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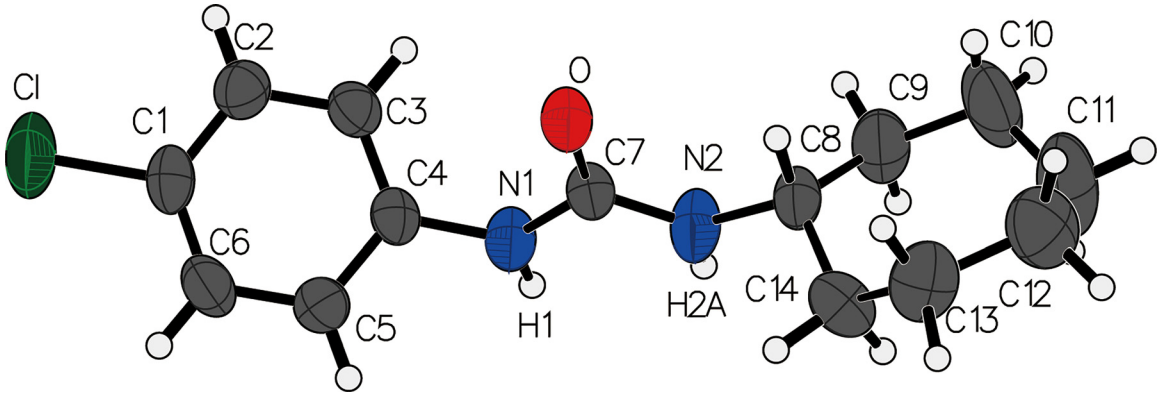
The crystal structure of 1-(4-chlorophenyl)-3-cycloheptylurea, $C_{14}H_{19}ClN_2O$



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Abstract

$C_{14}H_{19}ClN_2O$, monoclinic, $P2_1/c$ (no. 14), a = 12.8823(9) Å, b = 9.1617(6) Å, c = 11.7421(9) Å, β = 98.377(7)°, V = 1371.06(17) Å^3, Z = 4, $R_{gt}(F)$ = 0.0678, $wR_{ref}(F^2)$ = 0.1912, T = 293 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.43 × 0.37 × 0.33 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	29.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6456, 3150, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2039
$N(param)_{refined}$:	164
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

Source of materials

1-Chloro-4-isocyanatobenzene and cycloheptylamine were purchased from Aladdin Co. Ltd. and used as received. The 1-(4-chlorophenyl)-3-cycloheptylurea was synthesized using a modified literature procedure [5]. In a dry flask, a mixture of 1-chloro-4-isocyanatobenzene (0.15 g, 1 mmol) and cycloheptylamine (0.11 g, 1 mmol) was stirred in dichloromethane (15 mL). The reaction was monitored by thin-layer chromatography (TLC) on silica gel plates. The resulting precipitate was dried under vacuum at room temperature. The solution was prepared by dissolving 1-(4-chlorophenyl)-3-cycloheptylurea (0.05 g) in ethanol (10 mL) and stirring the solution at room temperature for 20 minutes. The solution was then placed in a vial and left to evaporate at room temperature. The crystals were obtained and collected after three days.

Experimental details

The structure was solved by Direct Methods and refined using the SHELXL program [3]. The structure refinement, and the graphical representation of the structure were performed using the OLEX2 software package [2].

Comment

Urea derivatives have been used as a starting material in the synthesis of a variety of heterocyclic compounds [6]. Many

