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Ethyl 2-{5-[(3-oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl)methyl]-1*H*-1,2,3-triazol-1-yl}acetate

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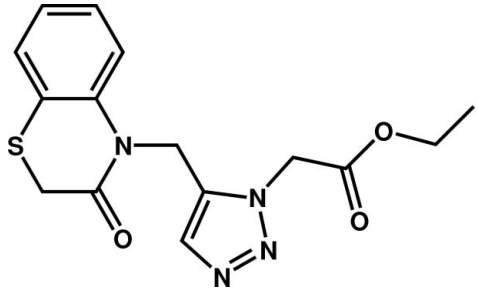
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.124; data-to-parameter ratio = 17.1.

In the title compound, $C_{15}H_{16}N_4O_3S$, the six-membered heterocycle of the benzothiazine fragment exhibits a screw boat conformation. The dihedral angle between the planes through the triazole ring and the benzene ring fused to the 1,4-thiazine ring is 62.98 (11)°. The mean plane formed by the atoms belonging to the acetate group is nearly perpendicular to the triazole ring [dihedral angle = 74.65 (12)°]. In the crystal, molecules are linked by pairs of C—H···O interactions, forming dimeric aggregates.

Related literature

For the pharmacological activity of benzothiazine derivatives, see: Fringuelli *et al.* (1998); Lopatina *et al.* (1982); Rathore & Kumar (2006). For related structures, see: Keita *et al.* (2000); Zerzouf *et al.* (2001); Barryala *et al.* (2011). For puckering calculation see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{15}H_{16}N_4O_3S$
M_r = 332.38
Triclinic, $P\overline{1}$
a = 5.6414 (2) Å
b = 11.1604 (4) Å
c = 13.3724 (5) Å
α = 73.823 (2)°
β = 87.226 (2)°
γ = 88.566 (2)°
V = 807.59 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 296 K
0.37 × 0.34 × 0.28 mm

Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.692, T_{max} = 0.747
16305 measured reflections
3560 independent reflections
2963 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.124
S = 1.05
3560 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.93	2.59	3.445 (3)	154

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5283).

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Acta Cryst. (2014). E**70**, o116 [doi:10.1107/S1600536813034697]

Ethyl 2-{5-[(3-oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl)methyl]-1*H*-1,2,3-triazol-1-yl}acetate

Nada Kheira Sebbar, Abdelfettah Zerzouf, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari

S1. Experimental

S1.1. Synthesis and crystallization

To a solution of 4-(prop-2-yn-1-yl)-2*H*-1, 4-benzothiazin-3-one (0.2 g, 0.7 mmol) in ethanol (15 ml) was added azide ethyl acetate (0.20 ml, 1.89 mmol). The mixture was stirred under reflux for 24 h. After completion of reaction (monitored by TLC), the solution was concentrated and the residue was purified by column chromatography on silica gel by using a mixture (hexane/ethyl acetate 2/1). Crystals were obtained when the solvent was allowed to evaporate. The solid product was purified by recrystallization from ethanol to afford yellow crystals in 75% yield.

S1.2. Refinement

The H atoms were located in a difference map and treated as riding with C—H = 0.93 Å (aromatic), C—H = 0.97 Å (methylene) and C—H = 0.96 Å, (methyl), and with U_{iso}(H) = 1.2 U_{eq} (methylene and ammonium) and U_{iso}(H) = 1.5 U_{eq} for the methyl. Owing to poor aggreement, three reflections, *i.e.* (0 1 1), (0 1 0) and (0 0 1), were omitted from the final cycles of refinement.

S2. Results and discussion

Several derivatives of benzothiazines form an important class of bioactive molecules in the field of drugs and pharmaceuticals. Applications in various therapeutic areas, include their use as anti-depressants (Lopatina *et al.*, 1982); anti-fungals (Fringuelli *et al.*, 1998) and anti-microbials (Rathore & Kumar, 2006). The present work is a continuation of the investigation of the benzothiazine derivatives published recently by our team (Keita *et al.*, 2000; Zerzouf *et al.*, 2001; Barryala *et al.*, 2011). The aim of the present paper was to study the recently synthesized ethyl2-{5-[(3-oxo-2*H*-1,4-benzothiazin-4-yl)methyl]- 1,2,3-triazol-1-yl}acetate crystal structure by X-ray crystallography at room temperature.

