

**3,3,6,6-Tetramethyl-9-(2-oxo-1,2-dihydroquinolin-3-yl)-1,2,3,4,5,6,7,8-octahydro-9H-xanthene-1,8-dione****S. Selvanayagam,<sup>a</sup> Jasmin Paul,<sup>b</sup>  
D. Velmurugan,<sup>a\*</sup> K. Ravikumar<sup>c</sup>  
and R. Rathna Durga<sup>d</sup>**<sup>a</sup>Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, <sup>b</sup>Bioinformatics Research Institute, Ashok Nagar, Chennai 600 083, India, <sup>c</sup>Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and <sup>d</sup>Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

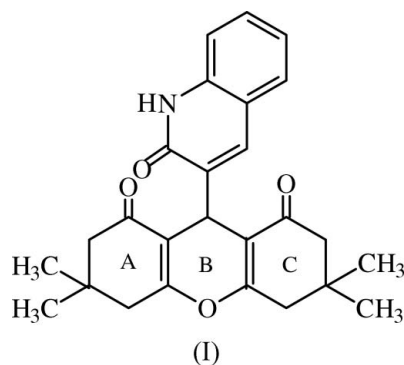
Correspondence e-mail: d\_velu@yahoo.com

**Key indicators**Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.050  
 $wR$  factor = 0.145  
Data-to-parameter ratio = 18.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound,  $\text{C}_{26}\text{H}_{27}\text{NO}_4$ , the central ring of the xanthene moiety is planar and the two outer rings are in half-boat conformations. The molecular packing in the crystal structure is stabilized by  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds in addition to van der Waals forces.

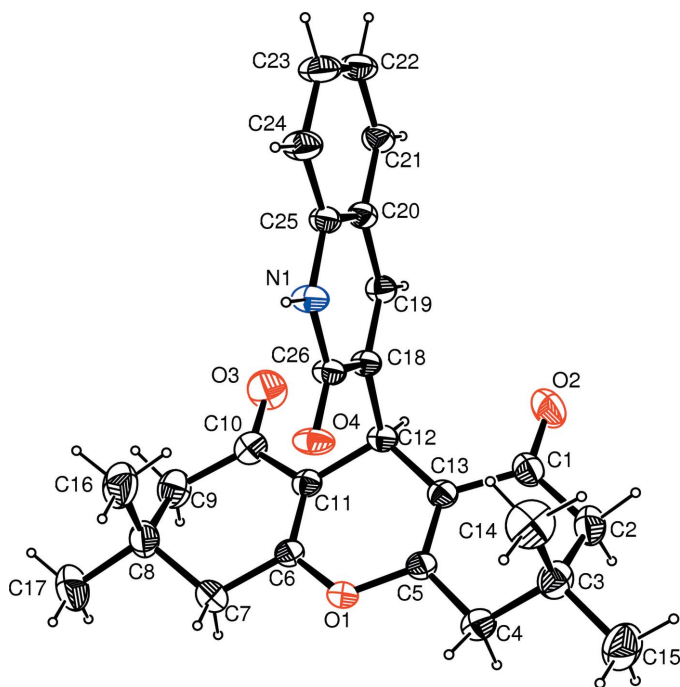
**Comment**

Structural elucidation of xanthenes is essential for understanding the wide range of biological and pharmacological activities described for these compounds (Gales *et al.*, 2005). These derivatives possess antitumour (Filippatos *et al.*, 1994; Gobbi *et al.*, 2002) and cytotoxic activities (Sittisombut *et al.*, 2001). They also act as novel CCR1 receptor antagonists (Naya *et al.*, 2003). Quinolones are valuable synthetic intermediates for other heterocyclic bioactive compounds (Gustavo Pozzi *et al.*, 2003). In view of its importance in this field, and in order to obtain more detailed information on the structure and conformation of the molecule, the crystal structure of the title compound, (I), was determined and the results are presented here.

