

(2*E*,3*E*)-2,3-Bis(1,3-dithiolan-2-ylidene-hydrazono)butaneLing-Juan Yang,^a Zhi-Gang Li,^b Xiao-Lan Liu^c and Yong-Hong Liu^{c*}^aCollege of Life Sciences and Chemistry, Tianshui Normal University, Tianshui 741000, People's Republic of China, ^bTianshui Institute for Drug Control, Tianshui 741018, People's Republic of China, and ^cCollege of Chemistry and Chemical Engineering, Yangzhou University, Yangzhou, 225002, People's Republic of China
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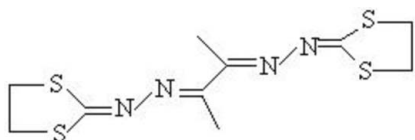
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Key indicators: single-crystal X-ray study; *T* = 294 K; mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$; disorder in main residue; *R* factor = 0.041; *wR* factor = 0.117; data-to-parameter ratio = 16.1.

The title compound, $\text{C}_{10}\text{H}_{14}\text{N}_4\text{S}_4$, which exists as the 2*E*,3*E* isomer, does not contain a center of inversion; the conformations of the two halves are different. The conformation of the 1,3-dithiolane ring on one side of the molecule is in the half-chair form but that on the other side is in the envelope form, in which the C atoms are disordered over two positions with relative occupancies of 0.635 (19) and 0.365 (19). There are C—H···N intramolecular hydrogen bonds, and C—H···N and C—H···S intermolecular hydrogen bonds, which stabilize the crystal structure.

Related literature

For related literature, see: Beghidja *et al.* (2006); Bernstein *et al.* (1995); Cremer & Pople (1975); Gou *et al.* (2004); Liu *et al.* (2007); Wang *et al.* (1994); Xu *et al.* (2005).

**Experimental***Crystal data*

$\text{C}_{10}\text{H}_{14}\text{N}_4\text{S}_4$ $V = 1448.6 (5) \text{ \AA}^3$
 $M_r = 318.49$ $Z = 4$
Monoclinic, $P2_1/n$ Mo $K\alpha$ radiation
 $a = 8.0210 (17) \text{ \AA}$ $\mu = 0.64 \text{ mm}^{-1}$
 $b = 17.939 (4) \text{ \AA}$ $T = 294 (2) \text{ K}$
 $c = 10.227 (2) \text{ \AA}$ $0.26 \times 0.20 \times 0.10 \text{ mm}$
 $\beta = 100.108 (3)^\circ$

Data collection

Bruker SMART 1000 CCD 8087 measured reflections
diffractometer 2963 independent reflections
Absorption correction: multi-scan 2041 reflections with $I > 2\sigma(I)$
(SADABS; Bruker, 1997; $R_{\text{int}} = 0.030$
Blessing, 1995)
 $T_{\text{min}} = 0.749$, $T_{\text{max}} = 0.957$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$ 2 restraints
 $wR(F^2) = 0.117$ H-atom parameters constrained
 $S = 1.02$ $\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
2963 reflections $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
184 parameters

Table 1

Table 1.

Hydrogen-bond geometry (\AA , $^\circ$)

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···N1	0.96	2.32	2.737 (4)	105
C7—H7A···N4	0.96	2.32	2.745 (3)	106
C9'—H9'A···N4 ⁱ	0.97	2.57	3.457 (10)	152
C9'—H9'A···S4 ⁱ	0.97	2.967	3.890 (5)	160
C10—H10A···N1 ⁱⁱ	0.97	2.593	3.641 (5)	163
C2—H2A ⁱⁱⁱ ···N4	0.97	2.628	3.563 (5)	162

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXTL (Bruker, 1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2077).

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supplementary materials

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(2*E*,3*E*)-2,3-Bis(1,3-dithiolan-2-ylidenehydrazono)butane

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Comment

The Schiff's bases and carbonyl derivatives of 2-hydrazono-1, 3-dithiolane have been abstracted for their coordination chemistry and biological activity (Beghidja *et al.*, 2006; Wang *et al.*, 1994, Gou *et al.*, 2004; Xu *et al.*, 2005). As ongoing research (Liu *et al.*, 2007) we report herein the synthesis and structure of a novel bi-Schiff's base, derived from condensation of butanedione and 2-hydrazono-1, 3-dithiolane.

