

2-Acetyl-3'-chloroacetanilide

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.041

wR factor = 0.075

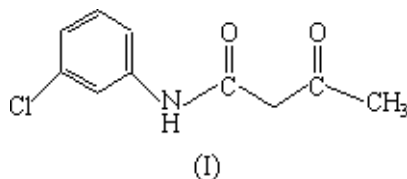
Data-to-parameter ratio = 15.5

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

In the title compound [alternatively called *N*-(3-chlorophenyl)-3-oxobutanamide], $\text{C}_{10}\text{H}_{10}\text{ClNO}_2$, the $\text{C}=\text{O}$ bond lengths are 1.2108 (19) and 1.179 (2) \AA , which implies that the molecule is in the keto form. The crystal structure is stabilized by an $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond.

Comment

Europium(III) and terbium(III) complexes with conjugated ligands having a β -diketonate group have been studied as emitting materials for organic electroluminescent diodes (OLED) (Kido *et al.*, 1991, 1993; Takada *et al.*, 1994; Huang *et al.*, 2001), since the initial reports by Kido *et al.* (1990). However, the quantum efficiency of most of these complexes is unfortunately still low. This may be due mainly to the inefficiency of the energy transfer, in particular triplet-triplet transfer, in these complexes. Chemists have realised that it is essential to design ligands which have better energy-transfer properties to the lanthanide metal ion. In the present work, as part of our studies of the synthesis and characterization of β -diketonate-type ligands and their complexes, we have synthesized the title compound, (I), and determined its crystal structure. The crystal structures of similar compounds, namely 2'-chloroacetoacetanilide and 4'-chloroacetoacetanilide, were reported by Kubozono *et al.* (1992).



In the molecule of (I), the $\text{C}=\text{O}$ bond lengths are 1.2108 (19) and 1.179 (2) \AA , which confirms that the compound is in the keto form (Fig. 1).

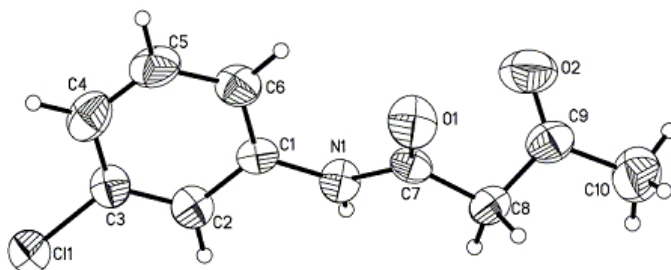


Figure 1

The molecule of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

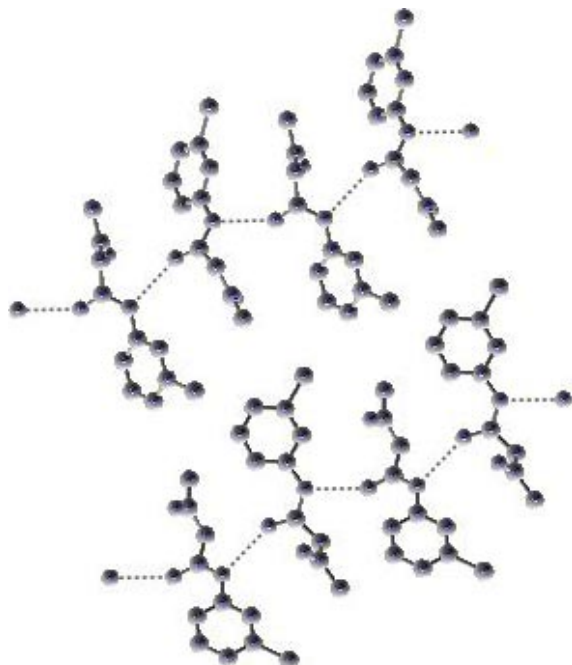


Figure 2
The packing of (I). N—H...O hydrogen bonds are indicated by dashed lines.

The crystal structure of (I) is stabilized by an N—H...O intermolecular hydrogen bond (Fig. 2).

Experimental

The title compound was prepared by a method similar to that of Lliopoulos *et al.* (1986). A solution of 3-chloroaniline (10 mmol) in benzene (30 ml) was added to a solution of ethyl acetoacetate (10 mmol) and the reaction mixture was refluxed for 2 h with stirring. The resulting pale precipitate was collected by filtration, washed several times with benzene and dried *in vacuo* (yield 89%). Analysis: MS for $C_{10}H_{10}ClNO_2$: $M+H = 212$ (found), $M = 211$ (calculated); calculated: C 56.74, H 4.73, N 6.62%; found: C 56.58, H 4.72, N 6.48%; IR (KBr, cm^{-1}): 3240 (*m*, N—H), 1722 (*s*, $CH_3C=O$), 1664 (*s*, amide $C=O$). An ethanol solution of the title compound was allowed to evaporate slowly and pale crystals of (I) were obtained after a week.

Crystal data

$C_{10}H_{10}ClNO_2$
 $M_r = 211.64$
Orthorhombic, *Pbca*
 $a = 9.5485$ (16) Å
 $b = 8.2197$ (13) Å
 $c = 25.940$ (4) Å
 $V = 2035.9$ (6) Å³
 $Z = 8$
 $D_x = 1.381$ Mg m⁻³

Mo $K\alpha$ radiation
Cell parameters from 702 reflections
 $\theta = 2.6$ – 18.3°
 $\mu = 0.35$ mm⁻¹
 $T = 293$ (2) K
Block, colourless
 $0.32 \times 0.26 \times 0.24$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
 $T_{min} = 0.90$, $T_{max} = 0.92$
10 164 measured reflections

1988 independent reflections
1285 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.046$
 $\theta_{max} = 26.0^\circ$
 $h = -11 \rightarrow 11$
 $k = -7 \rightarrow 10$
 $l = -29 \rightarrow 31$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.075$
 $S = 0.98$
1988 reflections
128 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.14$ e Å⁻³
 $\Delta\rho_{min} = -0.14$ e Å⁻³
Extinction correction: none

Table 1

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 ⁱ	0.86	2.09	2.9259 (18)	163

Symmetry code: (i) $x + \frac{1}{2}, y, -z + \frac{3}{2}$.

All H atoms were positioned geometrically, with C—H distances in the range 0.93–0.97 Å and an N—H distance of 0.86 Å, and they were treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C,N)$, or $1.5U_{eq}(C)$ for methyl H atoms.

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

The authors thank the National Natural Science Foundation of China (grant No. 20441002), NingXia Natural Gas Transferring Key Laboratory (grant No. 2004007) and Weifang University for research grants.

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