Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4a-Hydroxy-9-(3-nitrophenyl)-3,4,4a,5,-6,7,9,9a-octahydro-2*H*-xanthene-1,8-dione

Guoping Hua,[a] Jianing Xu,[b] Bo Jiang,[b] Yan Zhang[b] and Shujiang Tu[b]*

[a]Department of Chemistry, Xuzhou Institute of Architectural Technology, Xuzhou 221008, People's Republic of China, and [b]Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China

Correspondence e-mail: laotu2001@263.net

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.138
Data-to-parameter ratio = 14.8

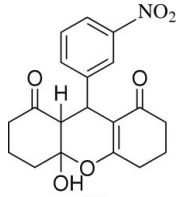
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 30 November 2005
Accepted 16 December 2005
Online 21 December 2005

The title compound, $C_{19}H_{19}NO_6$, has been synthesized by the reaction of 3-nitrobenzaldehyde with 1,3-cyclohexanedione in glycol under microwave irradiation. The compound is an intermediate of the target product 9-(3-nitrophenyl)-3,4,6,7-tetrahydro-2*H*-xanthene-1,8(5*H*,9*H*)-dione. X-ray crystal-structure analysis reveals that the central hydropyran ring adopts a half-chair conformation.

Comment

Pyrans and fused pyrans are biologically interesting compounds with antibacterial activitiy (El-Agrody *et al.*, 2000), antifungal activity (Ohira & Yatagai, 1993), antitumor activity (Mohr *et al.*, 1975) and hypotensive effects (Tandon *et al.*, 1991). Polyfuctionalized 4*H*-pyrans are the structural units of a number of natural products and are used as versatile synthons because of the inherent reactivity of the inbuilt pyran ring. 4*H*-Pyran rings can be transformed to pyridine systems related to pharmacologically important calcium antagonists of the DHP type (Ciller *et al.*, 1985; Marugan *et al.*, 1989; Gonzalez *et al.*, 1992).



(I)

In the course of our studies aimed at developing new approaches for the synthesis of xanthone derivatives, we

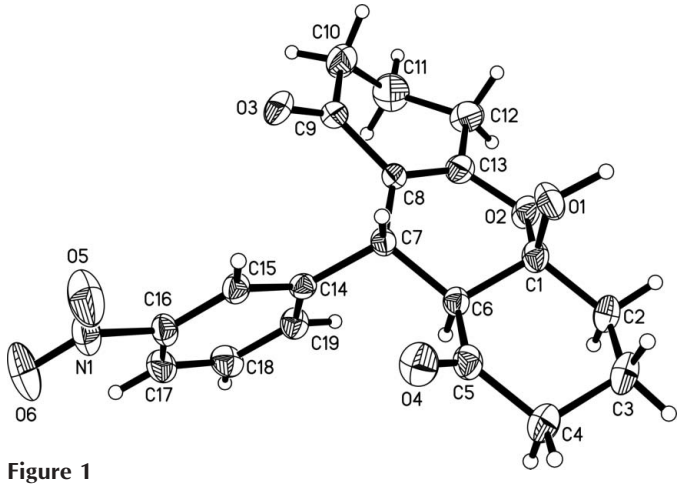


Figure 1
The molecular structure (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

synthesized the title compound, (I), as an intermediate reaction product. Its structure is reported here.

The central O2/C1/C6/C7/C8/C13 hydropyran ring in (I) displays a half-chair conformation, with atoms C1 and C6 displaced from the mean plane by −0.374 (3) and 0.283 (3) Å, respectively (Fig. 1). The C1–C6 ring is in a chair conformation, with atoms C1 and C4 deviating from the C2/C3/C5/C6 plane by −0.659 (3) and 0.614 (4) Å, respectively. The C8–C13 ring adopts a boat conformation, with atoms C8 and C11 displaced from the C9/C10/C12/C13 plane by 0.143 (3) and 0.628 (4) Å, respectively.

In the crystal structure, there is a strong O—H···O intermolecular hydrogen-bond interaction, stabilizing the structure (Table 1, Fig. 2).

