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2-(6-Phenyl-7*H*-1,2,4-triazolo[3,4-*b*]-[1,3,4]thiadiazin-3-yl)-1,3-benzothiazole

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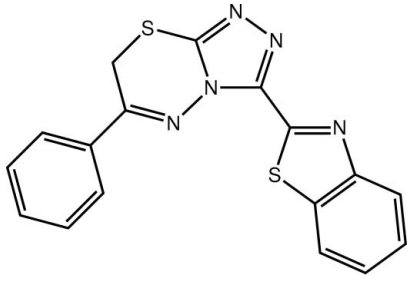
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.083; data-to-parameter ratio = 13.4.

In the title compound, $C_{17}H_{11}N_5S_2$, the dihedral angles formed between the triazole ring and the benzene ring and the 1,3-benzothiazole ring system are 8.67 (8) and 13.90 (9)°, respectively. The conformation of the triazolo-thiadiazin-3-yl fused ring system is a twisted half-chair. Overall, the molecule adopts a flattened shape. Supramolecular helical chains along the *a* axis sustained by C—H···N interactions are found in the crystal structure. These are linked *via* C—H···π contacts as well as π–π [centroid–centroid distance = 3.5911 (12) Å] interactions between the triazole and thiazole rings.

Related literature

For background to the synthesis and biological activity of benzothiazoles and [1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazines, see: Abdel-Aziz *et al.* (2007, 2010); Dawood *et al.* (2005).



Experimental

Crystal data

$C_{17}H_{11}N_5S_2$ M_r = 349.43
Orthorhombic, $P2_12_12$
a = 12.1437 (3) Å
b = 21.2950 (5) Å
c = 5.7946 (1) Å
V = 1498.48 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 3.29 mm^{-1}
T = 100 K
0.25 × 0.25 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.715, T_{max} = 1.000
5754 measured reflections
2902 independent reflections
2751 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.06
2902 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Flack (1983), 1150 Friedel pairs
Flack parameter: −0.006 (16)

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

*Cg*1 is the centroid of the C12–C17 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10a···N3^{i}	0.99	2.45	3.333 (3)	148
C3—H3···*Cg*1^{ii}	0.95	2.65	3.377 (2)	134

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

Data collection: *CrysAlis PRO* (Agilent, 2010); 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: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5092).

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supporting information

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2-(6-Phenyl-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazin-3-yl)-1,3-benzothiazole

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S1. Comment

The title compound, (I), was investigated in relation to the established biological activities exhibited by benzothiazoles and [1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazines (Abdel-Aziz *et al.* 2007; Abdel-Aziz *et al.* 2010; Dawood *et al.* 2005).

In (I), Fig. 1, the 1,3-benzothiazole ring is planar with r.m.s. deviations of 0.034 Å. By contrast, the triazolo-thiadiazin-3-yl fused ring system has a twisted half-chair form owing to the presence of the methylene-C10 group with the C10 atom lying 0.702 (3) Å out of the least-squares plane defined by the S2,N4,N5,C9,C11 atoms (r.m.s. deviation = 0.109 Å). The 1,3-benzothiazole ring forms dihedral angles of 13.90 (9) and 8.67 (8) °, respectively, with the triazole and benzene rings so that the entire molecule has a flattened shape.

In the crystal packing, C—H···N interactions, Table 1, lead to the formation of supramolecular chains along the *a* axis with an helical topology, Fig. 2. These assemble into zigzag layers in the *ac* plane with connections between them of the type C—H···π involving a methylene-H and the benzene ring, and π···π. The shortest interaction of the latter type of 3.5911 (12) Å occurs between the S1,N1,C1,C6,C7 and N2–N4,C8,C9 five-membered rings, Fig. 3.

S2. Experimental

The title compound was prepared according to the reported method (Abdel-Aziz *et al.*, 2007). Colourless crystals were obtained from an EtOH/DMF (*v*/*v* = 2/1) solution by slow evaporation at room temperature.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.99 Å, U_{iso}(H) 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation.

