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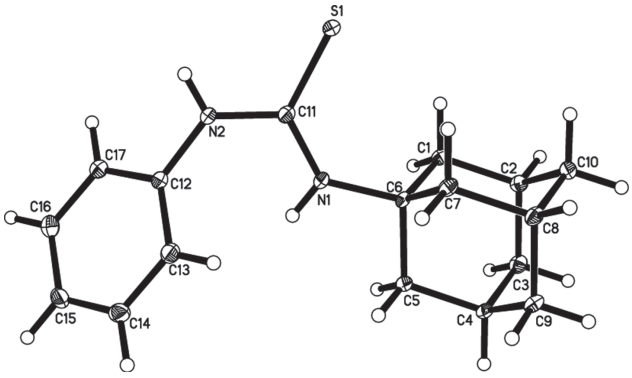
Crystal structure of 1-(adamantan-1-yl)-3-phenylthiourea, $C_{17}H_{22}N_2S$



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Abstract

$C_{17}H_{22}N_2S$, orthorhombic, *Pbca* (no. 61), $a = 12.0579(7)$ Å, $b = 11.12133(5)$ Å, $c = 21.9741(13)$ Å, $V = 2946.7(3)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0470$, $wR_{ref}(F^2) = 0.1125$, $T = 100$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	colourless, block, size 0.256×0.263×0.398 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.12 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8 venture, φ and ω scans
$2\theta_{max}$:	66.44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	75607, 5647
$N(param)_{refined}$:	189
Programs:	Bruker programs [21], SHELX [22]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	8*c*	0.1761	1.0092	0.3019	0.015
H(1B)	8*c*	0.2275	0.8773	0.2932	0.015
H(2A)	8*c*	0.2662	0.9974	0.2053	0.016
H(3A)	8*c*	0.4024	0.8491	0.2310	0.017
H(3B)	8*c*	0.4620	0.9633	0.2007	0.017
H(4A)	8*c*	0.5603	0.9046	0.2907	0.015
H(5A)	8*c*	0.4694	0.9182	0.3870	0.017
H(5B)	8*c*	0.4068	0.8219	0.3450	0.017
H(7A)	8*c*	0.3900	1.1240	0.4017	0.015
H(7B)	8*c*	0.2762	1.1614	0.3690	0.015
H(8A)	8*c*	0.4286	1.2449	0.3141	0.016
H(9A)	8*c*	0.5649	1.0971	0.3389	0.018
H(9B)	8*c*	0.5603	1.1148	0.2664	0.018
H(10A)	8*c*	0.2709	1.1887	0.2545	0.018
H(10B)	8*c*	0.3817	1.1707	0.2152	0.018
H(13A)	8*c*	0.3606	0.8937	0.5161	0.020
H(14A)	8*c*	0.4562	0.7413	0.5673	0.023
H(15A)	8*c*	0.3605	0.5779	0.6081	0.024
H(16A)	8*c*	0.1674	0.5679	0.5987	0.024
H(17)	8*c*	0.0720	0.7140	0.5434	0.021
H(1N2)	8*c*	0.078(1)	0.921(2)	0.5072(8)	0.023(4)
H(1N1)	8*c*	0.291(1)	0.868(2)	0.4235(7)	0.023(4)

Discussion

The adamantane nucleus represent an important structural motif in several biologically and pharmaceutically active drugs [1, 2]. Several adamantane-based drugs are currently used as efficient medications against influenza viral