Experimental

The title compound was prepared by the reaction of 3-nitrobenzaldehyde (1 mmol) with 1,3-cyclohexanedione (2 mmol) in glycol under microwave irradiation. The reaction was completed in 4 min. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution (m.p. 492 K).

Crystal data

$C_{19}H_{19}NO_6$
M_r = 357.35
Monoclinic, $P2_1/n$
a = 6.2983 (11) Å
b = 12.869 (2) Å
c = 21.619 (4) Å
β = 90.960 (4)°
V = 1752.1 (5) Å^3
Z = 4
D_x = 1.355 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1499 reflections
θ = 2.5–24.3°
μ = 0.10 mm^{-1}
T = 294 (2) K
Prism, colourless
0.22 × 0.18 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.994
9539 measured reflections
3530 independent reflections
1536 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 26.3°
$h = -7 \rightarrow 5$
$k = -14 \rightarrow 16$
$l = -24 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.138
S = 0.94
3530 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0535P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	1.00 (4)	1.66 (4)	2.661 (3)	175 (4)

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

The H atom bonded to O1 was located in a difference Fourier map and refined isotropically. All other H atoms were placed in geometrically idealized positions (C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) values set equal to 1.5U_{eq}(C) for the methyl H atoms and 1.2 U_{eq}(C) for other H atoms.

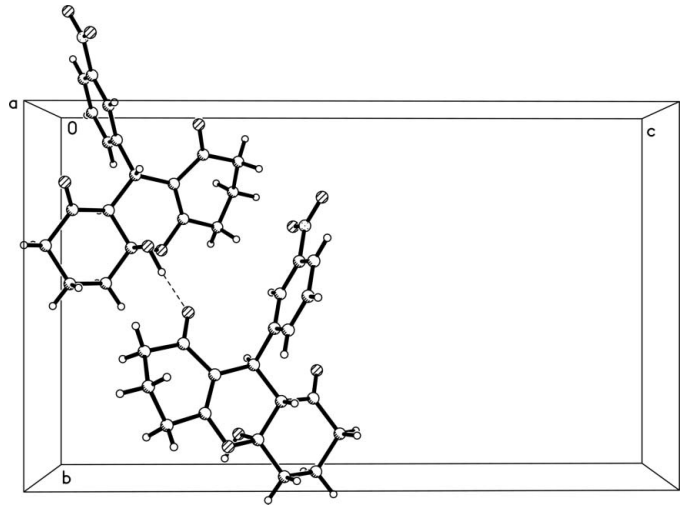


Figure 2
Partial packing diagram for (I), viewed along the *a* axis. The dashed line indicates a hydrogen bond.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

We thank the Natural Science Foundation of China (No. 20372057), the Key Laboratory of Organic Synthesis of Jiangsu Province, the College of Chemistry and Chemical Engineering, Suzhou University Open Foundation (No. JSK011), and the Key Lab of Biotechnology for Medicinal Plants of Jiangsu Province (No. 01AXL 14).

References

Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ciller, J. A., Martin, N., Seoane, C. & Soto, J. (1985). *J. Chem. Soc., Perkin Trans. 1*, pp. 2581–2584.
El-Agrody, A. M., El-Hakim, M. H., Abd El-Latif, M. S., Fakery, A. H., El-Sayed, E. S. M. & El-Ghareab, K. A. (2000). *Acta Pharm.* **50**, 111–120.
Gonzalez, R., Martin, N., Seoane, C., Macro, J. L., Albert, A. & Cano, F. H. (1992). *Tetrahedron Lett.* **33**, 3809–3812.
Marugan, M., Martin, N., Seoane, C. & Soto, J. (1989). *Liebigs Ann. Chem.* 145–149.
Mohr, S. J., Chirigos, M. A., Fuhrman, F. S. & Pryor, J. W. (1975). *Cancer Res.* **35**, 3750–3754.
Ohira, T. & Yatagai, M. (1993). *J. Jpn Wood Res. Soc.* **39**, 237–242.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Tandon, V. K., Vaish, M., Jain, S., Bhakuni, D. S. & Srimal, R. C. (1991). *Indian J. Pharm. Sci.* **53**, 22–23.