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4-Cyclohexyl-3-[(3,5-dimethyl-1*H*-pyrazol-1-yl)-methyl]-4,5-dihydro-1*H*-1,2,4-triazole-5-thione

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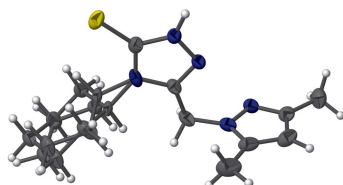
CCDC reference: 1525650

Structural data: full structural data are available from iucrdata.iucr.org

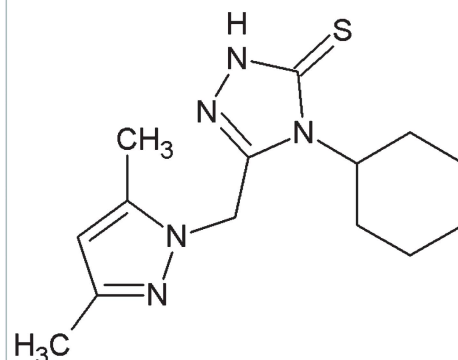
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In the title compound, C₁₄H₂₁N₅S, the dihedral angle between the triazole ring and the pyrazole ring is 88.16 (7)°. The cyclohexyl ring is disordered over two chair conformations in a 0.720 (3):0.280 (3) ratio. In the crystal, the molecules are linked by N—H···N hydrogen bonds to generate *C*(7) chains propagating in [001]. The chains are cross-linked by very weak C—H···S hydrogen bonds to generate (100) sheets.

3D view



Chemical scheme



Structure description

As part of our ongoing studies of pyrazole derivatives (Mague *et al.*, 2015), we report herein the synthesis and crystal structure of the title compound (Fig. 1).

The dihedral angle between the mean planes of the triazole and pyrazole rings is 88.16 (7)°. The cyclohexyl ring is disordered over two chair conformations in a 0.720 (3):0.280 (3) ratio. In the crystal, the molecules form (100) layers through a combination of N3—H3···N5ⁱ, C9—H9A···S1ⁱⁱ and C9—H9B···S1ⁱⁱⁱ (symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$) hydrogen bonds (Table 1 and Fig. 2).

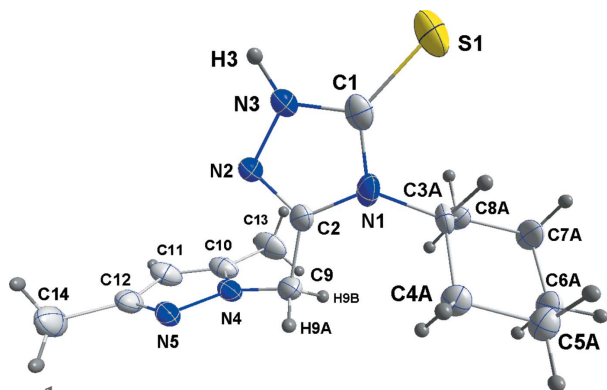


Figure 1
The title molecule with 50% probability ellipsoids. Only the major component of the disordered cyclohexane ring is shown.

Synthesis and crystallization

A solution of 1-[2-(3,5-dimethyl-1*H*-pyrazol-1-yl)acetyl]-4-cyclohexyl thiosemicarbazides (1.23 g, 4 mmol) in ethanol (50 ml) was added dropwise to 2 *N* sodium hydroxide solution (20 ml). The reaction mixture was refluxed for 2 h, cooled and filtered. The filtrate was acidified with 2 *N* hydrochloric acid solution. The separated solid was collected, washed with water and recrystallized from ethanol solution to yield colourless plates.

Refinement

Crystal and refinement details appear in Table 2. The cyclohexyl group is disordered in a 72:28 ratio over two chair conformations related by an approximately 180° rotation about the N1–C3 bond. The two components were refined with restraints that their geometries be comparable.

