

Gui-Qin Shang,^a Yu-Xing Gao,^b
Xia Gao,^a Zhi-Hui Yu^a and Hong
Zheng^{a*}^aThe Key Laboratory of Analytical Sciences,
Ministry of Education, Department of Chemistry,
College of Chemistry and Chemical Engineering,
Xiamen University, Xiamen 361005, People's
Republic of China, and ^bDepartment of
Chemistry, The Key Laboratory for Chemical
Biology of Fujian Province, College of Chemistry
and Chemical Engineering, Xiamen University,
Xiamen 361005, People's Republic of China

Correspondence e-mail: hzheng@xmu.edu.cn

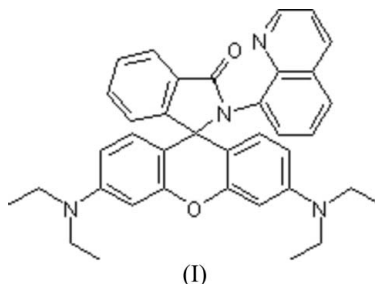
Key indicators

Single-crystal X-ray study
 $T = 223$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.048
 wR factor = 0.139
Data-to-parameter ratio = 14.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3',6'-Bis(diethylamino)-2-(quinolin-8-yl)spiro-
[1*H*-isoindoline-1,9'-xanthen]-3(2*H*)-oneThe title compound, $\text{C}_{37}\text{H}_{36}\text{N}_4\text{O}_2$, has been obtained by the
reaction of 2-[3,6-bis(diethylamino)-9*H*-xanthen-9-yl]benzoyl
chloride with 8-aminoquinoline. The molecular packing in the
crystal structure is stabilized by weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen
bonding.

Received 12 November 2006

Accepted 21 November 2006

Comment

The pH determination of a solution is very important in all
areas of chemistry and biochemistry, but the measurement of
pH at extreme values is still a problem (Safavi *et al.*, 2003). We
have therefore synthesized the title compound, (I), and
investigated its spectral responses to pH values in the range
0.8–4.0.The molecular structure of (I) is shown in Fig. 1. Bond
lengths and angles are in agreement with those reported for
similar compounds (Kwon *et al.*, 2005). The dihedral angle
between isoindoline and xanthene mean planes is 88.9 (3) $^\circ$.
The dihedral angle between the isoindoline mean planes is
 59.0 (2) $^\circ$. Weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding helps to stabi-
lize the crystal structure (Table 1).

Experimental

To a solution of 3',6'-bis(diethylamino)-3*H*-spiro[isobenzofuran-1,9'-
xanthen]-3-one (1.0 g, 2.3 mmol) in dry 1,2-dichloroethane (8.0 ml) at
room temperature, phosphorus oxychloride (1.1 g, 6.9 mmol) was
added dropwise over a period of 5 min. After being refluxed for 4 h,
the reaction mixture was cooled and concentrated under vacuum to
give 2-(3,6-bis(diethylamino)-9*H*-xanthen-9-yl)benzoyl chloride. The
chloride salt was dissolved in dry acetonitrile (10 ml). This solution
was added dropwise to a solution of 8-aminoquinoline (2.0 g,
14 mmol) in dry acetonitrile (6.0 ml) containing triethylamine
(8.0 ml). After stirring for 4 h at room temperature, the mixture was
concentrated under vacuum and the crude product was purified by
column chromatography (ethyl acetate–dichloromethane, 1:20) to
give (I) as a white solid in 70% yield. Single crystals of (I) were
obtained by slow evaporation of a dichloromethane/acetonitrile
solution (20:1 *v/v*).

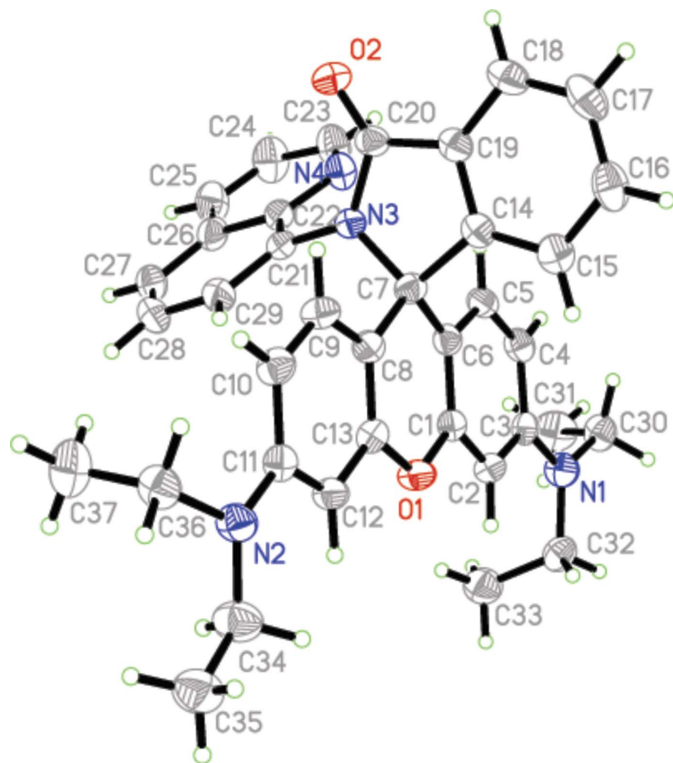


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

Crystal data

$C_{37}H_{36}N_4O_2$
 $M_r = 568.70$
 Monoclinic, $P2_1/c$
 $a = 10.634 (2) \text{ \AA}$
 $b = 23.577 (4) \text{ \AA}$
 $c = 12.614 (2) \text{ \AA}$
 $\beta = 111.222 (3)^\circ$
 $V = 2948.1 (9) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.281 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 223 (2) \text{ K}$
 Block, colorless
 $0.41 \times 0.41 \times 0.40 \text{ mm}$

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 15966 measured reflections

5771 independent reflections
 4748 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.025$
 $\theta_{max} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.139$
 $S = 1.00$
 5771 reflections
 392 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0876P)^2 + 0.4047P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.21 \text{ e \AA}^{-3}$

Table 1

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

<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>
C36—H36A...O2 ⁱ	0.98	2.56	3.521 (2)	166

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

Methyl H atoms were placed in calculated positions, with C—H = 0.96 \AA , and torsion angles were refined, $U_{iso}(H) = 1.5U_{eq}(C)$. Other H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.98 (methine) and 0.97 \AA (methylene), and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the Science Research Foundation of Xiamen University (No. E43011) and the National Natural Science Foundation of China (No. 20275033) for supporting this work. We also thank Mr S.-Y. Yang and Mr Z.-B. Wei for technical assistance.

References

Bruker (2001). *SAINT* (Version 6.22) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Kwon, J. Y., Jang, Y. J., Lee, Y. J., Kim, K. M., Seo, M. S., Nam, W. & Yoon, J. (2005). *J. Am. Chem. Soc.* **127**, 10107–10111.
 Safavi, A. & Bagheri, M. (2003). *Sens. Actuators B*, **90**, 143–150.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.