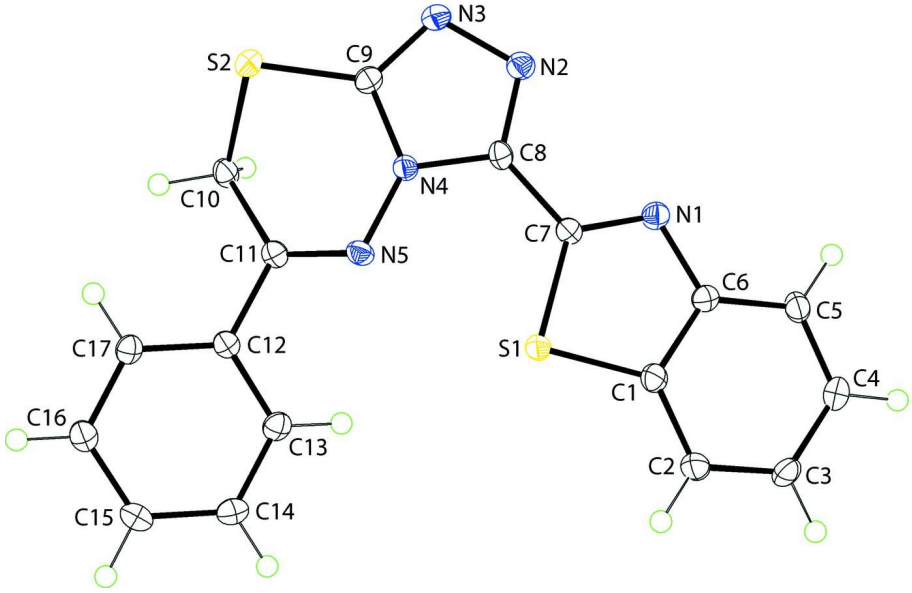

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

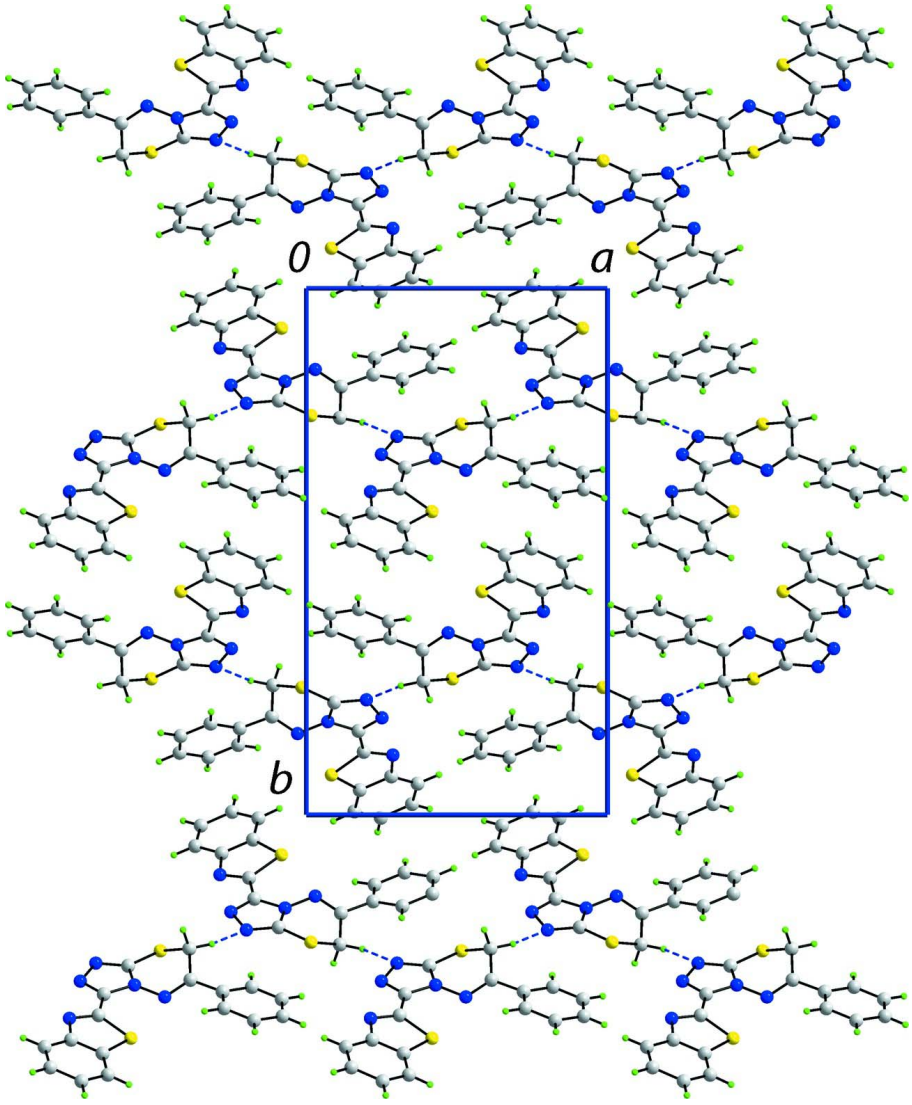

Figure 2

Supramolecular chains in (I) mediated by C—H···N interactions (blue dashed lines).

