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Structure of 3,4,5,6,7,8-Hexahydro-1-methyl-1*H*-2-benzoxecine-3,8-dione

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Abstract. $C_{14}H_{16}O_3$, $M_r = 232·28$, orthorhombic, $P2_12_12_1$, $a = 7·990\,(2)$, $b = 11·664\,(3)$, $c = 12·986\,(3)$ Å, $V = 1210·2$ Å^3, $Z = 4$, $D_x = 1·275$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 0·083$ mm^{-1}, $F(000) = 496$, $T = 200$ K, $R = 0·030$ for 978 data [$I \geq 2·5\sigma(I)$]. The conformation of the ten-membered ring is similar to that of 2,7-oxecanedione. The fused benzene ring affects the conformation of the attached segment of the oxecane ring, making it almost planar rather than *gauche*.

Introduction. The structure of the title compound was investigated to compare it with that of 2,7-oxecanedione (Fedeli & Dunitz, 1968) to study the effects on the conformation of the addition of a fused benzene ring.

Experimental. A colourless crystal of the title compound (Chow & Ouyang, 1990) was mounted on a Pyrex filament using five-minute epoxy resin. Intensity data were measured at 200 K with an Enraf–Nonius CAD-4F diffractometer having an extensively in-house-modified low-temperature attachment, using Mo $K\alpha$ radiation and a graphite monochromator. The unit cell was determined from 25 reflections, $15 \leq \theta \leq 21°$. Two standard intensities were measured every hour of acquisition time and showed no significant variations. An empirical absorption correction (0·96–1·00) was applied to the data (North, Phillips & Mathews, 1968). Data reduction was performed, including Lorentz and polarization corrections. $0 \leq h \leq 9$, $0 \leq k \leq 13$, $0 \leq l \leq 15$, 978 observed reflections [$I \geq 2·5\sigma(I)$].

The structure was solved by direct methods. The H atoms were included in calculated positions, 0·95 Å from their respective C atoms, and were recalculated prior to the final cycles of refinement. A weighting scheme, based on counting statistics, was applied such that $\langle w(|F_o| - |F_c|)^2\rangle$ was nearly constant as a function of $|F_o|$ and $(\sin\theta)/\lambda$. Final full-matrix least-squares refinement of 164 parameters included anisotropic thermal motion for the non-H atoms and extinction (Larson, 1970). While the observations to parameters ratio is somewhat low ($\simeq 6$) the resulting anisotropic thermal parameters are consistent with a model for rigid-body thermal motion (Schomaker & Trueblood, 1968). $R = 0·12$ for the agreement between experimental and calculated U_{ij}, with an r.m.s. discrepancy of 0·004 Å^2. The maximum Δ/σ was $\leq 0·01$ for the final cycle of refinement. The largest peak in the final difference map [0·13 (3) e Å^{-3}] occurred 0·80 Å from O(1), and the minimum was $-0·12$ (3) e Å^{-3}.

Complex scattering factors for neutral atoms (*International Tables for X-ray Crystallography*, 1974, Vol. IV) were used in the calculation of structure factors. The programs used for data reduction, structure solution and refinement were from the *NRCVAX* crystal structure system (Gabe, LePage, Charland, Lee & White, 1989). All computations were carried out on a MicroVAX-II computer.

Details of the data acquisition and structure refinement are summarized in Table 1. The final positional and equivalent isotropic thermal parameters for the non-H atoms are given in Table 2.*

Discussion. The structure is shown in Fig. 1. Selected bond distances and angles and bond torsion angles are given in Table 3. The conformation of the ten-membered ring is derived from that of 2,7-oxecanedione (Fedeli & Dunitz, 1968) which approximates a [2323] 'diamond-lattice' arrangement in that the fused benzene ring imposes near planarity on the C(7)—C(8)—C(9)—C(10) segment which would otherwise display a *gauche* relationship. This distortion of the ring is restricted to the torsion angles about the bonds C(7)—C(8), C(8)—C(9) and C(9)—C(10) while the remaining bond torsion angles about the ring closely parallel those in 2,7-oxecanedione. The conformation found is presumably favoured for two reasons. Firstly (*cf.* Dunitz, 1968), placement of the carbonyl and the ester linkage in the long sides of the ring eliminates cross-ring H···H nonbonded contacts and introduces a relatively favourable dipolar interaction between

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* Lists of coordinates and temperature factors for the H atoms, anisotropic temperature factors, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53596 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Experimental details for* 3,4,5,6,7,8-*hexahydro*-1-*methyl*-1*H*-2-*benzoxecine*-3,8-*dione*

Crystal size (mm)	0·35 × 0·40 × 0·44
Min.–max. 2θ (°)	4–50
Scan mode	ω–2θ
Scan width* (°)	1·0 + 0·35tanθ
Scan speed (° min^{-1})	0·87–3·30
Unique data	1236
Extinction, g†	0·41 (3)
$R(F)$	0·030
$wR(F)$‡	0·019
S	2·35

* The background intensities were estimated by extension of the predetermined scan angle by 25% on either side.

