$wR = 0.0582$ ($R_{all} = 0.0774$, $wR_{all} = 0.0611$) and a goodness of fit = 1·577. The maximum $|\Delta/\sigma| < 0.1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were -0.24 and 0.26 e Å^{-3}, respectively. Differentiation between enantiomorphs could not be made on the basis of the X-ray results ($wR = 0.0582$ for the enantiomorph). The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974).* Figures were generated using *SHELXTL-Plus* (Sheldrick, 1987). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The crystal structure of the related C(1)–C(10) subunit of the erythromycins has been reported (Lynch, Pacofsky, Martin & Davis, 1989). The total synthesis of erythronolide B has been

* Tables of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving H atoms, structure factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51865 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

reported (Sviridov, Ermolenko, Yashunsky, Borodkin & Kochetkov, 1987a,b).

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Structure of 3β,3aβ,6-Trimethyl-3a,7aβ-dihydro-2(3H),5(4H)-benzo[b]furandione

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Abstract. $C_{11}H_{14}O_3$, $M_r = 194.23$, orthorhombic, $P2_12_12_1$, $a = 6.648$ (2), $b = 6.710$ (1), $c = 22.941$ (3) Å, $V = 1023.4$ (4) Å^3, $Z = 4$, $D_x = 1.261$ g cm^{-3}, Mo $K\alpha$ radiation (graphite monochromator), $\lambda = 0.71073$ Å, $\mu = 0.85$ cm^{-1}, $F(000) = 416$, $T = 295$ (1) K, $R = 0.033$ for 183 variables and the 862 reflections having $I > 2\sigma(I)$. This structure confirms the stereochemistry at C(3a) and C(7a) of a model substrate used to evaluate a manganese triacetate oxidation step needed for a quassinoid synthesis. There are no unusual bond lengths or angles.

Experimental. The title compound was synthesized (Jeganathan, Richardson & Watt, 1989) and crystallized from ether as colorless, thick, rectangular plates; the dimensions of the data crystal were 0·22 ×

$C_{11}H_{14}O_3$

Table 1. Positional parameters and equivalent B values for 3β,3aβ,6-trimethyl-3a,7aβ-dihydro-2(3H),5(4H)-benzo[b]furandione

Starred atoms were refined isotropically. The equivalent displacement parameter B_{eq} is defined as $(4/3)Tr(\beta \cdot G)$, where $\beta_{ij} = 2\pi^2 a_i^* a_j^* U_{ij}$.

	x	y	z	B_{eq} or B_{iso} (Å^2)
O(1)	0·0091 (3)	−0·0758 (3)	−0·09965 (7)	4·07 (5)
O(2)	0·0787 (3)	−0·3916 (3)	−0·07830 (8)	5·27 (6)
O(3)	0·5775 (3)	0·1166 (3)	−0·24427 (7)	4·94 (5)
C(1)	0·1374 (4)	−0·2309 (4)	−0·09347 (10)	3·62 (7)
C(2)	0·3483 (4)	−0·1673 (4)	−0·10711 (10)	3·11 (6)
C(3)	0·5052 (6)	−0·2714 (5)	−0·06981 (13)	4·46 (9)
C(4)	0·3366 (4)	0·0619 (4)	−0·10360 (10)	3·00 (6)
C(5)	0·3682 (6)	0·1355 (5)	−0·04112 (12)	4·40 (9)
C(6)	0·4816 (5)	0·1659 (4)	−0·14534 (11)	3·82 (7)
C(7)	0·4408 (4)	0·1286 (4)	−0·20897 (10)	3·38 (6)
C(8)	0·2285 (4)	0·1156 (4)	−0·22693 (9)	3·28 (6)
C(9)	0·1844 (6)	0·1103 (6)	−0·29117 (13)	4·82 (9)
C(10)	0·0856 (4)	0·1057 (4)	−0·18665 (11)	3·66 (7)
C(11)	0·1188 (4)	0·0973 (4)	−0·12239 (11)	3·43 (7)
H(2)	0·372 (4)	−0·204 (3)	−0·1466 (10)	3·2 (5)*
H(3A)	0·629 (5)	−0·229 (4)	−0·0792 (11)	4·3 (7)*
H(3B)	0·510 (5)	−0·430 (6)	−0·0781 (12)	8·0 (9)*
H(3C)	0·481 (4)	−0·257 (4)	−0·0306 (12)	4·2 (6)*
H(5A)	0·502 (5)	0·108 (5)	−0·0283 (13)	6·9 (9)*
H(5B)	0·285 (4)	0·059 (4)	−0·0124 (11)	4·8 (6)*
H(5C)	0·342 (4)	0·274 (5)	−0·0374 (11)	4·8 (6)*
H(6A)	0·610 (4)	0·127 (4)	−0·1360 (10)	3·3 (6)*
H(6B)	0·459 (4)	0·315 (5)	−0·1409 (11)	5·2 (7)*
H(9A)	0·050 (6)	0·112 (5)	−0·2965 (13)	6·2 (9)*
H(9B)	0·264 (5)	0·001 (5)	−0·3109 (14)	6·5 (8)*
H(9C)	0·223 (5)	0·234 (5)	−0·3086 (15)	6·3 (8)*
H(10)	−0·055 (4)	0·093 (4)	−0·1979 (10)	3·6 (6)*
H(11)	0·072 (4)	0·216 (4)	−0·1027 (10)	4·0 (6)*

