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1-[(1*R*,2*R*)-2-(1,8-Naphthalimido)cyclohexyl]-3-phenylthiourea

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Key indicators

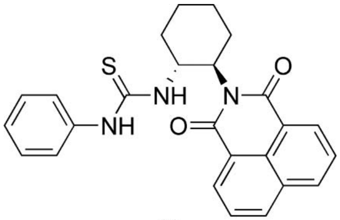
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.102
Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{25}H_{23}N_3O_2S$, the asymmetric unit consists of two molecules connected by N—H···S hydrogen bonds. The dihedral angles between the phenyl and naphthalimide ring systems in the scorpion-like molecules are 60.63 (7) and 61.09 (8)°. The cyclohexane rings adopt chair conformations.

Comment

Chiral recognition of carboxylic acids is an interesting and challenging topic as they are important structural components of many natural products and drugs (Coppola & Schuster, 1997; Hanessian *et al.*, 1983; Maier *et al.*, 2001; Valentine *et al.*, 1980). In a search for novel chiral CSAs (chiral solvating agents) (Chinchilla *et al.*, 1995; Parker & Taylor, 1987) for the recognition of chiral carboxylic acids, we have designed and synthesized the title compound, (I), and its structure is reported here.



(I)

The asymmetric unit of (I) (Fig. 1) consists of two molecules connected by N3—H3*A*···S2 and N6—H6···S1 intermolecular hydrogen bonds (Table 1). There is also an N—H···O intramolecular hydrogen bond between the cyclohexylamine H atom and a carbonyl O atom of the naphthalimide group in both molecules. The molecules adopt a scorpion-like shape with dihedral angles 60.63 (7) and 61.09 (8)° between the phenyl and naphthalimide ring planes of the two molecules. The cyclohexane rings exhibit chair conformations.

In the crystal structure, molecules link into pairs through N—H···S hydrogen bonds. Adjacent pairs interlock, forming chains running along the *a* axis (Fig. 2). These chains pack into columns down the *a* axis (Fig. 3).

Experimental

Phenyl isothiocyanate (0.59 g, 0.0044 mol) in dichloromethane (10 ml) was added slowly to (1*R*,2*R*)-1-(1,8-naphthalimido)-2-aminocyclohexane (1.30 g, 0.0044 mol) in dichloromethane (40 ml) and the mixture stirred at room temperature for 5 h. The solvent was

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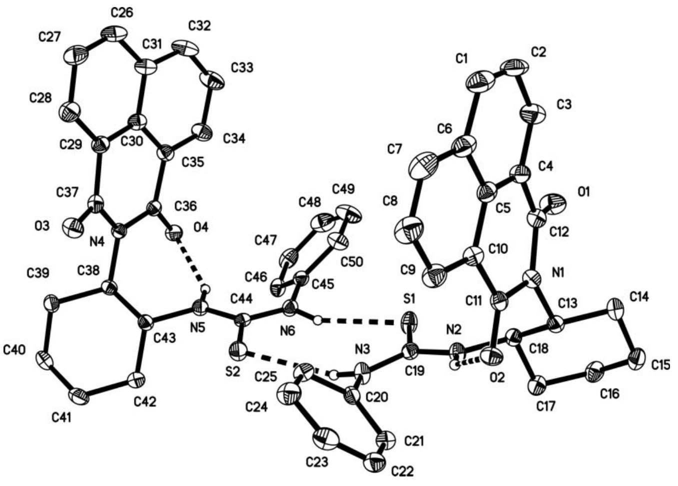

Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level, showing the hydrogen-bonding interactions. Hydrogen bonds are represented as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

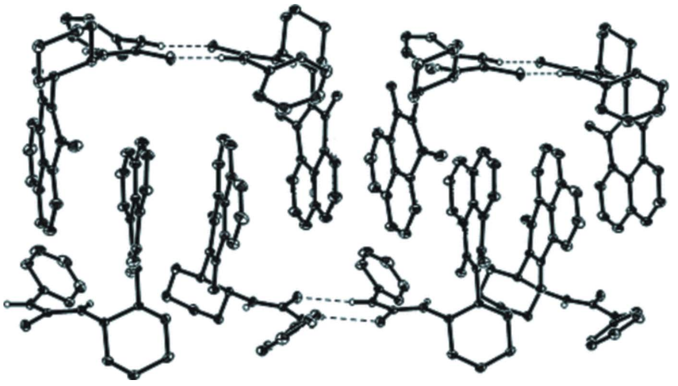

Figure 2
One-dimensional chains along the *a* axis. Hydrogen bonds are represented as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

evaporated to dryness under reduced pressure and the remaining residue recrystallized from ethanol to afford 1.61 g of yellow needle-like crystals (yield 85%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{25}H_{23}N_3O_2S$
$M_r = 429.52$
Orthorhombic, $P2_12_12_1$
$a = 14.8211$ (7) Å
$b = 16.8568$ (8) Å
$c = 17.3150$ (8) Å
$V = 4325.9$ (4) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.18$ mm^{-1}
$T = 173$ (2) K
$0.41 \times 0.30 \times 0.27$ mm

Data collection

Bruker AXS SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.931$, $T_{\max} = 0.954$
22367 measured reflections
9357 independent reflections
6724 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$

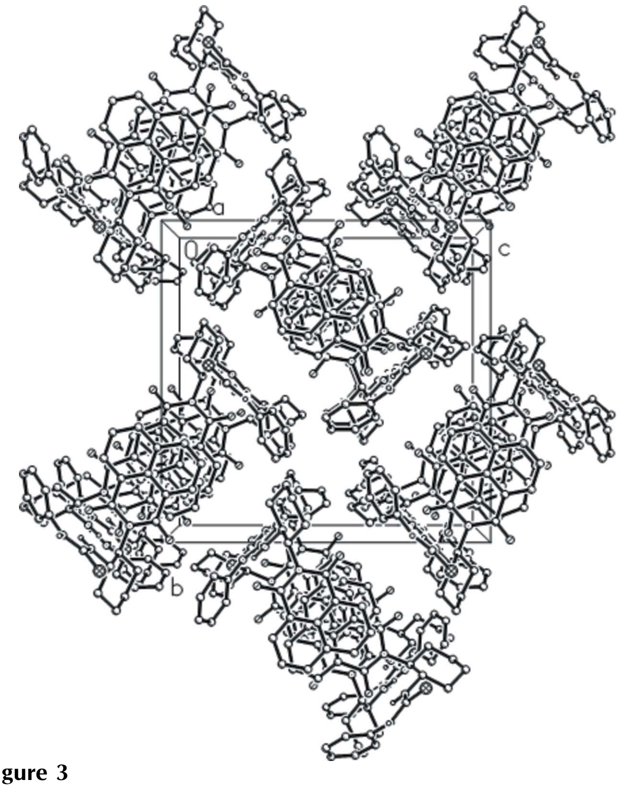

Figure 3
The crystal packing of (I). H atoms have been omitted for clarity.

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.102$
$S = 1.05$
9357 reflections
559 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Absolute structure: Flack (1983), 4114 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
N2—H2A···O2	0.88	2.11	2.876 (3)	145
N3—H3A···S2	0.88	2.50	3.306 (2)	152
N5—H5···O4	0.88	2.17	2.928 (3)	145
N6—H6···S1	0.88	2.46	3.295 (2)	159

All H atoms were positioned geometrically and refined using a riding model with, C—H = 0.95 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for aromatic, C—H = 1.00 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for CH, C—H = 0.99 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for CH_2, and N—H = 0.88 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(N).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2005); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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