

N-(4-Chlorophenyl)acrylamide

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Key indicators

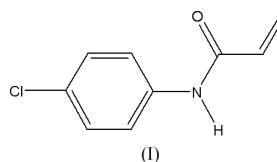
Single-crystal X-ray study
T = 298 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.047
wR factor = 0.126
Data-to-parameter ratio = 14.0

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

In the crystal structure of the title compound, $\text{C}_9\text{H}_8\text{ClNO}$, intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into one-dimensional molecular chains. These weak interactions generate a $C(4)$ chain and an $R_2^1(6)$ ring.

Comment

Recently, Lee and co-workers (Lee *et al.*, 2001) reported the Baylis–Hillman reaction, *i.e.* the coupling of activated alkenes with carbon electrophiles under the catalysis of tertiary amines (Baylis & Hillman, 1972), using α -naphthyl acrylates as the reaction substrates. We have now synthesized the title compound, (I), which is used to investigate the same reaction. Fortuitously, we also obtained single crystals of (I) and report its structure here.



The molecular configuration of (I) is shown in Fig. 1 and selected geometric parameters are listed in Table 1. All bond lengths are normal, but some bond angles are unusually large, *e.g.* $\text{C}3-\text{N}1-\text{C}4$ $129.25(16)$ and $\text{C}5-\text{C}4-\text{N}1$ $124.25(18)^\circ$ (Table 1). These effects can be caused by intermolecular interactions.

Hydrogen-bonding information is given in Table 2 and a crystal-packing diagram is shown in Fig. 2. Intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Desiraju, 1996; Steiner, 1997) link the molecules into one-dimensional molecular chains along the *a* axis. The $\text{N}-\text{H}\cdots\text{O}^i$ hydrogen bond, with $\text{N}\cdots\text{O}$ $2.864(2) \text{ \AA}$, has the carbonyl O atom of one molecule acting as an acceptor and the N atom from the other molecule acting as a donor [symmetry code: (i) $x - \frac{1}{2}, y, \frac{1}{2} - z$]. The $\text{C}-\text{H}\cdots\text{O}^i$ interaction, with $\text{H}\cdots\text{O}$ 2.53 \AA , is formed between the same two molecules, the only difference being

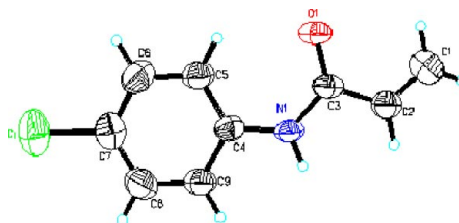


Figure 1

A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Received 24 February 2004

Accepted 18 March 2004

Online 27 March 2004

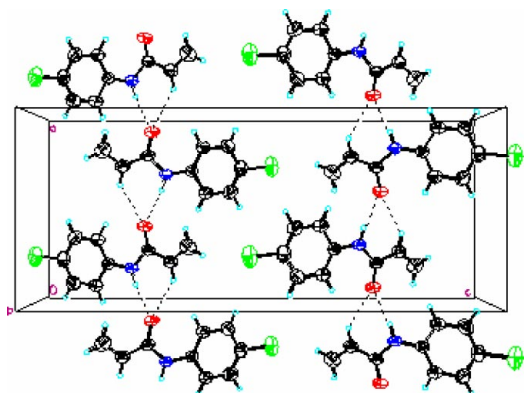


Figure 2

A view, down the *b* axis, of the one-dimensional molecular chains in (I), formed *via* N—H···O and C—H···O hydrogen bonds.

that the C atom of the CH₂ group acts as a donor here (Fig. 2). These strong and weak interactions generate graph-set motifs of *C*(4) and *R*₂¹(6) (Bernstein *et al.*, 1995).

Remarkably, a moderate intramolecular C—H···O interaction, with H···O 2.27 Å (Corey & Lee, 2001; Pálínkó, 1999; Table 2), occurs in the molecular structure of (I), with a ring pattern of *S*(6), and with the carbonyl O atom acting as an acceptor and one of the aromatic CH groups acting as a donor. Its existence is likely to make (I) participate in the Baylis–Hillman reaction more easily than aliphatic acrylamides. Similarly, this factor can offer a convincing proof that α -naphthyl acrylate is a more effective substrate than alkyl acrylates (Lee *et al.*, 2001).

Experimental

The title compound was prepared by the acylation of 4-chloroaniline. To 4-chloroaniline in dry CH₂Cl₂ solution containing NEt₃, acrylyl chloride was added. The mixture was stirred at room temperature for 10 h. Filtration and recrystallization from EtOAc provided the compound with good yield. The title compound (100 mg) was dissolved in CH₃COOCH₂CH₃ (2 ml). The solution was allowed to evaporate slowly at room temperature for several days. Colourless crystals suitable for X-ray crystallography were formed.

Crystal data

C ₉ H ₈ ClNO	Mo <i>K</i> α radiation
<i>M_r</i> = 181.61	Cell parameters from 9886 reflections
Orthorhombic, <i>Pbca</i>	θ = 2.8–26.8°
<i>a</i> = 9.552 (3) Å	μ = 0.39 mm ⁻¹
<i>b</i> = 8.028 (2) Å	<i>T</i> = 298 (2) K
<i>c</i> = 22.715 (7) Å	Pillar, colourless
<i>V</i> = 1741.7 (9) Å ³	0.20 × 0.20 × 0.10 mm
<i>Z</i> = 8	
<i>D_x</i> = 1.385 Mg m ⁻³	
Data collection	
Bruker SMART CCD area-detector diffractometer	1526 independent reflections
ω scans	1273 reflections with <i>I</i> > 2 σ (<i>I</i>)
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	<i>R</i> _{int} = 0.033
<i>T</i> _{min} = 0.927, <i>T</i> _{max} = 0.963	θ _{max} = 25.0°
7535 measured reflections	<i>h</i> = -11 → 11
	<i>k</i> = -9 → 8
	<i>l</i> = -27 → 24

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.5186P]$
$R[F^2 > 2\sigma(F^2)] = 0.047$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{\max} = 0.001$
<i>S</i> = 1.10	$\Delta\rho_{\max} = 0.30 \text{ e \AA}^{-3}$
1526 reflections	$\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$
109 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å).

O1—C3—N1	123.1 (2)	C3—N1—C4	129.25 (16)
O1—C3—C2	121.99 (19)	C5—C4—N1	124.25 (18)
N1—C3—C2	114.94 (17)	C9—C4—N1	117.33 (17)
C1—C2—C3	123.0 (2)		

Table 2

Hydrogen-bonding 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 ⁱ	0.86	2.01	2.864 (2)	172
C2—H2···O1 ⁱ	0.93	2.53	3.293 (3)	140
C5—H5···O1	0.93	2.27	2.869 (3)	122

Symmetry code: (i) $x - \frac{1}{2}, y, \frac{1}{2} - z$.

H atoms were placed in calculated positions and allowed to ride on their parent atoms, with *U*_{iso}(H) values set to 1.5*U*_{eq}(parent atom) for the H atoms of the CH₂ group and 1.2*U*_{eq}(parent atom) for the remaining H atoms. The N—H distance was fixed at 0.86 Å and the C—H distances were fixed at 0.93 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge the financial support of this work by the Natural Science Foundation of China (grant No. 20272034 to CZX). The authors also thank the Shanxi Provincial Natural Science Foundation for support.

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