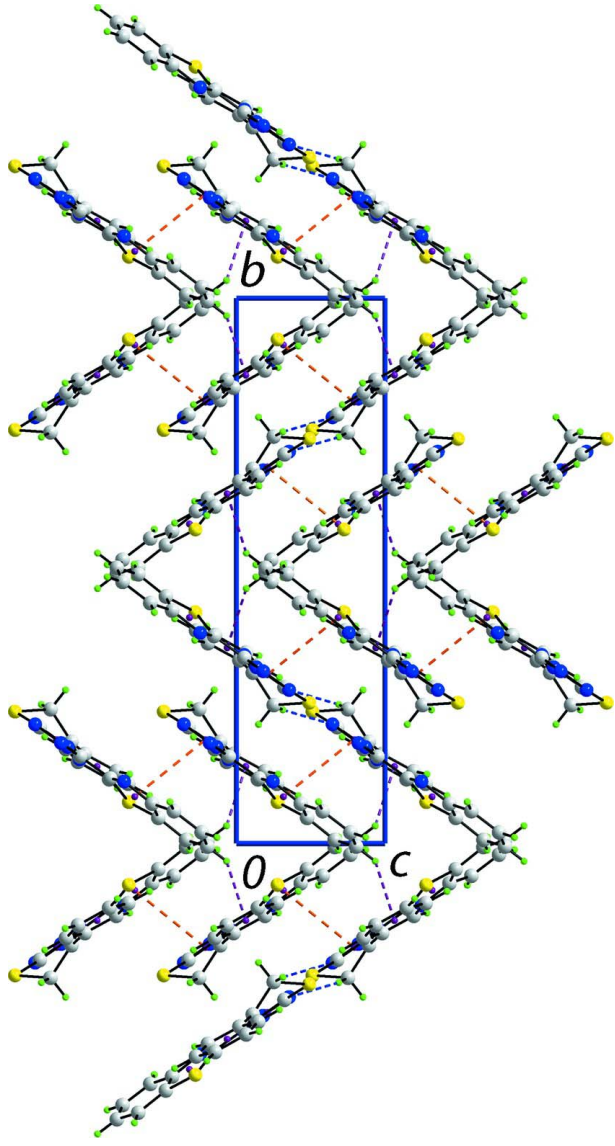

Figure 3

A view in projection down the *a* axis of the unit-cell contents of (I). The C—H···N, C—H···π and π···π interactions are shown as blue, purple and orange dashed lines, respectively.

2-(6-Phenyl-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazin-3-yl)-1,3- benzothiazole

Crystal data

$C_{17}H_{11}N_5S_2$
M_r = 349.43
Orthorhombic, $P2_12_12$
Hall symbol: P 2 2ab
a = 12.1437 (3) Å
b = 21.2950 (5) Å
c = 5.7946 (1) Å
V = 1498.48 (6) Å^3
Z = 4
$F(000)$ = 720
D_x = 1.549 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.5418 Å
Cell parameters from 3513 reflections
θ = 3.6–74.3°
μ = 3.29 mm^{-1}
T = 100 K
Plate, light-brown
0.25 × 0.25 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.715, T_{max} = 1.000
5754 measured reflections
2902 independent reflections
2751 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 74.5°, θ_{min} = 4.2°
h = −15→13
k = −13→26
l = −6→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.06
2902 reflections
217 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
$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Flack (1983), 1150 Friedel pairs
Absolute structure parameter: −0.006 (16)