The molecule of the title compound exists as the most stable configuration of (*E*, *E*)-isomer (Fig.1). In the molecule there are two intramolecular C—H···N hydrogen-bonds forming two rings with graph sets $S(5)$ (Bernstein *et al.*, 1995) and directly influencing the coplanarity of the atoms involved (Fig. 1, Table 2). Due to conjugation and intramolecular hydrogen-bonds, the atoms C3, N1, N2, C4, C5, C6, C7, N3, N4 and C8 are coplanar. The dihedral angles to the S1- C3 - S2 and S1- C3 - S2 planes are 17.02 (2) and 6.62 (18)°, respectively.

In the two 1,3-dithiolane rings of the molecule, the conformation of the ring defined by S1, C3, S2, C1 and C2 is in the half-chair form (Cremer & Pople, 1975; Xu *et al.*, 2005) and atom C1, C2 deviates by -0.292 (4) Å, 0.353 (4) Å from this plane. In the other ring C9 and C10 are disordered over two positions with relative occupancies of 0.635 (19) and 0.365 (19) for the major and minor components. The ring for the major component is also the half-chair form, but that for the minor component is in the envelope form similar to that for (3*E*)-3-(1,3-dithiolan-2-ylidenehydrazono)butane (Liu *et al.*, 2007). This is shown by the fact that atoms C9, C10, C9' and C10' deviate by -0.37 (2), 0.33 (2), 0.236 (10) Å and -0.376 (11) Å from the ring plane. The molecule does not possess a center of inversion and the solid crystal is stabilized by C—H···N intramolecular hydrogen-bonds, C—H···N and C—H···S intermolecular hydrogen-bonds (Table 1).

Experimental

2-Hydrazono-1, 3-dithiolane (38.5 mmol) and butanedione (19.0 mmol) in EtOH (35 cm³) were refluxed for 4 h. Then solvent was removed on a vacuum rotary evaporator. Crude product (2.85 g, 90% yield) was recrystallized from CH₂Cl₂–EtOH to give crystals of suitable for single-crystal X-ray diffraction (yield 82%, m.p. 474 – 476 K). ¹H NMR (600 MHz, CDCl₃, δ, p.p.m.): 2.31 (3H, s, CH₃), 3.49 (S, 4H, CH₂CH₂). ¹³C NMR (600 MHz, CDCl₃, δ, p.p.m.): 177.017 (N=N=C), 162.956 (CS₂), 36.454, 34.858 (CH₂CH₂), 11.605 (CH₃).

Refinement

The C9 and C10 atoms were refined as disordered with a refined occupancy of 68 (2)% for the major component. The anisotropic displacement parameters of C9, C9', C10 and C10' were constrained to be equal. After their location in a difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C atoms, with C—H distances of 0.96 (methyl) or 0.97 Å (CH₂), and with $U_{\text{iso}}(\text{H})$ values of 1.2U_{eq}(C), or 1.5U_{eq}(C) for the methyl groups.

Figures

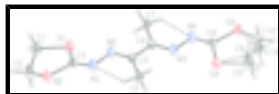


Fig. 1. The molecular of (I) structure of the title compound, showing 40% probability ellipsoids. The C — H \cdots N intramolecular hydrogen bond is shown dashed.

(2E,3E)-2,3-Bis(1,3-dithiolan-2-ylidenehydrazono)butane

Crystal data

$C_{10}H_{14}N_4S_4$	$F_{000} = 664$
$M_r = 318.49$	$D_x = 1.460 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Melting point: 392 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation
$a = 8.0210 (17) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 17.939 (4) \text{ \AA}$	Cell parameters from 2725 reflections
$c = 10.227 (2) \text{ \AA}$	$\theta = 2.8\text{--}25.8^\circ$
$\beta = 100.108 (3)^\circ$	$\mu = 0.64 \text{ mm}^{-1}$
$V = 1448.6 (5) \text{ \AA}^3$	$T = 294 (2) \text{ K}$
$Z = 4$	Block, light yellow
	$0.26 \times 0.20 \times 0.10 \text{ mm}$

Data collection

Bruker SMART 1000 CCD diffractometer	2963 independent reflections
Radiation source: fine-focus sealed tube	2041 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.030$
$T = 294(2) \text{ K}$	$\theta_{\text{max}} = 26.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 1997; Blessing, 1995)	$h = -10 \rightarrow 8$
$T_{\text{min}} = 0.749$, $T_{\text{max}} = 0.957$	$k = -11 \rightarrow 22$
8087 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.5471P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
2963 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
184 parameters	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
2 restraints	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
	Extinction correction: none

