

1-(4-Chlorophenyl)-2-(imidazolidin-2-ylidene)-ethanone

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ (C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

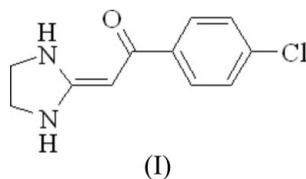
At 294 K, the C=C(H)–C=O grouping and the imidazoline ring of the title compound, C₁₁H₁₁ClN₂O, are coplanar as a result of an intramolecular N–H···O hydrogen bond and the conjugation, while the 4-chlorophenyl ring is twisted from the plane by 23.5 (2)°. Intermolecular N–H···O hydrogen bonding helps to establish the crystal packing.

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Comment

Heterocyclic ketene aminals (HKAs) (Huang & Wang, 1994, 2002), also known as cyclic 1,1-enediamines, are versatile synthons for heterocyclic synthesis. The title compound, (I) (Fig. 1), which possesses an imidazolidine ring, is a member of this family. The crystallographic data for (I) will provide valuable information for assessing its electronic conjugation properties and a possible intramolecular hydrogen bond (Wang *et al.*, 1987), which may be correlated with the reactivity of the secondary amine and the α -C atom (Huang & Wamhoff, 1984).



The abnormal geometrical parameters for (I) (Table 1) reflect the conjugation present, viz. the length of the C5=O1 double bond is greater than that of a normal C=O double bond, and the nominal C3=C4 double bond is longer even than that of the C4–C5 single bond in the conjugated system. On the other hand, the C3–N1 and C3–N2 bond lengths are shorter than those of normal C–N single bonds. The heterocyclic ring atoms C4, C5 and O1 are coplanar as a result of an intramolecular hydrogen bond between one of the NH groups and the carbonyl O atom (Table 2); the 4-chlorophenyl ring is twisted from this plane by an angle of 23.5 (2)°. The crystal packing is stabilized by an intermolecular hydrogen bond between the second NH group and the carbonyl O atom of an adjacent molecule (Fig. 2).

Experimental

Compound (I) was prepared according to the procedure of Huang & Liu (1987) and recrystallized from ethanol in 75% yield (m.p. 511 K). Analysis calculated for C₁₁H₁₁ClN₂O: C 59.33, H 4.98, N 12.58%; found: C 59.30, H 4.52, N 12.30%.

Crystal data

C₁₁H₁₁ClN₂O
M_r = 222.67
 Monoclinic, *P*2₁
a = 6.5657 (17) Å
b = 6.976 (2) Å
c = 11.602 (3) Å
 β = 94.981 (4)°
V = 529.4 (2) Å³

Z = 2
D_x = 1.397 Mg m⁻³
 Mo *K*α radiation
 μ = 0.33 mm⁻¹
T = 294 (2) K
 Prism, colourless
 0.24 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2002)
T_{min} = 0.924, *T_{max}* = 0.949

3013 measured reflections
 1996 independent reflections
 1676 reflections with *I* > 2σ(*I*)
R_{int} = 0.020
 θ_{max} = 26.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.036
wR(*F*²) = 0.095
S = 1.05
 1996 reflections
 146 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.064P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.18 e Å⁻³
 Δρ_{min} = -0.20 e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.040 (6)
 Absolute structure: Flack (1983),
 804 Friedel pairs
 Flack parameter: 0.35 (9)

Table 1
 Selected bond lengths (Å).

C3—C4	1.400 (3)	C3—N2	1.333 (3)
C4—C5	1.387 (3)	C5—O1	1.263 (2)
C3—N1	1.322 (3)		

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O1	0.74 (3)	2.12 (3)	2.685 (3)	133 (3)
N2—H2...O1 ⁱ	0.78 (3)	2.04 (3)	2.743 (3)	152 (3)

Symmetry code: (i) *x* - 1, *y*, *z*.

The N-bound H atoms were located in difference maps and their positions and *U*_{iso}(H) values were freely refined. The C-bound H atoms were placed in geometrically idealized positions (C—H = 0.95–1.00 Å) and refined as riding, with *U*_{iso}(H) = 1.2*U*_{eq}(C). The crystal studied is an inversion twin with volume fractions of 0.65 (9) and 0.35 (9) for the twin components.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

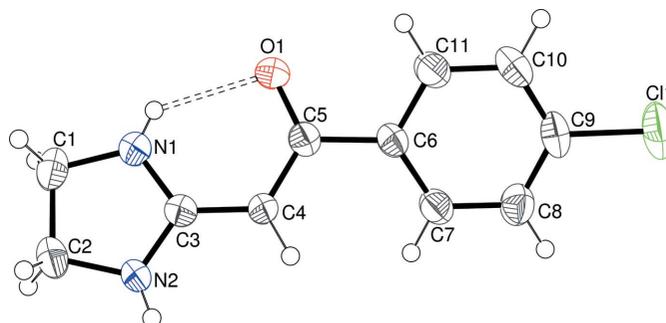


Figure 1
 View of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The intramolecular hydrogen bond is indicated by a dashed line.

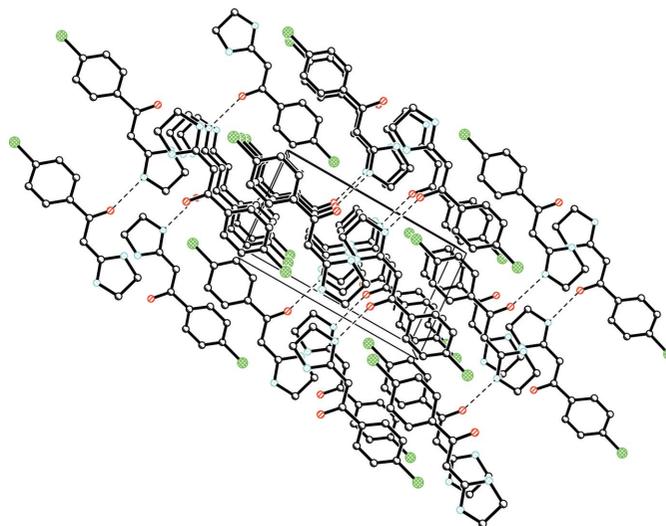


Figure 2
 The molecular packing of (I), viewed along the *b* axis. H atoms bonded to C atoms have been omitted for clarity. Dashed lines indicate the hydrogen-bonding interactions.

SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXTL.

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