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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	0.1781 (2)	0.4030 (3)	0.4453 (3)	0.0486 (7)
C2	0.2794 (2)	0.4315 (3)	0.4254 (3)	0.0486 (7)
H2	0.291555	0.476607	0.357692	0.058*
C3	0.3621 (2)	0.3917 (3)	0.5081 (2)	0.0446 (7)
H3	0.430601	0.408966	0.495354	0.053*
C4	0.34414 (19)	0.3265 (3)	0.6092 (2)	0.0379 (6)
C5	0.2420 (2)	0.3018 (3)	0.6283 (2)	0.0479 (7)
H5	0.229353	0.258863	0.696696	0.057*
C6	0.1587 (2)	0.3408 (3)	0.5457 (3)	0.0532 (8)
H6	0.090075	0.324678	0.558506	0.064*
C7	0.50912 (18)	0.3610 (3)	0.7407 (2)	0.0370 (6)
C8	0.6651 (2)	0.3622 (3)	0.8919 (2)	0.0451 (7)
H8	0.669120	0.463511	0.866093	0.054*
C9	0.7669 (2)	0.2875 (4)	0.8757 (3)	0.0643 (9)
H9A	0.773082	0.288147	0.794352	0.077*
H9B	0.763358	0.186385	0.899297	0.077*
C10	0.8661 (3)	0.3563 (6)	0.9422 (4)	0.1024 (15)
H10A	0.925935	0.303451	0.921673	0.123*
H10B	0.870803	0.455419	0.914420	0.123*
C11	0.8777 (3)	0.3624 (6)	1.0671 (4)	0.1075 (16)
H11A	0.859929	0.266700	1.093934	0.129*
H11B	0.951504	0.378125	1.095035	0.129*
C12	0.8192 (3)	0.4684 (5)	1.1226 (4)	0.0837 (12)
H12A	0.848163	0.563538	1.109187	0.100*
H12B	0.834174	0.450403	1.204743	0.100*
C13	0.7024 (3)	0.4804 (4)	1.0927 (3)	0.0719 (10)
H13A	0.687135	0.574357	1.055995	0.086*
H13B	0.673207	0.481949	1.164285	0.086*
C14	0.6436 (2)	0.3654 (4)	1.0160 (3)	0.0612 (9)
H14A	0.568988	0.380620	1.015417	0.073*
H14B	0.660849	0.270429	1.050067	0.073*
Cl	0.07362 (7)	0.44725 (11)	0.33836 (8)	0.0803 (4)
N1	0.42754 (17)	0.2769 (2)	0.6918 (2)	0.0471 (6)
H1	0.426672	0.186938	0.712617	0.057*
N2	0.57934 (18)	0.2915 (2)	0.8172 (2)	0.0553 (7)
H2A	0.573433	0.198385	0.822662	0.066*
O	0.51665 (14)	0.49159 (18)	0.71584 (17)	0.0465 (5)

urea derivatives crystals have been reported [7–12]. In these crystal structures, the hydrogen bond between molecules is the main force for the stacking of molecules into crystals.

1-(4-Chlorophenyl)-3-cycloheptylurea crystallizes in the monoclinic space group $P2_1/c$ with a unit cell containing four molecules. The structure is stabilized by intermolecular hydrogen bonds. The CPCU molecule consists of a cycloheptylurea ring, with a 4-chlorophenyl group attached to the nitrogen atom. The 4-chlorophenyl group is twisted away from the ring plane with a dihedral angle of 55.4°.

When compared to the isostructural bromocompound of the title compound, 1-(4-bromophenyl)-3-cycloheptylurea (BPBU) [13], the crystal structure of CPCU is similar in many respects. Both molecules contain a cycloheptylurea ring with a phenyl group attached to the nitrogen atom. In CPCU and BPBU, the 4-chlorophenyl group is twisted away from the ring plane with a similar angle. However, the crystal structures of the two molecules differ in the C–X (X=Cl or Br) bonds. The C–Cl bond (1.749(3) Å) is shorter than that of the C–Br bond (1.905(4) Å).

The crystal structure is stabilized by strong intermolecular interactions from hydrogen bonds (N1–H1–O) and (N2–H2A–O). The hydrogen bonds are formed between the hydrogen atoms attached to the nitrogen atoms of the cycloheptylurea ring. The angles of these two hydrogen bonds are 158.07(15)° and 151.13(16)°, and the length are 2.0648(17) Å and 2.2333(17) Å.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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