The molecule of the title compound is build up from two fused six-membered rings linked to a triazole ring which is attached to an ethylacetate group as shown in Fig.1. The 1,4-thiazine ring adopts a screw boat conformation as indicated by the puckering amplitude Q = 0.6536 (17) Å, and spherical polar angle θ = 112.04 (16)°, with φ = 152.14 (18)° (Cremer & Pople, 1975). The benzene ring (C1 to C6) makes a dihedral angle of 62.98 (11)° with the triazole ring (N2N3N4C10C11) which is nearly perpendicular to the mean plane through the acetate atoms (C12C13O2O3) as indicated by the dihedral angle between them of 74.65 (12)°. In the crystal, the molecules are linked by weak inter-molecular C4–H4···O2 interactions to form dimers (see Fig.2 and Table 1).

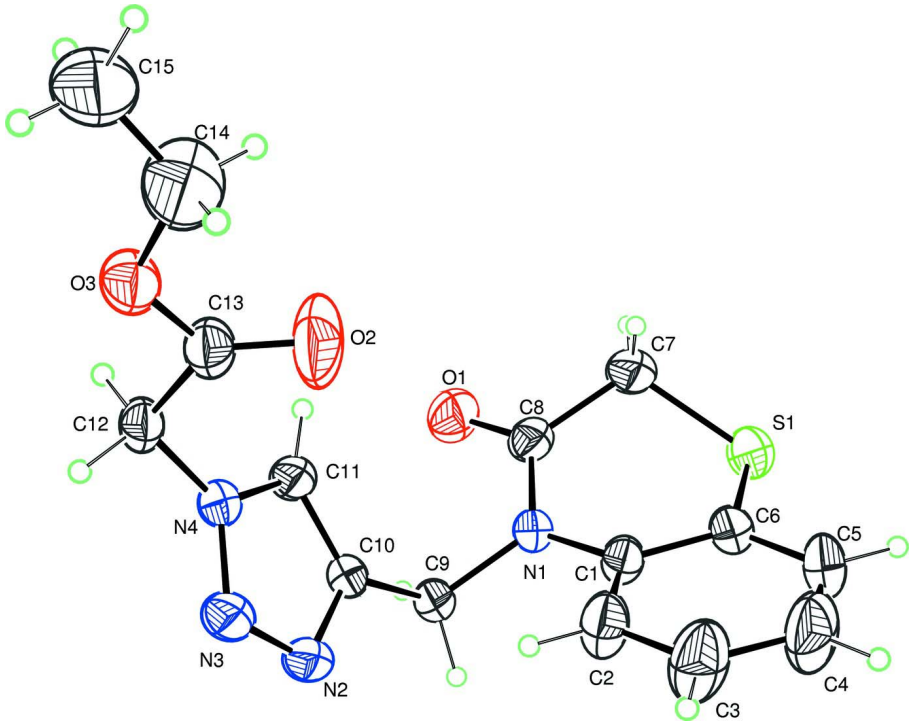

Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

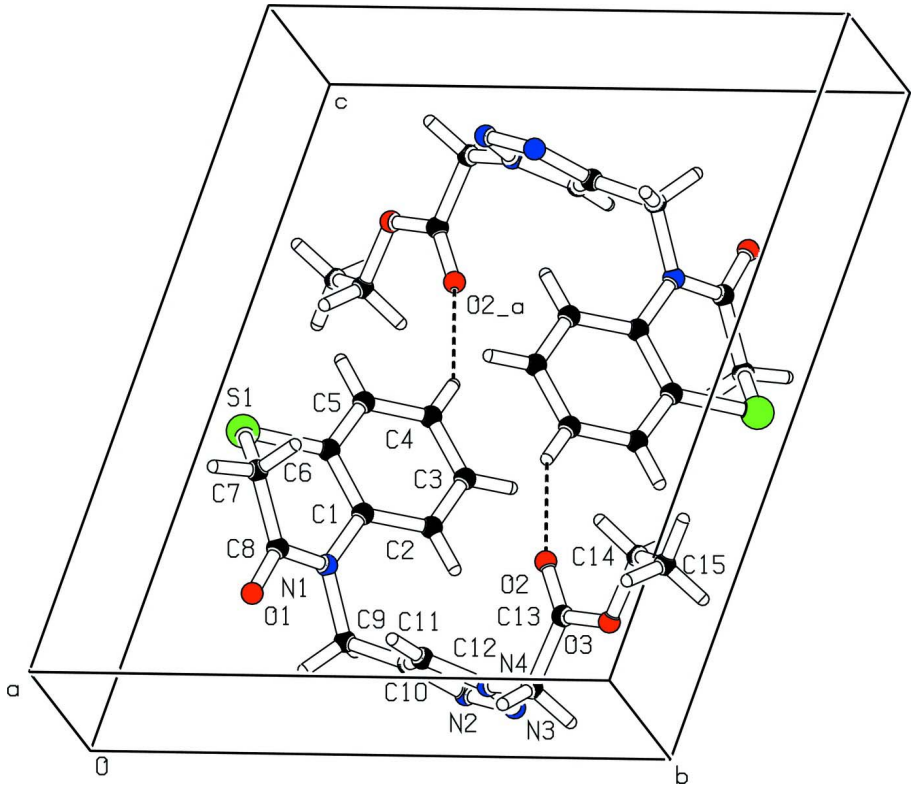

Figure 2

Three dimensional plot of the title compound, showing molecules linked through C4–H4···O2 hydrogen bonds (dashed lines).

Ethyl 2-{5-[(3-oxo-3,4-dihydro-2*H*-1,4-benzothiazin-4-yl)methyl]-1*H*-1,2,3-triazol-1-yl}acetate

Crystal data

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$\alpha = 73.823$ (2)°
$\beta = 87.226$ (2)°
$\gamma = 88.566$ (2)°
$V = 807.59$ (5) Å^3
$Z = 2$
$F(000) = 348$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3560 reflections
$\theta = 2.8$–27.1°
$\mu = 0.22$ mm^{-1}
$T = 296$ K
Block, yellow
