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N-Cyclohexylcarbonyl-*N*′-phenylthiourea

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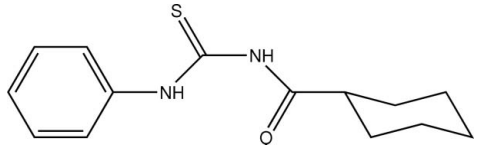
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.092; data-to-parameter ratio = 15.6.

The title compound, $C_{14}H_{18}N_2OS$, adopts a *trans–cis* configuration of the cyclohexylcarbonyl and phenyl groups with respect to the thione S atom across the thiourea C—N bonds. There is an intramolecular N—H···O hydrogen bond, and intermolecular N—H···S hydrogen bonds generate a dimer.

Related literature

For crystal structures of related compounds, see: Yamin & Yusof (2003); Yusof *et al.* (2007). For details of potential applications in materials and biological activities, see: Wei *et al.* (2004); Baruah *et al.* (2002).



Experimental

Crystal data

$C_{14}H_{18}N_2OS$
M_r = 262.36
Triclinic, $P\overline{1}$
a = 6.696 (2) Å
b = 9.395 (3) Å
c = 12.578 (4) Å
α = 100.262 (5)°
β = 104.720 (5)°
γ = 110.075 (5)°
V = 687.4 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 293 (2) K
0.46 × 0.42 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.903, T_{max} = 0.952
6848 measured reflections
2547 independent reflections
2207 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.07
2547 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	1.95	2.6471 (18)	137
N1—H1A···S1^{i}	0.86	2.68	3.4997 (19)	160

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2104).

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supplementary materials

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N-Cyclohexylcarbonyl-*N*'-phenylthiourea

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Comment

Thiourea derivatives have received considerable attention because of their potential applications in materials science (Wei *et al.*, 2004) and their biological activities (Baruah *et al.*, 2002). The title compound, (I), is similar to *N*-benzoyl-*N*'-phenylthiourea (II) (Yamin & Yusof, 2003), except that one of the phenyl groups is replaced by a cyclohexane group (Fig. 1). The molecule maintains its *trans-cis* configuration with respect to the positions of the cyclohexylcarbonyl and phenyl groups relative to the thiono S1 atom across the two C—N bonds. The bond lengths and angles are in normal ranges and comparable to those in (II) and other thiourea derivatives (Yusof *et al.*, 2007). The central carbonylthiourea (S1/N1/N2/C7/O1/C8) fragment and phenyl ring (C9—C14) are all planar, with a maximum deviation of 0.038 (1)Å from the least-squares plane for atom N1. The central carbonylthiourea fragment makes a dihedral angle of 62.68 (6)° with the phenyl ring, which is larger than the equivalent dihedral angle in (II) (28.78 (9)°).

There is an intramolecular hydrogen bond, N2—H2A···O1 (Table 1), which results in a pseudo-six-membered ring, O1···H2A—N2—C8—N1—C7—O1. In the crystal structure, the molecules are linked by two equivalent N—H···S intermolecular interactions, (symmetry codes as in Table 1) to form dimers (Fig. 2).

Experimental

To a stirred acetone solution (75 ml) of cyclohexylcarbonyl chloride (2.0 g, 14 mmol) and ammoniumthiocyanate (1.04 g, 14 mmol), aniline (1.27 g, 14 mmol) in 40 ml of acetone was added dropwise. The reaction mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before drying under vacuum. Good quality crystals were obtained by recrystallization from methanol.

Refinement

After their location in a difference map, all H-atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms with C—H = 0.93–0.97Å and N—H = 0.86Å with U_{iso}(H)= 1.2 (CH_2 and NH).

Figures

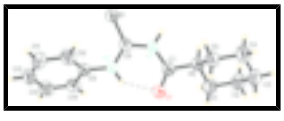

Fig. 1. The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

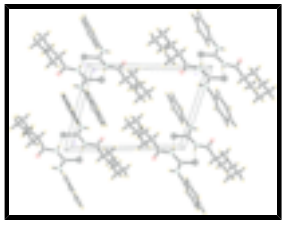

Fig. 2. Packing diagram of (I) viewed down the *a* axis. The dashed lines denote the N—H···S hydrogen bonds.

