

Yu-Lin Zhu, Shen-Lin Huang and  
Yuan-Jiang Pan\*Department of Chemistry, Zhejiang University,  
Hangzhou 310027, People's Republic of China

Correspondence e-mail: cheyjpan@zju.edu.cn

## Key indicators

Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.039  
 $wR$  factor = 0.089  
Data-to-parameter ratio = 9.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.9-Butyl-3,4,5,6,7,9-hexahydro-2H-  
xanthene-1,8-dione

The title compound,  $\text{C}_{17}\text{H}_{22}\text{O}_3$ , which was synthesized by the condensation of cyclohexane-1,3-dione and *n*-valeraldehyde, includes a partially hydrogenated xanthene ring system. The molecule has crystallographic mirror symmetry. The central ring adopts a very shallow boat conformation while the symmetry-related outer six-membered rings have sofa conformations. Molecules form extended tapes in the *c*-axis direction though weak  $\text{C}-\text{H}\cdots\text{O}$  interactions.

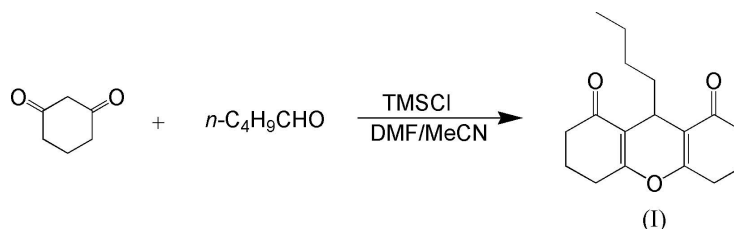
Received 3 December 2004

Accepted 2 March 2005

Online 31 March 2005

## Comment

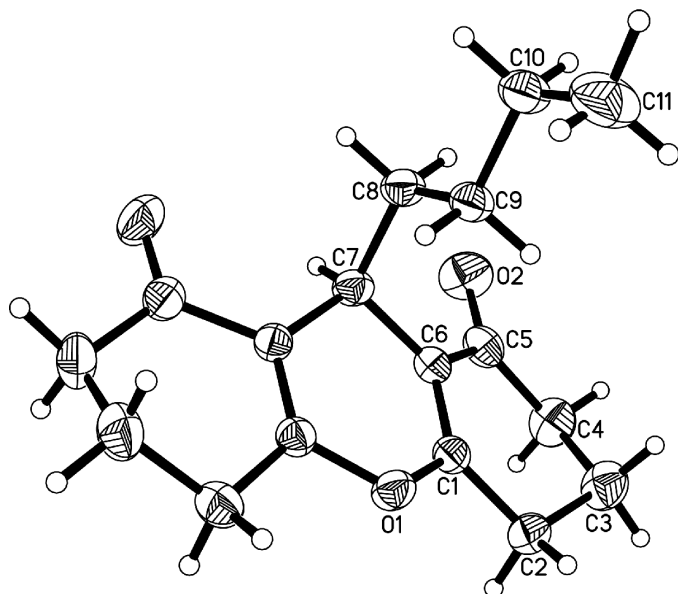
The synthesis of 9-butyl-3,4,5,6,7,9-hexahydro-2H-xanthene-1,8-dione, (I), was initially reported in 1962 (Hellmann & Schroeder, 1962). In our experiment, the reaction of cyclohexane-1,3-dione and *n*-valeraldehyde in the presence of trimethylsilyl chloride (TMSCl) affords (I) in 78% yield. Fig. 1 shows the molecular structure of (I).



The central ring is in a very shallow boat conformation, with atoms C1, C6, C1<sup>i</sup> and C6<sup>i</sup> [symmetry code: (i)  $1 - x, y, z$ ] exactly coplanar by symmetry and atoms O1 and C7 0.156 (4) and 0.263 (6) Å from this plane. Atoms O1 and C7–C11 lie on a crystallographic mirror plane at  $x = \frac{1}{2}$ . The two outer symmetry-related six-membered rings are each in a sofa conformation, with atoms C1, C2, C4, C5 and C6 forming a plane (r.m.s. deviation = 0.024 Å) and atom C3 0.595 (4) Å from this plane. The bond lengths and angles in (I) are normal. The O2–C5–C6–C1 torsion angle of 176.2 (2)° and the C5–C6 bond length of 1.458 (4) Å indicate conjugation between the O2=C5 and C6=C1 bonds. The torsion angle of 61.8 (2)° for C6–C7–C8–C9 defines the orientation of the *n*-butyl group. Molecules are linked by weak  $\text{C}-\text{H}\cdots\text{O}$  interactions [ $\text{H}3\text{A}\cdots\text{O}2^{\text{ii}} = 2.59\text{ \AA}$ ,  $\text{C}3\cdots\text{O}2^{\text{ii}} = 3.316(4)\text{ \AA}$  and  $\text{C}3-\text{H}3\text{A}\cdots\text{O}2^{\text{i}} = 132^\circ$ , symmetry code: (ii)  $x, 1 - y, \frac{1}{2} + z$ ], forming tapes in the *c*-axis direction (see Fig. 2).