0.37 × 0.34 × 0.28 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.692$, $T_{max} = 0.747$
16305 measured reflections
3560 independent reflections
2963 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.1°$, $\theta_{min} = 2.8°$
$h = -7 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.124$
$S = 1.05$
3560 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 0.3602P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
C1	0.0414 (3)	0.32734 (16)	0.35494 (13)	0.0404 (4)
C2	−0.1005 (4)	0.43140 (19)	0.35277 (16)	0.0584 (5)
H2	−0.1086	0.4960	0.2916	0.070*
C3	−0.2296 (5)	0.4392 (3)	0.4410 (2)	0.0816 (8)
H3	−0.3226	0.5097	0.4391	0.098*
C4	−0.2224 (5)	0.3437 (3)	0.5320 (2)	0.0848 (8)
H4	−0.3130	0.3488	0.5907	0.102*
C5	−0.0814 (4)	0.2415 (3)	0.53555 (16)	0.0681 (6)
H5	−0.0755	0.1775	0.5972	0.082*
C6	0.0537 (3)	0.23176 (18)	0.44807 (14)	0.0469 (4)
C7	0.4681 (3)	0.1852 (2)	0.36729 (17)	0.0589 (5)
H7A	0.5360	0.2444	0.3986	0.071*
H7B	0.5927	0.1272	0.3581	0.071*
C8	0.3767 (3)	0.25404 (16)	0.26244 (15)	0.0435 (4)
C9	0.0720 (3)	0.38039 (16)	0.16053 (13)	0.0386 (4)
H9A	−0.0999	0.3767	0.1659	0.046*
H9B	0.1268	0.3362	0.1106	0.046*
C10	0.1439 (3)	0.51384 (15)	0.12016 (12)	0.0351 (3)
C11	0.3619 (3)	0.56739 (15)	0.10351 (14)	0.0399 (4)
H11	0.5090	0.5274	0.1139	0.048*
C12	0.4837 (3)	0.79233 (16)	0.04712 (13)	0.0422 (4)
H12A	0.4078	0.8677	0.0059	0.051*
H12B	0.6199	0.7737	0.0066	0.051*
C13	0.5656 (4)	0.81404 (19)	0.14565 (15)	0.0498 (4)
C14	0.8094 (7)	0.9430 (4)	0.2083 (2)	0.1208 (14)