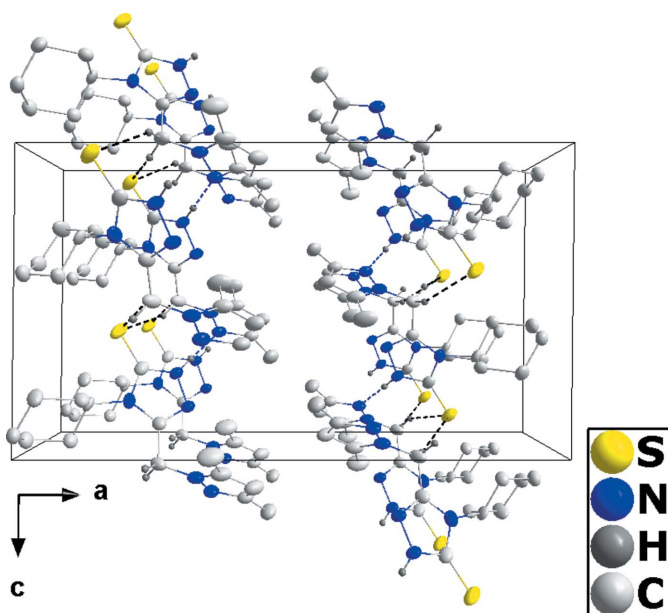


Figure 2
Packing viewed along the *b* axis. N–H···N and C–H···S hydrogen bonds are shown, respectively, as blue and black dotted lines.

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N3–H3···N5 ⁱ	0.90 (3)	1.91 (3)	2.805 (2)	175 (3)
C9–H9A···S1 ⁱⁱ	0.96 (3)	2.93 (3)	3.766 (3)	147 (2)
C9–H9B···S1 ⁱⁱⁱ	0.98 (3)	2.88 (3)	3.761 (2)	151 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₄ H ₂₁ N ₅ S
<i>M_r</i>	291.42
Crystal system, space group	Monoclinic, <i>P</i> ₂ / <i>c</i>
Temperature (K)	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	20.7954 (6), 6.2107 (2), 11.7871 (3)
β (°)	90.653 (1)
<i>V</i> (Å ³)	1522.25 (8)
<i>Z</i>	4
Radiation type	Cu Kα
μ (mm ^{−1})	1.87
Crystal size (mm)	0.34 × 0.19 × 0.02
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T_{min}</i> , <i>T_{max}</i>	0.83, 0.97
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	11034, 2951, 2589
<i>R_{int}</i>	0.034
(sin θ/λ) _{max} (Å ^{−1})	0.618
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.048, 0.115, 1.05
No. of reflections	2951
No. of parameters	215
No. of restraints	13
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ^{−3})	0.65, −0.82

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL 20147* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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full crystallographic data

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4-Cyclohexyl-3-[(3,5-dimethyl-1*H*-pyrazol-1-yl)methyl]-4,5-dihydro-1*H*-1,2,4-triazole-5-thione

Joel T. Mague, Shaaban K. Mohamed, Mehmet Akkurt, Adel A. Marzouk, Hamdy M. Abdel-Rahman and Farouq E. Hawaiz

4-Cyclohexyl-3-[(3,5-dimethyl-1*H*-pyrazol-1-yl)methyl]-4,5-dihydro-1-*H*-1,2,4-triazole-5-thione

Crystal data

C₁₄H₂₁N₃S

M_r = 291.42

Monoclinic, *P*2₁/*c*

a = 20.7954 (6) Å

b = 6.2107 (2) Å

c = 11.7871 (3) Å

β = 90.653 (1)°

V = 1522.25 (8) Å³

Z = 4

F(000) = 624

D_x = 1.272 Mg m⁻³

Cu *K*α radiation, λ = 1.54178 Å

Cell parameters from 8361 reflections

θ = 4.3–72.4°

μ = 1.87 mm⁻¹

T = 150 K

Plate, colourless

0.34 × 0.19 × 0.02 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer

Radiation source: INCOATEC IμS micro-focus source

Mirror monochromator

Detector resolution: 10.4167 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2016)

T_{min} = 0.83, *T_{max}* = 0.97

11034 measured reflections

2951 independent reflections

2589 reflections with *I* > 2σ(*I*)

R_{int} = 0.034

θ_{max} = 72.4°, θ_{min} = 4.3°

h = -25→25

k = -7→7

l = -14→14

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.048

wR(*F*²) = 0.115

S = 1.05

2951 reflections

215 parameters

13 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ²(*F_o*²) + (0.0372*P*)² + 1.6394*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.65 e Å⁻³

Δρ_{min} = -0.82 e Å⁻³

Extinction correction: *SHELXL* 2014/7

(Sheldrick, 2015*b*),

*F_c** = *kF_c*[1 + 0.001*xF_c*²λ³/sin(2θ)]^{-1/4}

Extinction coefficient: 0.0022 (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.