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.41692 (4)	0.42415 (2)	0.72377 (10)	0.01863 (14)
S2	0.51497 (4)	0.25815 (2)	1.48903 (10)	0.01803 (13)
N1	0.21298 (15)	0.38633 (8)	0.7555 (3)	0.0165 (4)
N2	0.24914 (15)	0.31575 (9)	1.1796 (3)	0.0170 (4)
N3	0.30068 (16)	0.28150 (8)	1.3539 (3)	0.0177 (4)
N4	0.42913 (15)	0.32303 (8)	1.1339 (3)	0.0142 (4)
N5	0.52940 (15)	0.34569 (8)	1.0531 (3)	0.0155 (4)
C1	0.32276 (18)	0.45294 (9)	0.5251 (4)	0.0173 (4)
C2	0.34046 (19)	0.49351 (11)	0.3396 (4)	0.0214 (5)
H2	0.4105	0.5121	0.3138	0.026*
C3	0.25298 (18)	0.50565 (10)	0.1955 (4)	0.0204 (5)
H3	0.2635	0.5325	0.0664	0.024*
C4	0.14936 (18)	0.47958 (9)	0.2342 (4)	0.0202 (5)
H4	0.0908	0.4887	0.1309	0.024*

C5	0.13077 (18)	0.44076 (10)	0.4203 (4)	0.0191 (5)
H5	0.0597	0.4237	0.4478	0.023*
C6	0.21838 (18)	0.42704 (9)	0.5676 (4)	0.0149 (4)
C7	0.30956 (18)	0.38046 (9)	0.8490 (4)	0.0139 (4)
C8	0.32714 (17)	0.34057 (9)	1.0515 (4)	0.0149 (4)
C9	0.40792 (19)	0.28660 (9)	1.3227 (4)	0.0153 (4)
C10	0.61131 (16)	0.25386 (9)	1.2505 (4)	0.0175 (4)
H10A	0.6859	0.2451	1.3118	0.021*
H10B	0.5904	0.2184	1.1489	0.021*
C11	0.61482 (18)	0.31330 (9)	1.1098 (4)	0.0143 (4)
C12	0.72157 (16)	0.33574 (8)	1.0201 (4)	0.0140 (4)
C13	0.72667 (19)	0.36733 (9)	0.8082 (4)	0.0171 (4)
H13	0.6617	0.3728	0.7193	0.021*
C14	0.82592 (18)	0.39051 (9)	0.7279 (4)	0.0180 (4)
H14	0.8288	0.4122	0.5846	0.022*
C15	0.92123 (19)	0.38220 (10)	0.8562 (4)	0.0186 (4)
H15	0.9891	0.3985	0.8010	0.022*
C16	0.91816 (19)	0.35013 (10)	1.0651 (4)	0.0184 (4)
H16	0.9838	0.3443	1.1518	0.022*
C17	0.81865 (18)	0.32671 (9)	1.1466 (4)	0.0159 (4)
H17	0.8164	0.3045	1.2886	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0133 (2)	0.0176 (2)	0.0250 (3)	−0.00121 (19)	−0.0008 (2)	0.0079 (2)
S2	0.0186 (3)	0.0185 (3)	0.0170 (3)	0.00037 (19)	−0.0012 (2)	0.00396 (19)
N1	0.0172 (9)	0.0139 (8)	0.0184 (9)	−0.0010 (7)	−0.0006 (8)	0.0007 (8)
N2	0.0168 (9)	0.0155 (8)	0.0186 (9)	−0.0002 (7)	0.0009 (8)	0.0010 (7)
N3	0.0185 (9)	0.0171 (9)	0.0175 (10)	−0.0008 (7)	0.0015 (7)	0.0035 (7)
N4	0.0129 (9)	0.0131 (8)	0.0167 (9)	−0.0003 (7)	−0.0003 (7)	0.0014 (7)
N5	0.0134 (9)	0.0136 (8)	0.0195 (10)	−0.0017 (7)	0.0019 (7)	0.0015 (7)
C1	0.0158 (10)	0.0138 (9)	0.0224 (11)	0.0024 (8)	0.0009 (9)	0.0014 (9)
C2	0.0168 (11)	0.0181 (10)	0.0295 (13)	−0.0004 (9)	0.0018 (10)	0.0066 (9)
C3	0.0231 (12)	0.0158 (10)	0.0223 (11)	0.0039 (9)	0.0017 (10)	0.0065 (9)
C4	0.0201 (11)	0.0195 (10)	0.0211 (12)	0.0042 (8)	−0.0038 (10)	0.0012 (9)
C5	0.0139 (11)	0.0198 (10)	0.0237 (12)	0.0009 (8)	−0.0021 (9)	0.0014 (9)
C6	0.0173 (11)	0.0118 (8)	0.0157 (10)	0.0011 (8)	0.0013 (8)	−0.0008 (8)
C7	0.0137 (9)	0.0105 (9)	0.0175 (11)	−0.0002 (8)	0.0008 (8)	−0.0011 (8)
C8	0.0127 (10)	0.0138 (9)	0.0183 (11)	0.0017 (8)	−0.0010 (8)	−0.0012 (8)
C9	0.0176 (10)	0.0121 (9)	0.0161 (10)	0.0003 (8)	0.0001 (8)	0.0004 (7)
C10	0.0139 (9)	0.0134 (9)	0.0252 (11)	0.0001 (8)	−0.0016 (9)	0.0024 (9)
C11	0.0145 (10)	0.0122 (9)	0.0161 (10)	−0.0022 (8)	−0.0012 (8)	−0.0024 (8)
C12	0.0143 (10)	0.0093 (8)	0.0184 (10)	−0.0001 (7)	−0.0001 (8)	−0.0015 (8)
C13	0.0199 (11)	0.0124 (9)	0.0191 (11)	−0.0002 (8)	−0.0015 (9)	0.0005 (8)
C14	0.0218 (11)	0.0159 (9)	0.0161 (11)	0.0000 (8)	0.0033 (9)	0.0007 (9)
C15	0.0160 (10)	0.0180 (10)	0.0219 (11)	−0.0031 (9)	0.0039 (9)	−0.0027 (9)
C16	0.0161 (10)	0.0169 (9)	0.0221 (12)	0.0003 (8)	−0.0015 (9)	−0.0010 (8)

