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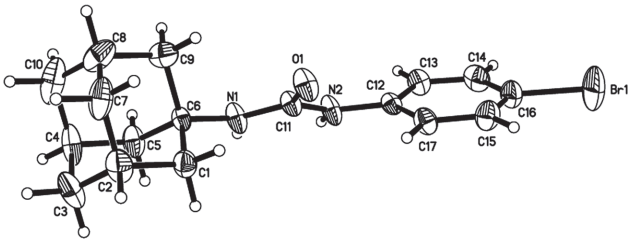
Crystal structure of 3-(adamantan-1-yl)-1-(4-bromophenyl)urea, $C_{17}H_{21}BrN_2O$



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Abstract

$C_{17}H_{21}BrN_2O$, orthorhombic, $Pna2_1$ (no. 33), $a = 9.2558(12)$ Å, $b = 13.0186(17)$ Å, $c = 13.4684(18)$ Å, $V = 1622.9(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0471$, $wR_{ref}(F^2) = 0.1059$, $T = 100$ K.

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The asymmetric unit of the title 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.

Table 1: Data collection and handling.

Crystal:	Colourless blocks Size 0.49 × 0.22 × 0.18 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	25.3 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness:	55°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$	3525, 3525
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1934
$N(param)_{refined}$:	199
Programs:	SHELX [25], Bruker programs [26]

Source of material

4-Bromophenyl isocyanate (1.98 g, 0.01 mol) was added to a solution of 1-adamantylamine (1.51 g, 0.01 mol), in ethanol (10 mL), and the mixture was heated under reflux for 3 hours. On cooling, the precipitated crude product was filtered, dried and crystallized from ethanol to yield 3.11 g (89%) of the title compound ($C_{17}H_{21}BrN_2O$) as transparent crystals. m.p.: 545–546 K. Single crystals were obtained by slow evaporation of a solution of the title compound in EtOH/$CHCl_3$ (1:1) at room temperature. **^{1}H NMR** (DMSO-d_6, 700.17 MHz): δ 1.62–1.64 (m, 6H, Adamantane-H), 2.03–2.05 (m, 6H, Adamantane-H), 2.08–2.10 (m, 3H, Adamantane-H), 5.90 (s, 1H, NH), 7.31–7.36 (m, 4H, Ar—H), 8.39 (s, 1H, NH). **^{13}C NMR** (DMSO-d_6, 176.08 MHz): δ 29.35, 36.48, 42.06, 50.37 (Adamantane-C), 112.37, 119.68, 131.78, 140.51 (Ar—C), 154.17 (C = O). **ESI-MS**, m/z: 349.2 [M + 2H, 100%]$^-$, 347.2 [M—H, 97%]$^-$.

Experimental details

Carbon-bound hydrogen 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 N-bound hydrogen atoms were located on a difference Fourier map and refined freely.

Discussion

1,3-Disubstituted urea derivatives were early recognized as important leads possessing various biological activities. Various 1-(1-adamantyl)-3-substituted urea derivatives were identified as potent inhibitors of *Mycobacterium tuberculosis* epoxide hydrolase B [1–4]. In addition, several 1,3-disubstituted urea analogues were reported to exhibit marked anticancer [5–8], antifungal [9], antibacterial [10, 11], antiviral [12] and herbicidal [13] activities. On the other hand, adamantane derivatives were early proved to possess marked antiviral [14, 15], antimalarial [16], hypoglycemic [17], antitubercular [18] and central nervous [19, 20] avtivities. As a part of ongoing research project on chemotherapeutic [21, 22] and structural [23, 24] properties of adamantane derivatives,

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.36803(8)	0.67620(5)	0.73438(7)	0.1262(3)
O1	0.4429(3)	0.2152(2)	0.4914(2)	0.0583(8)
N1	0.6674(4)	0.1564(2)	0.4538(3)	0.0513(11)
N2	0.6388(4)	0.3150(2)	0.5210(3)	0.0516(10)
C11	0.5742(3)	0.2260(3)	0.4884(3)	0.0408(9)
C17	0.4583(4)	0.3833(3)	0.6352(3)	0.0509(12)
H17A	0.4226	0.3162	0.6480	0.061*
C12	0.5709(4)	0.3975(3)	0.5688(3)	0.0398(10)
C1	0.5598(4)	−0.0106(3)	0.4993(3)	0.0532(11)
H1A	0.4675	0.0223	0.5185	0.064*
H1B	0.6223	−0.0143	0.5588	0.064*
C6	0.6339(3)	0.0532(3)	0.4190(3)	0.0390(9)
C16	0.3977(5)	0.4672(4)	0.6831(4)	0.0609(12)
H16A	0.3192	0.4574	0.7276	0.073*
C13	0.6198(4)	0.4966(3)	0.5520(3)	0.0509(11)
H13A	0.6953	0.5079	0.5055	0.061*
C15	0.4501(6)	0.5634(4)	0.6667(4)	0.0629(13)
C5	0.7776(4)	0.0024(3)	0.3932(4)	0.0593(13)
H5A	0.8273	0.0428	0.3411	0.071*
H5B	0.8404	0.0008	0.4527	0.071*
C14	0.5608(5)	0.5787(3)	0.6016(4)	0.0622(13)
H14A	0.5970	0.6460	0.5906	0.075*
C2	0.5306(5)	−0.1202(3)	0.4595(4)	0.0662(14)
H2A	0.4799	−0.1613	0.5117	0.079*
C7	0.4375(5)	−0.1155(4)	0.3679(5)	0.0766(15)
H7A	0.3436	−0.0830	0.3840	0.092*
H7B	0.4185	−0.1858	0.3431	0.092*
C8	0.5140(6)	−0.0536(4)	0.2890(4)	0.0759(15)
H8A	0.4528	−0.0512	0.2279	0.091*
C4	0.7517(5)	−0.1064(4)	0.3563(5)	0.0779(17)
H4A	0.8465	−0.1388	0.3393	0.094*
C9	0.5387(5)	0.0552(4)	0.3265(4)	0.0630(13)
H9A	0.4447	0.0875	0.3425	0.076*
H9B	0.5858	0.0966	0.2741	0.076*
C3	0.6766(6)	−0.1704(4)	0.4360(5)	0.0858(17)
H3A	0.7370	−0.1731	0.4967	0.103*
H3B	0.6618	−0.2414	0.4120	0.103*
C10	0.6576(6)	−0.1027(4)	0.2641(4)	0.097(2)
H10A	0.7066	−0.0625	0.2116	0.116*
H10B	0.6417	−0.1732	0.2389	0.116*
H1N1	0.744(4)	0.171(2)	0.467(2)	0.017(9)*
H1N2	0.723(4)	0.320(2)	0.519(2)	0.019(9)*

we report herein the crystal structure of the title adamantyl urea derivative.

The asymmetric unit of the title structure contains one molecule. The molecules are connected *via* two strong classical intermolecular hydrogen bonds, N1—H1N1$\cdots$O1^{i} and N2—H1N2$\cdots$O1^{i}. The H$\cdots$A distances are 2.39(3) and 2.12(4) Å, respectively and the angles are 156(3) and 161(3)°, respectively. Symmetry codes: (i) $x+1/2$, $-y+1/2$, z.

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