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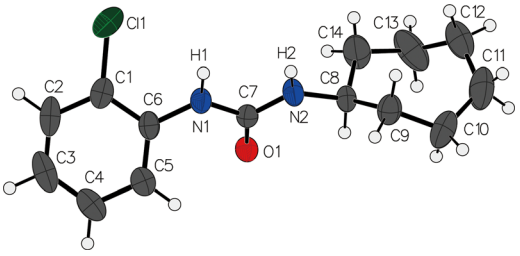
The crystal structure of 1-(2-chlorophenyl)-3-cycloheptylurea, $C_{14}H_{19}ClN_2O$



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Abstract

$C_{14}H_{19}ClN_2O$, orthorhombic, $P2_12_12_1$ (no. 19), a = 8.9579(3) Å, b = 12.2172(4) Å, c = 12.5669(5) Å, V = 1375.33(8) Å^3, Z = 4, $R_{gt}(F)$ = 0.0541, w$R_{ref}(F^2)$ = 0.1449, T = 193 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:	Colorless block
Size	0.57 × 0.48 × 0.41 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.27 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Discover, φ and ω
θ_{max}, completeness:	29.3°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14,926, 3749, 0.054
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3022
$N(param)_{refined}$:	163
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3, 4]

1 Source of materials

In a 3-neck round bottom flask, 1-chloro-2-isocyanatobenzene (0.15 g 1 mmol), cycloheptanamine (0.11 g 1 mmol), and dichloromethane (15 mL) were added and stirred under reflux for 5 h. The reaction mixture was cooled to room temperature and then concentrated under reduced pressure to give the crude product. Single crystals suitable for X-ray diffraction were obtained by slow evaporation technique. A solution of the pure compound in ethanol was prepared by slowly adding the crude product (50 mg) to a mixture of ethanol and dichloromethane (10 mL, 1:1 v/v) at room temperature and stirring overnight. The crystal of the title compound was obtained after three days with solution evaporation in the capless bottle.

2 Experimental details

The crystal structure was refined using the SHELXT package [3] in OLEX2 software [4].

3 Comment

Phenylurea herbicides are widely utilized in agriculture for the control of broadleaf weeds in cereal crops [5]. The molecular structure and intermolecular interaction, especially the hydrogen bonds, were the important factors affecting the properties of phenylurea herbicides. This highlights the need for further research on these herbicides. Many phenylurea derivative structures [6–14] and synthetic methods [15–18] were reported. In this paper, we show a phenylurea crystal structure named 1-(2-chlorophenyl)-3-cycloheptylurea.

It's also worth mentioning that crystal structures of other urea derivatives such as 1-[3-(hydroxymethyl) phenyl]-3-phenylurea [15], 1,1′-(propane-1,3-diyl)bis(3-phenylurea) [16], and *N*, *N*′–diphenylureas [9] have been reported. The molecular structure of 1-(2-chlorophenyl)-3-cycloheptylurea

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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	0.6087 (4)	0.0331 (3)	0.3092 (3)	0.0417 (8)
C2	0.6462 (4)	−0.0709 (3)	0.2741 (4)	0.0542 (10)
H2A	0.627276	−0.091697	0.202505	0.065*
C3	0.7108 (5)	−0.1435 (3)	0.3431 (4)	0.0663 (13)
H3	0.736201	−0.214952	0.319557	0.080*
C4	0.7390 (5)	−0.1129 (3)	0.4474 (4)	0.0579 (10)
H4	0.786016	−0.162825	0.494690	0.069*
C5	0.6988 (4)	−0.0094 (3)	0.4828 (3)	0.0448 (8)
H5	0.717223	0.010683	0.554672	0.054*
C6	0.6324 (3)	0.0647 (3)	0.4146 (3)	0.0350 (7)
C7	0.6605 (3)	0.2344 (2)	0.5182 (2)	0.0302 (6)
C8	0.6455 (4)	0.4074 (2)	0.6182 (3)	0.0341 (6)
H8	0.737524	0.376941	0.652107	0.041*
C9	0.5373 (4)	0.4343 (3)	0.7075 (3)	0.0459 (8)
H9A	0.450436	0.473370	0.676989	0.055*
H9B	0.500233	0.364915	0.738511	0.055*
C10	0.6026 (6)	0.5032 (4)	0.7954 (3)	0.0681 (13)
H10A	0.542887	0.490450	0.860527	0.082*
H10B	0.704873	0.476323	0.809831	0.082*
C11	0.6107 (9)	0.6213 (4)	0.7775 (5)	0.099 (2)
H11A	0.685074	0.651143	0.827981	0.119*
H11B	0.512727	0.652617	0.797490	0.119*
C12	0.6497 (6)	0.6651 (3)	0.6674 (4)	0.0704 (13)
H12A	0.556303	0.670449	0.625637	0.084*
H12B	0.689225	0.740258	0.675842	0.084*
C13	0.7598 (5)	0.6006 (3)	0.6041 (5)	0.0757 (16)
H13A	0.841249	0.576414	0.651921	0.091*
H13B	0.804188	0.648841	0.549364	0.091*
C14	0.6937 (6)	0.5005 (3)	0.5495 (4)	0.0642 (12)
H14A	0.606149	0.524849	0.507632	0.077*
H14B	0.768557	0.472473	0.498405	0.077*
Cl1	0.52770 (12)	0.12470 (9)	0.22082 (7)	0.0576 (3)
N1	0.5842 (3)	0.1682 (2)	0.4493 (2)	0.0371 (6)
H1	0.498270	0.192273	0.424702	0.045*
N2	0.5828 (3)	0.3211 (2)	0.5522 (2)	0.0394 (6)
H2	0.488357	0.326145	0.533656	0.047*
O1	0.7912 (2)	0.21490 (17)	0.54628 (17)	0.0347 (5)

consists of a cycloheptylurea moiety and a 2-chloroaniline moiety linked through a C–N single bond. The cycloheptyl ring adopts a chair conformation. The 2-chloroaniline moiety is planar, and the N–H bond is involved in hydrogen bonding interactions in the crystal.

The crystal structure of 1-(2-chlorophenyl)-3-cycloheptylurea is made up of a three-dimensional network of hydrogen bonds involving the carbonyl oxygen and the amide nitrogen atoms. The crystal structure can be described as layers of molecules stacked along the a-axis, with the layers held together by N–H···O hydrogen bonds (N2–H2···O1 and N1–H1···O1). The crystal structure of 1-(2-chlorophenyl)-3-cycloheptylurea is similar to other crystal structures of cycloheptylurea derivatives, where the same type of hydrogen bonding interactions is observed [19]. These comparisons can provide insight into the structural features that are important for the formation and stability of the crystal structure [20].

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