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Crystal structure of 4-cyclohexyl-1-(propan-2-ylidene)thiosemicarbazide

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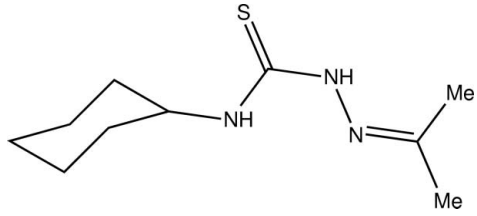
In the title compound, $C_{10}H_{19}N_3S$, the cyclohexyl group adopts a chair conformation and adopts a position approximately *syn* to the thione S atom. The CN_2S thiourea moiety makes dihedral angle of 13.13 (10)° with the propan-2-ylideneamino group. An intramolecular N—H···N hydrogen bond is noted. In the crystal, inversion dimers linked by pairs of N—H···S hydrogen bonds generate $R_2^2(8)$ loops.

Keywords: crystal structure; thiosemicarbazide; thiourea; biological activity; hydrogen bonding.

CCDC reference: 1023476

1. Related literature

For the applications and biological activity of thiosemicarbazide derivatives, see: Brokl *et al.* (1974), Jiang *et al.* (2006). For the crystal structures of related compounds, see: Affan *et al.* (2011); Miroslaw *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_{10}H_{19}N_3S$
M_r = 213.34
Orthorhombic, *Pbca*
a = 13.6668 (10) Å
b = 8.3356 (5) Å
c = 21.4683 (16) Å
V = 2445.7 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 301 K
0.50 × 0.19 × 0.19 mm

2.2. Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.892, T_{max} = 0.957
31768 measured reflections
3028 independent reflections
2051 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.135
S = 1.04
3028 reflections
133 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1D···N3	0.86	2.18	2.592 (2)	109
N2—H2C···S1^{i}	0.86	2.80	3.6170 (18)	158

Symmetry code: (i) $-x+1, -y, -z+1$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5341).

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supporting information

Acta Cryst. (2014). E**70**, o1109 [doi:10.1107/S160053681402025X]

Crystal structure of 4-cyclohexyl-1-(propan-2-ylidene)thiosemicarbazide

Bohari M Yamin, Monica Lulo Rodis and Dayang N. B. A Chee

S1. Chemical context

S2. Structural commentary

S3. Supramolecular features

S4. Database survey

S5. Synthesis and crystallization

A mixture of 4-cyclohexylthiosemicarbazide (0.174 g, 1 mmol), KOH (0.112 g, 0.05 mmol) and diphenyltin(IV) chloride (0.344 g, 1 mmol) in methanol was heated under reflux for 4–5 h. The reaction mixture was allowed to cool to room temperature for 1 h. The white precipitate formed was filtered and washed with acetone. Crystals suitable for X-ray study were obtained by recrystallization from acetone (0.240 g, 42% yield). *M*.pt = 390–393 K. IR (KBr): $\nu_{NH\text{-}cyclohexyl}$ (3336), $\nu_{S=C—NH}$ (3221), $\nu_{cyclohexyl}$ (2929,2850), $\nu_{C=N}$ (1527), $\nu_{C=S}$ (1263,881), $\nu_{N—N}$ (1106) cm^{-1}. All the chemicals were purchased from Sigma Aldrich (Germany).

S6. Refinement

Non-methine C-bound H atoms were positioned geometrically with C—H = 0.96–0.97 Å, and with U_{iso}(H)= 1.2–1.5U_{eq}(C). The N-bound H atoms were positioned geometrically with N—H = 0.86 Å, and with U_{iso}(H)= 1.2U_{eq}(N). The methine-bound H atom was located from a Fourier map and refined isotropically. A rotating model was applied in the refinement of the methyl hydrogen atoms.