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. The H-atoms of the methyl groups and the disordered cyclohexyl group were placed in calculated positions (C—H = 0.98 - 1.00 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. The cyclohexyl group is disordered in a 72:28 ratio over two chair conformations related by an approximately 180° rotation about the N1—C3 bond. The two components were refined with restraints that their geometries be comparable.

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	0.81796 (3)	0.38503 (9)	0.90485 (5)	0.03905 (19)	
N1	0.78874 (8)	0.5335 (3)	0.68895 (14)	0.0306 (4)	
N2	0.68920 (8)	0.4124 (3)	0.66233 (13)	0.0288 (4)	
N3	0.71376 (8)	0.3535 (3)	0.76686 (14)	0.0294 (4)	
H3	0.6906 (13)	0.264 (5)	0.810 (2)	0.051 (8)*	
N4	0.66611 (9)	0.6146 (3)	0.45832 (13)	0.0299 (4)	
N5	0.64407 (8)	0.4451 (3)	0.39492 (13)	0.0288 (4)	
C1	0.77356 (10)	0.4235 (3)	0.78605 (17)	0.0297 (4)	
C2	0.73585 (9)	0.5221 (3)	0.61729 (16)	0.0281 (4)	
C3A	0.85297 (11)	0.6382 (5)	0.6856 (2)	0.0252 (6)	0.720 (3)
H3A	0.8808	0.5706	0.7454	0.030*	0.720 (3)
C4A	0.88585 (13)	0.6111 (4)	0.5723 (2)	0.0277 (6)	0.720 (3)
H4A1	0.8608	0.6850	0.5119	0.033*	0.720 (3)
H4A2	0.8889	0.4564	0.5527	0.033*	0.720 (3)
C5A	0.9536 (2)	0.7096 (8)	0.5823 (7)	0.0329 (6)	0.720 (3)
H5A1	0.9791	0.6286	0.6394	0.039*	0.720 (3)
H5A2	0.9755	0.6973	0.5085	0.039*	0.720 (3)
C6A	0.95014 (14)	0.9463 (5)	0.6170 (3)	0.0322 (6)	0.720 (3)
H6A1	0.9943	1.0031	0.6277	0.039*	0.720 (3)
H6A2	0.9291	1.0300	0.5555	0.039*	0.720 (3)
C7A	0.91282 (15)	0.9761 (6)	0.7263 (3)	0.0386 (9)	0.720 (3)
H7A1	0.9092	1.1315	0.7438	0.046*	0.720 (3)
H7A2	0.9362	0.9057	0.7898	0.046*	0.720 (3)
C8A	0.84542 (19)	0.8779 (7)	0.7143 (6)	0.0279 (9)	0.720 (3)
H8A1	0.8210	0.9520	0.6532	0.033*	0.720 (3)
H8A2	0.8217	0.8947	0.7861	0.033*	0.720 (3)
C3B	0.8402 (2)	0.6828 (9)	0.6466 (6)	0.0252 (6)	0.280 (3)
H3B	0.8259	0.7426	0.5717	0.030*	0.280 (3)
C4B	0.9015 (3)	0.5558 (9)	0.6290 (7)	0.0277 (6)	0.280 (3)
H4B1	0.8935	0.4395	0.5732	0.033*	0.280 (3)
H4B2	0.9151	0.4886	0.7016	0.033*	0.280 (3)