H14A	0.6953	0.9796	0.2482	0.145*
H14B	0.8669	0.8653	0.2541	0.145*
C15	0.9975 (6)	1.0233 (3)	0.1746 (3)	0.1089 (12)
H15A	1.0693	1.0380	0.2337	0.163*
H15B	0.9412	1.1010	0.1303	0.163*
H15C	1.1128	0.9867	0.1364	0.163*
N1	0.1656 (2)	0.31606 (12)	0.26288 (10)	0.0359 (3)
N2	−0.0215 (3)	0.60571 (14)	0.09455 (13)	0.0471 (4)
N3	0.0854 (3)	0.71393 (14)	0.06368 (13)	0.0486 (4)
N4	0.3184 (2)	0.69022 (13)	0.06899 (11)	0.0380 (3)
O1	0.4877 (3)	0.25525 (15)	0.18177 (12)	0.0619 (4)
O2	0.5302 (4)	0.7457 (2)	0.23075 (13)	0.1013 (8)
O3	0.6924 (3)	0.91691 (13)	0.12300 (11)	0.0560 (4)
S1	0.23551 (10)	0.10089 (5)	0.45316 (4)	0.06117 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0407 (9)	0.0437 (9)	0.0341 (8)	−0.0073 (7)	0.0014 (7)	−0.0060 (7)
C2	0.0686 (13)	0.0517 (11)	0.0476 (11)	0.0061 (10)	0.0149 (10)	−0.0052 (9)
C3	0.097 (2)	0.0739 (16)	0.0688 (16)	0.0123 (14)	0.0299 (14)	−0.0176 (13)
C4	0.098 (2)	0.104 (2)	0.0478 (13)	0.0025 (17)	0.0262 (13)	−0.0187 (13)
C5	0.0732 (15)	0.0872 (17)	0.0330 (10)	−0.0082 (13)	0.0021 (10)	0.0015 (10)
C6	0.0438 (9)	0.0539 (10)	0.0369 (9)	−0.0068 (8)	−0.0049 (7)	−0.0015 (8)
C7	0.0386 (10)	0.0640 (13)	0.0608 (13)	0.0032 (9)	−0.0069 (9)	0.0052 (10)
C8	0.0369 (9)	0.0409 (9)	0.0481 (10)	−0.0043 (7)	−0.0002 (7)	−0.0046 (7)
C9	0.0370 (8)	0.0419 (9)	0.0338 (8)	−0.0049 (7)	−0.0042 (6)	−0.0049 (7)
C10	0.0326 (8)	0.0403 (8)	0.0291 (8)	0.0007 (6)	−0.0027 (6)	−0.0039 (6)
C11	0.0327 (8)	0.0367 (8)	0.0453 (9)	0.0027 (6)	−0.0022 (7)	−0.0031 (7)
C12	0.0479 (10)	0.0376 (8)	0.0360 (9)	−0.0069 (7)	−0.0020 (7)	−0.0014 (7)
C13	0.0559 (11)	0.0526 (11)	0.0380 (10)	−0.0085 (9)	−0.0009 (8)	−0.0073 (8)
C14	0.153 (3)	0.155 (3)	0.0655 (18)	−0.077 (3)	−0.0210 (19)	−0.039 (2)
C15	0.117 (3)	0.116 (3)	0.096 (2)	−0.047 (2)	−0.038 (2)	−0.0240 (19)
N1	0.0348 (7)	0.0363 (7)	0.0326 (7)	−0.0022 (5)	−0.0014 (5)	−0.0028 (5)
N2	0.0332 (7)	0.0468 (8)	0.0536 (9)	0.0020 (6)	−0.0060 (6)	−0.0009 (7)
N3	0.0394 (8)	0.0432 (8)	0.0555 (10)	0.0059 (6)	−0.0070 (7)	−0.0008 (7)
N4	0.0358 (7)	0.0362 (7)	0.0368 (7)	−0.0004 (5)	−0.0022 (5)	−0.0018 (6)
O1	0.0508 (8)	0.0707 (10)	0.0586 (9)	0.0096 (7)	0.0109 (7)	−0.0119 (7)
O2	0.1505 (19)	0.1102 (15)	0.0355 (9)	−0.0617 (14)	−0.0002 (10)	−0.0028 (9)
O3	0.0673 (9)	0.0560 (8)	0.0456 (8)	−0.0148 (7)	−0.0102 (6)	−0.0131 (6)
S1	0.0544 (3)	0.0533 (3)	0.0578 (3)	−0.0006 (2)	−0.0062 (2)	0.0150 (2)