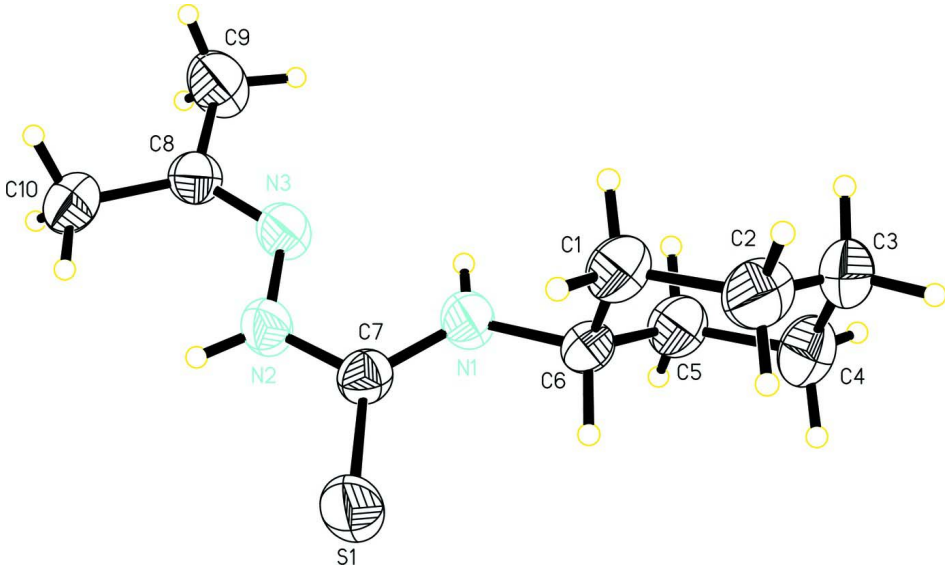

Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at 50% probability level.

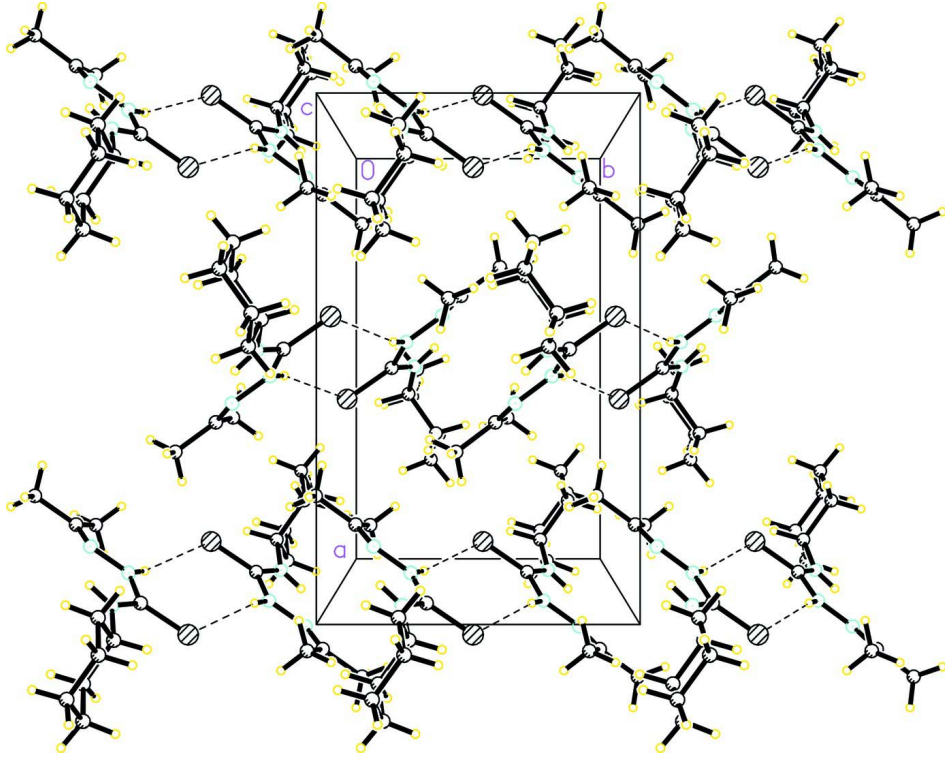

Figure 2

The crystal packing of (I) viewed down the *c* axis. The dashed lines indicate intermolecular hydrogen bonds

4-Cyclohexyl-1-(propan-2-ylidene)thiosemicarbazide

Crystal data

$C_{10}H_{19}N_3S$
$M_r = 213.34$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.6668$ (10) Å
$b = 8.3356$ (5) Å
$c = 21.4683$ (16) Å
$V = 2445.7$ (3) Å^3
$Z = 8$
$F(000) = 928$
$D_x = 1.159$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7621 reflections
$\theta = 2.9$–$28.3°$
$\mu = 0.24$ mm^{-1}
$T = 301$ K
Block, yellow
0.50 × 0.19 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 83.66 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.892$, $T_{max} = 0.957$
31768 measured reflections
3028 independent reflections
2051 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.9°$
$h = -18 \rightarrow 16$
$k = -11 \rightarrow 10$
$l = -28 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.135$
$S = 1.04$
3028 reflections
133 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.9515P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.59037 (4)	0.01795 (7)	0.41721 (3)	0.0582 (2)
N1	0.51795 (12)	0.26158 (19)	0.35150 (7)	0.0477 (4)
H1D	0.4748	0.3363	0.3483	0.057*