0·35 × 0·35 mm. Cell constants were determined from the setting angles of 21 reflections having $12·2 < \theta < 12·8°$. Data in a quadrant of reciprocal space with $2\theta \leq 55°$ and having $-8 \leq h \leq 8$, $0 \leq k \leq 8$, and $0 \leq l \leq 29$ were measured on an Enraf–Nonius CAD-4 diffractometer using $\omega/2\theta$ scans. After averaging (1201 reflections observed more than once; R factor on I for averaging of 0·037) there were 1402 unique reflections. The data were corrected for decomposition (3·2%); no correction was necessary for absorption or extinction. The structure was solved using direct methods; the absolute chirality was assigned on the basis of the synthetic pathway. The H atoms were located in difference Fourier maps and refined isotropically. The structure converged (maximum $\Delta/\sigma = 0·02$) to agreement factors R and wR on F_o of 0·033 and 0·032 for 183 variables and the 862 observations having $I > 2\sigma(I)$; the error in an observation of unit weight is 1·40. Weights used calculated as $4I/[\sigma^2(I)]$, where $[\sigma^2(I)]$ includes a term $(0·03I)^2$. Atomic scattering factors were taken from Cromer & Waber (1974); computer programs have been described previously (Brock & Webster, 1976). The largest features of the final difference Fourier map have heights $< 0·12$ and $> -0·16$ e Å^{-3}.

Table 2. Bond lengths (Å) and selected bond angles and torsion angles (°) for 3β,3aβ,6-trimethyl-3a, 7aβ-dihydro-2(3H),5(4H)-benzo[b]furandione

Numbers in parentheses are estimated standard deviations in the least significant digits.

