K). Analysis for $C_{13}H_{10}N_4SO$, found (calculated) in %: C 57.63 (57.77), H 3.62 (3.7), N 20.6 (20.74), S 11.79 (11.85).

S3. Refinement

The N-bound H atom was located in a difference map and refined with a N-H distance restraint of 0.85 (1) Å. C-bound H atoms were positioned geometrically [C-H = 0.93%A] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

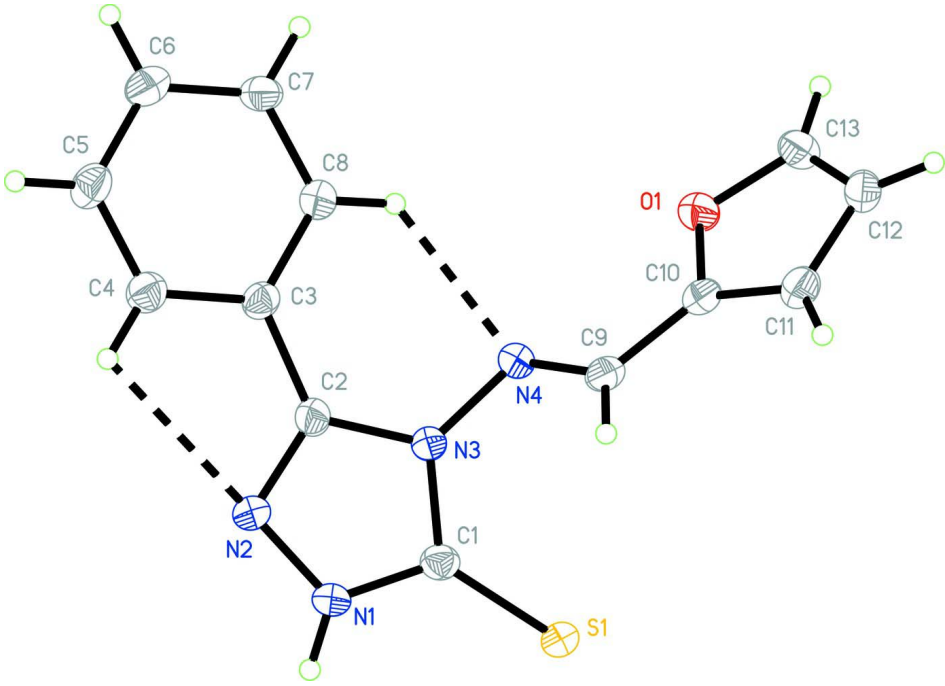

Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

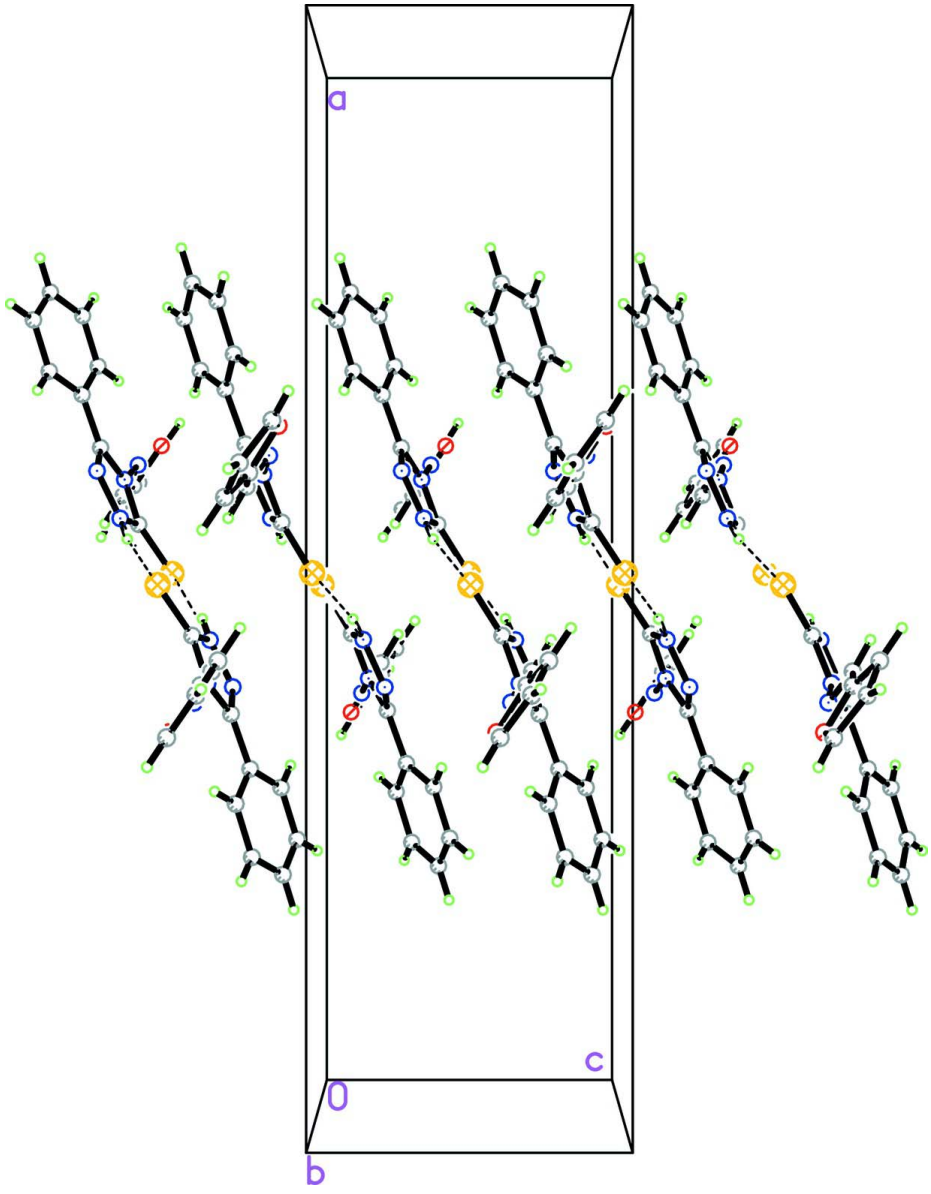

Figure 2

The crystal packing of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{13}H_{10}N_4OS$
$M_r = 270.31$
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
$a = 27.4006\ (6)$ Å
$b = 11.4940\ (3)$ Å
$c = 7.7886\ (2)$ Å
$V = 2452.96\ (10)$ Å^3
$Z = 8$
$F(000) = 1120$
$D_x = 1.464$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5448 reflections
$\theta = 2.9–27.9°$
$\mu = 0.26$ mm^{-1}
$T = 100$ K
Block, orange
0.40 × 0.13 × 0.10 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, 2005)
T_{min} = 0.829, T_{max} = 0.974