N-Cyclohexylcarbonyl-*N*'-phenylthiourea

Crystal data

$C_{14}H_{18}N_2OS$	$Z = 2$
$M_r = 262.36$	$F_{000} = 280$
Triclinic, $P\overline{1}$	$D_x = 1.268$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.696$ (2) Å	Cell parameters from 837 reflections
$b = 9.395$ (3) Å	$\theta = 1.7$–25.4°
$c = 12.578$ (4) Å	$\mu = 0.23$ mm^{-1}
$\alpha = 100.262$ (5)°	$T = 293$ (2) K
$\beta = 104.720$ (5)°	Block, colourless
$\gamma = 110.075$ (5)°	0.46 × 0.42 × 0.22 mm
$V = 687.4$ (4) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2547 independent reflections
Radiation source: fine-focus sealed tube	2207 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
Detector resolution: 83.66 pixels mm^{-1}	$\theta_{max} = 25.4^\circ$
$T = 293(2)$ K	$\theta_{min} = 1.7^\circ$
ω scan	$h = -8 \rightarrow 8$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$k = -11 \rightarrow 11$
$T_{min} = 0.903$, $T_{max} = 0.952$	$l = -15 \rightarrow 15$
6848 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.1063P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = <0.001$

2547 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
163 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.34074 (6)	0.15250 (5)	0.11564 (3)	0.05336 (15)
N2	0.56387 (19)	0.24546 (14)	−0.02447 (10)	0.0459 (3)
H2A	0.5697	0.2301	−0.0929	0.055*
O1	0.37126 (19)	0.08767 (14)	−0.24588 (9)	0.0631 (3)
N1	0.21478 (19)	0.03681 (14)	−0.10875 (10)	0.0458 (3)
H1A	0.0942	−0.0228	−0.0995	0.055*
C9	0.7521 (2)	0.37186 (16)	0.06468 (12)	0.0414 (3)
C7	0.2163 (2)	0.00689 (17)	−0.21976 (12)	0.0462 (3)
C6	0.0093 (2)	−0.13047 (17)	−0.30580 (12)	0.0457 (3)
H6A	−0.0568	−0.2029	−0.2647	0.055*
C8	0.3803 (2)	0.14953 (16)	−0.01007 (12)	0.0416 (3)
C10	0.7245 (3)	0.49819 (19)	0.12396 (14)	0.0541 (4)
H10A	0.5819	0.4995	0.1082	0.065*
C14	0.9628 (2)	0.37063 (18)	0.08647 (13)	0.0477 (4)
H14A	0.9818	0.2867	0.0452	0.057*
C13	1.1471 (3)	0.4958 (2)	0.17066 (14)	0.0583 (4)
H13A	1.2899	0.4946	0.1868	0.070*
C1	0.0713 (3)	−0.2208 (2)	−0.39591 (14)	0.0575 (4)
H1B	0.1462	−0.1492	−0.4341	0.069*
H1C	0.1759	−0.2618	−0.3587	0.069*
C12	1.1208 (3)	0.6209 (2)	0.23018 (14)	0.0620 (5)
H12A	1.2451	0.7047	0.2863	0.074*
C3	−0.3147 (3)	−0.3017 (2)	−0.53876 (14)	0.0632 (5)
H3A	−0.4496	−0.3927	−0.5901	0.076*
H3B	−0.2580	−0.2347	−0.5842	0.076*
C5	−0.1659 (3)	−0.07249 (19)	−0.36170 (14)	0.0531 (4)
H5A	−0.1008	0.0042	−0.3992	0.064*
H5B	−0.2087	−0.0203	−0.3031	0.064*

