Crystal structure of 2-(benzo[*d*]thiazol-2-yl)-3,3-bis(ethylsulfanyl)acrylonitrile

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In the title compound, $C_{14}H_{14}N_2S_3$, the double-bond system of the acrylonitrile moiety is significantly non-planar, with absolute *cis* torsion angles of 13.9 (2) and 15.1 (2)°. The ring system and the double bond system subtend an interplanar angle of 11.16 (4)°. The wide angle $C-C(CN)=C$ of 129.40 (12)° may be associated with a balance between planarity and avoidance of a very short S···S contact.

1. Chemical context

Research into medicinal chemistry based on benzothiazoles has become a fast developing and progressively more active topic. The high degree of structural diversity has proved to be important in the search for new effective treatments (Ammazzalorso *et al.*, 2020; Elgemeie, 1989). A large number of therapeutic agents based on benzothiazole systems have been synthesized and evaluated in terms of their pharmacological properties (Gill *et al.*, 2015; Fathy *et al.*, 1988). Much information about benzothiazoles has been reported in the scientific literature, describing their anti-inflammatory, anti-microbial, neuroprotective, anticonvulsant and anti-proliferative effects (Seenaiah *et al.*, 2014). The molecular mechanisms responsible for this variety of pharmacological activity have not been completely established, and various biological pathways have been indicated as possible targets of this class of molecules (Keri *et al.*, 2015). We are engaged in developing synthetic strategies for benzothaizole systems that show important biological activity as novel antimicrobial and antiviral agents (Azzam *et al.* 2017*a*,*b*, 2020*a*,*b*,*c*, 2021; Elgemeie *et al.*, 2000*a*,*b*; 2020).

As an extension of this research (Fathy & Elgemeie, 1988; Elgemeie & Elghandour, 1990), we report here a novel benzothiazole cyanoketene dithioacetal (**2**). Compound **2** was synthesized by the reaction of 2-cyanomethylbenzothiazole **1** with carbon disulfide in the presence of sodium ethoxide, followed by alkylation with ethyl iodide. The structure of **2** was originally based on its elemental analysis and spectroscopic data (see *Experimental*). In order to establish the structure of the compound unambiguously, the crystal structure was determined.

2. Structural commentary

The molecule of **2** is shown in Fig. 1. The heterocyclic system is coplanar to within an r.m.s. deviation of only 0.007 Å, and its
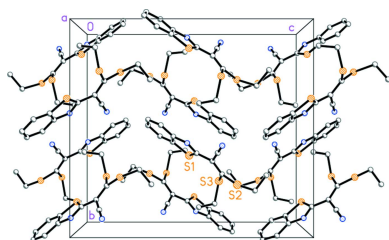
dimensions are as expected (a selection of molecular dimensions are presented in Table 1). There is appreciable twisting of *ca* 14° about the double bond C8=C9 (see torsion angles in Table 1), so that the 'plane' of the atoms C2, C8, C9, C10, S2 and S3 displays an r.m.s. deviation of 0.14 Å; the two planes subtend an interplanar angle of 11.16 (4)°. The angle C2—C8=C9 (formally sp^2) is strikingly wide, at 129.40 (12)°; for comparison, the corresponding angles in the five structures mentioned below (with refcodes) range from 122–126°. One might speculate that this large angle and the deviation from planarity about the double bond represent aspects of a compromise between (i) achieving coplanarity of the heterocycle with the double-bond system and (ii) avoiding too short an S⋯S contact. The intramolecular S⋯S distances are S1⋯S3 = 3.1155 (5) and S2⋯S3 = 3.0496 (5) Å. The ethyl groups project to opposite sides of the molecule.
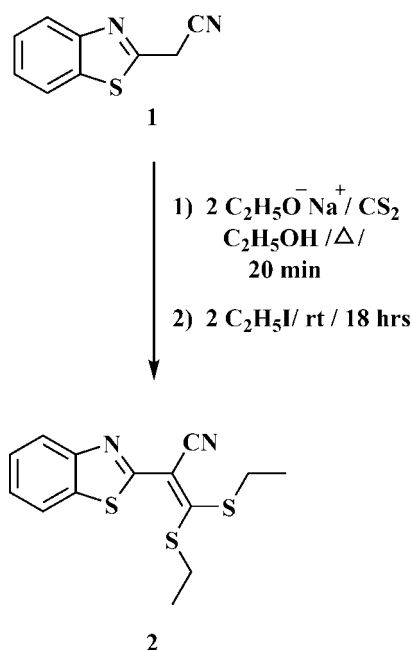
3. Supramolecular features