40042 measured reflections
3627 independent reflections
2573 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 30.2°, θ_{min} = 2.9°
h = −38→38
k = −16→16
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.125
S = 1.06
3627 reflections
176 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 0.3858P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.494570 (15)	0.69781 (4)	0.50297 (6)	0.02259 (13)
O1	0.36669 (4)	1.02453 (10)	0.58350 (18)	0.0258 (3)
N1	0.44316 (5)	0.52097 (12)	0.6503 (2)	0.0225 (3)
N2	0.39895 (5)	0.49428 (12)	0.7222 (2)	0.0228 (3)
N3	0.40473 (5)	0.68093 (11)	0.66047 (19)	0.0190 (3)
N4	0.38912 (5)	0.79579 (12)	0.6319 (2)	0.0206 (3)
C1	0.44803 (6)	0.63262 (15)	0.6058 (2)	0.0203 (3)
C2	0.37554 (6)	0.59359 (14)	0.7269 (2)	0.0204 (4)
C3	0.32509 (6)	0.60514 (14)	0.7901 (2)	0.0198 (3)
C4	0.30539 (6)	0.51246 (15)	0.8824 (2)	0.0244 (4)
H4A	0.3247	0.4484	0.9086	0.029*
C5	0.25719 (6)	0.51543 (16)	0.9351 (3)	0.0267 (4)
H5A	0.2444	0.4533	0.9971	0.032*
C6	0.22781 (6)	0.60973 (16)	0.8967 (2)	0.0259 (4)