N2	0.46292 (11)	0.23807 (19)	0.45107 (7)	0.0447 (4)
H2C	0.4630	0.1942	0.4873	0.054*
N3	0.40374 (11)	0.36924 (19)	0.43836 (7)	0.0462 (4)
C1	0.67225 (14)	0.3396 (2)	0.30141 (9)	0.0489 (5)
H1A	0.7093	0.3119	0.3384	0.059*
H1B	0.6530	0.4513	0.3048	0.059*
C2	0.73660 (16)	0.3179 (3)	0.24406 (11)	0.0580 (6)
H2A	0.7914	0.3916	0.2463	0.070*
H2B	0.7625	0.2096	0.2436	0.070*
C3	0.68044 (18)	0.3477 (3)	0.18483 (10)	0.0623 (6)
H3A	0.7223	0.3261	0.1493	0.075*
H3B	0.6609	0.4595	0.1830	0.075*
C4	0.59057 (17)	0.2423 (3)	0.18135 (10)	0.0589 (6)
H4A	0.6104	0.1308	0.1786	0.071*
H4B	0.5538	0.2682	0.1440	0.071*
C5	0.52547 (14)	0.2654 (3)	0.23839 (9)	0.0488 (5)
H5A	0.5001	0.3741	0.2387	0.059*
H5B	0.4704	0.1923	0.2361	0.059*
C6	0.58140 (13)	0.2352 (2)	0.29786 (8)	0.0393 (4)
H1C	0.6010 (13)	0.123 (2)	0.2991 (8)	0.041 (5)*
C7	0.52063 (13)	0.1809 (2)	0.40478 (8)	0.0387 (4)
C8	0.36147 (14)	0.4386 (2)	0.48408 (9)	0.0426 (4)
C9	0.29760 (19)	0.5788 (3)	0.46846 (11)	0.0714 (7)
H9A	0.2964	0.5937	0.4241	0.107*
H9B	0.3231	0.6736	0.4880	0.107*
H9C	0.2324	0.5591	0.4832	0.107*
C10	0.36946 (17)	0.3939 (3)	0.55073 (9)	0.0549 (5)
H10A	0.4361	0.3671	0.5602	0.082*
H10B	0.3283	0.3030	0.5590	0.082*
H10C	0.3491	0.4825	0.5761	0.082*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0625 (4)	0.0585 (3)	0.0536 (3)	0.0258 (3)	0.0105 (3)	0.0124 (2)
N1	0.0531 (9)	0.0453 (9)	0.0446 (9)	0.0189 (7)	0.0155 (8)	0.0074 (7)
N2	0.0499 (9)	0.0451 (9)	0.0391 (9)	0.0104 (7)	0.0105 (7)	0.0057 (7)
N3	0.0469 (9)	0.0448 (9)	0.0468 (9)	0.0105 (7)	0.0142 (7)	0.0068 (7)
C1	0.0468 (11)	0.0532 (11)	0.0467 (11)	0.0001 (9)	−0.0012 (9)	−0.0088 (9)
C2	0.0457 (11)	0.0557 (12)	0.0725 (15)	−0.0087 (10)	0.0167 (11)	−0.0103 (11)
C3	0.0777 (15)	0.0583 (13)	0.0509 (13)	−0.0037 (11)	0.0270 (12)	−0.0024 (10)
C4	0.0682 (14)	0.0682 (14)	0.0403 (11)	0.0013 (11)	−0.0001 (10)	−0.0082 (10)
C5	0.0443 (11)	0.0535 (11)	0.0486 (12)	0.0024 (9)	0.0008 (9)	−0.0040 (9)
C6	0.0428 (10)	0.0348 (9)	0.0403 (10)	0.0084 (8)	0.0083 (8)	−0.0016 (7)
C7	0.0358 (9)	0.0391 (9)	0.0410 (10)	0.0015 (7)	0.0050 (8)	−0.0017 (8)
C8	0.0429 (10)	0.0390 (9)	0.0460 (11)	0.0000 (8)	0.0128 (8)	0.0011 (8)
C9	0.0828 (16)	0.0633 (14)	0.0682 (15)	0.0310 (13)	0.0326 (13)	0.0157 (12)
C10	0.0661 (13)	0.0528 (11)	0.0458 (12)	0.0046 (11)	0.0055 (10)	−0.0092 (9)

Geometric parameters (Å, º)

