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6,7-Dimethyl-3,4,4a,7,8,8a-hexahydroisochromen-1-one

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The title compound, $C_{11}H_{16}O_2$, crystallized in space group $P2_1/n$ with two very similar molecules in the asymmetric unit. In each molecule, the two rings are fused in the *endo* configuration.

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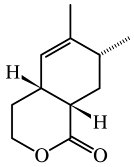
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.100
Data-to-parameter ratio = 14.7

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

Comment

The title lactone, (I), was prepared as an intermediate in the synthesis of an isomer of the oxaspirobicyclic tetronic acid unit of the CCK-B receptor antagonist tetronothiodin.



(I)

The asymmetric unit of (I) contains two independent molecules (Fig. 1) which are essentially superimposable. The structure determination confirms the expected geometry at atoms C2, C4 and C9 in each molecule and there are no unusual bond lengths or angles.

Fig. 2 shows the unit-cell packing; the molecules are stacked parallel to the *b* axis. There are indications of weak C—H···O hydrogen bonds between neighbouring molecules; these are listed in Table 1 (Desiraju, 1996; Taylor & Kennard, 1982).

Experimental

The title lactone was formed by oxidation of the analogous lactol, using pyridinium dichromate, and recrystallized from dichloromethane solution by evaporation. The synthesis of the compound has been reported elsewhere (Page *et al.*, 2003).

Crystal data

$C_{11}H_{16}O_2$
M_r = 180.24
Monoclinic, $P2_1/n$
a = 14.2924 (17) Å
b = 5.4060 (6) Å
c = 25.969 (3) Å
β = 96.530 (2)°
V = 1993.4 (4) Å^3
Z = 8
D_x = 1.201 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3712 reflections
θ = 2.6–27.8°
μ = 0.08 mm^{-1}
T = 150 (2) K
Lath, colourless
0.48 × 0.11 × 0.03 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SHELXTL*/*SADABS*; Sheldrick, 1998)
T_{min} = 0.938, T_{max} = 1.000
13 507 measured reflections
3517 independent reflections
2526 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.0°
$h = -16 \rightarrow 16$
$k = -6 \rightarrow 6$
$l = -30 \rightarrow 30$

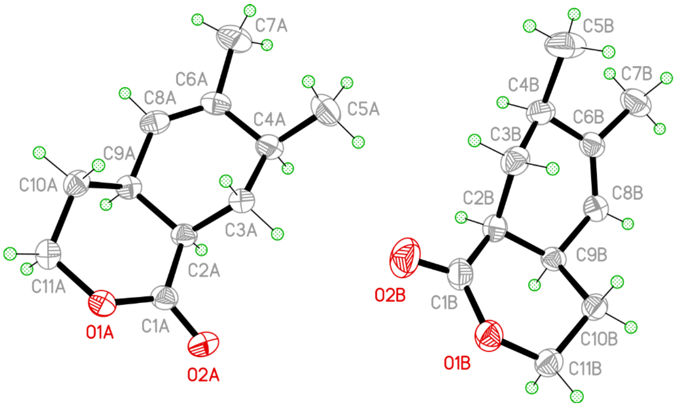


Figure 1
Perspective view of the two independent molecules in the asymmetric unit of (I), showing 50% probability displacement ellipsoids.

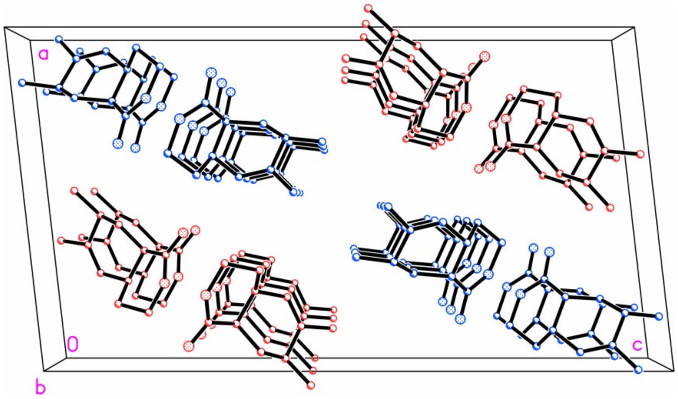


Figure 2
Projection of the unit-cell contents along *b*. Atoms labelled *A* in are shown in red, those labelled *B* are in blue.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.100$
$S = 1.04$
3517 reflections
239 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.7308P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8B—H8$B\cdots$O2$A^{\rm i}$	0.95	2.48	3.304 (2)	144
C9A—H9$A\cdots$O1$A^{\rm ii}$	1.00	2.80	3.577 (2)	135
C11A—H11$A\cdots$O2$A^{\rm iii}$	0.99	2.82	3.455 (2)	122
C11A—H11$B\cdots$O2$A^{\rm iv}$	0.99	2.55	3.510 (2)	162
C9B—H9$B\cdots$O1$B^{\rm i}$	1.00	2.88	3.569 (2)	127
C9B—H9$B\cdots$O1$B^{\rm ii}$	1.00	2.76	3.529 (2)	134
C11B—H11$C\cdots$O2$B^{\rm i}$	0.99	2.61	3.567 (2)	164

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

H atoms were introduced at calculated positions; the constrained C—H distances were 0.95, 0.98, 0.99 and 1.00 Å for H atoms bonded to sp^2, methylene, methyl and tertiary C atoms, respectively. H atoms were refined with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$, except for the methyl groups where $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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