† See Larson (1970).

‡ $w = [\sigma^2(F_o) + 0{\cdot}0001F_o^2]^{-1}$.

Table 2. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *for the non*-H *atoms of* 3,4,5,6,7,8-*hexahydro*-1-*methyl*-1*H*-2-*benzoxecine*-3,8-*dione*

	x	y	z	B_{eq}*
O(1)	0·64718 (22)	0·68803 (13)	0·15609 (12)	2·49
O(2)	0·8288 (3)	0·81801 (16)	0·09396 (15)	4·72
O(7)	0·66624 (24)	0·53659 (15)	0·34320 (14)	3·11
C(2)	0·7861 (4)	0·7202 (3)	0·10414 (19)	3·12
C(3)	0·8756 (4)	0·6172 (3)	0·05907 (20)	3·78
C(4)	0·8615 (4)	0·50440 (23)	0·11894 (22)	3·44
C(5)	0·9636 (4)	0·4973 (3)	0·21812 (23)	3·93
C(6)	0·9388 (4)	0·5981 (3)	0·29180 (20)	3·39
C(7)	0·7638 (3)	0·61420 (23)	0·33307 (19)	2·40
C(8)	0·7231 (3)	0·73474 (22)	0·36772 (18)	2·28
C(9)	0·6254 (3)	0·80990 (21)	0·30914 (18)	2·20
C(10)	0·5508 (4)	0·77830 (22)	0·20582 (20)	2·68
C(11)	0·3732 (4)	0·73253 (23)	0·21499 (21)	3·39
C(12)	0·7904 (4)	0·77205 (25)	0·46117 (18)	2·85
C(13)	0·7576 (4)	0·88106 (25)	0·49721 (20)	3·28
C(14)	0·6565 (4)	0·95379 (23)	0·44180 (20)	3·21
C(15)	0·5932 (3)	0·91867 (21)	0·34775 (20)	2·62

* B_{eq} is the mean of the principal axes of the thermal ellipsoids.

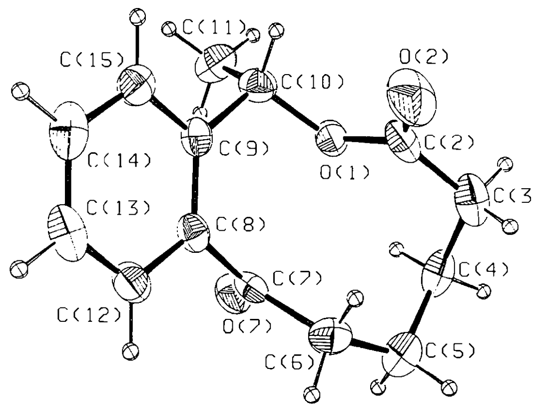

Fig. 1. Molecular structure of 3,4,5,6,7,8-hexahydro-1-methyl-1*H*-2-benzoxecine-3,8-dione.

Table 3. *Selected bond distances* (Å), *angles and torsion angles* (°) *for* 3,4,5,6,7,8-*hexahydro*-1-*methyl*-1*H*-2-*benzoxecine*-3,8-*dione*

O(7)—C(7)	1·202 (3)	C(14)—C(15)	1·384 (4)
O(2)—C(2)	1·198 (4)	C(15)—C(9)	1·388 (4)
O(1)—C(10)	1·456 (3)	C(9)—C(10)	1·514 (4)
O(1)—C(2)	1·352 (3)	C(10)—C(11)	1·521 (4)
C(8)—C(12)	1·397 (4)	C(2)—C(3)	1·515 (4)
C(8)—C(9)	1·399 (4)	C(3)—C(4)	1·533 (4)
C(8)—C(7)	1·512 (4)	C(4)—C(5)	1·527 (4)
C(12)—C(13)	1·380 (4)	C(5)—C(6)	1·528 (4)
C(13)—C(14)	1·375 (4)	C(6)—C(7)	1·509 (4)