C(1)—O(1)	1·353 (3)	C(6)—C(7)	1·506 (3)
C(1)—O(2)	1·198 (3)	C(7)—O(3)	1·220 (3)
C(1)—C(2)	1·499 (4)	C(7)—C(8)	1·473 (3)
C(2)—C(3)	1·519 (4)	C(8)—C(9)	1·503 (4)
C(2)—C(4)	1·542 (3)	C(8)—C(10)	1·327 (3)
C(4)—C(5)	1·530 (4)	C(10)—C(11)	1·492 (4)
C(4)—C(6)	1·528 (4)	C(11)—O(1)	1·468 (3)
C(4)—C(11)	1·529 (4)		
C(2)—H(2)	0·95 (2)	C(6)—H(6A)	0·92 (3)
C(3)—H(3A)	0·90 (3)	C(6)—H(6B)	1·02 (3)
C(3)—H(3B)	1·08 (4)	C(9)—H(9A)	0·90 (3)
C(3)—H(3C)	0·92 (3)	C(9)—H(9B)	1·01 (3)
C(5)—H(5A)	0·95 (3)	C(9)—H(9C)	0·96 (4)
C(5)—H(5B)	1·00 (3)	C(10)—H(10)	0·97 (3)
C(5)—H(5C)	0·95 (3)	C(11)—H(11)	0·97 (3)
C(1)—O(1)—C(11)	109·4 (2)	C(6)—C(4)—C(11)	110·5 (2)
O(1)—C(1)—O(2)	121·1 (2)	C(4)—C(6)—C(7)	114·7 (2)
O(1)—C(1)—C(2)	110·4 (2)	O(3)—C(7)—C(6)	121·3 (2)
O(2)—C(1)—C(2)	128·4 (3)	O(3)—C(7)—C(8)	121·6 (2)
C(1)—C(2)—C(3)	113·2 (2)	C(6)—C(7)—C(8)	117·0 (2)
C(1)—C(2)—C(4)	103·0 (2)	C(7)—C(8)—C(9)	117·6 (2)
C(3)—C(2)—C(4)	117·6 (2)	C(7)—C(8)—C(10)	119·6 (2)
C(2)—C(4)—C(5)	111·3 (2)	C(9)—C(8)—C(10)	122·8 (3)
C(2)—C(4)—C(6)	113·0 (2)	C(8)—C(10)—C(11)	125·7 (2)
C(2)—C(4)—C(11)	100·9 (2)	O(1)—C(11)—C(4)	104·3 (2)
C(5)—C(4)—C(6)	110·7 (2)	O(1)—C(11)—C(10)	107·9 (2)
C(5)—C(4)—C(11)	110·1 (2)	C(4)—C(11)—C(10)	115·1 (2)

O(1)—C(1)—C(2)—C(4)	17·2 (2)	C(4)—C(6)—C(7)—C(8)	37·0 (3)
C(1)—C(2)—C(4)—C(11)	−30·4 (2)	C(6)—C(7)—C(8)—C(10)	−10·4 (4)
C(2)—C(4)—C(11)—O(1)	33·6 (2)	C(7)—C(8)—C(10)—C(11)	−2·9 (4)
C(4)—C(11)—O(1)—C(1)	−25·0 (2)	C(8)—C(10)—C(11)—C(4)	−10·8 (4)
C(11)—O(1)—C(1)—C(2)	4·7 (3)	C(10)—C(11)—C(4)—C(6)	35·3 (3)
		C(11)—C(4)—C(6)—C(7)	−48·4 (3)
O(2)—C(1)—C(2)—C(3)	−33·8 (4)		
O(3)—C(7)—C(8)—C(9)	−6·8 (4)	C(6)—C(4)—C(11)—O(1)	153·4 (2)
C(3)—C(2)—C(4)—C(5)	−38·9 (4)	C(2)—C(4)—C(11)—C(10)	−84·4 (2)

The molecular structure and the atomic numbering scheme are shown in Fig. 1; the atomic coordinates are given in Table 1, and the bond lengths and some bond angles in Table 2.*

Related literature. Jeganathan, Richardson & Watt (1989) reported oxidation by manganese triacetate of the hindered α' position in certain enones as a route for the introduction of the α'-hydroxyenone functionality characteristic of the A ring in certain quassinoids. The structure of the title compound, a model enone for this synthetic route, was solved in order to determine the relative stereochemistry at C(3a) and C(7a) [C(4) and C(11) according to the numbering scheme shown in Fig. 1]. The absolute stereo-

