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5-(4-Chlorophenyl)-1-phenylpyrazolidin-3-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.008 Å
R factor = 0.062
wR factor = 0.178
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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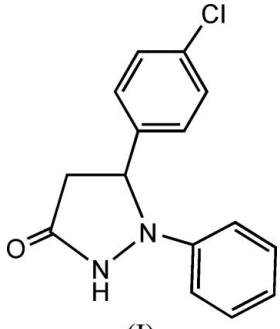
The title compound, $C_{15}H_{13}ClN_2O$, was synthesized by the reaction of ethyl 3-(4-chlorophenyl)acrylate and phenylhydrazine. Intermolecular N—H···O and intramolecular C—H···N hydrogen bonds are effective in the crystal structure. The intermolecular N—H···O hydrogen bonds link the molecules into dimers.

Comment

Some five-membered azole heterocycles, such as pyrazoles, thiazoles and pyrazole derivatives, are very important chemical and medical intermediates. 1,5-Diarylpyrazoles are particularly useful in the treatment of inflammation and related disorders (Reddy & Bell, 2003). We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. Pyra-

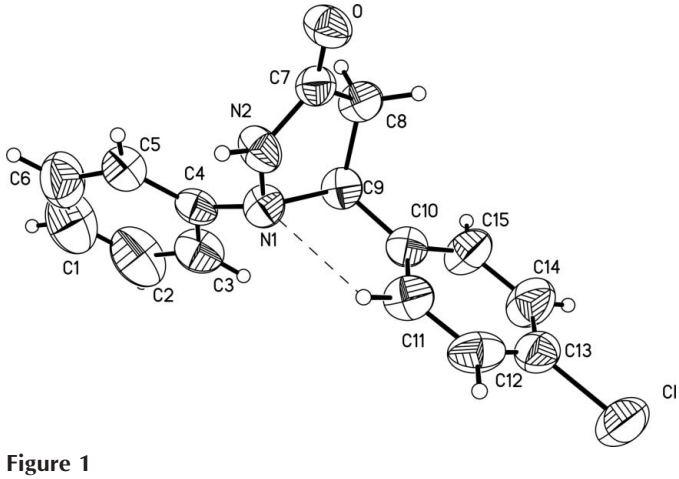


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. The intramolecular C—H···N hydrogen bond is indicated by a dashed line.

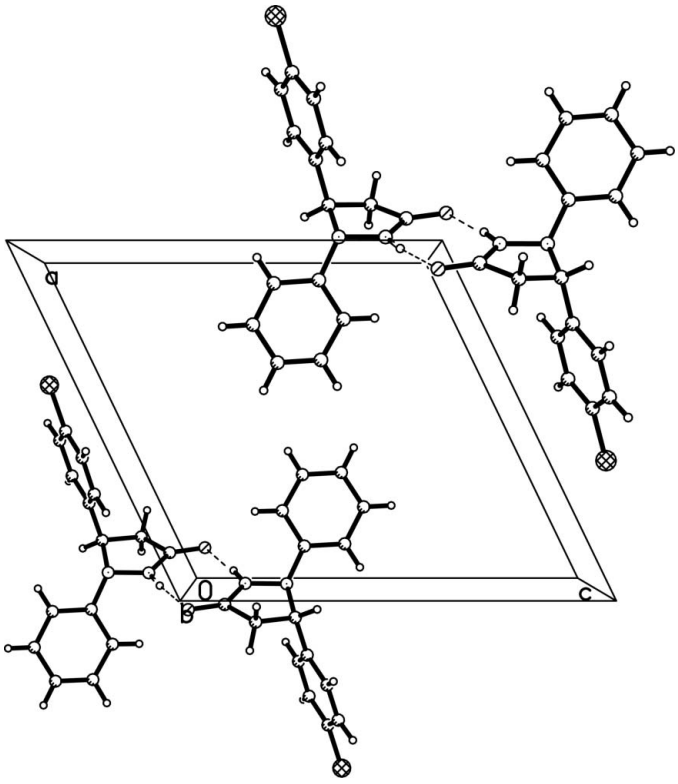


Figure 2
A packing diagram for (I). Dashed lines denote N—H···O hydrogen bonds.

zolidine ring *A* (N1/N2/C7–C9) adopts a twisted conformation (Low *et al.*, 2003); the dihedral angle between the C7/C8/C9 and C7/N2/N1/C9 planes is 21.6°. Because of the presence of the C═O group in ring *A*, the acidity of the H atoms on C8 may be increased (Zhu *et al.*, 2004). The dihedral angle between the two aromatic rings *B* (C1–C6) and *C* (C10–C15) [64.8 (3)°] may be compared with the corresponding value in 5-(4-methoxyphenyl)-1-phenylpyrazolidin-3-one [79.5 (1)°; Shi *et al.*, 2005].

Intramolecular C—H···N and intermolecular N—H···O hydrogen bonds (Table 2) are effective in stabilizing the crystal structure. The intermolecular N—H···O hydrogen bonds link the molecules into dimers (Fig. 2).

Experimental

Ethanolamine (4 ml) and *n*-butanol (20 ml) were added to a solution of sodium (40 mmol) in anhydrous methanol (9 mol). The methanol was removed by distillation and ethyl 3-(4-chlorophenyl)acrylate (30 mmol) was added. The resulting mixture was refluxed for 1 h at 373 K and then phenylhydrazine (4 ml) was added. The reaction mixture was refluxed for a further 7 h, left to cool to room temperature, acidified with acetic acid (36%), allowed to stand and then filtered; the filter cake was crystallized from ethyl acetate to give pure compound (I) (yield 5.63 g, 69%, m.p. 433–435 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{15}H_{13}ClN_2O$
M_r = 272.72
Monoclinic, $P2_1/c$
a = 11.826 (2) Å
b = 10.227 (2) Å
c = 12.867 (3) Å
β = 115.74 (3)°
V = 1401.8 (6) Å^3
Z = 4
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–12°
μ = 0.27 mm^{-1}
T = 296 (2) K
Block, colourless
0.30 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.925, T_{max} = 0.974
2581 measured reflections
2456 independent reflections
1395 reflections with $I > 2\sigma(I)$
R_{int} = 0.123
θ_{max} = 25.0°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 12$
$l = -15 \rightarrow 13$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.178
S = 1.00
2456 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl—C13	1.742 (5)	N1—C4	1.420 (6)
O—C7	1.220 (5)	N1—C9	1.496 (5)
N1—N2	1.392 (4)	N2—C7	1.334 (5)
N2—N1—C4	114.8 (3)	O—C7—C8	127.7 (4)
N2—N1—C9	104.2 (3)	N2—C7—C8	106.1 (4)
C4—N1—C9	116.8 (3)	N1—C9—C10	111.7 (3)
C7—N2—N1	116.1 (3)	N1—C9—C8	103.5 (3)
C3—C4—N1	119.2 (4)	C12—C13—Cl	120.1 (4)
C5—C4—N1	121.4 (4)	C14—C13—Cl	119.2 (4)
O—C7—N2	126.1 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O^{i}	0.86	1.94	2.799 (5)	174
C11—H11A···N1	0.93	2.56	2.875 (7)	101

Symmetry code: (i) $-x+2, -y+2, -z$.

The H atoms were positioned geometrically, with N—H = 0.86 Å, and C—H = 0.93 (aromatic) and 0.98 Å (CH) or 0.97 Å (CH_2), and constrained to ride on their parent atoms with U_{iso}(H) = 1.2 U_{eq}(C,N).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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