H6A	0.1954	0.6111	0.9320	0.031*
C7	0.24716 (6)	0.70213 (16)	0.8051 (3)	0.0255 (4)
H7A	0.2275	0.7656	0.7783	0.031*
C8	0.29558 (6)	0.70074 (15)	0.7530 (2)	0.0229 (4)
H8A	0.3084	0.7637	0.6931	0.028*
C9	0.41795 (6)	0.87250 (15)	0.6957 (2)	0.0212 (4)
H9A	0.4458	0.8494	0.7548	0.025*
C10	0.40718 (6)	0.99364 (15)	0.6757 (2)	0.0214 (4)
C11	0.42966 (7)	1.08948 (16)	0.7401 (3)	0.0268 (4)
H11A	0.4578	1.0913	0.8067	0.032*
C12	0.40148 (7)	1.18664 (15)	0.6854 (3)	0.0282 (4)
H12A	0.4075	1.2646	0.7095	0.034*
C13	0.36459 (7)	1.14332 (15)	0.5921 (3)	0.0289 (4)
H13A	0.3406	1.1883	0.5399	0.035*
H1N1	0.4628 (6)	0.4674 (14)	0.619 (3)	0.034 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0170 (2)	0.0210 (2)	0.0298 (3)	0.00050 (15)	0.00408 (17)	0.00147 (18)
O1	0.0210 (6)	0.0219 (6)	0.0344 (8)	0.0019 (5)	−0.0013 (5)	0.0001 (6)
N1	0.0169 (7)	0.0191 (7)	0.0316 (9)	0.0034 (5)	0.0022 (6)	0.0014 (6)
N2	0.0161 (7)	0.0206 (7)	0.0317 (9)	0.0010 (5)	0.0025 (6)	0.0013 (6)
N3	0.0153 (6)	0.0172 (7)	0.0244 (8)	0.0004 (5)	−0.0002 (6)	0.0008 (6)
N4	0.0185 (6)	0.0173 (7)	0.0260 (8)	0.0016 (5)	0.0010 (6)	0.0015 (6)
C1	0.0170 (7)	0.0208 (8)	0.0232 (9)	0.0014 (6)	−0.0020 (6)	−0.0018 (7)
C2	0.0182 (8)	0.0182 (8)	0.0247 (9)	−0.0013 (6)	−0.0014 (7)	0.0010 (7)
C3	0.0167 (7)	0.0215 (8)	0.0212 (8)	−0.0018 (6)	−0.0011 (6)	−0.0011 (7)
C4	0.0214 (8)	0.0256 (9)	0.0262 (9)	0.0001 (7)	0.0001 (7)	0.0040 (7)
C5	0.0240 (8)	0.0306 (10)	0.0255 (9)	−0.0038 (7)	0.0025 (7)	0.0058 (8)
C6	0.0199 (8)	0.0320 (10)	0.0259 (9)	−0.0012 (7)	0.0031 (7)	−0.0031 (8)
C7	0.0180 (8)	0.0231 (9)	0.0353 (10)	0.0026 (7)	0.0003 (7)	−0.0044 (8)
C8	0.0206 (8)	0.0204 (8)	0.0279 (10)	−0.0013 (6)	0.0008 (7)	0.0008 (7)
C9	0.0161 (7)	0.0240 (8)	0.0236 (9)	0.0006 (6)	0.0024 (7)	0.0003 (7)
C10	0.0169 (7)	0.0235 (9)	0.0238 (9)	−0.0008 (6)	0.0028 (7)	−0.0001 (7)
C11	0.0214 (8)	0.0254 (9)	0.0334 (10)	−0.0042 (7)	0.0033 (7)	−0.0027 (8)
C12	0.0280 (9)	0.0199 (9)	0.0366 (11)	−0.0031 (7)	0.0106 (8)	−0.0019 (8)
C13	0.0280 (9)	0.0216 (9)	0.0372 (11)	0.0054 (7)	0.0072 (8)	0.0050 (8)

Geometric parameters (Å, º)

S1—C1	1.6820 (17)	C5—C6	1.383 (3)
O1—C13	1.368 (2)	C5—H5A	0.93
O1—C10	1.368 (2)	C6—C7	1.385 (3)
N1—C1	1.336 (2)	C6—H6A	0.93
N1—N2	1.369 (2)	C7—C8	1.388 (2)
N1—H1N1	0.853 (9)	C7—H7A	0.93
N2—C2	1.310 (2)	C8—H8A	0.93

N3—C1	1.377 (2)	C9—C10	1.432 (2)
N3—C2	1.384 (2)	C9—H9A	0.93
N3—N4	1.4054 (18)	C10—C11	1.358 (2)
N4—C9	1.284 (2)	C11—C12	1.423 (3)
C2—C3	1.474 (2)	C11—H11A	0.93
C3—C4	1.394 (2)	C12—C13	1.341 (3)
C3—C8	1.395 (2)	C12—H12A	0.93
C4—C5	1.384 (2)	C13—H13A	0.93
C4—H4A	0.93		

