Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Redetermination of 3,3,6,6-tetramethyl-4a-hydroxy-9-(*ortho*-methoxyphenyl)-2,3,4,4a,5,6,7,8,9,9a-decahydro-1*H*-xanthene-1,8-dione at 173 K

Michael Bolte,* Alexander Degen and Stephan Rühl

Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

The title compound, $C_{24}H_{30}O_5$, has been redetermined at 173 K. In contrast to the structure determination of Tu *et al.* [*Chin. J. Struct. Chem.* (*Jiegou Huaxue*) (1998), **17**, 409–412] at room temperature, we were able to locate all H atoms. There is an intermolecular hydrogen bond between the hydroxyl group and the carbonyl O atom of the cyclohexenone ring.

Received 16 January 2001
Accepted 22 January 2001
Online 30 January 2001

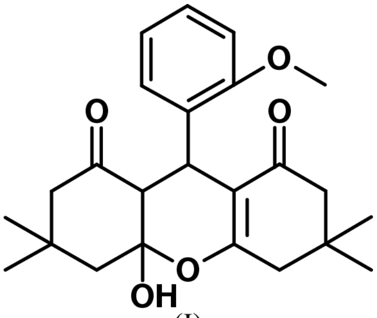
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.111
Data-to-parameter ratio = 18.0

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

Comment

The title compound, (I), was synthesized in the course of a search for twinned crystal structures (Bolte *et al.*, 1997, 2001*b*; Bolte & Scholtyssik, 1997). These compounds can easily be prepared (Hünig *et al.*, 1979). We hoped to find more twinned crystal structures in order to understand why some of these simple compounds containing the common bis-dimedone skeleton are twinned. Unfortunately, neither any of these nor the title compound turned out to be twinned. Whereas the reaction of dimedone with 2,6-dichlorobenzaldehyde leads to four stereoisomers (two enantiomeric pairs of diastereomers) of 3,3,6,6-tetramethyl-4a-hydroxy-9-(2,6-dichlorophenyl)-1,8-dioxo-2,3,4,4a,5,6,7,8,9,9a-decahydro-1*H*-xanthene which can all be found in the same crystal (Bolte *et al.*, 2001*a*), the crystals of the title compound just contain two stereomers. The conformation of the cyclohexane ring can be described as a chair. The other two rings display an approximate sofa conformation with five atoms in a common plane and one atom [C14 0.653 (2) Å; C21 0.592 (2) Å] deviating from this plane. The crystal packing is stabilized by a hydrogen bond between the hydroxyl group and the carbonyl group attached to the cyclohexene ring of an adjacent molecule translated along *y*. The crystal structure of the title compound agrees well with the structure of Tu *et al.* (1998) recently determined at room temperature, however, we have also determined the positions of the H atoms, including the hydroxyl group.



(I)

DOI: 101107/S160053680100160X

Experimental

The title compound was synthesized according to Cremlyn & Saunders (1993).

Crystal data

$C_{24}H_{30}O_5$
$M_r = 398.48$
Monoclinic, $C2/c$
$a = 33.845\ (4)$ Å
$b = 7.214\ (1)$ Å
$c = 22.605\ (3)$ Å
$\beta = 128.82\ (1)^\circ$
$V = 4300.1\ (10)$ Å^3
$Z = 8$

$D_x = 1.231$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7436 reflections
$\theta = 0$–25°
$\mu = 0.09$ mm^{-1}
$T = 173\ (2)$ K
Block, colourless
$0.60 \times 0.50 \times 0.50$ mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.951$, $T_{\max} = 0.959$
39326 measured reflections
4750 independent reflections
4193 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.024$
$\theta_{\max} = 27.1^\circ$
$h = -43 \rightarrow 43$
$k = -9 \rightarrow 9$
$l = -28 \rightarrow 28$
623 standard reflections
frequency: 1500 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.111$
$S = 1.04$
4750 reflections
264 parameters
H atoms constrained

$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 3.6403P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O22—H22···O16$^{\rm i}$	0.84	1.98	2.814 (1)	171

Symmetry code: (i) $x, y - 1, z$.

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [U(H) = 1.5$U_{\rm eq}$(C$_{\rm methyl}$), U(H) = 1.2$U_{\rm eq}$(C) or U(H) = 1.2$U_{\rm eq}$(O)] using a riding model with O—H = 0.84, C—H(aromatic) = 0.95, C—H(methyl) = 0.98, C—H(secondary) = 0.99 or C—H(tertiary) = 1.00 Å. Additionally the torsion angle about the C—O bond of the hydroxyl group was refined.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

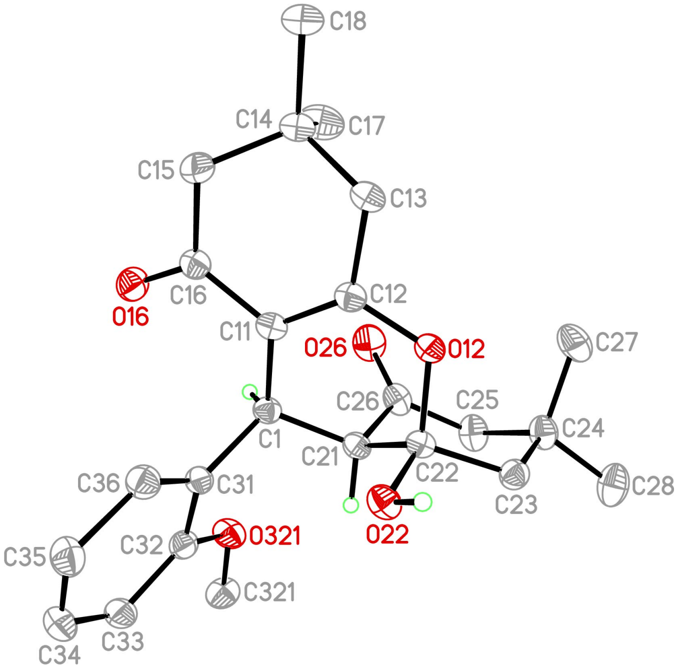


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level; only tertiary and hydroxyl H atoms are shown.

References

Bolte, M., Degen, A. & Rühl, S. (1997). *Acta Cryst.* C**53**, 340–342.
Bolte, M., Degen, A. & Rühl, S. (2001*a*). *Acta Cryst.* C**57**. In the press.
Bolte, M., Degen, A. & Rühl, S. (2001*b*). *Acta Cryst.* C**57**. Accepted.
Bolte, M. & Scholtyssik, M. (1997). *Acta Cryst.* C**53**, 1869–1871.
Cremlyn, R. J. & Saunders, D. (1993). *Phosphorus Sulfur Silicon*, **81**, 73–82.
Hünig, S., Märkl, G. & Sauer, J. (1979). *Integriertes Organisches Praktikum*. Weinheim: VCH.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tu, S.-J., Shi, D.-Q., Lu, Z.-S., Wang, S.-H. & Dai, G.-Y. (1998). *Chin. J. Struct. Chem.* (*Jiegou Huaxue*), **17**, 409–412.