Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(*N*,*N*-Dimethylamino)-3-(2,2-diphenylethenyl)-coumarin

Anwar Usman,[a] Ibrahim Abdul Razak,[a] Hoong-Kun Fun,[a]* Suchada Chantrapromma,[a]† Bao-Guo Zhao[b] and Jian-Hua Xu[b]

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

† Permanent address: Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.002 Å
R factor = 0.054
wR factor = 0.141
Data-to-parameter ratio = 15.0

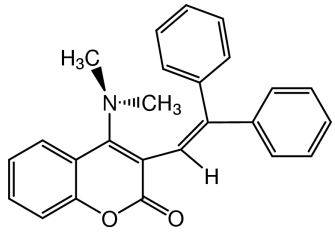
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 June 2002
Accepted 10 June 2002
Online 21 June 2002

In the title compound, $C_{25}H_{21}NO_2$, the coumarin moiety is planar and the attached dimethylamino group is twisted around the N—C bond. The dihedral angle between the two phenyl rings is 75.45 (9)°. The crystal structure is stabilized by weak intermolecular C—H···O interactions and van der Waals forces.

Comment

The photoinduced reactions of coumarin derivatives have been widely investigated (Lewis & Barancyk, 1989). In order to extend the scope of the photoinduced reactions of coumarin derivatives, we have been intensively investigating the photoinduced reactions of coumarins with phenylethenes. In an earlier study, we structurally characterized a benzo[*b*]-naphtho[1,2-*d*]pyran derivative (Usman *et al.*, 2002) which was obtained from the reaction of 3-ethoxycarbonylcoumarin with 1,1-diphenylethene. In this paper, we report the structure of 4-(*N*,*N*-dimethylamino)-3-(2,2-diphenylethenyl)coumarin, (I), obtained by the photo-induced reaction of 3,4-dichloro-coumarin with 1,1-diphenylethene.



(I)

The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The coumarin moiety is planar within ±0.075 (2) Å. The dihedral angle between the pyran and the fused benzene ring is 5.4 (1)°. The C1—O2 [1.215 (2) Å], C1—C9 [1.444 (2) Å] and C9—C8 [1.376 (2) Å] distances show π-conjugation of the C═O, C—C and C═C bonds. Though the dimethylamino group is usually coplanar with the attached coumarin moiety (Jasinski & Paight, 1994), in the title compound it is twisted around the N1—C8 bond [C24—N1—C8—C9 −33.3 (3)° and C25—N1—C8—C7 −57.7 (2)°]. The C9/C10/C11/C12/C18 plane containing the ethylene double bond is planar and it forms dihedral angles of 70.97 (7), 59.42 (9) and 27.57 (8)° with the coumarin moiety and phenyl rings C12/C17 and C18/C23, respectively (Fig. 1). The dihedral angle between the two phenyl rings is 75.45 (9)°. The carbonyl O atom is involved in weak C—H···O intermolecular interactions (see Table 2) and these interactions link the molecules into infinite molecular chains parallel to the *c* direction (Fig.

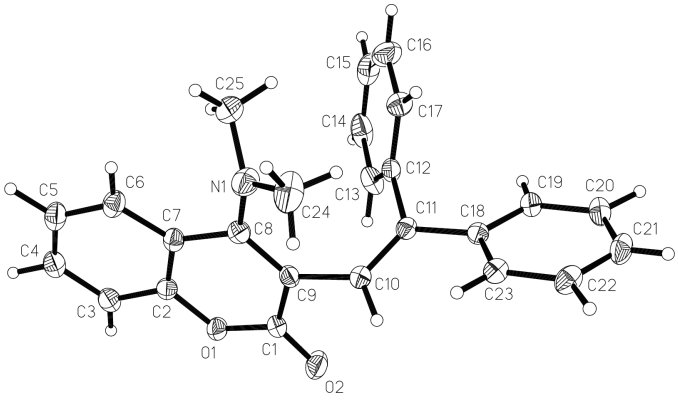


Figure 1
The structure of the title molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

2). These interactions, along with van der Waals interactions, stabilize the molecular packing in the crystal.

Experimental

The title compound was prepared by a photoinduced reaction of 3,4-dichlorocoumarin with an excess of 1,1-diphenylethene, followed by refluxing the cyclobutane product in *N,N*-dimethylacetamide. Single crystals were grown by slow evaporation from a petroleum ether/acetone (1:1 volume ratio) solution.

Crystal data

$C_{25}H_{21}NO_2$
M_r = 367.43
Monoclinic, $P2_1/c$
a = 12.4191 (3) Å
b = 8.8571 (2) Å
c = 17.6617 (3) Å
β = 96.313 (1)°
V = 1930.96 (7) Å^3
Z = 4
D_x = 1.264 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7060 reflections
θ = 2.6–28.3°
μ = 0.08 mm^{-1}
T = 213 (2) K
Block, yellow
0.44 × 0.38 × 0.26 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
11 044 measured reflections
4609 independent reflections
2883 reflections with $I > 2\sigma(I)$
R_{int} = 0.076
θ_{max} = 28.3°
$h = -16 \rightarrow 12$
$k = -11 \rightarrow 11$
$l = -22 \rightarrow 23$

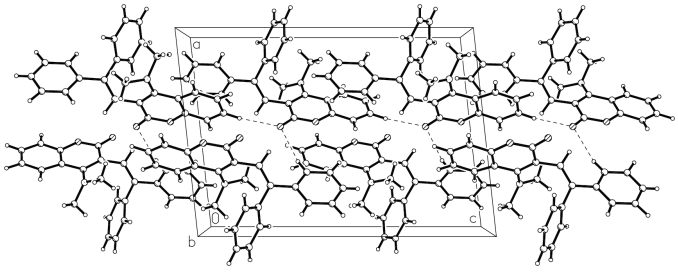


Figure 2
A view of the molecular packing, showing the formation of the molecular chain parallel to the *c* direction.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.141
S = 0.90
4609 reflections
308 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.022 (2)

Table 1
Selected geometric parameters (Å, °).

O2—C1	1.215 (2)	N1—C25	1.462 (2)
N1—C8	1.388 (2)	C1—C9	1.444 (2)
N1—C24	1.453 (2)	C8—C9	1.376 (2)
C24—N1—C8—C9	−33.3 (3)	C25—N1—C8—C7	−57.7 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	1.01 (2)	2.54 (2)	3.550 (2)	174 (1)
C23—H23···O2^{ii}	0.96 (2)	2.55 (2)	3.487 (2)	165 (1)

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

13 H atoms were located from a difference Fourier map and refined isotropically; the remaining eight H atoms were fixed geometrically and allowed to ride on their attached atoms. For the refined H atoms, the C—H distances range from 0.87 (2) to 1.05 (2) Å and U_{iso} values range from 0.031 (5) to 0.064 (7) Å^2.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

The authors thank the Malaysian Government and Universiti Sains Malaysia for research grant R & D No. 305/PFIZIK/610961. AU thanks Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Jasinski, J. P. & Paight, E. S. (1994). *Acta Cryst.* C**50**, 1928–1930.
Lewis, F. D. & Barancyk, S. V. (1989). *J. Am. Chem. Soc.* **111**, 8653–8661.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S., Zhao, B.-G. & Xu, J.-H. (2002). *Acta Cryst.* C**58**, o57–o58.