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5-(Adamantan-1-yl)-*N*-methyl-1,3,4-thiadiazol-2-amine

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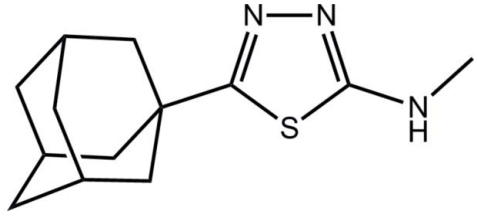
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.144; data-to-parameter ratio = 19.0.

In the title compound, $C_{13}H_{19}N_3S$, the methylamine substituent is coplanar with the thiadiazole ring to which it is attached [C—N—C—S torsion angle = 175.9 (2)°] and the amine H atom is *syn* to the thiadiazole S atom. Supramolecular chains along [101], sustained by N—H···N hydrogen bonding, feature in the crystal packing.

Related literature

For the biological activity of 1,3,4-thiadiazol-2-amine derivatives, see: Carvalho *et al.* (2008); Foroumadi *et al.* (1999), and of adamantane derivatives, see: Togo *et al.* (1968); El-Emam *et al.* (2004). For related structures, see: El-Emam *et al.* (2012); Almutairi *et al.* (2012). For the synthesis of the title compound, see: El-Emam & Lehmann (1994).



Experimental

Crystal data

$C_{13}H_{19}N_3S$
M_r = 249.37
Monoclinic, $P2_1/n$
a = 10.4394 (12) Å
b = 13.0910 (13) Å
c = 10.8871 (15) Å
β = 118.008 (16)°
V = 1313.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 295 K
0.30 × 0.20 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.887, T_{max} = 1.000
6791 measured reflections
3027 independent reflections
1975 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.144
S = 1.04
3027 reflections
159 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N1^{i}	0.87 (1)	2.15 (1)	3.021 (3)	179 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2012); 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 for Windows* (Farrugia, 2012) 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: HG5305).

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supplementary materials

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5-(Adamantan-1-yl)-*N*-methyl-1,3,4-thiadiazol-2-amine

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Comment

Derivatives of adamantane have long been known for their diverse biological activities including anti-viral activity against influenza (Togo *et al.*, 1968) and HIV viruses (El-Emam *et al.*, 2004). Moreover, 1,3,4-thiadiazole derivatives were reported to exhibit marked anti-trypanosomal (Carvalho *et al.*, 2008) and anti-microbial activities (Foroumadi *et al.*, 1999). In continuation of our interest in the chemical and pharmacological properties of adamantane derivatives, and as part of on-going structural studies of these (El-Emam *et al.*, 2012; Almutairi *et al.*, 2012), we report herein the X-ray crystallographic data of the title compound, (I).

In (I), Fig. 1, the five-membered ring is planar (r.m.s. deviation = 0.009 Å) and the methylamine substituent is co-planar: the C13—N3—C2—S1 torsion angle is 175.9 (2)°. The amine-H atom is *syn* to the thiadiazole-S1 atom. N—H···N hydrogen bonds feature in the crystal packing, leading to supramolecular chains along [1 0 1], Fig. 2 and Table 1. Chains pack with no specific intermolecular interactions between them. Globally, the crystal structure comprises alternating layers of hydrophilic and hydrophobic regions, Fig. 3.

Experimental

The title compound was prepared by dehydrative cyclization of 1-(1-adamantylcarbonyl)-4-methylthiosemicarbazide using sulfuric acid at room temperature for 24 h as previously described (El-Emam & Lehmann, 1994). Single crystals were obtained by slow evaporation from its $CHCl_3$:EtOH solution at room temperature; *M*.pt: 441–443 K.

Refinement

The C-bound H-atoms were placed in calculated positions [C—H = 0.96 to 0.98 Å, U_{iso}(H) = 1.2 or 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation. The N-bound H-atom was refined with N—H = 0.88±0.01 Å.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); 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) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

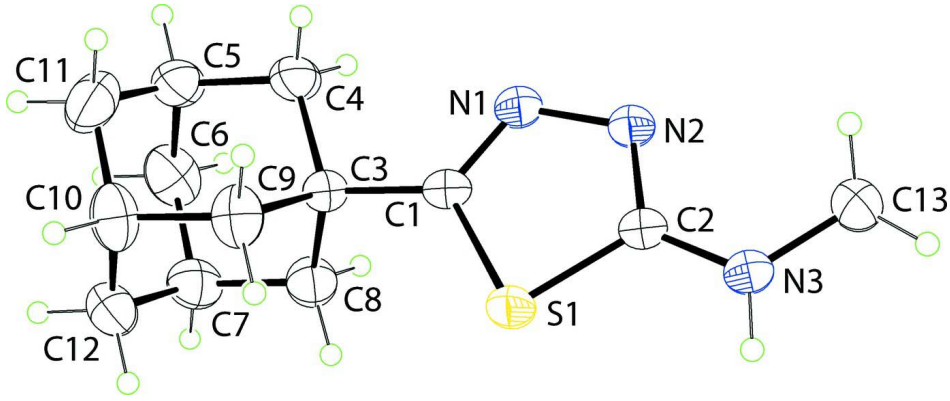

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 35% probability level.