The molecular packing is fairly featureless; a general view is given in Fig. 2 and some borderline possible 'weak' hydrogen bonds are listed in Table 2. The main feature is the loose association of pairs of molecules across inversion centres, whereby the heterocyclic systems face each other; however, there is a considerable offset. The centroids of the five-membered rings lie 3.72 Å apart, and the shortest contact is C7A⋯C7A′ (operator 1 − x, 1 − y, 1 − z) 3.741 (2) Å. The sulfur atom S1 lies 3.61 Å from the centroid of the six-membered ring in the facing molecule; such potential S⋯π interactions have been discussed by *e.g.* Ringer *et al.* (2007) and Silva *et al.* (2018).

4. Database survey

Searches of the Cambridge Structural Database (Groom *et al.*, 2016) were performed using *ConQuest* Version 2021.3.0. A

Table 1
Selected geometric parameters (Å, °).

S1—C7A	1.7371 (13)	C3A—C7A	1.4057 (17)
S1—C2	1.7519 (13)	C9—S3	1.7489 (13)
C2—N3	1.3078 (16)	C9—S2	1.7526 (13)
N3—C3A	1.3813 (16)		
C7A—S1—C2	88.97 (6)	C9—C8—C2	129.40 (12)
N3—C2—S1	115.52 (9)	C10—C8—C2	111.90 (10)
C2—N3—C3A	110.98 (11)	C8—C9—S3	121.13 (10)
N3—C3A—C7A	115.03 (11)	C8—C9—S2	117.68 (10)
C3A—C7A—S1	109.49 (9)	S3—C9—S2	121.14 (7)
C9—C8—C10	118.69 (11)		
C2—C8—C9—S3	13.90 (19)	C2—C8—C9—S2	−163.46 (10)
C10—C8—C9—S2	15.10 (16)	C8—C9—S2—C11	−146.43 (10)

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7⋯S2^i	0.95	3.02	3.6083 (13)	122
C12—H12B⋯S1^{ii}	0.98	3.03	3.9677 (15)	161
C13—H13A⋯N3^i	0.99	2.68	3.5746 (17)	151
C14—H14A⋯S1^{iii}	0.98	2.91	3.7648 (15)	146
C14—H14B⋯N3^{iv}	0.98	2.63	3.5277 (18)	152

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

search for the moiety benzo[*d*]thiazol-2-yl joined to C(CN)=C gave 27 hits, but none in which any further atom at the double bond was sulfur. A search for the group C—C(CN)=C(S—C)$_2$, with the first carbon atom three-coordinate, both sulfur atoms two-coordinate and not involving cyclicity, gave only five hits. The refcodes, references and absolute *cis* torsion angles NC—C=C—S were as follows: CIYDIY, Kumar *et al.* (2008), 9.9°; MTBCEY, Abrahamsson *et al.* (1974), 15.4°; VAPJAA, Azzam *et al.* (2017c), 7.3°; VELSIP, Peng *et al.* (2006), 3.6°; ZEDJEX, Osaka *et al.* (1994), 10.5°.