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* Lists of refined atomic displacement parameters, bond angles involving H atoms, and the observed and calculated structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51824 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
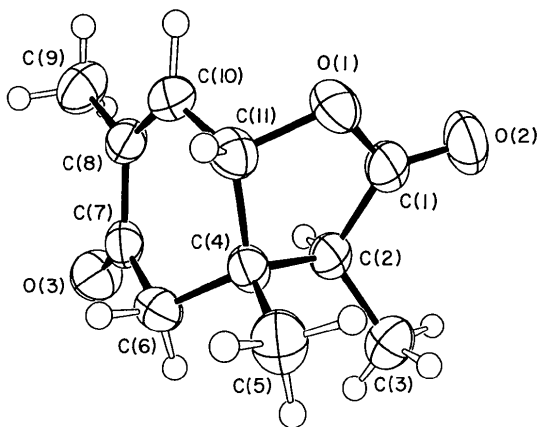
Fig. 1. Perspective drawing of the molecular structure of 3β,3aβ,6-trimethyl-3a,7aβ-dihydro-2(3H),5(4H)-benzo[b]furan-dione. The shapes of the ellipsoids of the C and O atoms correspond to 50% probability contours of atomic displacement.

chemistry was assigned on the basis of the revised structure (Norin, 1962; Cox, Koch, Whalley, Hursthouse & Rogers, 1967; Kulkarni, Eisenbraun & Marsh, 1968) of the precursor (−)-methyl-isopulegone.

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Structure of 3-(4-Bromophenyl)-5-n-butylglutaramic Acid

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Abstract. 3-(4-Bromophenyl)-5-(n-butylamino)-5-oxopentanoic acid, $C_{15}H_{20}BrNO_3$, $M_r = 342.2$, monoclinic, $P2_1/c$, $a = 16.000 (5)$, $b = 8.363 (1)$, $c = 12.713 (9)$ Å, $\beta = 107.55 (5)°$, $V = 1622 (1)$ Å^3, $Z = 4$, $D_x = 1.401$ Mg m^{-3}, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 3.527$ mm^{-1}, $F(000) = 704$, $T = 293$ K. Of the 3065 unique reflections, 1000 were observed with $F_o > 4\sigma(F_o)$ giving $R = 0.066$. The bond distance $N(1)—C(11) = 1.317 (14)$ Å and $C(11)—O(3) = 1.259 (14)$ Å. The H atoms of the carboxyl group and of the amido linkage are located; they participate in the intermolecular hydrogen-bonding scheme, through hydrogen bonds $N(1)—H(N1)\cdots O(1)$, $N\cdots O = 2.968 (16)$ Å, and $O(2)—H(O2)\cdots O(3)$, $O\cdots O = 2.570 (11)$ Å.

Experimental. Colourless crystals from dilute ethanol; crystal of approximate dimensions 0·25 × 0·04 × 0·05 mm; Cu $K\alpha$ radiation ($\lambda = 1.54184$ Å) was used with a graphite-crystal monochromator on a Nonius CAD-4 single-crystal diffractometer. Unit-cell dimensions from the angular settings of 25 reflections with $18 < \theta < 37°$. The space group was determined from the systematic extinctions and the structure determination. The intensity data of 6219 reflections were measured (half a sphere up to $\theta = 58°$), using the $\omega-2\theta$ scan technique, with a scan angle of 1·5° and a variable scan rate with a maximum scan time of 20 s per reflection. The maximum indices (*hkl*) were 19, 10 and 15, respectively. The intensity of the primary beam was checked throughout the data collection by monitoring three standard reflections every 30 min. The final drift-correction factors were between 0·97 and 1·03. Profile analysis (Lehmann & Larsen, 1974; Grant & Gabe, 1978) was performed on all reflections. Lorentz and polarization corrections were applied and the data were reduced to F_o values. Symmetry-equivalent reflections were averaged, $R_{int} = \Sigma(|F_o - \langle F_o \rangle|)\Sigma F_o = 0.129$ for all reflections and 0·026 for the observed reflections only, resulting in 3065 unique reflections of which 1000 were observed with $F_o > 4\sigma(F_o)$.

A *p*-bromotolyl skeleton (eight atoms) was input to the vector-search rotation-function program *ORIENT* (Beurskens, Beurskens, Strumpel &