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The crystal structure of *N*-(adamantan-1-yl)-piperidine-1-carbothioamide, C₁₆H₂₆N₂S

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Abstract

C₁₆H₂₆N₂S, monoclinic, *P*₂₁/*n* (no. 14), *a* = 6.4855(4) Å, *b* = 20.1110(12) Å, *c* = 11.2575(7) Å, *β* = 92.014(2)°, *V* = 1467.41(16) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0324, *wR*_{ref}(*F*²) = 0.0889, *T* = 200 K.

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The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

To a solution of 1-adamantylisothiocyanate (1 mmol) in ethanol was added piperidine (1.1 mmol). The mixture was

Table 1: Data collection and handling.

Crystal:	Colourless rods
Size:	0.63 × 0.15 × 0.13 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
<i>μ</i> :	0.21 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, <i>φ</i> and <i>ω</i>
<i>θ</i> _{max} , completeness:	28.4°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	31,238, 3649, 0.020
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3197
<i>N</i> (<i>param</i>) _{refined} :	176
Programs:	Bruker [1], SHELX [2–4], WinGX/ ORTEP [5], PLATON [6], Mercury [7]

refluxed for 3 h and after completion of the reaction, as indicated by TLC, the reaction mixture was poured into crushed ice, and the solid separated was filtered under suction, washed with cold ethanol (50 ml), and subsequently dried to afford the pure products. The melting point recorded is 185 °C and is uncorrected.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 *U*_{eq}(C). The nitrogen-bound H atom was located on a difference map and refined freely.

Comment

The titled compound discussed here is a result of continuation in the use of adamantyl isothiocyanates for the synthesis of promising adamantyl compounds which have recently shown to possess anti-microbial, anti-fungal and anti-proliferative cancer activities [8].

The crystal structure of the title compound contains one molecule in the asymmetric unit. The bond lengths and angles are in the expected ranges [9, 10]. The piperidine ring has a chair conformation. The atoms of the isothiocyanate group, the piperidine nitrogen and its two adjacent

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
S1	0.14338 (4)	0.62127 (2)	0.49443 (3)	0.03099 (10)
N1	0.51615 (15)	0.65768 (5)	0.58063 (9)	0.0258 (2)
N2	0.42327 (15)	0.54964 (5)	0.61764 (9)	0.0269 (2)
C1	0.53564 (15)	0.72243 (5)	0.51914 (9)	0.0186 (2)
C2	0.36573 (16)	0.77204 (5)	0.54965 (9)	0.0214 (2)
H2A	0.361900	0.777542	0.636934	0.026*
H2B	0.229918	0.754948	0.520676	0.026*
C3	0.40885 (16)	0.83942 (5)	0.49113 (10)	0.0227 (2)
H3	0.296723	0.871442	0.510118	0.027*
C4	0.41424 (17)	0.83028 (6)	0.35653 (10)	0.0252 (2)
H4A	0.439906	0.873645	0.318139	0.030*
H4B	0.279379	0.813273	0.325836	0.030*
C5	0.58507 (16)	0.78119 (6)	0.32631 (9)	0.0236 (2)
H5	0.588147	0.775612	0.238121	0.028*
C6	0.79301 (17)	0.80836 (6)	0.37298 (10)	0.0271 (2)
H6A	0.904487	0.776923	0.353787	0.033*
H6B	0.821693	0.851436	0.334330	0.033*
C7	0.78747 (16)	0.81778 (5)	0.50745 (10)	0.0230 (2)
H7	0.923717	0.835082	0.538171	0.028*
C8	0.74292 (16)	0.75089 (5)	0.56604 (10)	0.0220 (2)
H8A	0.739149	0.756802	0.653241	0.026*
H8B	0.854957	0.719170	0.549298	0.026*
C9	0.54378 (16)	0.71377 (5)	0.38415 (9)	0.0221 (2)
H9A	0.654715	0.682095	0.365122	0.027*
H9B	0.411094	0.695517	0.352578	0.027*
C10	0.61694 (17)	0.86709 (5)	0.53740 (11)	0.0254 (2)
H10A	0.643561	0.910629	0.499866	0.030*
H10B	0.614575	0.873635	0.624503	0.030*
C11	0.37282 (17)	0.60922 (5)	0.56799 (9)	0.0222 (2)
C12	0.27274 (19)	0.49579 (6)	0.62979 (11)	0.0308 (3)
H12A	0.322315	0.455324	0.589646	0.037*
H12B	0.139363	0.508913	0.591210	0.037*
C13	0.2426 (2)	0.48108 (7)	0.76015 (12)	0.0354 (3)
H13A	0.147168	0.442992	0.767247	0.042*
H13B	0.179208	0.520143	0.797987	0.042*
C14	0.4463 (2)	0.46491 (7)	0.82473 (12)	0.0390 (3)
H14A	0.498674	0.421797	0.795882	0.047*
H14B	0.424012	0.460654	0.910940	0.047*
C15	0.6063 (2)	0.51906 (6)	0.80432 (11)	0.0315 (3)
H15A	0.564713	0.560596	0.844144	0.038*
H15B	0.741749	0.505107	0.839137	0.038*
C16	0.62444 (17)	0.53150 (6)	0.67231 (10)	0.0264 (2)
H16A	0.724389	0.567835	0.659914	0.032*
H16B	0.676761	0.490925	0.633714	0.032*
H1	0.619 (2)	0.6474 (8)	0.6210 (14)	0.038 (4)*

carbon atoms are remarkably planar with a least square r.m.s. deviation of only 0.0795. As a consequence these forces lead to a relatively close H···H contact of 1.79 Å

between the amine hydrogen and a piperidine hydrogen. This shielding of the amine hydrogen prevents its involvement in any hydrogen bond interactions. There are two intramolecular C–H···S interactions of 2.76 and 2.83 Å. The shortest intermolecular interaction is C8–H8B···S1 (symmetry code: $x + 1, y, z$) of 2.80 Å, which links adjacent molecules parallel to the *a* axis.

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