C5B	0.9551 (5)	0.703 (2)	0.5861 (16)	0.0329 (6)	0.280 (3)
H5B1	0.9951	0.6191	0.5771	0.039*	0.280 (3)
H5B2	0.9427	0.7626	0.5110	0.039*	0.280 (3)
C6B	0.9673 (3)	0.8887 (11)	0.6694 (7)	0.0322 (6)	0.280 (3)
H6B1	0.9820	0.8306	0.7435	0.039*	0.280 (3)
H6B2	1.0013	0.9842	0.6399	0.039*	0.280 (3)
C7B	0.9054 (3)	1.0165 (10)	0.6848 (8)	0.0386 (9)	0.280 (3)
H7B1	0.8920	1.0805	0.6113	0.046*	0.280 (3)
H7B2	0.9131	1.1353	0.7393	0.046*	0.280 (3)
C8B	0.8516 (5)	0.8710 (18)	0.7286 (15)	0.0279 (9)	0.280 (3)
H8B1	0.8115	0.9555	0.7361	0.033*	0.280 (3)
H8B2	0.8637	0.8146	0.8045	0.033*	0.280 (3)
C9	0.73190 (11)	0.6154 (4)	0.50055 (17)	0.0335 (5)	
H9A	0.7575 (12)	0.531 (4)	0.450 (2)	0.040 (7)*	
H9B	0.7465 (12)	0.765 (5)	0.502 (2)	0.049 (7)*	
C10	0.61843 (12)	0.7525 (3)	0.48504 (17)	0.0371 (5)	
C11	0.56341 (12)	0.6719 (4)	0.43495 (19)	0.0408 (5)	
H11	0.5216	0.7330	0.4370	0.049*	
C12	0.58160 (10)	0.4813 (3)	0.38033 (16)	0.0322 (5)	
C13	0.63050 (16)	0.9470 (4)	0.5565 (2)	0.0550 (7)	
H13A	0.6662	1.0298	0.5249	0.083*	
H13B	0.6415	0.9023	0.6341	0.083*	
H13C	0.5917	1.0366	0.5575	0.083*	
C14	0.54153 (11)	0.3250 (4)	0.3135 (2)	0.0436 (6)	
H14A	0.5054	0.4012	0.2778	0.065*	
H14B	0.5251	0.2134	0.3643	0.065*	
H14C	0.5679	0.2582	0.2548	0.065*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0416 (3)	0.0356 (3)	0.0395 (3)	0.0083 (2)	-0.0179 (2)	-0.0107 (2)
N1	0.0252 (8)	0.0328 (9)	0.0339 (9)	-0.0084 (7)	0.0007 (7)	-0.0110 (7)
N2	0.0313 (9)	0.0318 (9)	0.0230 (8)	-0.0086 (7)	-0.0048 (6)	0.0040 (7)
N3	0.0313 (9)	0.0328 (9)	0.0240 (8)	-0.0045 (7)	-0.0065 (7)	0.0030 (7)
N4	0.0439 (10)	0.0247 (9)	0.0212 (8)	-0.0062 (7)	-0.0003 (7)	-0.0013 (6)
N5	0.0377 (9)	0.0260 (8)	0.0227 (8)	-0.0044 (7)	-0.0013 (7)	-0.0036 (7)
C1	0.0310 (10)	0.0274 (10)	0.0307 (10)	0.0022 (8)	-0.0039 (8)	-0.0089 (8)
C2	0.0314 (10)	0.0271 (10)	0.0257 (9)	-0.0076 (8)	0.0005 (7)	-0.0049 (8)
C3A	0.0185 (12)	0.0288 (14)	0.0280 (17)	-0.0030 (10)	-0.0098 (10)	-0.0019 (11)
C4A	0.0282 (13)	0.0230 (13)	0.0321 (16)	0.0004 (10)	0.0022 (11)	-0.0052 (11)
C5A	0.0267 (10)	0.0341 (12)	0.0378 (12)	0.0011 (9)	0.0029 (9)	-0.0017 (10)
C6A	0.0279 (14)	0.0344 (16)	0.0343 (17)	-0.0093 (12)	-0.0010 (12)	-0.0006 (12)
C7A	0.0365 (15)	0.0430 (18)	0.036 (2)	-0.0149 (13)	-0.0011 (15)	-0.0158 (16)
C8A	0.0307 (13)	0.0267 (11)	0.0263 (19)	-0.0035 (9)	0.0038 (13)	-0.0061 (10)
C3B	0.0185 (12)	0.0288 (14)	0.0280 (17)	-0.0030 (10)	-0.0098 (10)	-0.0019 (11)
C4B	0.0282 (13)	0.0230 (13)	0.0321 (16)	0.0004 (10)	0.0022 (11)	-0.0052 (11)
C5B	0.0267 (10)	0.0341 (12)	0.0378 (12)	0.0011 (9)	0.0029 (9)	-0.0017 (10)