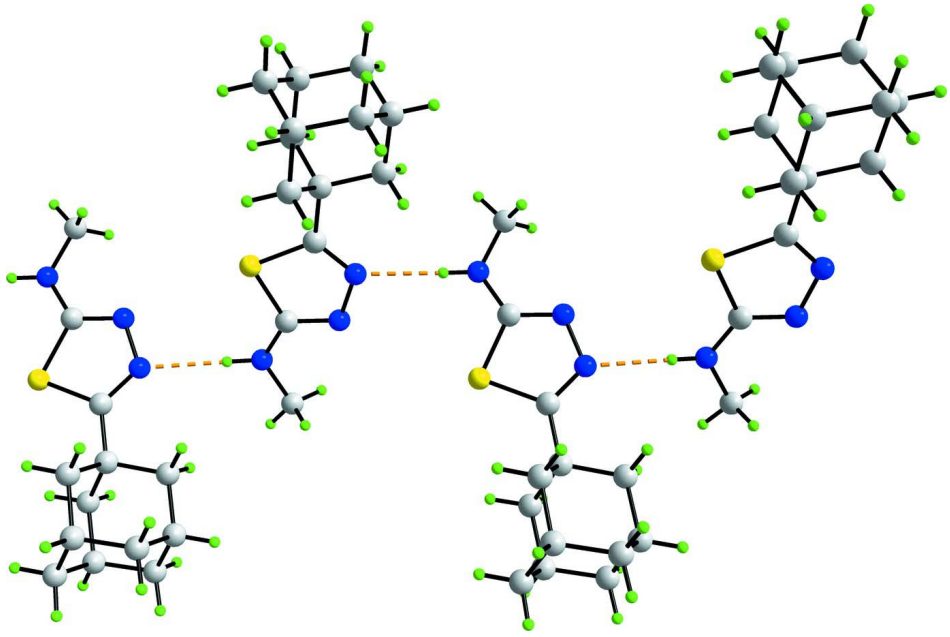

Figure 2

A view of the supramolecular chain in (I) sustained by N—H···N hydrogen bonds shown as orange dashed lines.

