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Key indicators

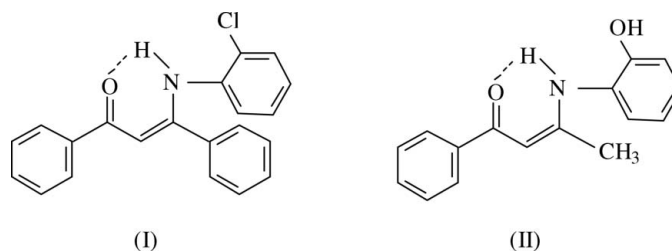
Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.053
 wR factor = 0.186
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-[(2-Chlorophenyl)amino]-1,3-diphenyl-
prop-2-en-1-one

The title compound, $\text{C}_{21}\text{H}_{16}\text{ClNO}$, has been synthesized by the condensation reaction of dibenzoylmethane and 2-chloroaniline in the presence of *p*-TsOH. The enaminone structure is stabilized by a strong intramolecular $\text{N}-\text{H} \cdots \text{O}=\text{C}$ hydrogen bond.

Received 3 October 2005
Accepted 12 October 2005
Online 19 October 2005

Comment

Enaminones have been studied extensively not only because they are widely used in chemical reactions (Christoffers *et al.*, 2003; Tietze *et al.*, 1989) but also owing to their applications in coordination chemistry (Kim *et al.*, 2001; Doherty *et al.*, 1999), as well as chiral auxiliaries in organic synthesis (Christoffers, 2003). As part of our continuing interest in the chemistry of enaminones, we have prepared the title compound, (I), by the condensation of dibenzoylmethane and 2-chloroaniline (Shi *et al.*, 2004, 2005).



The title compound has been shown by ^1H NMR spectroscopy to exist in solution in the enaminone and not the enol-imine form (Shi *et al.*, 2004, 2005). IR spectroscopy shows only the enaminone form in the solid state, and the crystal structure of (I) (Fig. 1) fully confirms these results.

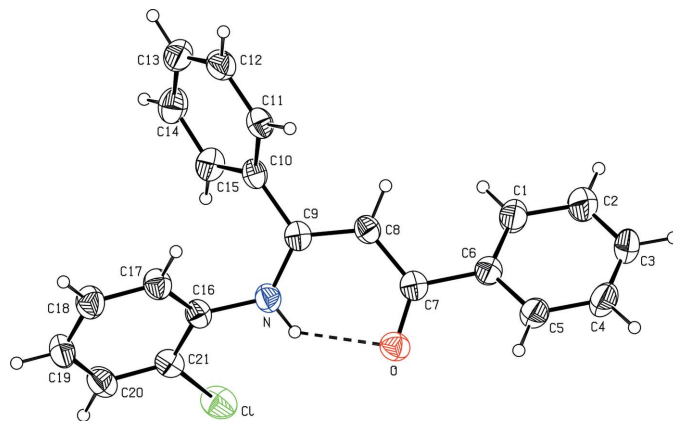


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

As also observed in the related compound 3-(2-hydroxyphenyl)-1-phenylbut-2-en-1-one, (II) (Shi, 2005), the O=C—C=C—N fragment of molecule (I) is planar; the bond lengths indicate electron delocalization (Gilli *et al.*, 2000). The three benzene rings C1—C6, C10—C15 and C16—C21 are twisted with respect to the O=C—C=C—N plane, resulting in a propeller-like arrangement with dihedral angles of 36.61 (17), 55.61 (15) and 49.74 (15)°, respectively. The C6—C7 [1.495 (4) Å] and C9—C10 [1.487 (4) Å] bond lengths are typical of a single bond (Csp^2-Csp^2) and suggest that the C1—C6 and C10—C15 rings are not involved in the conjugation of the O=C—C=C—N group.

As in (II), a strong intramolecular hydrogen bond [N—H...O=C] is present in the crystal structure of (I) (Fig. 1 and Table 1).

Experimental

A toluene solution of dibenzoylmethane and 2-chloroaniline (1:1) in the presence of a catalytic trace amount of *p*-TsOH was refluxed with a Dean–Stark apparatus to remove water for 48 h. After removal of the solvent, the residue was chromatographed on silica gel plates with CH₂Cl₂ and petroleum ether (1:2, *v/v*). The yellow band was collected and further recrystallized from a petroleum ether solution at 278 K to afford the title compound (m.p. 375.7–376.5 K). IR (KBr): 1541 (s, C=C), 1599 (*vs.*, C=O), 3059 (*w*, NH) cm⁻¹; ¹H NMR (600 MHz, CDCl₃, p.p.m.): δ 6.212 (*s*, 1H, CH), 6.470–6.483, 6.856–6.881, 6.929–6.954, 7.354–7.379 (*d*, 1H, *t*, 1H, *t*, 1H, *t*, 2H, C₆H₅CN), 7.403–7.425 (*m*, 4H, C₆H₄Cl), 7.460–7.485, 7.509–7.533, 8.014–8.026 (*t*, 2H, *t*, 1H, *d*, 2H, C₆H₅CO), 12.816 (*s*, 1H, NH).

Crystal data

C ₂₁ H ₁₆ ClNO	Z = 2
<i>M_r</i> = 333.80	<i>D_x</i> = 1.277 Mg m ⁻³
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 8.7720 (18) Å	Cell parameters from 25 reflections
<i>b</i> = 9.7380 (19) Å	<i>θ</i> = 10–12°
<i>c</i> = 11.306 (2) Å	<i>μ</i> = 0.23 mm ⁻¹
<i>α</i> = 86.02 (3)°	<i>T</i> = 295 K
<i>β</i> = 74.08 (3)°	Block, yellow
<i>γ</i> = 69.26 (3)°	0.40 × 0.30 × 0.20 mm
<i>V</i> = 868.1 (4) Å ³	

Data collection

Enraf–Nonius CAD-4 diffractometer	<i>R</i> _{int} = 0.026
<i>ω/2θ</i> scans	<i>θ</i> _{max} = 25.0°
Absorption correction: <i>ψ</i> scan (North <i>et al.</i> , 1968)	<i>h</i> = 0 → 10
<i>T</i> _{min} = 0.915, <i>T</i> _{max} = 0.947	<i>k</i> = -10 → 11
3273 measured reflections	<i>l</i> = -12 → 13
3058 independent reflections	3 standard reflections
2235 reflections with <i>I</i> > 2σ(<i>I</i>)	every 200 reflections
	intensity decay: none

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.114P)^2 + 0.0942P]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.186$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.09	$\Delta\rho_{max} = 0.31 \text{ e \AA}^{-3}$
3058 reflections	$\Delta\rho_{min} = -0.33 \text{ e \AA}^{-3}$
218 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.055 (10)

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>
N—H1N...O	0.86	2.01	2.670 (3)	132

All H atoms were placed at geometrically idealized positions and were treated as riding on their parent atoms, with C—H = 0.93 Å and N—H = 0.86 Å. *U*_{iso}(H) values were set at 1.2*U*_{eq}(C,N) or 1.5*U*_{eq}(methyl C,O).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

The authors thank the Natural Science Foundation of Jiangsu Province (05KJB150151) and Yangzhou University for financial support.

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