C6B	0.0279 (14)	0.0344 (16)	0.0343 (17)	-0.0093 (12)	-0.0010 (12)	-0.0006 (12)
C7B	0.0365 (15)	0.0430 (18)	0.036 (2)	-0.0149 (13)	-0.0011 (15)	-0.0158 (16)
C8B	0.0307 (13)	0.0267 (11)	0.0263 (19)	-0.0035 (9)	0.0038 (13)	-0.0061 (10)
C9	0.0403 (12)	0.0349 (12)	0.0252 (10)	-0.0149 (9)	0.0034 (8)	-0.0014 (8)
C10	0.0592 (14)	0.0267 (11)	0.0253 (10)	0.0048 (10)	-0.0010 (9)	0.0008 (8)
C11	0.0512 (13)	0.0419 (13)	0.0293 (11)	0.0150 (11)	-0.0032 (9)	-0.0016 (9)
C12	0.0393 (11)	0.0347 (11)	0.0224 (9)	0.0004 (9)	-0.0025 (8)	0.0008 (8)
C13	0.090 (2)	0.0327 (13)	0.0417 (14)	0.0082 (13)	-0.0048 (13)	-0.0115 (11)
C14	0.0403 (12)	0.0544 (15)	0.0358 (12)	-0.0051 (11)	-0.0074 (9)	-0.0073 (11)

Geometric parameters (Å, °)

S1—C1	1.686 (2)	C3B—C4B	1.514 (4)
N1—C1	1.373 (3)	C3B—C8B	1.534 (5)
N1—C2	1.381 (3)	C3B—H3B	1.0000
N1—C3A	1.487 (3)	C4B—C5B	1.534 (5)
N1—C3B	1.506 (4)	C4B—H4B1	0.9900
N2—C2	1.303 (2)	C4B—H4B2	0.9900
N2—N3	1.378 (2)	C5B—C6B	1.532 (5)
N3—C1	1.334 (3)	C5B—H5B1	0.9900
N3—H3	0.90 (3)	C5B—H5B2	0.9900
N4—C10	1.350 (3)	C6B—C7B	1.525 (4)
N4—N5	1.367 (2)	C6B—H6B1	0.9900
N4—C9	1.451 (3)	C6B—H6B2	0.9900
N5—C12	1.328 (3)	C7B—C8B	1.531 (5)
C2—C9	1.495 (3)	C7B—H7B1	0.9900
C3A—C4A	1.516 (3)	C7B—H7B2	0.9900
C3A—C8A	1.536 (4)	C8B—H8B1	0.9900
C3A—H3A	1.0000	C8B—H8B2	0.9900
C4A—C5A	1.539 (4)	C9—H9A	0.96 (3)
C4A—H4A1	0.9900	C9—H9B	0.98 (3)
C4A—H4A2	0.9900	C10—C11	1.376 (3)
C5A—C6A	1.528 (4)	C10—C13	1.492 (3)
C5A—H5A1	0.9900	C11—C12	1.402 (3)
C5A—H5A2	0.9900	C11—H11	0.9500
C6A—C7A	1.523 (4)	C12—C14	1.497 (3)
C6A—H6A1	0.9900	C13—H13A	0.9800
C6A—H6A2	0.9900	C13—H13B	0.9800
C7A—C8A	1.533 (4)	C13—H13C	0.9800
C7A—H7A1	0.9900	C14—H14A	0.9800
C7A—H7A2	0.9900	C14—H14B	0.9800
C8A—H8A1	0.9900	C14—H14C	0.9800
C8A—H8A2	0.9900		
C1—N1—C2	107.18 (16)	C4B—C3B—H3B	108.4
C1—N1—C3A	117.09 (18)	C8B—C3B—H3B	108.4
C2—N1—C3A	135.69 (19)	C3B—C4B—C5B	110.5 (5)
C1—N1—C3B	138.9 (3)	C3B—C4B—H4B1	109.6

