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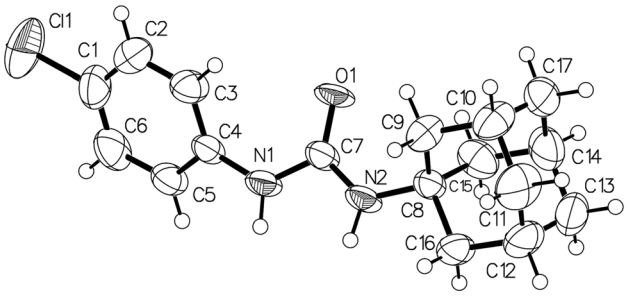
The crystal structure of 1-(adamantan-1-yl)-3-(4-chlorophenyl)urea, $C_{17}H_{21}ClN_2O$



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Abstract

$C_{17}H_{21}ClN_2O$, orthorhombic, $Pna2_1$ (no. 33), a = 9.2750(10) Å, b = 12.8640(17) Å, c = 13.483(2) Å, β = 90°, V = 1608.8(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0681, w$R_{ref}(F^2)$ = 0.1361, T = 293 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

A mixture of 4-chlorophenyl isocyanate (153 mg, 1 mmol), amantadine (166 mg, 1.1 mmol), and dichloromethane (10 mL) was heated at 313 K under reflux for 2 h. On cooling, the mixture was stirred for 24 h. After dichloromethane volatilization, the crude product of the title compound was obtained. Subsequently, a 50 mg product sample was dissolved in 10 mL EtOH/$CHCl_3$ (1:1) solvent. Crystals were obtained after four days from solvent at room temperature.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.18 × 0.16 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.24 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	29.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4764, 2883, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1586
$N(param)_{refined}$:	190
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], Olex2 [4]

Experimental details

The structure was solved with the ShelXT [2] structure solution program and refined with the ShelXL [3] refinement package [4]. All hydrogen atoms were placed in calculated positions with fixed isotropic thermal parameters.

Comment

Many 1-adamantyl-3-phenyl ureas, such as 1-(adamantan-2-yl)-3-(2,3,4-trifluorophenyl)urea, 1-(adamantan-2-yl)-3-(4-cyanoph-enyl)urea, and methyl 4-(3-(1-adamantyl) ureido)-2-hydroxy-benzoate have been identified as potent anti-tuberculosis activity compounds [5, 6]. The detailed crystal structure analysis of similar compounds is helpful to the development of innovative drugs for tuberculosis. There are many phenyl urea crystal structures have been reported [7–12]. A series of interesting solid-state systems and functional solution aggregates were designed via hydrogen bonds between urea groups. However, only a few 1-(1-adamantyl)-3-substituted urea derivatives have been reported [13].

The same space group and close cell paraments of the compound in this contribution and its isostructural bromo derivative must be mentioned [13]. The molecules formed zigzag polymeric chains along the (*a*) axis via two classical intermolecular hydrogen bonds, N1–H1···O1 with an H···A distance of 2.072(3) Å, and an angle of 158.1(4)°, and N2–H2A···O1 with an H···A distance of 2.225(4) Å, and an angle of 154.0(3)°. These two hydrogen bonds are more symmetrical than the corresponding hydrogen bonds with distances of 2.39(3) and

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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}
C1	0.5550 (8)	0.5704 (6)	0.6735 (5)	0.074 (2)
C2	0.6058 (7)	0.4717 (6)	0.6901 (5)	0.073 (2)
H2	0.681302	0.460949	0.734323	0.087*
C3	0.5447 (6)	0.3887 (5)	0.6411 (4)	0.0563 (16)
H3	0.578796	0.321825	0.653013	0.068*
C4	0.4329 (5)	0.4036 (4)	0.5742 (4)	0.0451 (14)
C5	0.3855 (6)	0.5034 (4)	0.5584 (5)	0.0544 (15)
H5	0.312336	0.515252	0.512593	0.065*
C6	0.4437 (7)	0.5859 (5)	0.6087 (5)	0.070 (2)
H6	0.407469	0.652513	0.598776	0.084*
C7	0.4277 (5)	0.2308 (4)	0.4942 (4)	0.0494 (14)
C8	0.3687 (4)	0.0555 (4)	0.4228 (4)	0.0438 (12)
C9	0.4424 (6)	−0.0101 (4)	0.5014 (4)	0.0575 (15)
H9A	0.381307	−0.014659	0.559641	0.069*
H9B	0.532412	0.022561	0.520828	0.069*
C10	0.4725 (7)	−0.1192 (5)	0.4615 (5)	0.0680 (18)
H10	0.522624	−0.160006	0.512355	0.082*
C11	0.3270 (7)	−0.1704 (5)	0.4381 (6)	0.091 (2)
H11A	0.268451	−0.173828	0.497584	0.110*
H11B	0.342069	−0.240570	0.413967	0.110*
C12	0.2500 (7)	−0.1050 (5)	0.3585 (6)	0.082 (2)
H12	0.157160	−0.136849	0.341646	0.098*
C13	0.3450 (9)	−0.0994 (6)	0.2668 (6)	0.099 (3)
H13A	0.296444	−0.059527	0.215647	0.119*
H13B	0.361510	−0.168934	0.241457	0.119*
C14	0.4891 (8)	−0.0484 (6)	0.2914 (5)	0.079 (2)
H14	0.548755	−0.044880	0.231510	0.094*
C15	0.4633 (7)	0.0604 (5)	0.3310 (4)	0.0642 (17)
H15A	0.416632	0.102330	0.280541	0.077*
H15B	0.554760	0.092645	0.347384	0.077*
C16	0.2264 (5)	0.0046 (4)	0.3968 (5)	0.0652 (18)
H16A	0.177663	0.045545	0.346469	0.078*
H16B	0.165236	0.002476	0.455093	0.078*
C17	0.5647 (7)	−0.1137 (5)	0.3694 (6)	0.078 (2)
H17A	0.581266	−0.183240	0.343873	0.094*
H17B	0.657377	−0.083088	0.385312	0.094*
Cl1	0.6316 (3)	0.67399 (19)	0.7355 (2)	0.1461 (11)
N1	0.3642 (4)	0.3206 (4)	0.5260 (4)	0.0587 (14)
H1	0.273159	0.326898	0.515494	0.070*
N2	0.3342 (4)	0.1599 (3)	0.4606 (4)	0.0563 (13)
H2A	0.244606	0.177157	0.461102	0.068*
O1	0.5605 (3)	0.2186 (3)	0.4972 (3)	0.0615 (12)

2.12(4) Å and the angles 156(3)° and 161(3)° in its isostructural bromo compound. The crystal configuration of molecules combining via hydrogen bonds was similar to the reported urea compound crystals [7–12]. In addition, the molecule conformation, including bond lengths, angles, and torsion angles of the adamantane ring system and the 4-chlorophenyl group, are consistent with previously published structures [14, 15].

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