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tert-Butyl *N*-[(1*R*,2*R*)-2-(isothiocyanato)-cyclohexyl]carbamate

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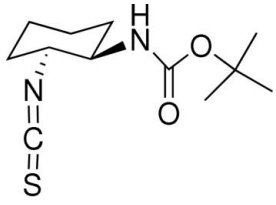
Key indicators: single-crystal X-ray study; T = 90 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.083; data-to-parameter ratio = 20.3.

In the crystal structure of the title compound, $C_{12}H_{20}N_2O_2S$, the molecules form one-dimensional chains along the [100] direction, based on a $C(4)$ hydrogen-bonding motif between the carbamate NH group of one molecule and the carbamate C=O group of a neighbouring molecule.

Related literature

For related literature, see: Allen *et al.* (1987); Drobnica *et al.* (1977); Etter (1990); Smith *et al.* (1996).



Experimental

Crystal data

$C_{12}H_{20}N_2O_2S$
M_r = 256.36
Monoclinic, $P2_1$
a = 5.1684 (2) Å
b = 8.5494 (3) Å
c = 15.9227 (5) Å
β = 95.518 (2)°
V = 700.31 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 90.0 (2) K
0.40 × 0.25 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.92, T_{max} = 0.98
12160 measured reflections
3181 independent reflections
2852 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.083
S = 1.07
3181 reflections
157 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), with 1476 Friedel pairs
Flack parameter: 0.04 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.88	2.15	2.9727 (17)	155

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

SL is grateful to Dr Sean Parkin for providing support and laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2075).

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supplementary materials

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tert-Butyl *N*-[(1*R*,2*R*)-2-(isothiocyanato)cyclohexyl]carbamate

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Comment

Isothiocyanates are important linkers for the facile synthesis of thioureas. The title compound (I), $C_{12}H_{20}N_2O_2S$, is a chiral isothiocyanate which can form a thiourea bond with another amine (Drobnica, *et al.*, 1977; and Smith, *et al.*, 1996).

The asymmetric unit of (I), (Fig. 1), contains one molecule and the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) exist bewteen the NH of the carbamate of one molecule and the carbonyl O of the carbamate of another molecule. The molecules form one-dimensional chains along the [100] direction, based on a C(4) (Etter, 1990) hydrogen bonding motif (Fig. 2).

Experimental

In a 50-ml round-bottom flask under N_2 was placed dicyclohexylcarbodiimide (0.72 g, 3.50 mmol), carbon disulfide (1.5 ml, 24.9 mmol), and THF (20 ml). The solution was cooled to −5 °C. To this was added a solution of *tert*-butyl (1*R*,2*R*)-2-aminocyclohexylcarbamate (0.75 g, 3.50 mmol) in THF (10 ml) over a period of 30 min. This mixture was allowed to stir at ambient temperature overnight. THF was removed under reduced pressure, and the resulting white solid was resuspended in diethyl ether (20 ml) and filtered. The filtrate was concentrated to give a white solid which was further purified by flash chromatography (hexanes/ethyl acetate, 3:1) to afford 0.70 g of a white crystalline material (78%) (Smith, *et al.*, 1996).

Refinement

H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained C—H distances of 0.98 Å (CH_3), 0.99 Å (CH_2), 1.00 Å (CH_1), and 0.88 Å (N—H). U_{iso}(H) values were set to either $1.5U_{eq}$ of the attached C atom (CH_3) or $1.2U_{eq}$ for all other H atoms.

Figures

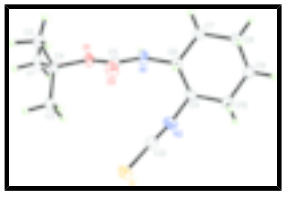

Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

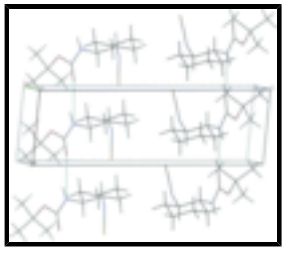

Fig. 2. A packing diagram of (I) viewed along the *b* axis. In broken lines, the H bonds defining the [100] chains.