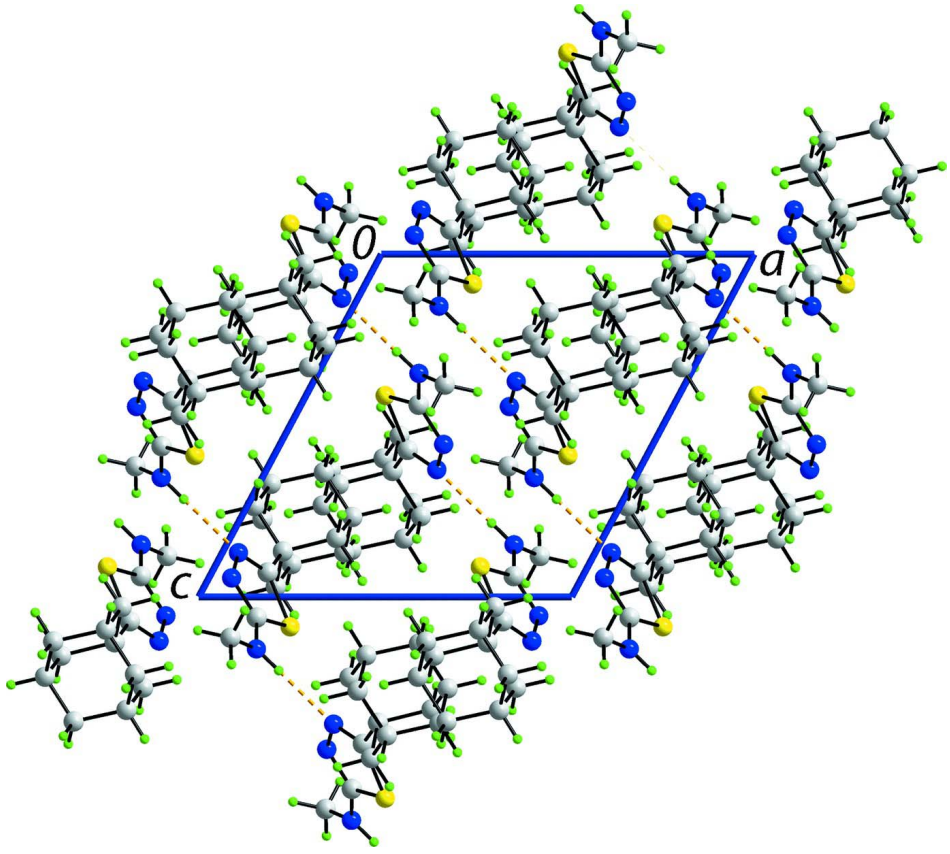

Figure 3

A view in projection down the *b* axis of the unit-cell contents for (I).

5-(Adamantan-1-yl)-*N*-methyl-1,3,4-thiadiazol-2-amine

Crystal data

$C_{13}H_{19}N_3S$
$M_r = 249.37$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.4394$ (12) Å
$b = 13.0910$ (13) Å
$c = 10.8871$ (15) Å
$\beta = 118.008$ (16)°
$V = 1313.6$ (3) Å^3
$Z = 4$

$F(000) = 536$
$D_x = 1.261$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1515 reflections
$\theta = 3.1$–27.5°
$\mu = 0.23$ mm^{-1}
$T = 295$ K
Prism, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)

$T_{min} = 0.887$, $T_{max} = 1.000$
6791 measured reflections
3027 independent reflections
1975 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.1°$
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 17$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.144$
$S = 1.04$
3027 reflections
159 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.1967P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.21237 (6)	0.64435 (5)	0.41269 (6)	0.0480 (2)
N1	0.45652 (19)	0.66351 (15)	0.62872 (19)	0.0446 (5)
N2	0.43886 (18)	0.75355 (15)	0.55520 (19)	0.0455 (5)
N3	0.2711 (2)	0.83210 (16)	0.3481 (2)	0.0514 (5)
H3	0.1801 (12)	0.8326 (19)	0.2850 (19)	0.047 (7)*
C1	0.3500 (2)	0.60016 (19)	0.5713 (2)	0.0398 (5)
C2	0.3152 (2)	0.75433 (18)	0.4397 (2)	0.0404 (5)
C3	0.3392 (2)	0.49988 (18)	0.6325 (2)	0.0398 (5)
C4	0.4805 (3)	0.4770 (2)	0.7646 (3)	0.0614 (7)
H4A	0.5016	0.5321	0.8308	0.074*
H4B	0.5598	0.4725	0.7420	0.074*
C5	0.4676 (3)	0.3761 (2)	0.8298 (3)	0.0698 (9)
H5	0.5580	0.3627	0.9150	0.084*
C6	0.3426 (3)	0.3824 (3)	0.8635 (3)	0.0744 (9)
H6A	0.3359	0.3190	0.9062	0.089*
H6B	0.3600	0.4373	0.9292	0.089*
C7	0.2019 (3)	0.4013 (2)	0.7331 (3)	0.0614 (7)
H7	0.1221	0.4047	0.7564	0.074*
C8	0.2149 (3)	0.5035 (2)	0.6717 (3)	0.0566 (7)
H8A	0.2337	0.5575	0.7391	0.068*
H8B	0.1242	0.5188	0.5896	0.068*
C9	0.3080 (3)	0.4115 (2)	0.5300 (3)	0.0605 (7)
H9A	0.3848	0.4073	0.5040	0.073*
H9B	0.2175	0.4242	0.4465	0.073*
C10	0.2977 (3)	0.3098 (2)	0.5956 (3)	0.0682 (8)