S1—C7	1.6803 (18)	C3—H3B	0.9700
N1—C7	1.328 (2)	C4—C5	1.526 (3)
N1—C6	1.458 (2)	C4—H4A	0.9700
N1—H1D	0.8600	C4—H4B	0.9700
N2—C7	1.355 (2)	C5—C6	1.509 (3)
N2—N3	1.387 (2)	C5—H5A	0.9700
N2—H2C	0.8600	C5—H5B	0.9700
N3—C8	1.277 (2)	C6—H1C	0.973 (19)
C1—C6	1.518 (3)	C8—C10	1.483 (3)
C1—C2	1.524 (3)	C8—C9	1.497 (3)
C1—H1A	0.9700	C9—H9A	0.9600
C1—H1B	0.9700	C9—H9B	0.9600
C2—C3	1.506 (3)	C9—H9C	0.9600
C2—H2A	0.9700	C10—H10A	0.9600
C2—H2B	0.9700	C10—H10B	0.9600
C3—C4	1.512 (3)	C10—H10C	0.9600
C3—H3A	0.9700		
C7—N1—C6	125.98 (15)	C6—C5—C4	111.24 (16)
C7—N1—H1D	117.0	C6—C5—H5A	109.4
C6—N1—H1D	117.0	C4—C5—H5A	109.4
C7—N2—N3	118.20 (15)	C6—C5—H5B	109.4
C7—N2—H2C	120.9	C4—C5—H5B	109.4
N3—N2—H2C	120.9	H5A—C5—H5B	108.0
C8—N3—N2	118.00 (16)	N1—C6—C5	109.97 (14)
C6—C1—C2	111.30 (16)	N1—C6—C1	111.11 (15)
C6—C1—H1A	109.4	C5—C6—C1	111.16 (16)
C2—C1—H1A	109.4	N1—C6—H1C	106.7 (11)
C6—C1—H1B	109.4	C5—C6—H1C	108.9 (11)
C2—C1—H1B	109.4	C1—C6—H1C	108.9 (11)
H1A—C1—H1B	108.0	N1—C7—N2	115.93 (16)
C3—C2—C1	111.62 (18)	N1—C7—S1	124.19 (14)
C3—C2—H2A	109.3	N2—C7—S1	119.87 (14)
C1—C2—H2A	109.3	N3—C8—C10	126.46 (18)
C3—C2—H2B	109.3	N3—C8—C9	116.44 (18)
C1—C2—H2B	109.3	C10—C8—C9	117.10 (17)
H2A—C2—H2B	108.0	C8—C9—H9A	109.5
C2—C3—C4	111.10 (18)	C8—C9—H9B	109.5
C2—C3—H3A	109.4	H9A—C9—H9B	109.5
C4—C3—H3A	109.4	C8—C9—H9C	109.5
C2—C3—H3B	109.4	H9A—C9—H9C	109.5
C4—C3—H3B	109.4	H9B—C9—H9C	109.5
H3A—C3—H3B	108.0	C8—C10—H10A	109.5
C3—C4—C5	111.13 (17)	C8—C10—H10B	109.5
C3—C4—H4A	109.4	H10A—C10—H10B	109.5
C5—C4—H4A	109.4	C8—C10—H10C	109.5

C3—C4—H4B	109.4	H10A—C10—H10C	109.5
C5—C4—H4B	109.4	H10B—C10—H10C	109.5
H4A—C4—H4B	108.0		

C7—N2—N3—C8	−169.08 (17)	C2—C1—C6—N1	−177.51 (16)
C6—C1—C2—C3	54.9 (2)	C2—C1—C6—C5	−54.7 (2)
C1—C2—C3—C4	−55.4 (2)	C6—N1—C7—N2	172.31 (17)
C2—C3—C4—C5	55.8 (2)	C6—N1—C7—S1	−7.2 (3)
C3—C4—C5—C6	−55.9 (2)	N3—N2—C7—N1	2.8 (2)
C7—N1—C6—C5	145.50 (19)	N3—N2—C7—S1	−177.70 (13)
C7—N1—C6—C1	−91.0 (2)	N2—N3—C8—C10	0.4 (3)
C4—C5—C6—N1	178.77 (16)	N2—N3—C8—C9	−179.53 (18)
C4—C5—C6—C1	55.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*D*···N3	0.86	2.18	2.592 (2)	109
C6—H1*C*···S1	0.97 (3)	2.69 (2)	3.142 (2)	108.9 (12)
C10—H10*A*···N2	0.96	2.40	2.811 (3)	106
N2—H2*C*···S1^{i}	0.86	2.80	3.6170 (18)	158

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