C13—O1—C10	105.50 (14)	C5—C6—C7	119.33 (16)
C1—N1—N2	114.19 (14)	C5—C6—H6A	120.3
C1—N1—H1N1	123.8 (15)	C7—C6—H6A	120.3
N2—N1—H1N1	120.9 (15)	C6—C7—C8	120.52 (17)
C2—N2—N1	104.44 (13)	C6—C7—H7A	119.7
C1—N3—C2	108.72 (13)	C8—C7—H7A	119.7
C1—N3—N4	126.29 (13)	C7—C8—C3	120.18 (16)
C2—N3—N4	124.37 (13)	C7—C8—H8A	119.9
C9—N4—N3	113.36 (14)	C3—C8—H8A	119.9
N1—C1—N3	102.77 (14)	N4—C9—C10	119.92 (16)
N1—C1—S1	128.84 (13)	N4—C9—H9A	120.0
N3—C1—S1	128.36 (13)	C10—C9—H9A	120.0
N2—C2—N3	109.79 (14)	C11—C10—O1	110.56 (15)
N2—C2—C3	123.18 (15)	C11—C10—C9	130.92 (17)
N3—C2—C3	127.01 (15)	O1—C10—C9	118.46 (15)
C4—C3—C8	119.00 (15)	C10—C11—C12	106.24 (17)
C4—C3—C2	117.83 (15)	C10—C11—H11A	126.9
C8—C3—C2	123.05 (15)	C12—C11—H11A	126.9
C5—C4—C3	120.26 (16)	C13—C12—C11	106.25 (16)
C5—C4—H4A	119.9	C13—C12—H12A	126.9
C3—C4—H4A	119.9	C11—C12—H12A	126.9
C6—C5—C4	120.71 (17)	C12—C13—O1	111.45 (16)
C6—C5—H5A	119.6	C12—C13—H13A	124.3
C4—C5—H5A	119.6	O1—C13—H13A	124.3

C1—N1—N2—C2	−1.5 (2)	C8—C3—C4—C5	0.4 (3)
C1—N3—N4—C9	−60.1 (2)	C2—C3—C4—C5	−175.78 (17)
C2—N3—N4—C9	129.83 (18)	C3—C4—C5—C6	0.3 (3)
N2—N1—C1—N3	2.8 (2)	C4—C5—C6—C7	−0.3 (3)
N2—N1—C1—S1	−175.45 (13)	C5—C6—C7—C8	−0.3 (3)
C2—N3—C1—N1	−2.91 (19)	C6—C7—C8—C3	1.0 (3)
N4—N3—C1—N1	−174.24 (15)	C4—C3—C8—C7	−1.0 (3)
C2—N3—C1—S1	175.32 (14)	C2—C3—C8—C7	174.91 (17)
N4—N3—C1—S1	4.0 (3)	N3—N4—C9—C10	179.42 (14)
N1—N2—C2—N3	−0.50 (19)	C13—O1—C10—C11	0.1 (2)
N1—N2—C2—C3	177.88 (17)	C13—O1—C10—C9	177.54 (16)
C1—N3—C2—N2	2.2 (2)	N4—C9—C10—C11	174.88 (19)
N4—N3—C2—N2	173.76 (15)	N4—C9—C10—O1	−2.0 (3)

C1—N3—C2—C3	−176.07 (17)	O1—C10—C11—C12	0.1 (2)
N4—N3—C2—C3	−4.5 (3)	C9—C10—C11—C12	−176.88 (18)
N2—C2—C3—C4	14.2 (3)	C10—C11—C12—C13	−0.3 (2)
N3—C2—C3—C4	−167.70 (17)	C11—C12—C13—O1	0.4 (2)
N2—C2—C3—C8	−161.76 (17)	C10—O1—C13—C12	−0.3 (2)
N3—C2—C3—C8	16.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···S1^{i}	0.85 (2)	2.42 (2)	3.265 (2)	169 (2)
C4—H4*A*···N2	0.93	2.55	2.859 (2)	100
C6—H6*A*···O1^{ii}	0.93	2.59	3.347 (2)	139
C8—H8*A*···N4	0.93	2.29	2.942 (2)	126
C5—H5*A*···*Cg*1^{iii}	0.93	2.92	3.522 (2)	123

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1/2, −*y*+3/2, *z*+1/2; (iii) −*x*−1/2, *y*+1/2, *z*.