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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.

## 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).
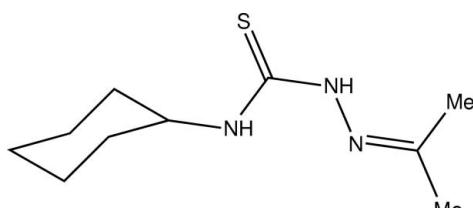


## 2. Experimental

### 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
$\mu = 0.24$ mm$^{-1}$
$T = 301$ K
$0.50 \times 0.19 \times 0.19$ 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—H1$D$⋯N3 | 0.86 | 2.18 | 2.592 (2) | 109 |
| N2—H2$C$⋯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).

## Acknowledgements

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

## References

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# supporting information

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

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

### S1. 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): $v_{NH\text{-}cyclohexyl}$ (3336), $v_{S=C-NH}$ (3221), $v_{cyclohexyl}$ (2929,2850), $v_{C=N}$ (1527), $v_{C=S}$ (1263,881), $v_{N-N}$ (1106) cm$^{-1}$. All the chemicals were purchased from Sigma Aldrich (Germany).

### S2. Refinement

Non-methine C-bound H atoms were positioned geometrically with C—H = 0.96–0.97 Å, and with $U_{iso}$(H)= 1.2–1.5$U_{eq}$(C). The N-bound H atoms were positioned geometrically with N—H = 0.86 Å, and with $U_{iso}$(H)= 1.2$U_{eq}$(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.
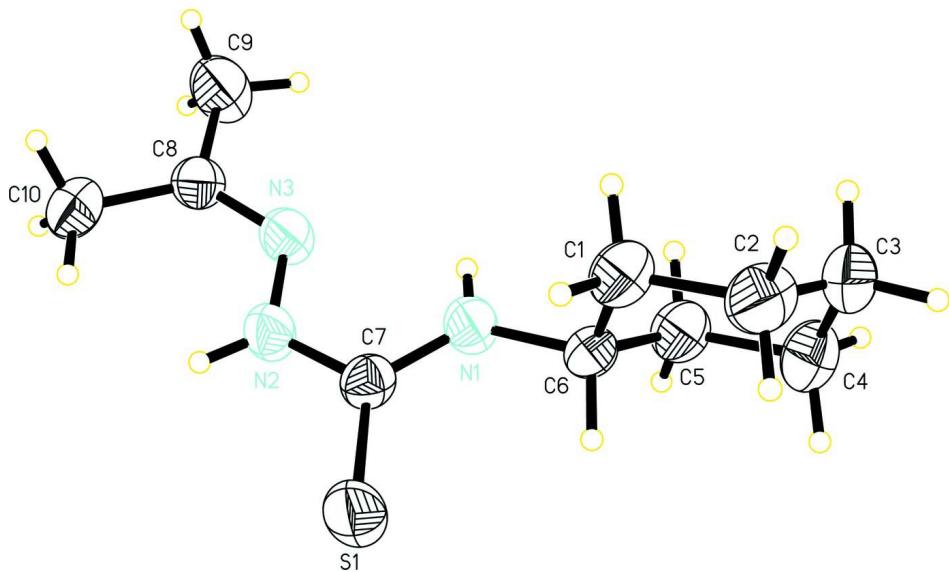


**Figure 1**

The molecular structure of (I) with displacement ellipsoids drawn at 50% probability level.
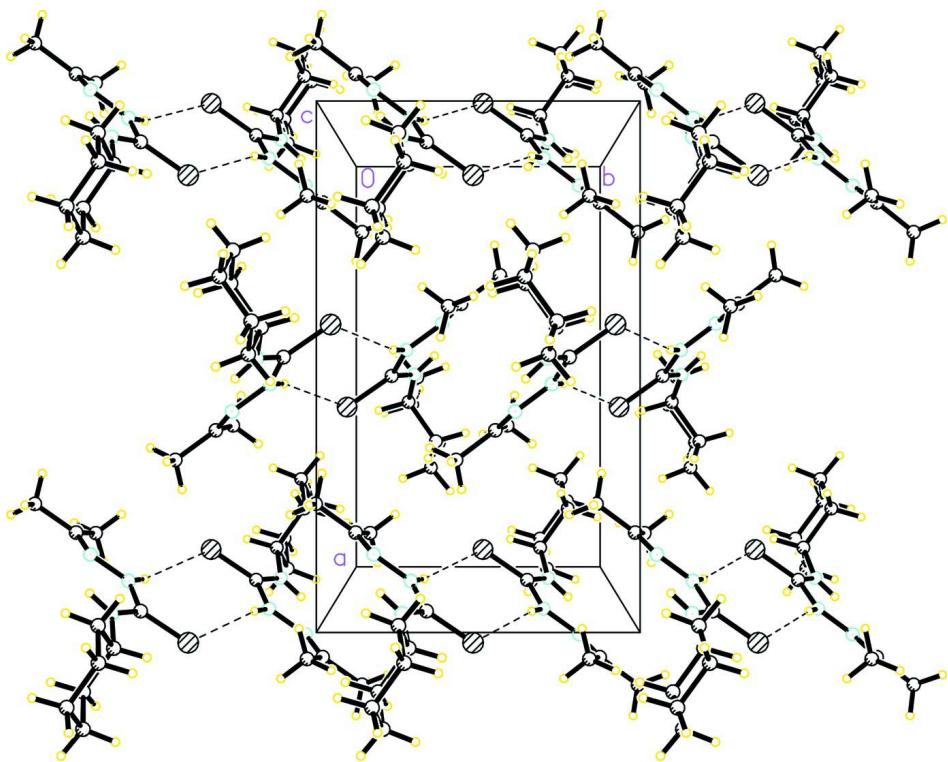
**Figure 2**

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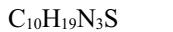
| | |
|---|---|
| $C_{10}H_{19}N_3S$ | $F(000) = 928$ |
| $M_r = 213.34$ | $D_x = 1.159$ Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Mo *Kα* radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 7621 reflections |
| $a = 13.6668$ (10) Å | $\theta = 2.9–28.3°$ |
| $b = 8.3356$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

*Data collection*

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

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.053$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.135$ | |
| $S = 1.04$ | H atoms treated by a mixture of independent and constrained refinement |
| 3028 reflections | |
| 133 parameters | $w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.9515P]$ |
| 0 restraints | where $P = (F_o^2 + 2F_c^2)/3$ |
| Primary atom site location: structure-invariant direct methods | $(\Delta/\sigma)_{max} = 0.001$ |
| | $\Delta\rho_{max} = 0.25$ 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) $-x+1, -y, -z+1$.