C11	0.9092 (3)	0.6221 (2)	0.20657 (15)	0.0622 (4)
H11A	0.8910	0.7073	0.2467	0.075*
C2	−0.1381 (3)	−0.3573 (2)	−0.48445 (15)	0.0637 (5)
H2B	−0.0954	−0.4078	−0.5438	0.076*
H2C	−0.2018	−0.4354	−0.4477	0.076*
C4	−0.3753 (3)	−0.2105 (2)	−0.44985 (15)	0.0643 (5)
H4A	−0.4496	−0.2811	−0.4109	0.077*
H4B	−0.4805	−0.1703	−0.4877	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0464 (2)	0.0612 (3)	0.0426 (2)	0.01045 (19)	0.01690 (17)	0.01272 (18)
N2	0.0388 (6)	0.0484 (7)	0.0397 (6)	0.0069 (5)	0.0138 (5)	0.0089 (5)
O1	0.0529 (7)	0.0675 (7)	0.0465 (6)	−0.0014 (6)	0.0184 (5)	0.0149 (5)
N1	0.0371 (6)	0.0461 (7)	0.0422 (7)	0.0046 (5)	0.0134 (5)	0.0106 (5)
C9	0.0378 (7)	0.0429 (7)	0.0387 (7)	0.0104 (6)	0.0127 (6)	0.0134 (6)
C7	0.0425 (8)	0.0467 (8)	0.0431 (8)	0.0113 (7)	0.0133 (6)	0.0147 (7)
C6	0.0430 (8)	0.0441 (8)	0.0409 (8)	0.0078 (6)	0.0130 (6)	0.0130 (6)
C8	0.0364 (7)	0.0404 (7)	0.0449 (8)	0.0135 (6)	0.0119 (6)	0.0124 (6)
C10	0.0436 (8)	0.0550 (9)	0.0583 (10)	0.0184 (7)	0.0157 (7)	0.0098 (8)
C14	0.0431 (8)	0.0511 (8)	0.0514 (9)	0.0188 (7)	0.0180 (7)	0.0189 (7)
C13	0.0370 (8)	0.0733 (11)	0.0570 (10)	0.0152 (8)	0.0113 (7)	0.0226 (9)
C1	0.0529 (9)	0.0603 (10)	0.0541 (10)	0.0231 (8)	0.0147 (8)	0.0095 (8)
C12	0.0498 (10)	0.0605 (10)	0.0476 (9)	0.0013 (8)	0.0061 (7)	0.0085 (8)
C3	0.0563 (10)	0.0647 (11)	0.0449 (9)	0.0105 (8)	0.0059 (7)	0.0061 (8)
C5	0.0474 (9)	0.0518 (9)	0.0525 (9)	0.0155 (7)	0.0153 (7)	0.0099 (7)
C11	0.0659 (11)	0.0517 (9)	0.0569 (10)	0.0164 (8)	0.0202 (9)	0.0041 (8)
C2	0.0690 (11)	0.0554 (10)	0.0544 (10)	0.0208 (9)	0.0164 (8)	0.0035 (8)
C4	0.0448 (9)	0.0695 (11)	0.0622 (11)	0.0168 (8)	0.0078 (8)	0.0091 (9)

Geometric parameters (Å, °)

S1—C8	1.6653 (15)	C13—H13A	0.9300
N2—C8	1.3292 (18)	C1—C2	1.521 (2)
N2—C9	1.4268 (17)	C1—H1B	0.9700
N2—H2A	0.8600	C1—H1C	0.9700
O1—C7	1.2172 (17)	C12—C11	1.376 (3)
N1—C7	1.3777 (19)	C12—H12A	0.9300
N1—C8	1.3846 (18)	C3—C2	1.509 (3)
N1—H1A	0.8600	C3—C4	1.509 (2)
C9—C14	1.372 (2)	C3—H3A	0.9700
C9—C10	1.380 (2)	C3—H3B	0.9700
C7—C6	1.507 (2)	C5—C4	1.525 (2)
C6—C1	1.522 (2)	C5—H5A	0.9700
C6—C5	1.524 (2)	C5—H5B	0.9700
C6—H6A	0.9800	C11—H11A	0.9300
C10—C11	1.376 (2)	C2—H2B	0.9700
C10—H10A	0.9300	C2—H2C	0.9700