C17	0.0175 (11)	0.0123 (9)	0.0177 (11)	0.0013 (8)	−0.0017 (9)	−0.0002 (8)

Geometric parameters (Å, º)

S1—C1	1.734 (2)	C4—H4	0.9500
S1—C7	1.758 (2)	C5—C6	1.395 (3)
S2—C9	1.728 (2)	C5—H5	0.9500
S2—C10	1.813 (2)	C7—C8	1.464 (3)
N1—C7	1.298 (3)	C10—C11	1.507 (3)
N1—C6	1.393 (3)	C10—H10A	0.9900
N2—C8	1.314 (3)	C10—H10B	0.9900
N2—N3	1.394 (3)	C11—C12	1.476 (3)
N3—C9	1.319 (3)	C12—C13	1.402 (3)
N4—C9	1.366 (3)	C12—C17	1.401 (3)
N4—C8	1.379 (3)	C13—C14	1.383 (3)
N4—N5	1.391 (2)	C13—H13	0.9500
N5—C11	1.288 (3)	C14—C15	1.387 (3)
C1—C2	1.396 (3)	C14—H14	0.9500
C1—C6	1.404 (3)	C15—C16	1.391 (3)
C2—C3	1.376 (3)	C15—H15	0.9500
C2—H2	0.9500	C16—C17	1.390 (3)
C3—C4	1.394 (3)	C16—H16	0.9500
C3—H3	0.9500	C17—H17	0.9500
C4—C5	1.377 (3)		

C1—S1—C7	88.40 (10)	N4—C8—C7	124.43 (19)
C9—S2—C10	94.47 (10)	N3—C9—N4	110.04 (19)
C7—N1—C6	110.09 (18)	N3—C9—S2	129.60 (17)
C8—N2—N3	107.21 (18)	N4—C9—S2	120.26 (17)
C9—N3—N2	107.51 (17)	C11—C10—S2	112.86 (14)
C9—N4—C8	105.17 (18)	C11—C10—H10A	109.0
C9—N4—N5	129.18 (19)	S2—C10—H10A	109.0
C8—N4—N5	125.17 (17)	C11—C10—H10B	109.0
C11—N5—N4	115.69 (17)	S2—C10—H10B	109.0
C2—C1—C6	121.1 (2)	H10A—C10—H10B	107.8
C2—C1—S1	128.93 (17)	N5—C11—C12	116.37 (18)
C6—C1—S1	109.87 (16)	N5—C11—C10	124.40 (19)
C3—C2—C1	117.7 (2)	C12—C11—C10	119.17 (18)
C3—C2—H2	121.1	C13—C12—C17	119.1 (2)
C1—C2—H2	121.1	C13—C12—C11	120.2 (2)
C2—C3—C4	121.6 (2)	C17—C12—C11	120.7 (2)
C2—C3—H3	119.2	C14—C13—C12	120.3 (2)
C4—C3—H3	119.2	C14—C13—H13	119.8
C5—C4—C3	120.9 (2)	C12—C13—H13	119.8
C5—C4—H4	119.6	C13—C14—C15	120.1 (2)
C3—C4—H4	119.6	C13—C14—H14	120.0
C4—C5—C6	118.7 (2)	C15—C14—H14	120.0
C4—C5—H5	120.7	C14—C15—C16	120.5 (2)

