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Crystal structure of 1-(cyclopentylidene-amino)-3-(prop-2-en-1-yl)thiourea

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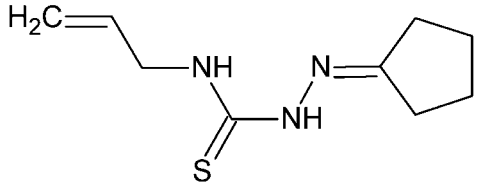
In the title compound, $C_9H_{15}N_3S$, the cyclopentyl ring adopts an envelope conformation with one of the methylene C atoms as the flap. The thiosemicarbazide fragment is almost planar (r.m.s. deviation = 0.038 Å) and a short intramolecular N—H···N contact occurs. In the crystal, molecules are linked into helical (4_1 symmetry) chains propagating in [001] by N—H···N and N—H···S hydrogen bonds. A very weak C—H···S interaction is also observed.

Keywords: crystal structure; thiosemicarbazides; hydrogen bonding.

CCDC reference: 1435175

1. Related literature

For the biological activities of thiosemicarbazide-containing compounds, see: Hu *et al.* (2010); da Costa *et al.* (2015). For the synthesis of the title compound, see: Mague *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_9H_{15}N_3S$ M_r = 197.30

Tetragonal, $P4_1$
a = 9.0124 (2) Å
c = 12.8200 (2) Å
V = 1041.28 (5) Å^3
Z = 4

Cu $K\alpha$ radiation
μ = 2.42 mm^{-1}
T = 150 K
0.21 × 0.16 × 0.10 mm

2.2. Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.67, T_{max} = 0.79
7869 measured reflections
1990 independent reflections
1901 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.10
1990 reflections
119 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack x determined using 826 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.04 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*C*···N1^{i}	0.91	2.29	3.194 (4)	177
N3—H3*C*···N1	0.91	2.25	2.642 (4)	106
N3—H3*C*···S1^{ii}	0.91	2.49	3.310 (3)	151
C3—H3*A*···S1^{iii}	0.99	2.86	3.663 (4)	139

Symmetry codes: (i) $-y+1, x, z+\frac{1}{4}$; (ii) $y, -x+1, z-\frac{1}{4}$; (iii) $-y, x, z+\frac{1}{4}$.

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7535).

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supporting information

Acta Cryst. (2015). E**71**, o924–o925 [https://doi.org/10.1107/S2056989015021003]

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S1. Comment

The thiosemicarbazones comprise a class of molecules known for their diverse biological activities (Hu *et al.*, 2010; Costa *et al.*, 2015). In this context we report here the synthesis and crystal structure determination of the title compound.

In the title compound (Fig. 1), a Cremer-Pople analysis of the conformation of the 5-membered ring gave puckering parameters Q(2) = 0.380 (4) Å and φ(2) = 285.3 (6)°. The molecules pack in helical chains about the 4_1 axis assisted by an N2—H2C···N1^{i} and an N3—H3C···S1^{ii} (i: 1 - *y*, *x*, 1/4 + *z*; ii: *y*, 1 - *x*, -1/4 + *z*) hydrogen bond between each pair of adjacent molecules (Table 1 and Fig. 2).

S2. Experimental

The title compound was prepared according to our recently reported method (Mague *et al.*, 2014). Colourless blocks were grown from ethanol solution by slow evaporation. *M*. p. 374–375 K; 89% yield.

S3. Refinement

H atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å) while those attached to nitrogen were placed in locations derived from a difference map and their parameters adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

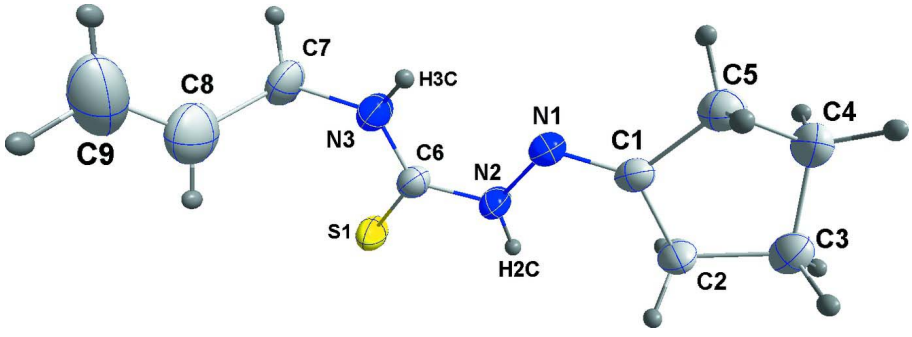

Figure 1

The title molecule, shown with 50% probability ellipsoids.