Geometric parameters (Å, º)

C1—C2	1.388 (3)	C9—H9B	0.9700
C1—C6	1.399 (2)	C10—N2	1.351 (2)
C1—N1	1.421 (2)	C10—C11	1.362 (2)
C2—C3	1.379 (3)	C11—N4	1.339 (2)

C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.378 (4)	C12—N4	1.448 (2)
C3—H3	0.9300	C12—C13	1.500 (3)
C4—C5	1.365 (4)	C12—H12A	0.9700
C4—H4	0.9300	C12—H12B	0.9700
C5—C6	1.394 (3)	C13—O2	1.191 (2)
C5—H5	0.9300	C13—O3	1.322 (2)
C6—S1	1.751 (2)	C14—C15	1.381 (4)
C7—C8	1.507 (3)	C14—O3	1.446 (3)
C7—S1	1.800 (2)	C14—H14A	0.9700
C7—H7A	0.9700	C14—H14B	0.9700
C7—H7B	0.9700	C15—H15A	0.9600
C8—O1	1.217 (2)	C15—H15B	0.9600
C8—N1	1.363 (2)	C15—H15C	0.9600
C9—N1	1.473 (2)	N2—N3	1.314 (2)
C9—C10	1.495 (2)	N3—N4	1.335 (2)
C9—H9A	0.9700		

C2—C1—C6	119.07 (17)	C11—C10—C9	131.21 (15)
C2—C1—N1	120.41 (15)	N4—C11—C10	104.97 (14)
C6—C1—N1	120.48 (17)	N4—C11—H11	127.5
C3—C2—C1	120.2 (2)	C10—C11—H11	127.5
C3—C2—H2	119.9	N4—C12—C13	111.39 (14)
C1—C2—H2	119.9	N4—C12—H12A	109.4
C4—C3—C2	120.8 (2)	C13—C12—H12A	109.4
C4—C3—H3	119.6	N4—C12—H12B	109.4
C2—C3—H3	119.6	C13—C12—H12B	109.4
C5—C4—C3	119.6 (2)	H12A—C12—H12B	108.0
C5—C4—H4	120.2	O2—C13—O3	125.37 (19)
C3—C4—H4	120.2	O2—C13—C12	124.90 (19)
C4—C5—C6	120.9 (2)	O3—C13—C12	109.65 (15)
C4—C5—H5	119.5	C15—C14—O3	112.4 (3)
C6—C5—H5	119.5	C15—C14—H14A	109.1
C5—C6—C1	119.4 (2)	O3—C14—H14A	109.1
C5—C6—S1	120.78 (16)	C15—C14—H14B	109.1
C1—C6—S1	119.84 (15)	O3—C14—H14B	109.1
C8—C7—S1	111.53 (13)	H14A—C14—H14B	107.8
C8—C7—H7A	109.3	C14—C15—H15A	109.5
S1—C7—H7A	109.3	C14—C15—H15B	109.5
C8—C7—H7B	109.3	H15A—C15—H15B	109.5
S1—C7—H7B	109.3	C14—C15—H15C	109.5
H7A—C7—H7B	108.0	H15A—C15—H15C	109.5
O1—C8—N1	121.92 (17)	H15B—C15—H15C	109.5
O1—C8—C7	121.56 (17)	C8—N1—C1	123.85 (14)
N1—C8—C7	116.51 (16)	C8—N1—C9	116.72 (14)
N1—C9—C10	113.91 (13)	C1—N1—C9	119.33 (14)
N1—C9—H9A	108.8	N3—N2—C10	109.05 (14)
C10—C9—H9A	108.8	N2—N3—N4	106.88 (14)

N1—C9—H9B	108.8	N3—N4—C11	111.00 (14)
C10—C9—H9B	108.8	N3—N4—C12	119.89 (14)
H9A—C9—H9B	107.7	C11—N4—C12	128.89 (14)
N2—C10—C11	108.10 (14)	C13—O3—C14	116.59 (19)
N2—C10—C9	120.68 (14)	C6—S1—C7	95.49 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.59	3.445 (3)	154

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.