C14—C13	1.387 (2)	C4—H4A	0.9700
C14—H14A	0.9300	C4—H4B	0.9700
C13—C12	1.367 (3)		
C8—N2—C9	125.13 (12)	C2—C1—H1C	109.4
C8—N2—H2A	117.4	C6—C1—H1C	109.4
C9—N2—H2A	117.4	H1B—C1—H1C	108.0
C7—N1—C8	129.00 (12)	C13—C12—C11	119.65 (15)
C7—N1—H1A	115.5	C13—C12—H12A	120.2
C8—N1—H1A	115.5	C11—C12—H12A	120.2
C14—C9—C10	120.34 (14)	C2—C3—C4	111.90 (14)
C14—C9—N2	119.24 (13)	C2—C3—H3A	109.2
C10—C9—N2	120.33 (13)	C4—C3—H3A	109.2
O1—C7—N1	122.25 (13)	C2—C3—H3B	109.2
O1—C7—C6	123.07 (13)	C4—C3—H3B	109.2
N1—C7—C6	114.66 (12)	H3A—C3—H3B	107.9
C7—C6—C1	110.87 (13)	C6—C5—C4	110.71 (13)
C7—C6—C5	110.31 (12)	C6—C5—H5A	109.5
C1—C6—C5	111.02 (13)	C4—C5—H5A	109.5
C7—C6—H6A	108.2	C6—C5—H5B	109.5
C1—C6—H6A	108.2	C4—C5—H5B	109.5
C5—C6—H6A	108.2	H5A—C5—H5B	108.1
N2—C8—N1	115.78 (13)	C10—C11—C12	120.41 (16)
N2—C8—S1	125.19 (11)	C10—C11—H11A	119.8
N1—C8—S1	119.00 (10)	C12—C11—H11A	119.8
C11—C10—C9	119.65 (15)	C3—C2—C1	111.72 (14)
C11—C10—H10A	120.2	C3—C2—H2B	109.3
C9—C10—H10A	120.2	C1—C2—H2B	109.3
C9—C14—C13	119.36 (15)	C3—C2—H2C	109.3
C9—C14—H14A	120.3	C1—C2—H2C	109.3
C13—C14—H14A	120.3	H2B—C2—H2C	107.9
C12—C13—C14	120.57 (15)	C3—C4—C5	111.35 (14)
C12—C13—H13A	119.7	C3—C4—H4A	109.4
C14—C13—H13A	119.7	C5—C4—H4A	109.4
C2—C1—C6	110.98 (14)	C3—C4—H4B	109.4
C2—C1—H1B	109.4	C5—C4—H4B	109.4
C6—C1—H1B	109.4	H4A—C4—H4B	108.0
C8—N2—C9—C14	120.74 (16)	C10—C9—C14—C13	1.6 (2)
C8—N2—C9—C10	−62.8 (2)	N2—C9—C14—C13	178.01 (13)
C8—N1—C7—O1	−3.9 (2)	C9—C14—C13—C12	−1.2 (2)
C8—N1—C7—C6	177.58 (13)	C7—C6—C1—C2	−178.70 (13)
O1—C7—C6—C1	37.5 (2)	C5—C6—C1—C2	−55.71 (18)
N1—C7—C6—C1	−143.95 (14)	C14—C13—C12—C11	0.2 (3)
O1—C7—C6—C5	−85.88 (18)	C7—C6—C5—C4	179.44 (13)
N1—C7—C6—C5	92.65 (15)	C1—C6—C5—C4	56.13 (18)
C9—N2—C8—N1	178.47 (12)	C9—C10—C11—C12	0.1 (3)
C9—N2—C8—S1	−3.4 (2)	C13—C12—C11—C10	0.3 (3)
C7—N1—C8—N2	2.1 (2)	C4—C3—C2—C1	−54.5 (2)
C7—N1—C8—S1	−176.13 (12)	C6—C1—C2—C3	54.8 (2)

C14—C9—C10—C11	−1.0 (2)	C2—C3—C4—C5	54.9 (2)
N2—C9—C10—C11	−177.45 (14)	C6—C5—C4—C3	−55.56 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.86	1.95	2.6471 (18)	137
N1—H1A···S1^{i}	0.86	2.68	3.4997 (19)	160

Symmetry codes: (i) −*x*, −*y*, −*z*.

Fig. 1

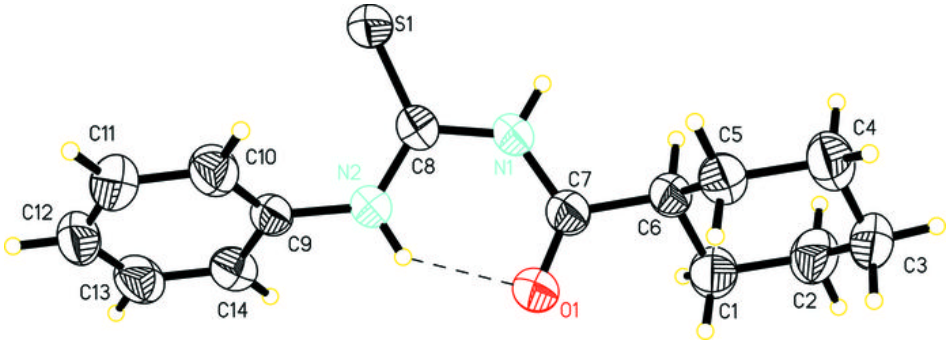

Fig. 2