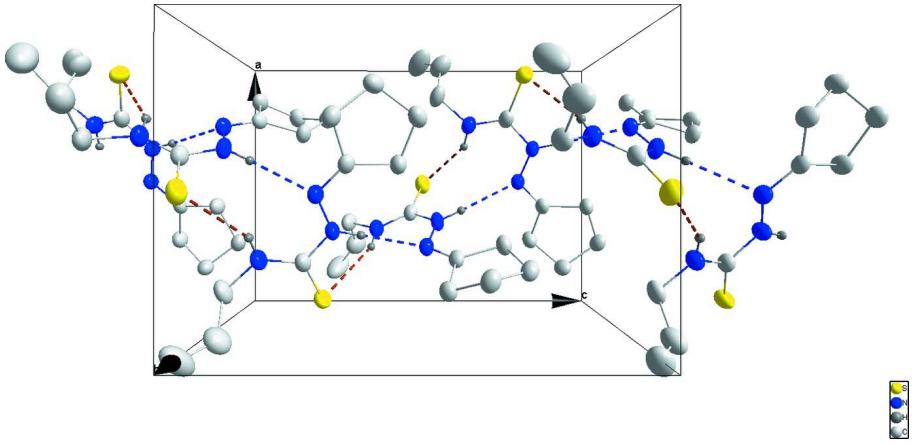

Figure 2

The packing in the title molecule, viewed down the *b* axis. N—H···N and N—H···S hydrogen bonds are shown, respectively, as blue and brown dotted lines.

1-(Cyclopentylideneamino)-3-(prop-2-en-1-yl)thiourea

Crystal data

$C_9H_{15}N_3S$
M_r = 197.30
Tetragonal, $P4_1$
Hall symbol: P 4w
a = 9.0124 (2) Å
c = 12.8200 (2) Å
V = 1041.28 (5) Å^3
Z = 4
$F(000)$ = 424
D_x = 1.259 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7005 reflections
θ = 3.5–72.4°
μ = 2.42 mm^{-1}
T = 150 K
Block, colourless
0.21 × 0.16 × 0.10 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, 2014)
T_{min} = 0.67, T_{max} = 0.79
7869 measured reflections
1990 independent reflections
1901 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 72.4°, θ_{min} = 4.9°
h = −10→11
k = −11→11
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.109
S = 1.10
1990 reflections
119 parameters
1 restraint
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.3211P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack x determined using 826 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.04 (3)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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	0.50830 (11)	0.10489 (10)	0.51268 (7)	0.0404 (3)
N1	0.2886 (3)	0.4807 (3)	0.5217 (2)	0.0312 (8)
N2	0.3701 (3)	0.3575 (3)	0.5510 (2)	0.0325 (8)
N3	0.3362 (3)	0.2660 (3)	0.3854 (2)	0.0357 (9)
C1	0.2584 (4)	0.5764 (4)	0.5926 (3)	0.0293 (9)
C2	0.3050 (4)	0.5761 (4)	0.7060 (3)	0.0339 (10)
C3	0.2402 (4)	0.7223 (4)	0.7498 (3)	0.0362 (10)
C4	0.2242 (4)	0.8220 (4)	0.6547 (3)	0.0389 (11)
C5	0.1737 (4)	0.7160 (4)	0.5686 (3)	0.0359 (11)
C6	0.3975 (4)	0.2491 (4)	0.4790 (3)	0.0331 (10)
C7	0.3533 (5)	0.1587 (5)	0.3012 (3)	0.0430 (11)
C8	0.2503 (6)	0.0262 (6)	0.3117 (4)	0.0617 (17)
C9	0.1462 (7)	−0.0066 (7)	0.2532 (5)	0.079 (2)
H2A	0.41450	0.57460	0.71240	0.0410*
H2B	0.26350	0.48910	0.74300	0.0410*
H2C	0.41060	0.34020	0.61480	0.0390*
H3A	0.14260	0.70480	0.78310	0.0430*
H3B	0.30820	0.76700	0.80170	0.0430*
H3C	0.27400	0.34430	0.37540	0.0430*
H4A	0.32000	0.86910	0.63660	0.0470*
H4B	0.14930	0.90050	0.66700	0.0470*
H5A	0.06530	0.69880	0.57190	0.0430*
H5B	0.19970	0.75490	0.49880	0.0430*
H7A	0.45720	0.12310	0.30000	0.0520*
H7B	0.33340	0.20860	0.23380	0.0520*
H8	0.26760	−0.03810	0.36910	0.0740*
H9A	0.12420	0.05410	0.19460	0.0950*
H9B	0.08830	−0.09250	0.26690	0.0950*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0529 (6)	0.0352 (4)	0.0332 (4)	0.0076 (4)	−0.0021 (4)	−0.0046 (4)
N1	0.0366 (14)	0.0334 (13)	0.0235 (12)	−0.0002 (11)	−0.0003 (12)	0.0003 (12)
N2	0.0433 (16)	0.0325 (15)	0.0217 (12)	0.0033 (12)	−0.0040 (12)	−0.0021 (11)
N3	0.0413 (17)	0.0387 (17)	0.0272 (15)	0.0005 (12)	−0.0016 (13)	−0.0047 (12)