tert-Butyl _N_-[(1_R_,2_R_)-2-(isothiocyanato)cyclohexyl]carbamate

Crystal data

$C_{12}H_{20}N_2O_2S$	F_{000} = 276
M_r = 256.36	D_x = 1.216 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1710 reflections
a = 5.1684 (2) Å	θ = 1.0–27.5º
b = 8.5494 (3) Å	μ = 0.23 mm^{-1}
c = 15.9227 (5) Å	T = 90.0 (2) K
β = 95.518 (2)º	Thick plate, colourless
V = 700.31 (4) Å^3	0.40 × 0.25 × 0.10 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	3181 independent reflections
Radiation source: fine-focus sealed tube	2852 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
Detector resolution: 18 pixels mm^{-1}	θ_{max} = 27.5º
T = 90.0(2) K	θ_{min} = 1.3º
ω scans at fixed χ = 55°	h = −6→6
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	k = −11→11
T_{min} = 0.92, T_{max} = 0.98	l = −20→20
12160 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 0.1871P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.083	$(\Delta/\sigma)_{max} < 0.001$
S = 1.07	$\Delta\rho_{max}$ = 0.19 e Å^{-3}

3181 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
157 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (6)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\sigma(F^2)$ 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}
C1	0.5682 (3)	0.9658 (2)	0.04080 (13)	0.0298 (4)
H1A	0.4673	0.9113	−0.0055	0.045*
H1B	0.6622	1.0534	0.0182	0.045*
H1C	0.4506	1.0056	0.0806	0.045*
C2	0.9247 (3)	0.7762 (2)	0.02368 (11)	0.0241 (4)
H2A	1.0591	0.7118	0.0545	0.036*
H2B	1.0073	0.8567	−0.0085	0.036*
H2C	0.8142	0.7099	−0.0151	0.036*
C3	0.9231 (3)	0.9369 (2)	0.15731 (11)	0.0257 (4)
H3A	0.8100	0.9727	0.1994	0.038*
H3B	1.0108	1.0270	0.1347	0.038*
H3C	1.0532	0.8643	0.1838	0.038*
C4	0.7601 (3)	0.8538 (2)	0.08578 (10)	0.0188 (3)
C5	0.6834 (3)	0.6210 (2)	0.17045 (10)	0.0186 (3)
C6	0.5257 (3)	0.4216 (2)	0.26417 (10)	0.0185 (3)
H6	0.7153	0.3958	0.2719	0.022*
C7	0.3768 (3)	0.2729 (2)	0.23807 (10)	0.0219 (4)
H7A	0.4390	0.2317	0.1855	0.026*
H7B	0.1897	0.2977	0.2264	0.026*
C8	0.4121 (4)	0.1481 (2)	0.30674 (12)	0.0274 (4)
H8A	0.3076	0.0548	0.2888	0.033*
H8B	0.5970	0.1164	0.3148	0.033*
C9	0.3281 (4)	0.2087 (2)	0.38992 (12)	0.0279 (4)
H9A	0.1396	0.2320	0.3832	0.033*
H9B	0.3595	0.1272	0.4339	0.033*
C10	0.4781 (4)	0.3554 (3)	0.41734 (11)	0.0286 (4)
H10A	0.6649	0.3298	0.4291	0.034*

H10B	0.4155	0.3960	0.4700	0.034*
C11	0.4441 (3)	0.4810 (2)	0.34895 (11)	0.0229 (4)
H11	0.2564	0.5112	0.3409	0.028*
C12	0.7622 (3)	0.7085 (2)	0.38407 (11)	0.0244 (4)
N1	0.4866 (3)	0.54204 (18)	0.20018 (9)	0.0212 (3)
H1	0.3265	0.5645	0.1798	0.025*
N2	0.5948 (3)	0.6181 (2)	0.37449 (11)	0.0332 (4)
O1	0.5876 (2)	0.73878 (14)	0.12093 (7)	0.0202 (3)
O2	0.9141 (2)	0.58898 (16)	0.18584 (8)	0.0231 (3)
S1	0.98280 (9)	0.83806 (6)	0.40018 (3)	0.03501 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0189 (8)	0.0283 (11)	0.0429 (11)	0.0002 (8)	0.0064 (8)	0.0156 (9)
C2	0.0187 (8)	0.0296 (10)	0.0248 (9)	−0.0032 (7)	0.0064 (7)	−0.0024 (8)
C3	0.0248 (9)	0.0263 (10)	0.0265 (9)	−0.0074 (8)	0.0057 (7)	−0.0034 (8)
C4	0.0116 (6)	0.0208 (9)	0.0246 (8)	−0.0029 (7)	0.0043 (6)	0.0024 (8)
C5	0.0154 (7)	0.0204 (9)	0.0203 (8)	−0.0007 (7)	0.0029 (6)	−0.0011 (7)
C6	0.0142 (7)	0.0201 (9)	0.0215 (8)	0.0009 (7)	0.0022 (6)	0.0022 (7)
C7	0.0235 (8)	0.0219 (9)	0.0202 (9)	−0.0007 (7)	0.0014 (7)	−0.0005 (7)
C8	0.0309 (10)	0.0209 (10)	0.0299 (10)	−0.0027 (8)	0.0008 (8)	0.0009 (8)
C9	0.0298 (9)	0.0305 (11)	0.0234 (9)	−0.0069 (8)	0.0025 (7)	0.0052 (8)
C10	0.0329 (9)	0.0336 (11)	0.0194 (8)	−0.0105 (9)	0.0033 (7)	−0.0009 (8)
C11	0.0209 (8)	0.0210 (9)	0.0276 (9)	−0.0064 (7)	0.0055 (7)	−0.0038 (7)
C12	0.0250 (9)	0.0270 (10)	0.0212 (8)	−0.0002 (8)	0.0022 (7)	−0.0001 (8)
N1	0.0104 (6)	0.0259 (9)	0.0272 (8)	0.0013 (6)	0.0016 (5)	0.0074 (6)
N2	0.0367 (9)	0.0285 (10)	0.0350 (9)	−0.0117 (8)	0.0070 (7)	−0.0077 (7)
O1	0.0117 (5)	0.0216 (7)	0.0278 (6)	−0.0004 (5)	0.0033 (4)	0.0067 (5)
O2	0.0115 (5)	0.0263 (7)	0.0317 (7)	0.0015 (5)	0.0025 (5)	0.0060 (6)
S1	0.0318 (2)	0.0396 (3)	0.0333 (3)	−0.0163 (2)	0.00155 (19)	−0.0024 (2)