5. Synthesis and crystallization

A mixture of sodium ethoxide (0.08 mol) and 2-cyanomethylbenzothiazole (0.04 mol) in absolute ethanol (100 ml) was refluxed for 20 min. After cooling, carbon disulfide
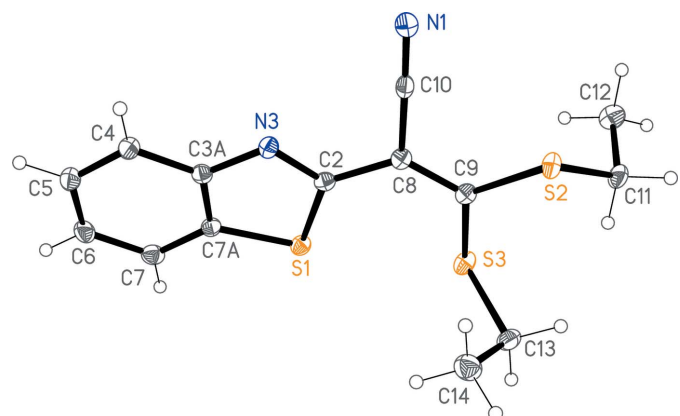
Figure 1
The molecule of **2** in the crystal. Ellipsoids represent 50% probability levels.
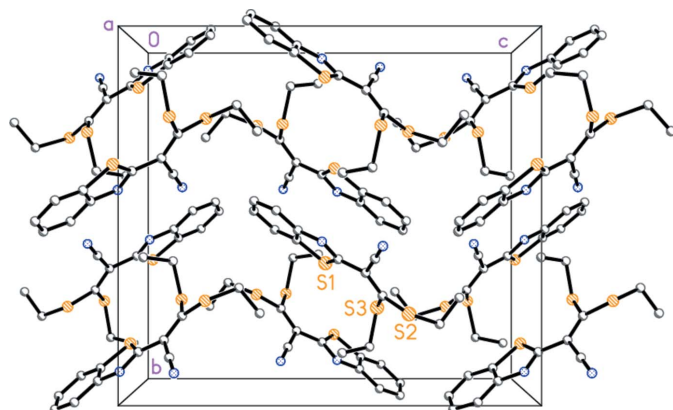
Figure 2
Crystal packing of **2** viewed parallel to the *a* axis (hydrogen atoms omitted for clarity). The loose association of the heterocyclic systems can be recognized in the central horizontal rows of rings.

(0.04 mol) was added gradually and then the solution was warmed for 20 min. Ethyl iodide (0.08 mol) was then added, and the reaction mixture was stirred overnight at room temperature. The solution was poured onto ice–water and the solid product thus formed was filtered off. The product was purified by dissolving it in hot petroleum ether, filtering, and allowing the solution to cool. The solid that formed was recrystallized from DMF to give pale-yellow crystals, m.p. = 366–368 K, yield 72%; IR (KBr, cm^{-1}): υ 3056 (ArCH), 2924 (CH$_3$), 2213 (CN), 1502 (C=N); ^{1}H NMR (300 MHz, DMSO-d_6): δ 1.27–1.34 (*m*, 6H, 2 SCH$_2$CH$_3$), 3.16–3.23 (*m*, 4H, 2 SCH$_2$CH$_3$), 7.50–7.57 (*m*, 2H, benzothiazole H), 8.04–8.15 (*m*, 2H, benzothiazole H); analysis, calculated for C$_{14}$H$_{14}$N$_2$S$_3$ (306.47): C% 54.87; H% 4.60; N% 9.14; S% 31.39; found: C% 54.85, H% 4.58; N% 9.16; MS *m/z* (%): 306 (*M*$^+$, 15%), 276 (100%), 273 (57%), 248 (26%), 217 (76%), 204 (26%), 146 (20%).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The methyl groups were refined as idealized rigid groups allowed to rotate but not tip, with C—H = 0.98 Å and H—C—H = 109.5°. Other hydrogen atoms were included using a riding model starting from calculated positions (C—H$_{aromatic}$ = 0.95, C—H$_{methylene}$ = 0.99 Å). The U(H) values were fixed at 1.5 or 1.2 times the equivalent U_{iso} value of the parent carbon atoms for methyl and non-methyl hydrogen atoms, respectively.

Acknowledgements

Table 3
Experimental details.

Crystal data	
Chemical formula	C$_{14}$H$_{14}$N$_2$S$_3$
M_r	306.45
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	100
a, *b*, *c* (Å)	10.0771 (3), 16.0292 (5), 17.8768 (6)
V (Å^3)	2887.58 (16)
Z	8
Radiation type	Mo *K*α
μ (mm^{-1})	0.50
Crystal size (mm)	0.4 × 0.4 × 0.15
Data collection	
Diffractometer	Oxford Diffraction Xcalibur, Eos
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.954, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	58593, 4475, 3679
R_{int}	0.053
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.729
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.077, 1.05
No. of reflections	4475
No. of parameters	174
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.33

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015) and *XP* (Siemens, 1994).

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Crystal structure of 2-(benzo[*d*]thiazol-2-yl)-3,3-bis(ethylsulfanyl)acrylonitrile

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL2018/3* (Sheldrick, 2015).