C(10)—O(1)—C(2)	117·07 (20)	O(1)—C(10)—C(11)	105·94 (20)
C(12)—C(8)—C(9)	119·47 (24)	C(9)—C(10)—C(11)	112·55 (21)
C(12)—C(8)—C(7)	117·75 (23)	O(2)—C(2)—O(1)	123·6 (3)
C(9)—C(8)—C(7)	122·76 (21)	O(2)—C(2)—C(3)	125·3 (3)
C(8)—C(12)—C(13)	120·57 (25)	O(1)—C(2)—C(3)	111·12 (24)
C(12)—C(13)—C(14)	120·18 (24)	C(2)—C(3)—C(4)	116·75 (22)
C(13)—C(14)—C(15)	119·61 (25)	C(3)—C(4)—C(5)	115·79 (24)
C(14)—C(15)—C(9)	121·42 (25)	C(4)—C(5)—C(6)	114·67 (23)
C(8)—C(9)—C(15)	118·67 (23)	C(5)—C(6)—C(7)	116·00 (24)
C(8)—C(9)—C(10)	123·31 (22)	O(7)—C(7)—C(8)	121·89 (24)
C(15)—C(9)—C(10)	118·02 (23)	O(7)—C(7)—C(6)	123·10 (25)
O(1)—C(10)—C(9)	111·16 (21)	C(8)—C(7)—C(6)	114·90 (23)

C(2)—O(1)—C(10)—C(9)	−82·2 (2)	C(2)—O(1)—C(10)—C(11)	155·2 (3)
C(10)—O(1)—C(2)—O(2)	3·0 (1)	C(10)—O(1)—C(2)—C(3)	178·5 (3)
C(9)—C(8)—C(12)—C(13)	1·7 (2)	C(7)—C(8)—C(12)—C(13)	−179·8 (3)
C(12)—C(8)—C(9)—C(15)	−2·3 (2)	C(12)—C(8)—C(9)—C(10)	178·5 (3)
C(7)—C(8)—C(9)—C(15)	179·4 (3)	C(7)—C(8)—C(9)—C(10)	0·1 (1)
C(12)—C(8)—C(7)—O(7)	101·0 (3)	C(12)—C(8)—C(7)—C(6)	−75·2 (2)
C(9)—C(8)—C(7)—O(7)	−80·6 (2)	C(9)—C(8)—C(7)—C(6)	103·2 (3)
C(8)—C(12)—C(13)—C(14)	0·7 (2)	C(12)—C(13)—C(14)—C(15)	−2·5 (2)
C(13)—C(14)—C(15)—C(9)	1·9 (2)	C(14)—C(15)—C(9)—C(8)	0·5 (2)
C(14)—C(15)—C(9)—C(10)	179·8 (3)	C(8)—C(9)—C(10)—O(1)	−25·1 (1)
C(8)—C(9)—C(10)—C(11)	93·5 (3)	C(15)—C(9)—C(10)—O(1)	155·6 (3)
C(15)—C(9)—C(10)—C(11)	−85·7 (2)	O(2)—C(2)—C(3)—C(4)	150·5 (4)
O(1)—C(2)—C(3)—C(4)	−31·1 (2)	C(2)—C(3)—C(4)—C(5)	−73·6 (2)
C(3)—C(4)—C(5)—C(6)	51·0 (2)	C(4)—C(5)—C(6)—C(7)	61·9 (2)
C(5)—C(6)—C(7)—O(7)	27·8 (2)	C(5)—C(6)—C(7)—C(8)	−156·0 (3)

O(1) and C(7) which are separated by only 2·625 (3) Å. The analogous distance is 2·826 (3) Å in 2,7-oxecanedione (Fedeli & Dunitz, 1968). Secondly, 1–4 nonbonded contacts involving the methyl group [C(11)] appear to be minimized [*cf.* C(11)—C(10)—C(9)—C(8) 93·5, C(11)—C(10)—C(9)—C(15) −85·7 (2), C(11)—C(10)—O(1)—C(2), 155·2 (3)°] in this conformer. The effect of these interactions clearly overrides any relative destabilization resulting from the disruption of conjugation between ketone function and the benzene ring [*cf.* −80·6 (2)° for C(9)—C(8)—C(7)—O(7)].

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