Geometric parameters (Å, °)

C1—C4	1.509 (2)	C6—H6	1.0000
C1—H1A	0.9800	C7—C8	1.526 (3)
C1—H1B	0.9800	C7—H7A	0.9900
C1—H1C	0.9800	C7—H7B	0.9900
C2—C4	1.518 (2)	C8—C9	1.524 (3)
C2—H2A	0.9800	C8—H8A	0.9900
C2—H2B	0.9800	C8—H8B	0.9900
C2—H2C	0.9800	C9—C10	1.517 (3)
C3—C4	1.526 (2)	C9—H9A	0.9900
C3—H3A	0.9800	C9—H9B	0.9900
C3—H3B	0.9800	C10—C11	1.527 (3)
C3—H3C	0.9800	C10—H10A	0.9900
C4—O1	1.473 (2)	C10—H10B	0.9900
C5—O2	1.2247 (19)	C11—N2	1.444 (2)
C5—O1	1.344 (2)	C11—H11	1.0000

C5—N1	1.344 (2)	C12—N2	1.159 (2)
C6—N1	1.449 (2)	C12—S1	1.5927 (19)
C6—C7	1.524 (2)	N1—H1	0.8800
C6—C11	1.539 (2)		
C4—C1—H1A	109.5	C8—C7—H7A	109.3
C4—C1—H1B	109.5	C6—C7—H7B	109.3
H1A—C1—H1B	109.5	C8—C7—H7B	109.3
C4—C1—H1C	109.5	H7A—C7—H7B	108.0
H1A—C1—H1C	109.5	C9—C8—C7	111.17 (16)
H1B—C1—H1C	109.5	C9—C8—H8A	109.4
C4—C2—H2A	109.5	C7—C8—H8A	109.4
C4—C2—H2B	109.5	C9—C8—H8B	109.4
H2A—C2—H2B	109.5	C7—C8—H8B	109.4
C4—C2—H2C	109.5	H8A—C8—H8B	108.0
H2A—C2—H2C	109.5	C10—C9—C8	110.54 (15)
H2B—C2—H2C	109.5	C10—C9—H9A	109.5
C4—C3—H3A	109.5	C8—C9—H9A	109.5
C4—C3—H3B	109.5	C10—C9—H9B	109.5
H3A—C3—H3B	109.5	C8—C9—H9B	109.5
C4—C3—H3C	109.5	H9A—C9—H9B	108.1
H3A—C3—H3C	109.5	C9—C10—C11	110.82 (14)
H3B—C3—H3C	109.5	C9—C10—H10A	109.5
O1—C4—C1	102.12 (12)	C11—C10—H10A	109.5
O1—C4—C2	110.70 (14)	C9—C10—H10B	109.5
C1—C4—C2	110.63 (14)	C11—C10—H10B	109.5
O1—C4—C3	109.76 (13)	H10A—C10—H10B	108.1
C1—C4—C3	110.44 (16)	N2—C11—C10	110.33 (15)
C2—C4—C3	112.70 (13)	N2—C11—C6	109.17 (14)
O2—C5—O1	125.56 (15)	C10—C11—C6	111.81 (16)
O2—C5—N1	124.94 (16)	N2—C11—H11	108.5
O1—C5—N1	109.50 (13)	C10—C11—H11	108.5
N1—C6—C7	111.61 (13)	C6—C11—H11	108.5
N1—C6—C11	110.57 (15)	N2—C12—S1	176.98 (18)
C7—C6—C11	109.91 (13)	C5—N1—C6	123.01 (14)
N1—C6—H6	108.2	C5—N1—H1	118.5
C7—C6—H6	108.2	C6—N1—H1	118.5
C11—C6—H6	108.2	C12—N2—C11	162.54 (19)
C6—C7—C8	111.45 (14)	C5—O1—C4	121.36 (12)
C6—C7—H7A	109.3		
N1—C6—C7—C8	−178.30 (14)	O2—C5—N1—C6	9.0 (3)
C11—C6—C7—C8	−55.25 (18)	O1—C5—N1—C6	−171.51 (14)
C6—C7—C8—C9	56.88 (19)	C7—C6—N1—C5	−130.07 (17)
C7—C8—C9—C10	−57.0 (2)	C11—C6—N1—C5	107.26 (18)
C8—C9—C10—C11	56.5 (2)	C10—C11—N2—C12	92.0 (7)
C9—C10—C11—N2	−178.01 (14)	C6—C11—N2—C12	−31.2 (7)
C9—C10—C11—C6	−56.31 (19)	O2—C5—O1—C4	−6.1 (3)
N1—C6—C11—N2	−58.75 (18)	N1—C5—O1—C4	174.42 (14)
C7—C6—C11—N2	177.59 (15)	C1—C4—O1—C5	−176.42 (15)

