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The crystal structure of ethyl-1-(*N*-(adamantan-1-yl)-carbamothioyl)piperidine-4-carboxylate, $C_{19}H_{30}N_2O_2S$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.54 × 0.38 × 0.33 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.19 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	28.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	38096, 4627, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3915
$N(\text{param})_{\text{refined}}$:	222
Programs:	Bruker [1], SHELX [2–4], WinGX/ORTEP [5], PLATON [6], Mercury [7]

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Abstract

$C_{19}H_{30}N_2O_2S$, monoclinic, $P2_1/n$ (no. 14), $a = 6.4977(5)$ Å, $b = 28.728(2)$ Å, $c = 10.1806(8)$ Å, $\beta = 101.549(3)^\circ$, $V = 1861.9(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0460$, $wR_{\text{ref}}(F^2) = 0.1213$, $T = 296$ 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 material

To a solution of 1-adamantylisothiocyanate (1 mmol) in ethanol was added ethyl-4-piperidine-4-carboxylate (1.1 mmol). The mixture was refluxed for 3 h and after completion of the reaction, indicated by TLC, the reaction mixture

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	−0.08922(6)	0.68167(2)	0.32236(5)	0.04541(13)
O2	0.47842(19)	0.87114(5)	0.27657(12)	0.0532(3)
O3	0.1549(2)	0.85269(5)	0.17229(14)	0.0668(4)
N1	0.3017(2)	0.64994(4)	0.41249(13)	0.0356(3)
N2	0.23567(19)	0.72341(4)	0.48022(13)	0.0358(3)
C1	0.3077(2)	0.61183(5)	0.31591(13)	0.0309(3)
C2	0.3057(3)	0.63121(6)	0.17554(15)	0.0401(3)
H2A	0.174577	0.647469	0.143052	0.048*
H2B	0.419829	0.653262	0.179395	0.048*
C3	0.3299(3)	0.59171(7)	0.07965(16)	0.0495(4)
H3	0.327111	0.604473	−0.009958	0.059*
C4	0.1492(3)	0.55698(7)	0.07318(17)	0.0500(4)
H4A	0.016065	0.572502	0.041184	0.060*
H4B	0.163024	0.532043	0.011285	0.060*
C5	0.1550(3)	0.53703(6)	0.21275(17)	0.0434(4)
H5	0.039704	0.514732	0.208588	0.052*
C6	0.3642(3)	0.51209(6)	0.2624(2)	0.0528(4)
H6A	0.380264	0.486920	0.201695	0.063*
H6B	0.367432	0.498962	0.350522	0.063*
C7	0.5438(3)	0.54720(6)	0.26891(19)	0.0490(4)
H7	0.678417	0.531529	0.300973	0.059*
C8	0.5173(2)	0.58653(5)	0.36550(16)	0.0383(3)
H8A	0.632462	0.608431	0.371660	0.046*
H8B	0.520452	0.573829	0.454154	0.046*
C9	0.1289(2)	0.57649(5)	0.30945(15)	0.0364(3)
H9A	0.131158	0.563848	0.398083	0.044*
H9B	−0.005190	0.591799	0.278978	0.044*
C10	0.5391(3)	0.56717(7)	0.1296(2)	0.0574(5)
H10A	0.556692	0.542301	0.068361	0.069*

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H10B	0.653605	0.589078	0.133078	0.069*
C11	0.1623(2)	0.68496(5)	0.40723(14)	0.0320(3)
C12	0.0935(3)	0.76145(5)	0.49793(16)	0.0413(3)
H12A	0.141777	0.775716	0.585001	0.050*
H12B	−0.045995	0.749010	0.495479	0.050*
C13	0.0830(2)	0.79812(5)	0.38937(16)	0.0399(3)
H13A	−0.004524	0.823777	0.407249	0.048*
H13B	0.019437	0.784805	0.303232	0.048*
C14	0.3019(2)	0.81622(5)	0.38405(15)	0.0369(3)
H14	0.354071	0.833905	0.466296	0.044*
C15	0.4544(2)	0.77579(6)	0.37774(17)	0.0416(4)
H15A	0.415907	0.760376	0.291543	0.050*
H15B	0.595624	0.787961	0.385735	0.050*
C16	0.4510(2)	0.74069(5)	0.48893(17)	0.0401(3)
H16A	0.543814	0.714903	0.480398	0.048*
H16B	0.500340	0.755275	0.575427	0.048*
C17	0.2985(3)	0.84820(5)	0.26607(16)	0.0409(3)
C18	0.4986(3)	0.90183(7)	0.16575(18)	0.0569(5)
H18A	0.389056	0.925287	0.152942	0.068*
H18B	0.486464	0.884106	0.083454	0.068*
C19	0.7070(4)	0.92400(8)	0.2005(2)	0.0664(6)
H19A	0.726774	0.944092	0.128690	0.100*
H19B	0.813922	0.900434	0.214425	0.100*
H19C	0.716113	0.941929	0.281049	0.100*
H1	0.412(3)	0.6516(6)	0.4702(18)	0.039(4)*

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. Melting point recorded is 380–385 °C.

Experimental details

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 H atoms of the methyl group were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the SHELX system [3]), with *U*_{iso}(H) set to 1.5 *U*_{eq}(C).

Comment

Due to the presence of anticancer, antiviral and other biological properties associated with adamantane, it has received considerable attention. Moreover, adamantyl isothiocyanates are versatile synthetic precursors for the synthesis of various promising compounds. The change in biological activity when adamantyl is attached to compound is due to relatively high lipophilicity associate with adamantane.

The crystal structure of the title compound C₁₉H₃₀N₂O₂S contains one molecule in the asymmetric unit. The bond lengths and angles are in the expected ranges which fit

with those of related compounds in literature [8–11]. The piperidine ring has a chair conformation with the ethylcarboxylate in the equatorial position and the carbamothioyl in the axial position. All the hydrogen interactions are long, the shortest being the intermolecular N1–H1···O3 (symmetry code: 1/2 + *x*, 3/2 − *y*, 1/2 + *z*) interaction of length 2.332(18) Å linking adjacent molecules in a chain along [1 0 1] and a graph set descriptor of C¹₁(9) [11]. The shortest intramolecular interaction is C16–H16A···N1 of length 2.45 Å.

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