C1	0.0316 (16)	0.0328 (17)	0.0236 (16)	−0.0033 (13)	0.0013 (13)	0.0030 (13)
C2	0.0415 (19)	0.0357 (18)	0.0245 (17)	0.0010 (15)	−0.0038 (14)	0.0002 (13)
C3	0.0379 (18)	0.0401 (18)	0.0307 (17)	−0.0040 (14)	0.0039 (15)	−0.0051 (15)
C4	0.047 (2)	0.0338 (18)	0.036 (2)	0.0042 (15)	0.0050 (17)	−0.0026 (15)
C5	0.0389 (18)	0.0392 (19)	0.0297 (18)	0.0042 (15)	−0.0013 (14)	0.0033 (15)
C6	0.0372 (17)	0.0361 (18)	0.0260 (16)	−0.0053 (13)	0.0023 (14)	−0.0018 (13)
C7	0.044 (2)	0.055 (2)	0.0299 (17)	−0.0055 (17)	0.0020 (15)	−0.0131 (17)
C8	0.065 (3)	0.061 (3)	0.059 (3)	−0.002 (2)	0.005 (2)	−0.018 (2)
C9	0.075 (3)	0.090 (4)	0.073 (4)	−0.040 (3)	0.019 (3)	−0.028 (3)

Geometric parameters (Å, º)

S1—C6	1.695 (4)	C8—C9	1.237 (8)
N1—N2	1.383 (4)	C2—H2A	0.9900
N1—C1	1.282 (5)	C2—H2B	0.9900
N2—C6	1.367 (5)	C3—H3A	0.9900
N3—C6	1.330 (5)	C3—H3B	0.9900
N3—C7	1.457 (5)	C4—H4A	0.9900
C1—C2	1.513 (5)	C4—H4B	0.9900
C1—C5	1.503 (5)	C5—H5A	0.9900
C2—C3	1.547 (5)	C5—H5B	0.9900
N2—H2C	0.9100	C7—H7A	0.9900
C3—C4	1.521 (5)	C7—H7B	0.9900
N3—H3C	0.9100	C8—H8	0.9500
C4—C5	1.529 (5)	C9—H9A	0.9500
C7—C8	1.519 (7)	C9—H9B	0.9500
N2—N1—C1	117.4 (3)	C2—C3—H3B	111.00
N1—N2—C6	119.1 (3)	C4—C3—H3A	111.00
C6—N3—C7	123.3 (3)	C4—C3—H3B	111.00
N1—C1—C2	128.4 (3)	H3A—C3—H3B	109.00
N1—C1—C5	121.7 (3)	C3—C4—H4A	111.00
C2—C1—C5	109.8 (3)	C3—C4—H4B	111.00
C1—C2—C3	104.0 (3)	C5—C4—H4A	111.00
N1—N2—H2C	127.00	C5—C4—H4B	111.00
C6—N2—H2C	114.00	H4A—C4—H4B	109.00
C2—C3—C4	104.4 (3)	C1—C5—H5A	111.00
C6—N3—H3C	118.00	C1—C5—H5B	111.00
C7—N3—H3C	118.00	C4—C5—H5A	111.00
C3—C4—C5	103.8 (3)	C4—C5—H5B	111.00
C1—C5—C4	102.9 (3)	H5A—C5—H5B	109.00
S1—C6—N2	118.9 (3)	N3—C7—H7A	109.00
N2—C6—N3	116.9 (3)	N3—C7—H7B	109.00
S1—C6—N3	124.2 (3)	C8—C7—H7A	109.00
N3—C7—C8	113.1 (3)	C8—C7—H7B	109.00
C7—C8—C9	126.7 (5)	H7A—C7—H7B	108.00
C1—C2—H2A	111.00	C7—C8—H8	117.00
C1—C2—H2B	111.00	C9—C8—H8	117.00

C3—C2—H2A	111.00	C8—C9—H9A	120.00
C3—C2—H2B	111.00	C8—C9—H9B	120.00
H2A—C2—H2B	109.00	H9A—C9—H9B	120.00
C2—C3—H3A	111.00		

C1—N1—N2—C6	176.4 (3)	N1—C1—C2—C3	177.8 (4)
N2—N1—C1—C2	2.3 (5)	C5—C1—C2—C3	1.3 (4)
N2—N1—C1—C5	178.5 (3)	N1—C1—C5—C4	−154.9 (3)
N1—N2—C6—S1	175.4 (2)	C2—C1—C5—C4	21.9 (4)
N1—N2—C6—N3	−3.5 (5)	C1—C2—C3—C4	−24.2 (4)
C7—N3—C6—S1	2.2 (5)	C2—C3—C4—C5	38.1 (3)
C7—N3—C6—N2	−179.0 (3)	C3—C4—C5—C1	−36.6 (3)
C6—N3—C7—C8	80.2 (5)	N3—C7—C8—C9	113.4 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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