H10	0.2792	0.2540	0.5294	0.082*
C11	0.1735 (3)	0.3176 (2)	0.6301 (3)	0.0675 (8)
H11A	0.0840	0.3314	0.5459	0.081*
H11B	0.1625	0.2533	0.6683	0.081*
C12	0.4399 (3)	0.2912 (3)	0.7263 (4)	0.0807 (10)
H12A	0.4358	0.2262	0.7671	0.097*
H12B	0.5187	0.2882	0.7028	0.097*
C13	0.3567 (3)	0.9237 (2)	0.3810 (3)	0.0619 (7)
H13A	0.3160	0.9693	0.3028	0.093*
H13B	0.4544	0.9068	0.4020	0.093*
H13C	0.3570	0.9562	0.4602	0.093*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0374 (3)	0.0496 (4)	0.0422 (4)	−0.0049 (3)	0.0063 (3)	0.0015 (3)
N1	0.0365 (9)	0.0478 (12)	0.0403 (11)	0.0009 (9)	0.0105 (8)	0.0020 (9)
N2	0.0361 (9)	0.0460 (12)	0.0435 (11)	−0.0023 (9)	0.0096 (8)	0.0033 (10)
N3	0.0380 (10)	0.0503 (13)	0.0490 (13)	−0.0012 (10)	0.0063 (9)	0.0077 (11)
C1	0.0314 (10)	0.0487 (14)	0.0366 (12)	0.0015 (10)	0.0138 (9)	−0.0020 (11)
C2	0.0327 (10)	0.0455 (14)	0.0396 (12)	0.0020 (10)	0.0142 (9)	0.0001 (11)
C3	0.0366 (11)	0.0435 (13)	0.0388 (12)	0.0010 (10)	0.0171 (9)	0.0007 (11)
C4	0.0435 (13)	0.0650 (18)	0.0611 (17)	−0.0039 (13)	0.0125 (12)	0.0184 (14)
C5	0.0500 (14)	0.070 (2)	0.0689 (19)	−0.0026 (15)	0.0110 (14)	0.0242 (17)
C6	0.089 (2)	0.081 (2)	0.0559 (18)	−0.0179 (18)	0.0364 (17)	0.0018 (16)
C7	0.0560 (15)	0.073 (2)	0.0647 (18)	−0.0040 (14)	0.0364 (14)	0.0026 (15)
C8	0.0556 (14)	0.0574 (17)	0.0643 (17)	0.0008 (13)	0.0344 (13)	−0.0009 (14)
C9	0.0785 (18)	0.0535 (16)	0.0597 (17)	−0.0018 (14)	0.0408 (15)	−0.0035 (14)
C10	0.095 (2)	0.0474 (16)	0.070 (2)	−0.0086 (16)	0.0448 (18)	−0.0112 (15)
C11	0.0611 (16)	0.0589 (18)	0.075 (2)	−0.0128 (15)	0.0254 (15)	0.0020 (16)
C12	0.081 (2)	0.066 (2)	0.115 (3)	0.0235 (18)	0.062 (2)	0.034 (2)
C13	0.0534 (14)	0.0537 (17)	0.0642 (18)	−0.0072 (13)	0.0157 (13)	0.0063 (14)

Geometric parameters (Å, º)

S1—C2	1.737 (2)	C6—H6B	0.9700
S1—C1	1.747 (2)	C7—C11	1.495 (4)
N1—C1	1.289 (3)	C7—C8	1.530 (4)
N1—N2	1.388 (3)	C7—H7	0.9800
N2—C2	1.313 (3)	C8—H8A	0.9700
N3—C2	1.346 (3)	C8—H8B	0.9700
N3—C13	1.437 (3)	C9—C10	1.538 (4)
N3—H3	0.872 (9)	C9—H9A	0.9700
C1—C3	1.500 (3)	C9—H9B	0.9700
C3—C4	1.530 (3)	C10—C11	1.513 (4)
C3—C9	1.532 (3)	C10—C12	1.518 (4)
C3—C8	1.545 (3)	C10—H10	0.9800
C4—C5	1.535 (4)	C11—H11A	0.9700
C4—H4A	0.9700	C11—H11B	0.9700
C4—H4B	0.9700	C12—H12A	0.9700

C5—C12	1.511 (4)	C12—H12B	0.9700
C5—C6	1.514 (4)	C13—H13A	0.9600
C5—H5	0.9800	C13—H13B	0.9600
C6—C7	1.509 (4)	C13—H13C	0.9600
C6—H6A	0.9700		

