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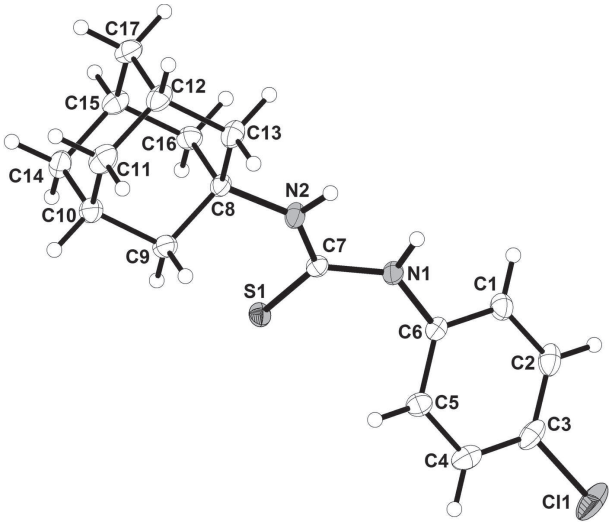
Crystal structure of 1-(adamantan-1-yl)-3-(4-chlorophenyl)thiourea, $C_{17}H_{21}ClN_2S$



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Abstract

$C_{17}H_{21}ClN_2S$, orthorhombic, *Pbca* (no. 61), $a = 17.2134(6)$ Å, $b = 8.2251(2)$ Å, $c = 22.5220(7)$ Å, $V = 3188.71(17)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0371$, $wR_{ref}(F^2) = 0.0933$, $T = 100$ 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 0.142×0.214×0.451 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.66 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	72.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14409, 7753
$N(param)_{refined}$:	198
Programs:	Bruker programs [22], SHELX [23]

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*	0.8917	0.7658	0.6787	0.017
H(2A)	8*c*	0.9304	0.9244	0.5973	0.020
H(4A)	8*c*	0.7052	0.9701	0.5470	0.020
H(5A)	8*c*	0.6667	0.8057	0.6269	0.016
H(9A)	8*c*	0.5368	0.6299	0.8014	0.016
H(9B)	8*c*	0.5347	0.4517	0.7725	0.016
H(10A)	8*c*	0.4399	0.4711	0.8502	0.018
H(11A)	8*c*	0.4963	0.2092	0.8353	0.021
H(11B)	8*c*	0.4762	0.2336	0.9043	0.021
H(12A)	8*c*	0.6051	0.1259	0.8937	0.018
H(13A)	8*c*	0.6345	0.2444	0.7997	0.018
H(13B)	8*c*	0.7020	0.2873	0.8461	0.018
H(14A)	8*c*	0.4816	0.5120	0.9499	0.019
H(14B)	8*c*	0.5060	0.6659	0.9104	0.019
H(15A)	8*c*	0.6132	0.5828	0.9706	0.017
H(16A)	8*c*	0.6446	0.6998	0.8765	0.016
H(16B)	8*c*	0.7081	0.5645	0.8930	0.016
H(17A)	8*c*	0.6705	0.3206	0.9546	0.020
H(17B)	8*c*	0.5827	0.2996	0.9772	0.020
H(1N1)	8*c*	0.7854(8)	0.578(2)	0.7114(6)	0.025(4)
H(1N2)	8*c*	0.7203(8)	0.464(2)	0.7694(6)	0.022(3)

Source of material

4-Chlorophenyl isothiocyanate (1.70 g, 0.01 mol) was added to a solution of 1-adamantylamine (1.51 mg, 0.01 mol), in ethanol (15 mL), and the mixture was heated under reflux for 2 h. The mixture was then concentrated to half the original

