

2-Anilino-3-benzoyl-4-(2-chlorophenyl)-7,7-dimethyl-7,8-dihydro-4*H*-chromen-5(6*H*)-oneMing Li,\* En-Tao Sun and  
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## Key indicators

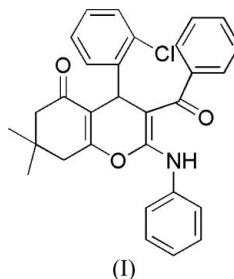
Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.127  
Data-to-parameter ratio = 14.1For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

There are two independent molecules in the asymmetric unit of the title compound,  $\text{C}_{30}\text{H}_{26}\text{ClNO}_3$ . Both molecules are linked through  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds into centrosymmetric dimers, providing stabilization.

Received 28 September 2006  
Accepted 23 October 2006

## Comment

4*H*-Benzo[*b*]pyran and its derivatives are a class of versatile building blocks for the synthesis of a variety of natural products (Hatokeyama *et al.*, 1998), many of which have been reported to show various biological properties (Hassanien *et al.*, 1999), such as anti-inflammatory and fungicidal activities (Jiang, 1994). In the course of our systematic studies aimed at the synthesis of new bioactive compounds, we synthesized the title compound, (I), and its structure is reported here.

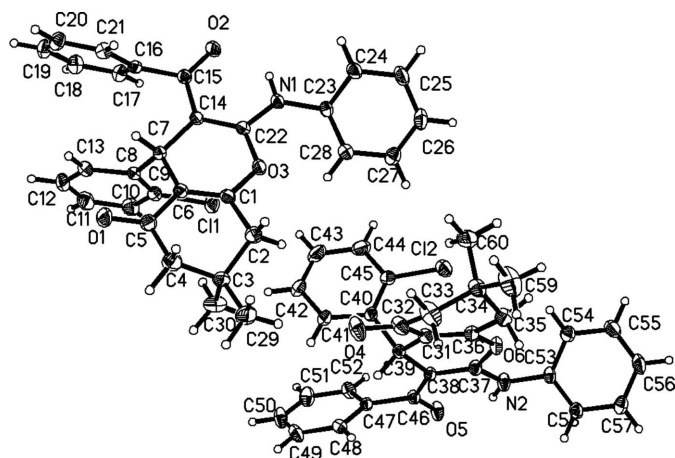


The structure of (I) consists of two crystallographically independent molecules (*A* and *B*). In the two molecules, the six-membered rings C1–C6 and C31–C36 adopt envelope conformations, the largest deviations being 0.317 (2) and 0.316 (3) Å for C3 and C33, respectively. The dihedral angle between the pyran ring and the phenyl ring C23–C28 in molecule *A* is 43.23 (7)°, while the dihedral angle between the pyran ring and phenyl ring C53–C58 in molecule *B* is 55.44 (7)°.

In the crystal structure, there are three intramolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, each of which forms a six-membered ring (Table 1). Both independent molecules are linked into centrosymmetric dimers through intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen-bonding interactions (Table 1 and Fig. 2).

## Experimental

A mixture of  $\beta$ -benzoylthioacetanilide (1 mmol, 0.255 g) synthesized according to a literature method (Gomper & Schaefer, 1967), 2-chlorobenzaldehyde (1 mmol, 0.141 g) and 5,5-dimethyl-1,3-cyclohexanedione (1 mmol, 0.140 g) was added to anhydrous ethanol (10 ml) and refluxed in the presence of three drops of triethylamine for 10 h. The crude solid product was collected by filtration. Colourless block-shaped crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution (m.p. 442 K).



**Figure 1**  
The asymmetric unit of the title compound, with 35% probability displacement ellipsoids.

*Crystal data*

$C_{30}H_{26}ClNO_3$   
 $M_r = 483.97$   
Monoclinic,  $P2_1/c$   
 $a = 19.982(5) \text{ \AA}$   
 $b = 13.768(4) \text{ \AA}$   
 $c = 19.018(5) \text{ \AA}$   
 $\beta = 103.956(3)^\circ$   
 $V = 5078(2) \text{ \AA}^3$

$Z = 8$   
 $D_x = 1.266 \text{ Mg m}^{-3}$   
Mo  $K\alpha$  radiation  
 $\mu = 0.18 \text{ mm}^{-1}$   
 $T = 293(2) \text{ K}$   
Block, colourless  
 $0.26 \times 0.22 \times 0.16 \text{ mm}$

*Data collection*

Bruker APEXII CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.954, T_{\max} = 0.971$

27044 measured reflections  
8974 independent reflections  
5707 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$   
 $\theta_{\text{max}} = 25.0^\circ$

*Refinement*

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.127$   
 $S = 1.02$   
8974 reflections  
635 parameters  
H-atom parameters constrained

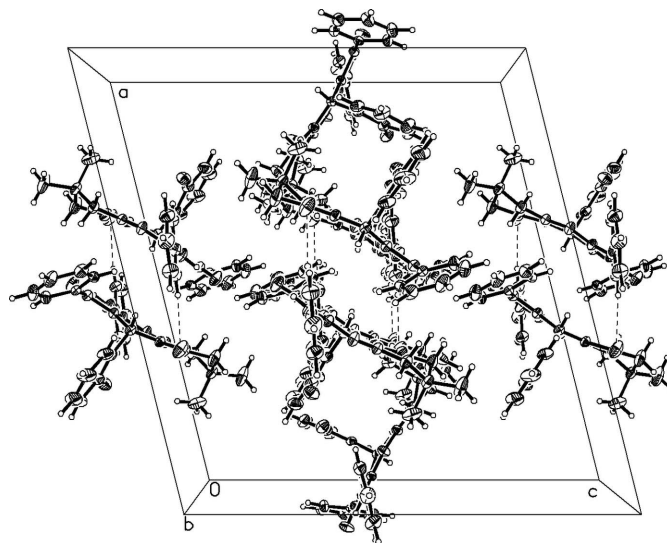
$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 1.1067P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ ).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C21-H21\cdots O3^i$	0.93	2.55	3.409 (3)	154
$C24-H24\cdots O1^i$	0.93	2.51	3.393 (3)	158
$N1-H1\cdots O2$	0.86	1.90	2.582 (2)	136
$N2-H2\cdots O5$	0.86	1.88	2.559 (2)	135
$C28-H28\cdots O3$	0.93	2.42	2.811 (3)	106
$C58-H58\cdots O4^{ii}$	0.93	2.52	3.446 (3)	175

Symmetry codes: (i)  $-x, -y + 1, -z + 1$ ; (ii)  $-x + 1, -y + 2, -z + 1$ .



**Figure 2**

The molecular packing of the title compound, viewed along the  $b$  axis. Hydrogen bonds are shown as dashed lines.

All H atoms were placed in calculated positions, with  $C-H = 0.93-0.98 \text{ \AA}$ ,  $N-H = 0.86 \text{ \AA}$ , and refined using a riding model, with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C, N)$  or  $1.5U_{\text{eq}}(\text{methyl C})$ .

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1999); 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*.

This project was supported by the National Natural Science Foundation of China (grant No. 20572057).

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