2-(Benzo[*d*]thiazol-2-yl)-3,3-bis(ethylsulfanyl)acrylonitrile

Crystal data

$C_{14}H_{14}N_2S_3$	$D_x = 1.410$ Mg m^{-3}
$M_r = 306.45$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, *Pbca*	Cell parameters from 10579 reflections
$a = 10.0771$ (3) Å	$\theta = 2.6$–$30.3°$
$b = 16.0292$ (5) Å	$\mu = 0.50$ mm^{-1}
$c = 17.8768$ (6) Å	$T = 100$ K
$V = 2887.58$ (16) Å^3	Tablet, pale yellow
$Z = 8$	$0.4 \times 0.4 \times 0.15$ mm
$F(000) = 1280$	

Data collection

Oxford Diffraction Xcalibur, Eos diffractometer	58593 measured reflections
Radiation source: Enhance (Mo) X-ray Source	4475 independent reflections
Graphite monochromator	3679 reflections with $I > 2\sigma(I)$
Detector resolution: 16.1419 pixels mm^{-1}	$R_{int} = 0.053$
ω–scan	$\theta_{max} = 31.2°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan (CrysAlisPro; Agilent, 2014)	$h = -14 \rightarrow 14$
	$k = -23 \rightarrow 22$
	$l = -25 \rightarrow 25$
$T_{min} = 0.954$, $T_{max} = 1.000$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.032$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.077$	
$S = 1.05$	H-atom parameters constrained
4475 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2 + 1.3474P]$ where $P = (F_o^2 + 2F_c^2)/3$
174 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.002$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.40$ e Å^{-3}
	$\Delta\rho_{min} = -0.32$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
S1	0.48621 (3)	0.63526 (2)	0.48864 (2)	0.01440 (8)
C2	0.64877 (12)	0.62079 (7)	0.52085 (7)	0.0129 (2)
N3	0.72242 (10)	0.57082 (6)	0.48059 (6)	0.0141 (2)
C3A	0.65225 (12)	0.54010 (7)	0.42020 (7)	0.0135 (2)
C4	0.70382 (14)	0.48489 (8)	0.36681 (7)	0.0169 (2)
H4	0.793137	0.466208	0.369750	0.020*
C5	0.62153 (14)	0.45833 (8)	0.30983 (7)	0.0194 (3)
H5	0.654822	0.420983	0.273082	0.023*
C6	0.48945 (14)	0.48568 (8)	0.30536 (7)	0.0198 (3)
H6	0.435044	0.466538	0.265499	0.024*
C7	0.43675 (14)	0.53990 (8)	0.35774 (7)	0.0179 (3)
H7	0.347076	0.557869	0.354695	0.022*
C7A	0.51995 (12)	0.56737 (7)	0.41532 (7)	0.0141 (2)
C8	0.70646 (12)	0.65707 (7)	0.58882 (7)	0.0135 (2)
C9	0.66224 (12)	0.72227 (7)	0.63140 (7)	0.0137 (2)
C10	0.82871 (13)	0.61618 (8)	0.60980 (7)	0.0147 (2)
N1	0.92273 (12)	0.58066 (7)	0.62713 (6)	0.0200 (2)
S2	0.77453 (3)	0.76792 (2)	0.69408 (2)	0.01682 (8)
C11	0.67428 (14)	0.80013 (8)	0.77350 (7)	0.0190 (3)
H11A	0.723828	0.842357	0.802710	0.023*
H11B	0.591943	0.826694	0.754915	0.023*
C12	0.63786 (15)	0.72831 (9)	0.82435 (8)	0.0224 (3)
H12A	0.584732	0.687626	0.796466	0.034*
H12B	0.586321	0.749299	0.866859	0.034*
H12C	0.718942	0.701444	0.842693	0.034*
S3	0.50142 (3)	0.76155 (2)	0.62083 (2)	0.01684 (8)
C13	0.53315 (14)	0.87150 (8)	0.60082 (8)	0.0192 (3)
H13A	0.447342	0.900815	0.594773	0.023*
H13B	0.579883	0.896822	0.643920	0.023*
C14	0.61552 (15)	0.88404 (9)	0.53106 (8)	0.0242 (3)
H14A	0.701489	0.856282	0.537151	0.036*
H14B	0.629538	0.943837	0.522807	0.036*
H14C	0.568927	0.860130	0.487976	0.036*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01247 (14)	0.01380 (15)	0.01694 (15)	0.00051 (11)	0.00032 (11)	−0.00103 (11)
C2	0.0125 (5)	0.0120 (5)	0.0143 (5)	−0.0005 (4)	0.0015 (4)	0.0019 (4)