Table 3: Fractional atomic coordinate and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	8*c*	0.08030(2)	1.07375(3)	0.41559(2)	0.0116(1)	0.0181(2)	0.0144(1)	0.0040(1)	0.0035(1)	0.0036(1)
N(1)	8*c*	0.26034(8)	0.9295(1)	0.41040(5)	0.0125(4)	0.0155(5)	0.0141(4)	0.0037(4)	0.0042(4)	0.0052(4)
N(2)	8*c*	0.14348(8)	0.9049(1)	0.49190(5)	0.0110(4)	0.0201(5)	0.0144(5)	0.0034(4)	0.0048(4)	0.0052(4)
C(1)	8*c*	0.24619(9)	0.9634(1)	0.29833(5)	0.0099(4)	0.0130(5)	0.0134(5)	−0.0011(4)	0.0006(4)	−0.0021(4)
C(2)	8*c*	0.31196(9)	1.0079(1)	0.24289(5)	0.0123(5)	0.0163(5)	0.0109(5)	0.0007(4)	−0.0005(4)	−0.0012(4)
C(3)	8*c*	0.4199(1)	0.9353(1)	0.23670(5)	0.0154(5)	0.0146(5)	0.0135(5)	0.0010(4)	0.0038(4)	−0.0028(4)
C(4)	8*c*	0.49018(9)	0.9521(1)	0.29439(5)	0.0102(5)	0.0141(5)	0.0142(5)	0.0022(4)	0.0037(4)	0.0005(4)
C(5)	8*c*	0.42416(9)	0.9084(1)	0.34975(5)	0.0112(5)	0.0159(6)	0.0148(5)	0.0033(4)	0.0030(4)	0.0036(4)
C(6)	8*c*	0.31567(9)	0.9803(1)	0.35617(5)	0.0095(5)	0.0133(5)	0.0095(5)	0.0001(4)	0.0027(4)	0.0009(4)
C(7)	8*c*	0.34492(9)	1.1135(1)	0.36445(5)	0.0111(5)	0.0141(5)	0.0123(5)	−0.0012(4)	0.0019(4)	−0.0034(4)
C(8)	8*c*	0.41048(9)	1.1578(1)	0.30882(5)	0.0120(5)	0.0109(5)	0.0171(5)	−0.0014(4)	0.0034(4)	−0.0011(4)
C(9)	8*c*	0.51822(9)	1.0858(1)	0.30224(6)	0.0099(5)	0.0170(6)	0.0168(5)	−0.0015(4)	0.0027(4)	−0.0006(4)
C(10)	8*c*	0.3401(1)	1.1413(1)	0.25103(6)	0.0147(5)	0.0142(5)	0.0148(5)	0.0026(4)	0.0024(4)	0.0032(4)
C(11)	8*c*	0.16826(9)	0.9648(1)	0.43974(5)	0.0109(5)	0.0142(5)	0.0124(5)	−0.0005(4)	0.0014(4)	0.0004(4)
C(12)	8*c*	0.20689(9)	0.8168(1)	0.52299(5)	0.0127(5)	0.0164(6)	0.0109(5)	0.0019(4)	0.0021(4)	0.0011(4)
C(13)	8*c*	0.3215(1)	0.8261(1)	0.53153(5)	0.0142(5)	0.0211(6)	0.0136(5)	−0.0016(4)	0.0004(4)	0.0007(4)
C(14)	8*c*	0.3780(1)	0.7362(1)	0.56271(6)	0.0143(5)	0.0280(7)	0.0141(5)	0.0026(5)	−0.0015(4)	−0.0022(5)
C(15)	8*c*	0.3214(1)	0.6394(1)	0.58715(6)	0.0215(6)	0.0247(7)	0.0130(5)	0.0074(5)	−0.0003(4)	0.0026(5)
C(16)	8*c*	0.2069(1)	0.6327(1)	0.58079(6)	0.0212(6)	0.0204(6)	0.0194(6)	0.0002(5)	0.0024(5)	0.0064(5)
C(17)	8*c*	0.1500(1)	0.7201(1)	0.54835(6)	0.0129(5)	0.0215(6)	0.0173(6)	−0.0002(4)	0.0019(4)	0.0043(5)

infections [3–5], malaria infections [6], central nervous disorders [7, 8], hyperglycaemia [9] and drug-resistant TB strain infections [10]. In addition, thiourea derivatives are recognized as potent anti-Malaria [11], anti-HIV [12], anticancer [13] and antimicrobial [14] agents. As a part of an ongoing research interest in the chemotherapeutic [15–17] and structure [18–20] of adamantane derivatives, we report herein, the crystal structural of the title compound. In the title structure the asymmetric unit of the crystal structure contains one molecule. The title molecule is a functionalized thiourea with an adamantyl and phenyl substituents attached to the two nitrogen atoms, N1 and N2. The molecules packing in the crystal structure is stabilized *via* one intermolecular hydrogen bond, of which S1 acts as the hydrogen bond acceptor and the NH group of N2 acts as hydrogen bond donor. The distance of the interactions between N2—H1N2$\cdots$S1^{i} is 2.554 (17) Å and the angle is 158.6(15) Symmetry code: (i) -*x*, -*y*+2, -*z*+1.

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT [21]. All hydrogen atoms were idealized and refined usind a riding model (AFIX 13, 23 or 43 option of the SHELX program [22]).

Source of material

A mixture of phenyl isothiocyanate (1.35 g, 0.01 mol) and 1-adamantylamine (1.51 mg, 0.01 mol), in ethanol (10 mL), was heated under reflux for 4 h. After cooling, the precipitated crude product was filtered, dried and crystallized from ethanol to yield 2.18 mg (76%) of the title compound ($C_{17}H_{22}N_2S$) as transparent block crystals. M.P.: 444–446 K. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in EtOH/$CHCl_3$ (1:1) at room temperature. ^{1}H NMR (DMSO-d_6, 500.13 MHz): δ 1.66–1.69 (m, 6H, Adamantane-H), 2.02 (s, 3H, Adamantane-H), 2.14–2.18 (m, 6H, Adamantane-H), 6.95 (d, 2H, Ar—H, $J = 7.0$ Hz), 7.25–7.27 (m, 1H, Ar—H), 7.41 (s, 1H, NH), 9.34 (s, 1H, NH). ^{13}C NMR (DMSO-d_6, 125.76 MHz): δ 28.50, 34.96, 41.82, 51.22 (Adamantane-C), 123.22, 126.84, 130.32, 138.74 (Ar—C), 180.26 (C = S). ESI-MS, *m/z*: 285.3 $(M-H)^-$.

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