C6—C5—H5	120.7	C14—C15—H15	119.8
C5—C6—N1	124.9 (2)	C16—C15—H15	119.8
C5—C6—C1	119.93 (19)	C17—C16—C15	119.7 (2)
N1—C6—C1	115.08 (19)	C17—C16—H16	120.2
N1—C7—C8	121.47 (19)	C15—C16—H16	120.2
N1—C7—S1	116.54 (16)	C16—C17—C12	120.3 (2)
C8—C7—S1	121.97 (16)	C16—C17—H17	119.8
N2—C8—N4	110.06 (18)	C12—C17—H17	119.8
N2—C8—C7	125.50 (19)		

C8—N2—N3—C9	−0.5 (2)	S1—C7—C8—N2	166.92 (17)
C9—N4—N5—C11	25.6 (3)	N1—C7—C8—N4	167.6 (2)
C8—N4—N5—C11	−163.6 (2)	S1—C7—C8—N4	−13.8 (3)
C7—S1—C1—C2	−177.9 (2)	N2—N3—C9—N4	0.0 (2)
C7—S1—C1—C6	−0.48 (16)	N2—N3—C9—S2	176.46 (16)
C6—C1—C2—C3	−2.0 (3)	C8—N4—C9—N3	0.5 (2)
S1—C1—C2—C3	175.07 (18)	N5—N4—C9—N3	172.72 (19)
C1—C2—C3—C4	1.2 (4)	C8—N4—C9—S2	−176.35 (15)
C2—C3—C4—C5	0.4 (4)	N5—N4—C9—S2	−4.1 (3)
C3—C4—C5—C6	−1.2 (3)	C10—S2—C9—N3	153.5 (2)
C4—C5—C6—N1	−176.8 (2)	C10—S2—C9—N4	−30.35 (18)
C4—C5—C6—C1	0.3 (3)	C9—S2—C10—C11	48.76 (17)
C7—N1—C6—C5	176.0 (2)	N4—N5—C11—C12	178.39 (18)
C7—N1—C6—C1	−1.3 (3)	N4—N5—C11—C10	1.0 (3)
C2—C1—C6—C5	1.3 (3)	S2—C10—C11—N5	−41.2 (3)
S1—C1—C6—C5	−176.30 (16)	S2—C10—C11—C12	141.45 (17)
C2—C1—C6—N1	178.7 (2)	N5—C11—C12—C13	−30.4 (3)
S1—C1—C6—N1	1.1 (2)	C10—C11—C12—C13	147.11 (19)
C6—N1—C7—C8	179.56 (17)	N5—C11—C12—C17	148.6 (2)
C6—N1—C7—S1	0.9 (2)	C10—C11—C12—C17	−33.9 (3)
C1—S1—C7—N1	−0.26 (18)	C17—C12—C13—C14	−1.5 (3)
C1—S1—C7—C8	−178.90 (18)	C11—C12—C13—C14	177.53 (19)
N3—N2—C8—N4	0.9 (2)	C12—C13—C14—C15	0.5 (3)
N3—N2—C8—C7	−179.81 (18)	C13—C14—C15—C16	0.6 (3)
C9—N4—C8—N2	−0.8 (2)	C14—C15—C16—C17	−0.5 (3)
N5—N4—C8—N2	−173.48 (19)	C15—C16—C17—C12	−0.5 (3)
C9—N4—C8—C7	179.81 (18)	C13—C12—C17—C16	1.5 (3)
N5—N4—C8—C7	7.2 (3)	C11—C12—C17—C16	−177.49 (19)
N1—C7—C8—N2	−11.7 (3)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C12–C17 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10a···N3^{i}	0.99	2.45	3.333 (3)	148
C3—H3···*Cg*1^{ii}	0.95	2.65	3.377 (2)	134

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