N3	0.0150 (5)	0.0129 (5)	0.0143 (5)	−0.0007 (4)	0.0011 (4)	−0.0009 (4)
C3A	0.0158 (6)	0.0119 (5)	0.0129 (5)	−0.0019 (4)	0.0006 (4)	0.0020 (4)
C4	0.0197 (6)	0.0152 (6)	0.0158 (6)	0.0014 (5)	0.0009 (5)	−0.0003 (5)
C5	0.0268 (7)	0.0158 (6)	0.0157 (6)	0.0009 (5)	−0.0002 (5)	−0.0016 (5)
C6	0.0261 (7)	0.0171 (6)	0.0163 (6)	−0.0023 (5)	−0.0067 (5)	−0.0011 (5)
C7	0.0184 (6)	0.0153 (6)	0.0200 (6)	−0.0014 (5)	−0.0039 (5)	0.0021 (5)
C7A	0.0173 (6)	0.0106 (5)	0.0144 (5)	−0.0013 (4)	0.0003 (5)	0.0012 (4)
C8	0.0131 (6)	0.0127 (5)	0.0145 (5)	−0.0018 (4)	0.0018 (4)	0.0008 (4)
C9	0.0134 (6)	0.0127 (5)	0.0150 (6)	−0.0017 (4)	0.0024 (4)	0.0010 (4)
C10	0.0182 (6)	0.0137 (5)	0.0122 (5)	−0.0011 (5)	0.0010 (4)	−0.0028 (4)
N1	0.0218 (6)	0.0205 (5)	0.0177 (5)	0.0028 (5)	−0.0026 (4)	−0.0029 (4)
S2	0.01627 (15)	0.01690 (16)	0.01730 (15)	−0.00122 (12)	0.00140 (11)	−0.00523 (12)
C11	0.0250 (7)	0.0161 (6)	0.0159 (6)	0.0035 (5)	0.0025 (5)	−0.0049 (5)
C12	0.0231 (7)	0.0208 (7)	0.0232 (7)	0.0015 (5)	0.0051 (5)	0.0012 (5)
S3	0.01226 (15)	0.01522 (15)	0.02304 (17)	0.00007 (11)	0.00338 (11)	−0.00326 (12)
C13	0.0198 (6)	0.0139 (6)	0.0240 (7)	0.0024 (5)	−0.0015 (5)	−0.0002 (5)
C14	0.0270 (7)	0.0238 (7)	0.0218 (7)	−0.0025 (6)	−0.0011 (6)	0.0031 (5)

Geometric parameters (Å, °)

S1—C7A	1.7371 (13)	C9—S3	1.7489 (13)
S1—C2	1.7519 (13)	C9—S2	1.7526 (13)
C2—N3	1.3078 (16)	C10—N1	1.1480 (17)
C2—C8	1.4672 (17)	S2—C11	1.8174 (13)
N3—C3A	1.3813 (16)	C11—C12	1.5121 (19)
C3A—C4	1.4015 (17)	C11—H11A	0.9900
C3A—C7A	1.4057 (17)	C11—H11B	0.9900
C4—C5	1.3807 (18)	C12—H12A	0.9800
C4—H4	0.9500	C12—H12B	0.9800
C5—C6	1.404 (2)	C12—H12C	0.9800
C5—H5	0.9500	S3—C13	1.8265 (14)
C6—C7	1.3835 (19)	C13—C14	1.512 (2)
C6—H6	0.9500	C13—H13A	0.9900
C7—C7A	1.3988 (18)	C13—H13B	0.9900
C7—H7	0.9500	C14—H14A	0.9800
C8—C9	1.3675 (17)	C14—H14B	0.9800
C8—C10	1.4449 (18)	C14—H14C	0.9800
C7A—S1—C2	88.97 (6)	S3—C9—S2	121.14 (7)
N3—C2—C8	118.28 (11)	N1—C10—C8	177.05 (14)
N3—C2—S1	115.52 (9)	C9—S2—C11	105.02 (6)
C8—C2—S1	126.18 (9)	C12—C11—S2	112.85 (9)
C2—N3—C3A	110.98 (11)	C12—C11—H11A	109.0
N3—C3A—C4	124.58 (12)	S2—C11—H11A	109.0
N3—C3A—C7A	115.03 (11)	C12—C11—H11B	109.0
C4—C3A—C7A	120.38 (12)	S2—C11—H11B	109.0
C5—C4—C3A	118.32 (12)	H11A—C11—H11B	107.8
C5—C4—H4	120.8	C11—C12—H12A	109.5