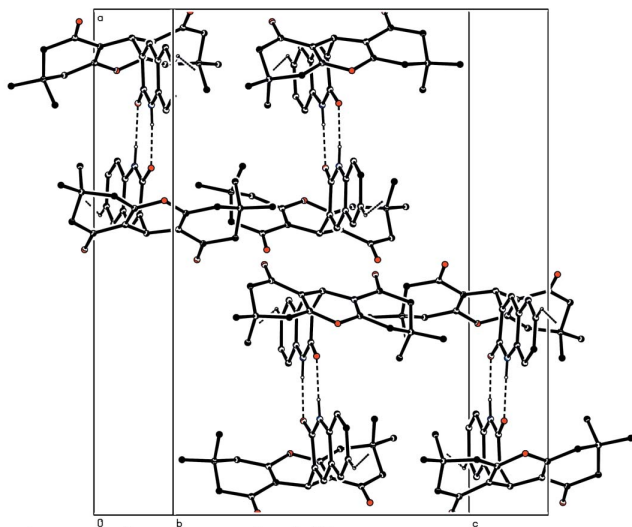


The present X-ray analysis has confirmed the molecular structure and atom connectivity for (I), as illustrated in Fig. 1. The geometry of the xanthene moiety is comparable with that in a related structure reported by Jeyakanthan *et al.* (1999). The bond lengths and angles within the quinolone moiety are comparable with the values reported for the related structure of 1-ethyl-4-hydroxyquinolin-2(1*H*)-one (Baumer *et al.*, 2004).

The quinolone moiety is planar, with a maximum deviation of 0.034 (1) Å for atom O4. The central ring (B) of the xanthene moiety is also planar, with a maximum deviation of 0.120 (1) Å for atom C12. These two planes make a dihedral angle of 86.4 (1)°, thus indicating that the quinolone moiety and ring B are almost perpendicular to each other. The mean planes of the two halves of the xanthene moiety (C1–C13/O1/



**Figure 1**  
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.



**Figure 2**  
A view of the molecular packing of (I), showing C—H...O and N—H...O interactions (dashed lines). H atoms have been omitted.

C12 and C6—C11/O1/C12) make a dihedral angle of 13.0 (1)°.

The two outer rings (A and C) of the xantheno moiety adopt half-boat conformations, as confirmed by the puckering parameters (Cremer & Pople, 1975):  $q_2 = 0.397$  (2),  $q_3 = 0.254$  (2) and  $Q_T = 0.472$  (2) Å for ring A, and  $q_2 = 0.394$  (2),  $q_3 = 0.260$  (2) and  $Q_T = 0.472$  (2) Å for ring C. This is also confirmed by the asymmetry parameters (Nardelli, 1983) of  $\Delta_S(C8) = 0.009$  (1) (ring A) and  $\Delta_S(C3) = 0.009$  (1) (ring C). In ring A, atom C8 deviates from the mean plane of the ring by 0.660 (2) Å, while in ring C, atom C3 deviates from the mean plane of the ring by 0.660 (2) Å.

In the molecular packing, an N1—H1...O4<sup>i</sup> hydrogen bond links symmetry-related molecules (Table 1; symmetry code as in Table 1), forming a dimer with graph set  $R_2^2(8)$  (Fig. 2). The molecules are further linked by C—H...O interactions (Table 1 and Fig. 2).

## Experimental

To a mixture of dimedone (0.5 mmol) and 2-(3-methyl-but-2-enylsulfonyl)quinolone-3-carbaldehyde (1 mmol) was added ethylenediamine acetic acid as catalyst in ethanol (20 ml). After completion of the reaction, as evidenced by thin-layer chromatography, the solvent was removed *in vacuo* and the residue was chromatographed on silica gel, using a mixture of hexane and ethyl acetate (4:1) as eluent, to give the title compound. The compound was recrystallized from a mixture of ethyl acetate and hexane (1:1) to yield diffraction quality crystals.

### Crystal data

$C_{26}H_{27}NO_4$   
 $M_r = 417.49$   
Orthorhombic, *Pccn*  
 $a = 22.2713$  (12) Å  
 $b = 11.9162$  (6) Å  
 $c = 17.2698$  (9) Å  
 $V = 4583.2$  (4) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.210$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
Cell parameters from 6390 reflections  
 $\theta = 2.3$ – $27.5^\circ$   
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
Block, colourless  
0.24 × 0.22 × 0.20 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 $\omega$  scans  
Absorption correction: none  
26484 measured reflections  
5325 independent reflections

3862 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.025$   
 $\theta_{max} = 28.0^\circ$   
 $h = -28 \rightarrow 26$   
 $k = -15 \rightarrow 15$   
 $l = -21 \rightarrow 22$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.050$   
 $wR(F^2) = 0.145$   
 $S = 1.02$   
5325 reflections  
284 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0719P)^2 + 0.8427P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.17$  e Å<sup>-3</sup>

**Table 1**  
Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1...O4 <sup>i</sup>	0.86	1.93	2.787 (2)	176
C22—H22...O1 <sup>ii</sup>	0.93	2.55	3.320 (2)	140

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

H atoms were positioned geometrically and treated as riding on their parent C and N atoms, with C—H distances of 0.93–0.98 Å and an N—H distance of 0.86 Å, and with  $U_{iso}(H) = 1.5U_{eq}(C)$  for methyl H or  $1.2U_{eq}(C, N)$  for other H.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; 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) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PARST (Nardelli, 1995).

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