C2—N1—C3B	113.1 (3)	C5B—C4B—H4B1	109.6
C2—N2—N3	103.48 (16)	C3B—C4B—H4B2	109.6
C1—N3—N2	113.59 (17)	C5B—C4B—H4B2	109.6
C1—N3—H3	127.6 (17)	H4B1—C4B—H4B2	108.1
N2—N3—H3	118.4 (17)	C6B—C5B—C4B	110.7 (6)
C10—N4—N5	111.93 (17)	C6B—C5B—H5B1	109.5
C10—N4—C9	127.58 (18)	C4B—C5B—H5B1	109.5
N5—N4—C9	119.98 (17)	C6B—C5B—H5B2	109.5
C12—N5—N4	105.21 (17)	C4B—C5B—H5B2	109.5
N3—C1—N1	104.10 (17)	H5B1—C5B—H5B2	108.1
N3—C1—S1	126.53 (17)	C7B—C6B—C5B	109.5 (5)
N1—C1—S1	129.36 (16)	C7B—C6B—H6B1	109.8
N2—C2—N1	111.65 (17)	C5B—C6B—H6B1	109.8
N2—C2—C9	122.99 (18)	C7B—C6B—H6B2	109.8
N1—C2—C9	125.33 (17)	C5B—C6B—H6B2	109.8
N1—C3A—C4A	112.9 (2)	H6B1—C6B—H6B2	108.2
N1—C3A—C8A	108.9 (2)	C6B—C7B—C8B	110.7 (5)
C4A—C3A—C8A	110.5 (3)	C6B—C7B—H7B1	109.5
N1—C3A—H3A	108.1	C8B—C7B—H7B1	109.5
C4A—C3A—H3A	108.1	C6B—C7B—H7B2	109.5
C8A—C3A—H3A	108.1	C8B—C7B—H7B2	109.5
C3A—C4A—C5A	108.1 (3)	H7B1—C7B—H7B2	108.1
C3A—C4A—H4A1	110.1	C7B—C8B—C3B	110.2 (5)
C5A—C4A—H4A1	110.1	C7B—C8B—H8B1	109.6
C3A—C4A—H4A2	110.1	C3B—C8B—H8B1	109.6
C5A—C4A—H4A2	110.1	C7B—C8B—H8B2	109.6
H4A1—C4A—H4A2	108.4	C3B—C8B—H8B2	109.6
C6A—C5A—C4A	110.9 (3)	H8B1—C8B—H8B2	108.1
C6A—C5A—H5A1	109.4	N4—C9—C2	110.86 (16)
C4A—C5A—H5A1	109.5	N4—C9—H9A	108.1 (15)
C6A—C5A—H5A2	109.5	C2—C9—H9A	109.8 (15)
C4A—C5A—H5A2	109.4	N4—C9—H9B	107.5 (15)
H5A1—C5A—H5A2	108.0	C2—C9—H9B	109.7 (16)
C7A—C6A—C5A	111.7 (3)	H9A—C9—H9B	111 (2)
C7A—C6A—H6A1	109.3	N4—C10—C11	106.15 (19)
C5A—C6A—H6A1	109.3	N4—C10—C13	121.7 (2)
C7A—C6A—H6A2	109.3	C11—C10—C13	132.1 (2)
C5A—C6A—H6A2	109.3	C10—C11—C12	106.1 (2)
H6A1—C6A—H6A2	107.9	C10—C11—H11	127.0
C6A—C7A—C8A	110.4 (3)	C12—C11—H11	127.0
C6A—C7A—H7A1	109.6	N5—C12—C11	110.61 (19)
C8A—C7A—H7A1	109.6	N5—C12—C14	119.78 (19)
C6A—C7A—H7A2	109.6	C11—C12—C14	129.6 (2)
C8A—C7A—H7A2	109.6	C10—C13—H13A	109.5
H7A1—C7A—H7A2	108.1	C10—C13—H13B	109.5
C7A—C8A—C3A	108.1 (3)	H13A—C13—H13B	109.5
C7A—C8A—H8A1	110.1	C10—C13—H13C	109.5
C3A—C8A—H8A1	110.1	H13A—C13—H13C	109.5