C3A—C4—H4	120.8	C11—C12—H12B	109.5
C4—C5—C6	121.01 (12)	H12A—C12—H12B	109.5
C4—C5—H5	119.5	C11—C12—H12C	109.5
C6—C5—H5	119.5	H12A—C12—H12C	109.5
C7—C6—C5	121.44 (12)	H12B—C12—H12C	109.5
C7—C6—H6	119.3	C9—S3—C13	101.91 (6)
C5—C6—H6	119.3	C14—C13—S3	112.70 (10)
C6—C7—C7A	117.76 (12)	C14—C13—H13A	109.1
C6—C7—H7	121.1	S3—C13—H13A	109.1
C7A—C7—H7	121.1	C14—C13—H13B	109.1
C7—C7A—C3A	121.08 (12)	S3—C13—H13B	109.1
C7—C7A—S1	129.43 (10)	H13A—C13—H13B	107.8
C3A—C7A—S1	109.49 (9)	C13—C14—H14A	109.5
C9—C8—C10	118.69 (11)	C13—C14—H14B	109.5
C9—C8—C2	129.40 (12)	H14A—C14—H14B	109.5
C10—C8—C2	111.90 (10)	C13—C14—H14C	109.5
C8—C9—S3	121.13 (10)	H14A—C14—H14C	109.5
C8—C9—S2	117.68 (10)	H14B—C14—H14C	109.5
C7A—S1—C2—N3	0.65 (10)	C2—S1—C7A—C7	178.99 (13)
C7A—S1—C2—C8	−177.35 (11)	C2—S1—C7A—C3A	−0.97 (9)
C8—C2—N3—C3A	178.07 (10)	N3—C2—C8—C9	165.41 (12)
S1—C2—N3—C3A	−0.10 (13)	S1—C2—C8—C9	−16.64 (19)
C2—N3—C3A—C4	−179.85 (12)	N3—C2—C8—C10	−13.23 (16)
C2—N3—C3A—C7A	−0.71 (15)	S1—C2—C8—C10	164.72 (9)
N3—C3A—C4—C5	179.09 (12)	C10—C8—C9—S3	−167.54 (9)
C7A—C3A—C4—C5	−0.01 (18)	C2—C8—C9—S3	13.90 (19)
C3A—C4—C5—C6	−0.11 (19)	C10—C8—C9—S2	15.10 (16)
C4—C5—C6—C7	−0.2 (2)	C2—C8—C9—S2	−163.46 (10)
C5—C6—C7—C7A	0.5 (2)	C8—C9—S2—C11	−146.43 (10)
C6—C7—C7A—C3A	−0.63 (19)	S3—C9—S2—C11	36.21 (9)
C6—C7—C7A—S1	179.41 (10)	C9—S2—C11—C12	77.62 (11)
N3—C3A—C7A—C7	−178.79 (11)	C8—C9—S3—C13	−123.70 (11)
C4—C3A—C7A—C7	0.39 (18)	S2—C9—S3—C13	53.57 (9)
N3—C3A—C7A—S1	1.17 (13)	C9—S3—C13—C14	59.99 (11)
C4—C3A—C7A—S1	−179.64 (10)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···S2[i]	0.95	3.02	3.6083 (13)	122
C12—H12B···S1[ii]	0.98	3.03	3.9677 (15)	161
C13—H13A···N3[i]	0.99	2.68	3.5746 (17)	151
C14—H14A···S1[iii]	0.98	2.91	3.7648 (15)	146
C14—H14B···N3[iv]	0.98	2.63	3.5277 (18)	152

Symmetry codes: (i) $x-1/2$, $-y+3/2$, $-z+1$; (ii) x, $-y+3/2$, $z+1/2$; (iii) $x+1/2$, $-y+3/2$, $-z+1$; (iv) $-x+3/2$, $y+1/2$, z.