Primary atom site location: structure-invariant direct methods

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.22062 (12)	0.49090 (4)	0.59864 (7)	0.0650 (3)	
S2	0.35239 (11)	0.64030 (4)	0.68331 (7)	0.0618 (2)	
S3	0.15131 (10)	0.32739 (4)	1.38926 (7)	0.0565 (2)	
S4	-0.00920 (10)	0.18337 (4)	1.29964 (7)	0.0551 (2)	
N1	0.2561 (3)	0.54408 (12)	0.8478 (2)	0.0537 (6)	
N2	0.1831 (3)	0.47375 (12)	0.8615 (2)	0.0483 (6)	
N3	0.1317 (3)	0.35516 (11)	1.1187 (2)	0.0466 (5)	
N4	0.0610 (3)	0.28473 (12)	1.1336 (2)	0.0502 (6)	
C1	0.2486 (5)	0.5562 (2)	0.4687 (3)	0.0820 (11)	
H1A	0.2807	0.5295	0.3944	0.098*	
H1B	0.1431	0.5821	0.4373	0.098*	
C2	0.3846 (5)	0.61137 (19)	0.5233 (3)	0.0748 (10)	
H2A	0.3794	0.6541	0.4646	0.090*	
H2B	0.4952	0.5885	0.5296	0.090*	
C3	0.2716 (3)	0.55535 (14)	0.7272 (2)	0.0416 (6)	
C4	0.1932 (3)	0.45185 (14)	0.9825 (2)	0.0406 (6)	
C5	0.2773 (4)	0.49329 (16)	1.1025 (3)	0.0584 (8)	
H5A	0.3003	0.5434	1.0781	0.088*	
H5B	0.3816	0.4690	1.1393	0.088*	
H5C	0.2040	0.4941	1.1673	0.088*	
C6	0.1144 (3)	0.37859 (13)	0.9983 (2)	0.0397 (6)	
C7	0.0266 (4)	0.33730 (15)	0.8782 (2)	0.0527 (7)	
H7A	-0.0031	0.2882	0.9037	0.079*	
H7B	0.1008	0.3335	0.8144	0.079*	
H7C	-0.0742	0.3637	0.8396	0.079*	
C8	0.0689 (3)	0.26931 (13)	1.2569 (2)	0.0401 (6)	
C9	0.069 (3)	0.2724 (7)	1.5139 (18)	0.061 (4)	0.365 (19)
H9A	0.1365	0.2809	1.6010	0.074*	0.365 (19)
H9B	-0.0471	0.2867	1.5166	0.074*	0.365 (19)
C10	0.076 (3)	0.1905 (7)	1.4760 (11)	0.058 (4)	0.365 (19)
H10A	0.0099	0.1606	1.5270	0.069*	0.365 (19)