C2—S1—C1	87.21 (11)	C11—C7—H7	109.5
C1—N1—N2	114.59 (18)	C6—C7—H7	109.5
C2—N2—N1	111.34 (18)	C8—C7—H7	109.5
C2—N3—C13	119.4 (2)	C7—C8—C3	110.6 (2)
C2—N3—H3	117.1 (16)	C7—C8—H8A	109.5
C13—N3—H3	120.7 (16)	C3—C8—H8A	109.5
N1—C1—C3	125.1 (2)	C7—C8—H8B	109.5
N1—C1—S1	112.82 (18)	C3—C8—H8B	109.5
C3—C1—S1	122.04 (16)	H8A—C8—H8B	108.1
N2—C2—N3	123.8 (2)	C3—C9—C10	110.7 (2)
N2—C2—S1	114.02 (17)	C3—C9—H9A	109.5
N3—C2—S1	122.18 (16)	C10—C9—H9A	109.5
C1—C3—C4	110.38 (18)	C3—C9—H9B	109.5
C1—C3—C9	111.84 (19)	C10—C9—H9B	109.5
C4—C3—C9	108.5 (2)	H9A—C9—H9B	108.1
C1—C3—C8	110.08 (19)	C11—C10—C12	110.7 (2)
C4—C3—C8	108.1 (2)	C11—C10—C9	108.1 (2)
C9—C3—C8	107.8 (2)	C12—C10—C9	108.9 (2)
C3—C4—C5	110.3 (2)	C11—C10—H10	109.7
C3—C4—H4A	109.6	C12—C10—H10	109.7
C5—C4—H4A	109.6	C9—C10—H10	109.7
C3—C4—H4B	109.6	C7—C11—C10	110.1 (2)
C5—C4—H4B	109.6	C7—C11—H11A	109.6
H4A—C4—H4B	108.1	C10—C11—H11A	109.6
C12—C5—C6	109.6 (2)	C7—C11—H11B	109.6
C12—C5—C4	108.4 (3)	C10—C11—H11B	109.6
C6—C5—C4	109.8 (3)	H11A—C11—H11B	108.1
C12—C5—H5	109.7	C5—C12—C10	109.9 (2)
C6—C5—H5	109.7	C5—C12—H12A	109.7
C4—C5—H5	109.7	C10—C12—H12A	109.7
C7—C6—C5	110.5 (2)	C5—C12—H12B	109.7
C7—C6—H6A	109.6	C10—C12—H12B	109.7
C5—C6—H6A	109.6	H12A—C12—H12B	108.2
C7—C6—H6B	109.6	N3—C13—H13A	109.5
C5—C6—H6B	109.6	N3—C13—H13B	109.5
H6A—C6—H6B	108.1	H13A—C13—H13B	109.5
C11—C7—C6	110.4 (3)	N3—C13—H13C	109.5
C11—C7—C8	109.9 (2)	H13A—C13—H13C	109.5
C6—C7—C8	108.0 (2)	H13B—C13—H13C	109.5

C1—N1—N2—C2	−0.7 (3)	C12—C5—C6—C7	−58.7 (3)
N2—N1—C1—C3	−177.1 (2)	C4—C5—C6—C7	60.3 (3)
N2—N1—C1—S1	1.3 (2)	C5—C6—C7—C11	58.9 (3)

C2—S1—C1—N1	−1.21 (18)	C5—C6—C7—C8	−61.2 (3)
C2—S1—C1—C3	177.24 (19)	C11—C7—C8—C3	−59.0 (3)
N1—N2—C2—N3	−179.5 (2)	C6—C7—C8—C3	61.4 (3)
N1—N2—C2—S1	−0.3 (2)	C1—C3—C8—C7	179.5 (2)
C13—N3—C2—N2	−5.0 (4)	C4—C3—C8—C7	−59.9 (3)
C13—N3—C2—S1	175.9 (2)	C9—C3—C8—C7	57.3 (3)
C1—S1—C2—N2	0.84 (18)	C1—C3—C9—C10	179.9 (2)
C1—S1—C2—N3	−180.0 (2)	C4—C3—C9—C10	57.9 (3)
N1—C1—C3—C4	−6.8 (3)	C8—C3—C9—C10	−58.9 (3)
S1—C1—C3—C4	174.97 (17)	C3—C9—C10—C11	61.2 (3)
N1—C1—C3—C9	−127.7 (2)	C3—C9—C10—C12	−59.1 (3)
S1—C1—C3—C9	54.0 (3)	C6—C7—C11—C10	−58.0 (3)
N1—C1—C3—C8	112.5 (2)	C8—C7—C11—C10	60.9 (3)
S1—C1—C3—C8	−65.8 (2)	C12—C10—C11—C7	57.7 (3)
C1—C3—C4—C5	178.1 (2)	C9—C10—C11—C7	−61.5 (3)
C9—C3—C4—C5	−59.0 (3)	C6—C5—C12—C10	57.9 (3)
C8—C3—C4—C5	57.7 (3)	C4—C5—C12—C10	−61.9 (3)
C3—C4—C5—C12	61.2 (3)	C11—C10—C12—C5	−57.8 (3)
C3—C4—C5—C6	−58.5 (3)	C9—C10—C12—C5	61.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N1^{i}	0.87 (1)	2.15 (1)	3.021 (3)	179 (2)

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