C7A—C8A—H8A2	110.1	H13B—C13—H13C	109.5
C3A—C8A—H8A2	110.1	C12—C14—H14A	109.5
H8A1—C8A—H8A2	108.4	C12—C14—H14B	109.5
N1—C3B—C4B	109.1 (4)	H14A—C14—H14B	109.5
N1—C3B—C8B	111.5 (5)	C12—C14—H14C	109.5
C4B—C3B—C8B	111.0 (5)	H14A—C14—H14C	109.5
N1—C3B—H3B	108.4	H14B—C14—H14C	109.5
C2—N2—N3—C1	-0.1 (2)	N1—C3A—C8A—C7A	172.4 (3)
C10—N4—N5—C12	1.2 (2)	C4A—C3A—C8A—C7A	-63.0 (6)
C9—N4—N5—C12	173.48 (17)	C1—N1—C3B—C4B	72.2 (7)
N2—N3—C1—N1	-0.1 (2)	C2—N1—C3B—C4B	-119.4 (4)
N2—N3—C1—S1	179.05 (15)	C1—N1—C3B—C8B	-50.7 (9)
C2—N1—C1—N3	0.3 (2)	C2—N1—C3B—C8B	117.7 (8)
C3A—N1—C1—N3	178.24 (18)	N1—C3B—C4B—C5B	-179.9 (8)
C3B—N1—C1—N3	169.1 (4)	C8B—C3B—C4B—C5B	-56.7 (10)
C2—N1—C1—S1	-178.83 (16)	C3B—C4B—C5B—C6B	57.6 (13)
C3A—N1—C1—S1	-0.8 (3)	C4B—C5B—C6B—C7B	-58.3 (13)
C3B—N1—C1—S1	-10.0 (5)	C5B—C6B—C7B—C8B	58.5 (9)
N3—N2—C2—N1	0.3 (2)	C6B—C7B—C8B—C3B	-57.8 (12)
N3—N2—C2—C9	178.33 (19)	N1—C3B—C8B—C7B	178.6 (8)
C1—N1—C2—N2	-0.4 (2)	C4B—C3B—C8B—C7B	56.7 (12)
C3A—N1—C2—N2	-177.8 (2)	C10—N4—C9—C2	78.1 (3)
C3B—N1—C2—N2	-172.4 (3)	N5—N4—C9—C2	-92.9 (2)
C1—N1—C2—C9	-178.3 (2)	N2—C2—C9—N4	14.3 (3)
C3A—N1—C2—C9	4.2 (4)	N1—C2—C9—N4	-167.98 (18)
C3B—N1—C2—C9	9.6 (4)	N5—N4—C10—C11	-1.3 (2)
C1—N1—C3A—C4A	138.3 (2)	C9—N4—C10—C11	-172.90 (19)
C2—N1—C3A—C4A	-44.5 (4)	N5—N4—C10—C13	178.3 (2)
C1—N1—C3A—C8A	-98.6 (4)	C9—N4—C10—C13	6.7 (3)
C2—N1—C3A—C8A	78.6 (4)	N4—C10—C11—C12	0.9 (2)
N1—C3A—C4A—C5A	-175.5 (4)	C13—C10—C11—C12	-178.7 (2)
C8A—C3A—C4A—C5A	62.2 (5)	N4—N5—C12—C11	-0.6 (2)
C3A—C4A—C5A—C6A	-57.5 (7)	N4—N5—C12—C14	179.97 (18)
C4A—C5A—C6A—C7A	55.3 (7)	C10—C11—C12—N5	-0.2 (2)
C5A—C6A—C7A—C8A	-55.8 (6)	C10—C11—C12—C14	179.2 (2)
C6A—C7A—C8A—C3A	58.4 (6)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3 \cdots N5 ⁱ	0.90 (3)	1.91 (3)	2.805 (2)	175 (3)
C9—H9A \cdots S1 ⁱⁱ	0.96 (3)	2.93 (3)	3.766 (3)	147 (2)
C9—H9B \cdots S1 ⁱⁱⁱ	0.98 (3)	2.88 (3)	3.761 (2)	151 (2)

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