Table 3: Fractional coordinates and atomic 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*	0.64104(2)	0.84027(2)	0.75533(2)	0.01178(8)	0.00966(8)	0.01413(9)	0.00131(6)	0.00193(7)	0.00147(6)
N(1)	8*c*	0.75609(4)	0.66253(9)	0.70713(3)	0.0125(3)	0.0101(3)	0.0127(3)	0.0019(2)	0.0031(2)	0.0025(2)
N(2)	8*c*	0.68581(4)	0.53178(9)	0.77633(3)	0.0147(3)	0.0101(3)	0.0141(3)	0.0026(2)	0.0057(2)	0.0025(2)
Cl(1)	8*c*	0.84980(2)	1.07615(3)	0.50415(2)	0.0436(2)	0.0160(1)	0.0165(1)	−0.00017(9)	0.01294(9)	0.00406(7)
C(1)	8*c*	0.85390(5)	0.8061(1)	0.65169(4)	0.0130(3)	0.0132(3)	0.0164(4)	0.0004(3)	0.0029(3)	0.0019(3)
C(2)	8*c*	0.87704(6)	0.9007(1)	0.60353(4)	0.0171(4)	0.0133(3)	0.0200(4)	−0.0006(3)	0.0077(3)	0.0013(3)
C(3)	8*c*	0.82123(6)	0.9597(1)	0.56492(4)	0.0270(5)	0.0101(3)	0.0119(3)	−0.0005(3)	0.0061(3)	0.0008(3)
C(4)	8*c*	0.74287(6)	0.9270(1)	0.57348(4)	0.0236(4)	0.0145(4)	0.0112(3)	0.0005(3)	−0.0013(3)	0.0003(3)
C(5)	8*c*	0.72005(5)	0.8305(1)	0.62117(4)	0.0153(3)	0.0131(3)	0.0116(3)	−0.0011(3)	−0.0014(3)	−0.0006(3)
C(6)	8*c*	0.77555(5)	0.77017(9)	0.66055(3)	0.0132(3)	0.0086(3)	0.0100(3)	−0.0003(3)	0.0016(2)	−0.0004(2)
C(7)	8*c*	0.69512(5)	0.6703(1)	0.74585(3)	0.0109(3)	0.0101(3)	0.0097(3)	−0.0010(2)	−0.0002(2)	0.0002(2)
C(8)	8*c*	0.63293(5)	0.4888(1)	0.82545(3)	0.0124(3)	0.0102(3)	0.0099(3)	0.0004(3)	0.0022(2)	0.0014(2)
C(9)	8*c*	0.54702(5)	0.5133(1)	0.80904(4)	0.0123(3)	0.0152(3)	0.0114(3)	−0.0019(3)	−0.0009(3)	0.0015(3)
C(10)	8*c*	0.49578(5)	0.4533(1)	0.86048(4)	0.0114(3)	0.0186(4)	0.0140(3)	−0.0024(3)	0.0006(3)	0.0030(3)
C(11)	8*c*	0.50970(6)	0.2719(1)	0.87140(4)	0.0195(4)	0.0165(4)	0.0164(4)	−0.0066(3)	0.0015(3)	0.0025(3)
C(12)	8*c*	0.59548(5)	0.2445(1)	0.88728(4)	0.0203(4)	0.0114(3)	0.0136(3)	−0.0005(3)	0.0033(3)	0.0029(3)
C(13)	8*c*	0.64664(5)	0.3060(1)	0.83629(4)	0.0192(4)	0.0107(3)	0.0141(3)	0.0020(3)	0.0050(3)	0.0019(3)
C(14)	8*c*	0.51544(5)	0.5485(1)	0.91697(4)	0.0163(4)	0.0181(4)	0.0134(3)	0.0024(3)	0.0042(3)	0.0007(3)
C(15)	8*c*	0.60081(5)	0.5210(1)	0.93354(4)	0.0183(4)	0.0152(4)	0.0101(3)	−0.0004(3)	−0.0005(3)	−0.0005(3)
C(16)	8*c*	0.65285(5)	0.5819(1)	0.88267(4)	0.0134(3)	0.0137(3)	0.0123(3)	−0.0011(3)	−0.0015(3)	0.0002(3)
C(17)	8*c*	0.61539(6)	0.3387(1)	0.94400(4)	0.0193(4)	0.0177(4)	0.0118(3)	0.0014(3)	0.0003(3)	0.0037(3)

volume and allowed to stand at room temperature for 3 h. The precipitated crude product was filtered, dried and crystallized from ethanol to yield 2.73 mg (85%) of the title compound ($C_{17}H_{21}ClN_2S$) as transparent block crystals. M.P.: 474–476 K. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in EtOH/$CHCl_3$ (1:2) at room temperature. ^{1}H NMR (DMSO-d_6, 500.13 MHz): δ 1.68–1.72 (m, 6H, adamantane-H), 2.04 (s, 3H, adamantane-H), 2.12–2.18 (m, 6H, adamantane-H), 6.82 (d, 2H, Ar-H, $J = 7.0$ Hz), 7.22 (d, 2H, Ar-H, $J = 7.0$ Hz), 7.82 (s, 1H, NH), 9.36 (s, 1H, NH). ^{13}C NMR (DMSO-d_6, 125.76 MHz): δ 28.65, 35.90, 41.87, 50.25 (adamantane-C), 127.23, 129.85, 131.30, 134.77 (Ar-C), 180.57 (C = S). ESI-MS, *m/z*: 319.2 (M−H, 100%)$^-$, 321.2 (M+2−H, 36%)$^-$.

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT [22]. The carbon-bound hydrogen atoms were idealized and refined using a riding model (AFIX 13, AFIX 23 or AFIX 43 option of the SHELX programm [23]. Nitrogen-bound hydrogen atoms were refined freely.

Discussion

Several adamantane-based drugs as amantadine [1, 2], rimantadine [3, 4] and tromantadine [5] are currently employed as efficient antiviral agents. In addition, numerous adamantane-based drugs are currently used as important medications against malaria [6], central nervous disorders [7–9], hyperglycaemia [10] and drug-resistant TB strain infections [11]. Moreover, thiourea derivatives were reported to possess marked anticancer [12], anti-HIV [13], antimalarial [14] and antimicrobial [15] activities. As a part of our current research interest in the pharmacological [16–18] and structural [19–21] properties of adamantane derivatives, we report herein the synthesis and crystal structure of the potentially bioactive title compound.

The molecules packing in the crystal structure is stabilized *via* two intermolecular hydrogen bonds, of which S1 work as hydrogen bond acceptor and the NH groups of N1 and N2 work as hydrogen bond donors. The distance of the interactions between N1—H1N1···S1^{i} and N2—H1N2···S1^{i} are 2.534(14) and 2.615(14) Å, respectively and the angles are 163.1(12) and 159.9(12)°, respectively. Symmetry codes: (i) $-x+3/2, y-1/2, z$.

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