## Experimental

Cyclohexane-1,3-dione (10 mmol), *n*-valeraldehyde (10 mmol) and dimethylformamide–acetonitrile (1:2 *v/v* 9 ml) were mixed in a 25 ml flask. TMSCl (10 mmol) was then added dropwise at room



**Figure 1**  
View of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Unlabeled atoms are related by the symmetry code  $(1 - x, y, z)$ .

temperature. The resulting reaction mixture was stirred at 353 K for 3 h, cooled to room temperature and precipitation was observed. The precipitate was isolated by filtering through a Buchner funnel, washed with ethanol and dried to give the crystalline powder. The powder was further purified by recrystallization from  $C_2H_5OH$ . The crystalline product was dissolved in a DMF solution and single crystals suitable for X-ray structure analysis were obtained by slow evaporation of the solution at room temperature.

#### Crystal data

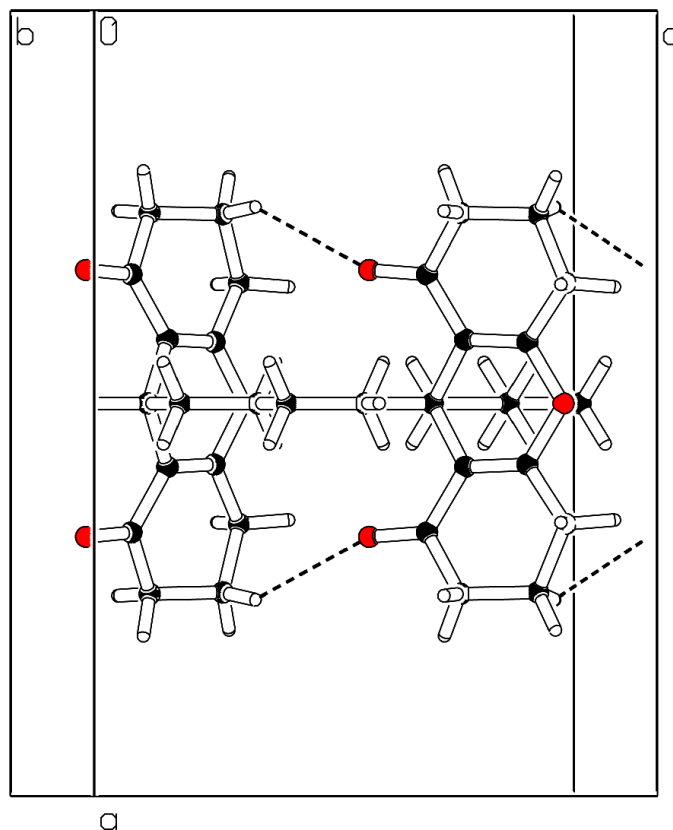
$C_{17}H_{22}O_3$	Mo $K\alpha$ radiation
$M_r = 274.35$	Cell parameters from 40 reflections
Orthorhombic, $Cmc2_1$	$\theta = 4.6\text{--}15.0^\circ$
$a = 15.121(4) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$b = 8.872(2) \text{ \AA}$	$T = 298(2) \text{ K}$
$c = 11.025(3) \text{ \AA}$	Block, colorless
$V = 1479.0(7) \text{ \AA}^3$	$0.50 \times 0.26 \times 0.26 \text{ mm}$
$Z = 4$	
$D_x = 1.232 \text{ Mg m}^{-3}$	

#### Data collection

Siemens P4 diffractometer	$\theta_{\max} = 27.8^\circ$
$\omega$ scans	$h = 0 \rightarrow 19$
Absorption correction: none	$k = 0 \rightarrow 11$
1011 measured reflections	$l = -14 \rightarrow 1$
955 independent reflections	3 standard reflections
638 reflections with $I > 2\sigma(I)$	every 97 reflections
$R_{\text{int}} = 0.010$	intensity decay: 3.2%

#### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.039$	$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2]$
$wR(F^2) = 0.089$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.88$	$(\Delta/\sigma)_{\max} < 0.001$
955 reflections	$\Delta\rho_{\max} = 0.15 \text{ e \AA}^{-3}$
100 parameters	$\Delta\rho_{\min} = -0.12 \text{ e \AA}^{-3}$



**Figure 2**  
Partial packing diagram (Spek, 2003), showing weak  $C-H \cdots O$  interactions as dashed lines.

H atoms were placed in calculated positions, with  $C-H = 0.96\text{--}0.98 \text{ \AA}$ , and refined in riding-model approximation with  $U_{\text{iso}}(H)$  values set equal to  $1.2U_{\text{eq}}(\text{carrier atom})$ . In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Data collection: *XSCANS* (Siemens, 1991); cell refinement: *XSCANS*; data reduction: *SHELXTL/PC* (Siemens, 1994); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

This research was funded by NSFC of China (No. 20375036).

#### References

- Hellmann, H. & Schroeder, M. (1962). *Gerany Ann.* **656**, 85–89.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.  
 Siemens (1991). *XSCANS*. Version 2.10b. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1994). *SHELXTL/PC*. Version 4.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.