N1—C6—C11—C10	178.88 (14)	C2—C4—O1—C5	65.79 (18)
C7—C6—C11—C10	55.22 (17)	C3—C4—O1—C5	−59.24 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.88	2.15	2.9727 (17)	155

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

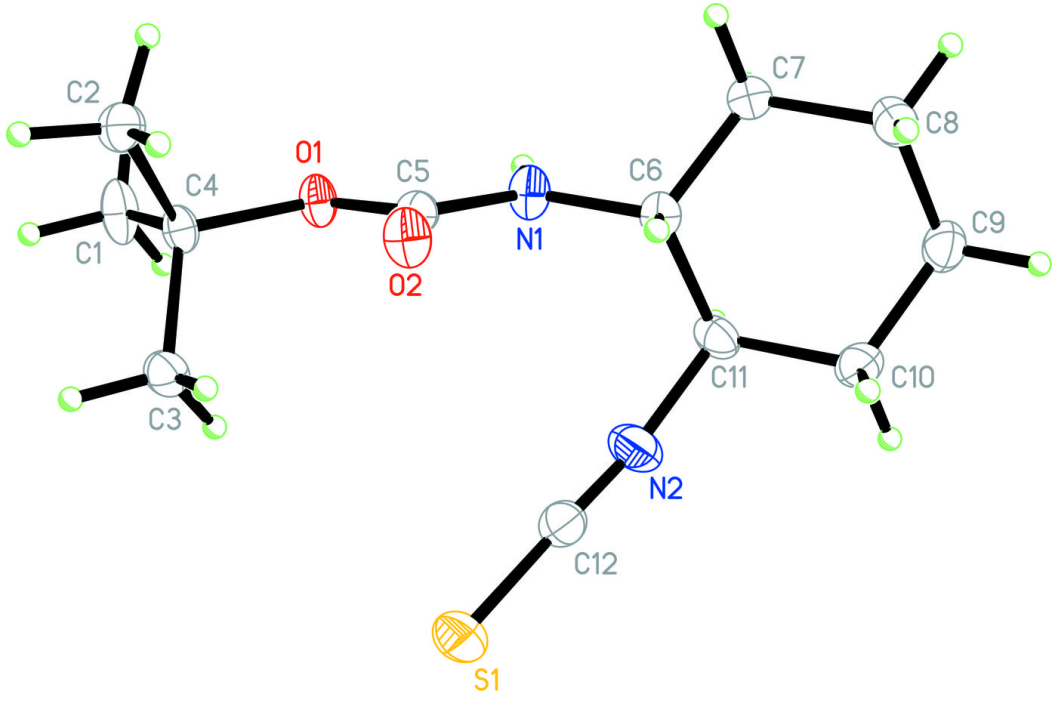

Fig. 2