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H10B	0.1925	0.1728	1.4939	0.069*	0.365 (19)
C9'	0.1410 (12)	0.2578 (7)	1.5165 (10)	0.062 (3)	0.635 (19)
H9'A	0.2456	0.2294	1.5331	0.075*	0.635 (19)
H9'B	0.1267	0.2820	1.5985	0.075*	0.635 (19)
C10'	-0.0065 (14)	0.2060 (6)	1.4707 (8)	0.062 (2)	0.635 (19)
H10C	-0.1118	0.2302	1.4806	0.075*	0.635 (19)
H10D	0.0055	0.1610	1.5239	0.075*	0.635 (19)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.1024 (7)	0.0511 (5)	0.0419 (4)	-0.0163 (4)	0.0141 (4)	-0.0022 (3)
S2	0.0877 (6)	0.0469 (4)	0.0525 (4)	-0.0160 (4)	0.0171 (4)	0.0085 (3)
S3	0.0695 (5)	0.0580 (5)	0.0412 (4)	-0.0175 (4)	0.0074 (3)	-0.0053 (3)
S4	0.0760 (5)	0.0417 (4)	0.0467 (4)	-0.0098 (3)	0.0081 (3)	0.0084 (3)
N1	0.0805 (17)	0.0404 (12)	0.0429 (12)	-0.0119 (11)	0.0178 (12)	0.0026 (10)
N2	0.0656 (15)	0.0409 (12)	0.0404 (11)	-0.0083 (10)	0.0145 (11)	0.0023 (9)
N3	0.0628 (15)	0.0406 (12)	0.0379 (11)	-0.0070 (10)	0.0129 (10)	0.0023 (9)
N4	0.0752 (16)	0.0385 (12)	0.0371 (12)	-0.0076 (11)	0.0101 (11)	0.0013 (9)
C1	0.123 (3)	0.087 (3)	0.0379 (16)	-0.018 (2)	0.0186 (18)	0.0094 (16)
C2	0.106 (3)	0.069 (2)	0.0579 (19)	-0.009 (2)	0.0368 (19)	0.0114 (16)
C3	0.0467 (15)	0.0375 (14)	0.0408 (13)	-0.0007 (11)	0.0080 (11)	0.0052 (11)
C4	0.0468 (15)	0.0397 (14)	0.0372 (13)	0.0042 (11)	0.0127 (11)	0.0007 (10)
C5	0.080 (2)	0.0497 (17)	0.0447 (15)	-0.0114 (15)	0.0072 (14)	0.0007 (12)
C6	0.0467 (15)	0.0378 (13)	0.0367 (13)	0.0024 (11)	0.0127 (11)	-0.0005 (10)
C7	0.072 (2)	0.0464 (15)	0.0402 (14)	-0.0066 (14)	0.0111 (13)	0.0022 (12)
C8	0.0454 (15)	0.0375 (14)	0.0373 (13)	0.0003 (11)	0.0068 (11)	0.0012 (10)
C9	0.080 (12)	0.068 (7)	0.036 (5)	0.019 (8)	0.010 (8)	0.012 (4)
C10	0.071 (10)	0.073 (7)	0.029 (4)	0.002 (7)	0.007 (6)	0.016 (4)
C9'	0.059 (5)	0.090 (7)	0.035 (3)	-0.008 (4)	0.001 (4)	0.007 (4)
C10'	0.062 (5)	0.074 (5)	0.050 (3)	-0.009 (4)	0.012 (4)	0.018 (3)

Geometric parameters (\AA , $^\circ$)

S1—C3	1.744 (3)	C2—H2B	0.9700
S1—C1	1.814 (3)	C4—C6	1.480 (3)
S2—C3	1.745 (3)	C4—C5	1.491 (4)
S2—C2	1.778 (3)	C5—H5A	0.9600
S3—C8	1.743 (2)	C5—H5B	0.9600
S3—C9'	1.816 (11)	C5—H5C	0.9600
S3—C9	1.825 (19)	C6—C7	1.500 (4)
S4—C8	1.748 (2)	C7—H7A	0.9600
S4—C10'	1.792 (8)	C7—H7B	0.9600
S4—C10	1.819 (12)	C7—H7C	0.9600
N1—C3	1.278 (3)	C9—C10	1.524 (9)
N1—N2	1.408 (3)	C9—H9A	0.9700
N2—C4	1.287 (3)	C9—H9B	0.9700
N3—C6	1.286 (3)	C10—H10A	0.9700
N3—N4	1.404 (3)	C10—H10B	0.9700

