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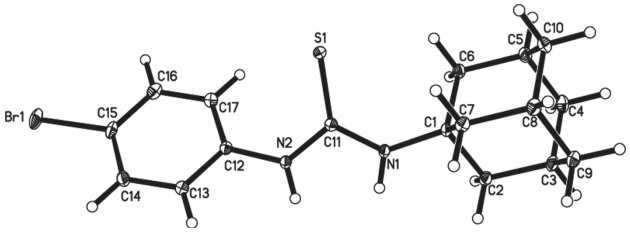
Crystal structure of 1-(adamantan-1-yl)-3-(4-bromophenyl)thiourea, $C_{17}H_{21}BrN_2S$



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Abstract

$C_{17}H_{21}BrN_2S$, orthorhombic, *Pbca* (No. 61), $a = 17.0675(7)$ Å, $b = 8.3422(3)$ Å, $c = 22.5970(8)$ Å, $V = 3217.4(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0329$, $wR_{ref}(F^2) = 0.0724$, $T = 100$ K.

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The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

To a solution of 1-adamantylamine (1.51 g, 0.01 mol) in ethanol (15 mL), 4-bromophenyl isothiocyanate (2.14 g, 0.01 mol) was added and the mixture was heated under reflux for three hours. On cooling, the precipitated crude product was filtered, dried and crystallized from ethanol to yield 3.36 g (92%) of the title compound ($C_{17}H_{21}BrN_2S$) as transparent block crystals. M.P.:

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Table 1: Data collection and handling.

Crystal:	Colourless blocks
	Size 0.51 × 0.27 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	26.8 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II , φ and ω
$2\theta_{max}$, completeness:	65.2°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	94777, 6159, 0.074
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4365
$N(param)_{refined}$:	198
Programs:	SHELX [23], Bruker programs [24]

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.85483(2)	1.08286(2)	0.49890(2)	0.02160(5)
S1	0.64189(2)	0.83748(4)	0.24363(2)	0.01154(7)
N1	0.68609(7)	0.53228(15)	0.22343(5)	0.0125(2)
N2	0.75715(7)	0.66164(14)	0.29236(5)	0.0112(2)
C1	0.63266(8)	0.48893(16)	0.17477(6)	0.0104(2)
C2	0.64562(9)	0.30820(17)	0.16505(6)	0.0138(3)
H2A	0.7014	0.2882	0.1554	0.017*
H2B	0.6330	0.2494	0.2019	0.017*
C3	0.59395(9)	0.24645(17)	0.11465(6)	0.0140(3)
H3A	0.6031	0.1291	0.1090	0.017*
C4	0.50768(9)	0.27545(18)	0.13038(7)	0.0160(3)
H4A	0.4737	0.2366	0.0979	0.019*
H4B	0.4940	0.2157	0.1668	0.019*
C5	0.49436(9)	0.45499(18)	0.14001(6)	0.0143(3)
H5A	0.4381	0.4740	0.1501	0.017*
C7	0.65316(8)	0.57777(17)	0.11726(6)	0.0128(3)
H7A	0.7089	0.5594	0.1072	0.015*
H7B	0.6452	0.6944	0.1226	0.015*
C6	0.54610(8)	0.51525(17)	0.19092(6)	0.0123(3)
H6A	0.5333	0.4562	0.2277	0.015*
H6B	0.5363	0.6307	0.1979	0.015*
C8	0.60047(9)	0.51604(17)	0.06686(6)	0.0137(3)
H8A	0.6133	0.5750	0.0296	0.016*
C9	0.61444(9)	0.33604(18)	0.05753(6)	0.0154(3)
H9A	0.5813	0.2966	0.0246	0.018*
H9B	0.6700	0.3168	0.0471	0.018*
C10	0.51467(9)	0.54595(19)	0.08309(6)	0.0156(3)
H10A	0.4803	0.5093	0.0505	0.019*
H10B	0.5058	0.6622	0.0890	0.019*
C11	0.69582(8)	0.66934(16)	0.25352(6)	0.0104(2)

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C12	0.77708(8)	0.76914(16)	0.33833(6)	0.0106(2)
C13	0.85588(9)	0.80530(17)	0.34672(6)	0.0137(3)
H13A	0.8939	0.7654	0.3198	0.016*
C14	0.87940(9)	0.89946(17)	0.39434(7)	0.0160(3)
H14A	0.9333	0.9232	0.4004	0.019*
C15	0.82321(10)	0.95804(17)	0.43279(6)	0.0146(3)
C16	0.74427(9)	0.92559(17)	0.42454(6)	0.0151(3)
H16A	0.7063	0.9687	0.4508	0.018*
C17	0.72139(9)	0.82936(17)	0.37747(6)	0.0127(3)
H17A	0.6676	0.8045	0.3720	0.015*
H1N1	0.7201(11)	0.465(2)	0.2302(8)	0.018(5)*
H1N2	0.7867(11)	0.576(2)	0.2892(8)	0.018(5)*

459–461 K. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in chloroform at room temperature. **^{1}H NMR** ($CDCl_3$, 500.13 MHz): δ 1.58–1.70 (m, 6H, Adamantane-H), 2.14 (s, 3H, Adamantane-H), 2.21 (s, 6H, Adamantane-H), 5.92 (s, 1H, NH), 7.11 (d, 2H, Ar-H, $J = 8.0$ Hz), 7.53 (d, 2H, Ar-H, $J = 0.0$ Hz), 7.65 (s, 1H, NH). **^{13}C NMR** ($CDCl_3$, 125.76 MHz): δ 29.55, 36.20, 41.58, 54.90 (Adamantane-C), 120.14, 126.50, 133.12, 135.88 (Ar-C), 179.58 (C = S). ESI-MS, *m/z*: 363.4 (M-H, 47%)$^-$, 365.4 (M+2-H, 52%)$^-$.

Experimental details

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

Discussion

The adamantanyl moiety represents the major pharmacophore of various drugs as amantadine [1–3], rimantadine [4, 5] and tromantadine [6], which are currently described as efficient antiviral therapies. Moreover, several adamantane-based drugs are currently employed as important medications against malaria infections [7], central nervous disorders [8–10], hyperglycaemia [11] and drug-resistant TB strain infections [12]. In addition, thiourea derivatives were reported to display marked antitumor [13], anti-HIV [14], antimalarial [15] and antimicrobial [16] activities. As a part of our current research interest in the pharmacological [17–19] and structural [20–22] properties of adamantane derivatives, we report herein the synthesis and crystal structure of the title compound as bioactive agent and as precursor for adamantane-based chemotherapeutic agents.

The asymmetric unit of the title structure contains one independent molecule. All bond lengths and angles of this molecule are in the expected ranges. The molecules packed in the crystal structure *via* two intermolecular strong classical hydrogen bonds, N1—H1N1···S1^{i} and N2—H1N2···S1^{i}. The H···A distances are 2.602(18) and 2.551(17) Å, respectively and the angles are 160.1(16) and 160.5(16)°, respectively. Symmetry codes: (i) -*x*+3/2, *y*-1/2, *z*.

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