N4—C8	1.282 (3)	C9'—C10'	1.512 (7)
C1—C2	1.506 (5)	C9'—H9'A	0.9700
C1—H1A	0.9700	C9'—H9'B	0.9700
C1—H1B	0.9700	C10'—H10C	0.9700
C2—H2A	0.9700	C10'—H10D	0.9700
C3—S1—C1	94.67 (14)	N3—C6—C4	114.7 (2)
C3—S2—C2	95.86 (14)	N3—C6—C7	125.4 (2)
C8—S3—C9'	94.9 (3)	C4—C6—C7	119.9 (2)
C8—S3—C9	94.8 (6)	C6—C7—H7A	109.5
C9'—S3—C9	20.0 (4)	C6—C7—H7B	109.5
C8—S4—C10'	95.8 (3)	H7A—C7—H7B	109.5
C8—S4—C10	95.6 (4)	C6—C7—H7C	109.5
C10'—S4—C10	22.8 (4)	H7A—C7—H7C	109.5
C3—N1—N2	110.5 (2)	H7B—C7—H7C	109.5
C4—N2—N1	114.3 (2)	N4—C8—S3	125.53 (19)
C6—N3—N4	114.9 (2)	N4—C8—S4	118.57 (19)
C8—N4—N3	110.5 (2)	S3—C8—S4	115.90 (14)
C2—C1—S1	109.1 (2)	C10—C9—S3	108.0 (11)
C2—C1—H1A	109.9	C10—C9—H9A	110.1
S1—C1—H1A	109.9	S3—C9—H9A	110.1
C2—C1—H1B	109.9	C10—C9—H9B	110.1
S1—C1—H1B	109.9	S3—C9—H9B	110.1
H1A—C1—H1B	108.3	H9A—C9—H9B	108.4
C1—C2—S2	108.7 (2)	C9—C10—S4	107.1 (11)
C1—C2—H2A	110.0	C9—C10—H10A	110.3
S2—C2—H2A	110.0	S4—C10—H10A	110.3
C1—C2—H2B	110.0	C9—C10—H10B	110.3
S2—C2—H2B	110.0	S4—C10—H10B	110.3
H2A—C2—H2B	108.3	H10A—C10—H10B	108.5
N1—C3—S1	125.0 (2)	C10'—C9'—S3	109.4 (7)
N1—C3—S2	119.3 (2)	C10'—C9'—H9'A	109.8
S1—C3—S2	115.71 (14)	S3—C9'—H9'A	109.8
N2—C4—C6	114.8 (2)	C10'—C9'—H9'B	109.8
N2—C4—C5	125.6 (2)	S3—C9'—H9'B	109.8
C6—C4—C5	119.6 (2)	H9'A—C9'—H9'B	108.2
C4—C5—H5A	109.5	C9'—C10'—S4	108.4 (7)
C4—C5—H5B	109.5	C9'—C10'—H10C	110.0
H5A—C5—H5B	109.5	S4—C10'—H10C	110.0
C4—C5—H5C	109.5	C9'—C10'—H10D	110.0
H5A—C5—H5C	109.5	S4—C10'—H10D	110.0
H5B—C5—H5C	109.5	H10C—C10'—H10D	108.4
C3—N1—N2—C4	167.0 (2)	N3—N4—C8—S4	-179.09 (17)
C6—N3—N4—C8	-173.4 (2)	C9'—S3—C8—N4	-171.9 (4)
C3—S1—C1—C2	31.8 (3)	C9—S3—C8—N4	168.1 (6)
S1—C1—C2—S2	-43.7 (3)	C9'—S3—C8—S4	8.3 (4)
C3—S2—C2—C1	33.1 (3)	C9—S3—C8—S4	-11.7 (6)
N2—N1—C3—S1	-3.2 (3)	C10'—S4—C8—N4	-167.6 (5)
N2—N1—C3—S2	177.76 (18)	C10—S4—C8—N4	169.5 (7)

supplementary materials

C1—S1—C3—N1	171.7 (3)	C10'—S4—C8—S3	12.2 (4)
C1—S1—C3—S2	-9.3 (2)	C10—S4—C8—S3	-10.7 (7)
C2—S2—C3—N1	167.6 (3)	C8—S3—C9—C10	35.3 (18)
C2—S2—C3—S1	-11.5 (2)	C9'—S3—C9—C10	-55.8 (16)
N1—N2—C4—C6	179.0 (2)	S3—C9—C10—S4	-46 (2)
N1—N2—C4—C5	-1.7 (4)	C8—S4—C10—C9	34.5 (18)
N4—N3—C6—C4	-178.3 (2)	C10'—S4—C10—C9	-57.1 (17)
N4—N3—C6—C7	0.3 (4)	C8—S3—C9'—C10'	-31.0 (11)
N2—C4—C6—N3	176.8 (2)	C9—S3—C9'—C10'	59.5 (19)
C5—C4—C6—N3	-2.5 (4)	S3—C9'—C10'—S4	43.1 (14)
N2—C4—C6—C7	-1.9 (4)	C8—S4—C10'—C9'	-33.1 (11)
C5—C4—C6—C7	178.8 (2)	C10—S4—C10'—C9'	57.6 (14)
N3—N4—C8—S3	1.2 (3)		

Table 1

Hydrogen-bond geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
C5—H5A...N1	0.96	2.32	2.737 (4)	105
C7—H7A...N4	0.96	2.32	2.745 (3)	106
C9'—H9'A...N4 ⁱ	0.97	2.57	3.457 (10)	152
C9'—H9'A...S4 ⁱ	0.97	2.967	3.890 (5)	160
C10—H10A...N1 ⁱⁱ	0.97	2.593	3.641 (5)	163
C2—H2A ⁱⁱⁱ ...N4	0.97	2.628	3.563 (5)	162

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

Fig. 1

