Table 2. *Bond lengths* (Å) *and angles* (°) *for the non*-H *atoms of* (1)

1	2	3	1—2	1—2—3
C2	C1	C10	1·548 (2)	112·86 (14)
C2	C1	C11		110·87 (14)
C10	C1	C11	1·530 (2)	109·68 (13)
C10	C1	O24		110·1 (2)
C11	C1	O24	1·559 (2)	104·51 (12)
O24	C1	C2	1·444 (2)	108·49 (13)
C3	C2	C1	1·519 (3)	116·97 (14)
C4	C3	O14	1·524 (3)	119·1 (2)
C4	C3	C2		120·5 (2)
O14	C3	C2	1·223 (2)	119·7 (2)
C5	C4	C15	1·528 (2)	108·6 (2)
C5	C4	C3		117·4 (2)
C15	C4	C3	1·536 (3)	107·5 (2)
C6	C5	C4	1·535 (2)	114·72 (14)
C7	C6	C5	1·516 (3)	110·83 (15)
C7	C6	S17		121·53 (12)
S17	C6	C5	1·835 (2)	103·31 (11)
C8	C7	C11	1·343 (2)	117·9 (2)
C8	C7	C6		117·8 (2)
C11	C7	C6	1·540 (3)	123·54 (13)
C9	C8	C16	1·511 (3)	111·1 (2)
C9	C8	C7		122·1 (2)
C16	C8	C7	1·509 (3)	126·6 (2)
C10	C9	C8	1·534 (3)	116·1 (2)
C1	C10	C9		114·5 (2)
C12	C11	C13	1·543 (2)	105·42 (14)
C12	C11	C1		110·9 (2)
C12	C11	C7		108·41 (14)
C13	C11	C1	1·538 (2)	109·99 (14)
C13	C11	C7		117·0 (2)
C1	C11	C7		105·16 (12)
C18	S17	C6	1·760 (2)	108·73 (8)
C19	C18	C23	1·395 (3)	118·9 (2)
C19	C18	S17		114·99 (13)
C23	C18	S17	1·381 (3)	126·11 (14)
C20	C19	C18	1·375 (3)	120·6 (2)
C21	C20	C19	1·376 (4)	120·4 (2)
C22	C21	C20	1·368 (4)	119·2 (2)
C23	C22	C21	1·385 (3)	121·4 (2)
C18	C23	C22		119·6 (2)
C25	O24	C1	1·418 (3)	118·56 (13)
C26	C25	O24	1·500 (3)	108·96 (15)
C27	C26	C31	1·379 (3)	117·7 (2)
C27	C26	C25		120·0 (2)
C31	C26	C25	1·375 (3)	122·3 (2)
C28	C27	C26	1·388 (4)	121·0 (2)
C29	C28	C27	1·366 (4)	119·9 (3)
C30	C29	C28	1·353 (5)	119·9 (3)
C31	C30	C29	1·386 (4)	120·3 (3)
C26	C31	C30		121·2 (2)

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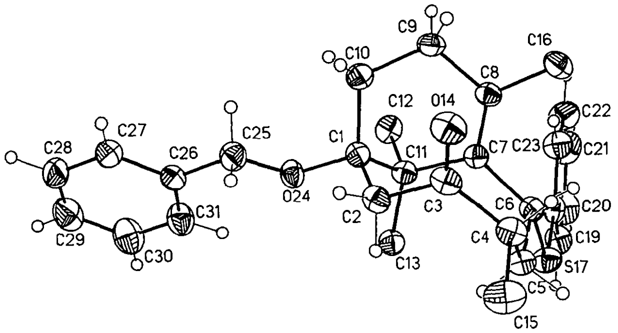

Fig. 1. View of (1) with the atomic labeling scheme showing the boat-chair conformation of the cyclooctane portion of the [5.3.1] ring system. The nearly parallel orientation of the carbonyl group and the alkene moiety can also be seen. The methyl-group H atoms were removed for clarity. Atom label for C18 has been omitted. Ellipsoids are scaled to the 30% probability level. H atoms are of arbitrary size.

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Structure of (±)-(1*S**,3*S**)-3-Hydroxy-8,11,11-trimethyl-9-oxobicyclo[5.3.1]undec-7-en-1-yl Benzoate

By V. M. Lynch, J. R. Fishpaugh, S. F. Martin and B. E. Davis

Department of Chemistry, University of Texas at Austin, Austin, TX 78712, *USA*

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Abstract. $C_{21}H_{26}O_4$, $M_r = 342{\cdot}43$, monoclinic, $P2_1/n$, $a = 7{\cdot}211\ (2)$, $b = 19{\cdot}521\ (6)$, $c = 12{\cdot}674\ (3)$ Å, $\beta = 95{\cdot}04\ (2)°$, $V = 1777{\cdot}2\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}28$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}8152$ cm^{-1}, $F(000) = 736$, $T = 163$ K, $R = 0{\cdot}0632$ for 3754 reflections $[F_o \geq 4\sigma(F_o)]$. The molecules exist

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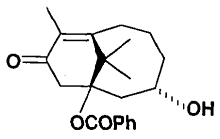
as hydrogen-bonded dimers [O14—H14···O16 (related by $-x$, $1-y$, $1-z$), O14···O16 2·775 (2), H14···O16 1·93 (2) Å, O14—H14···O16 175 (2)°] which stack in columns along **b**. Distortion at the bridgehead double bond is observed. Deviations from ideal values for the torsion angles around the double bond, C7—C8, are as large as 17·8 (2)°. The dihedral angle between planes through C6—C7—C8—C11 and C7—C8—C9— C15, the planar portions of the alkene moiety, is 13·4 (9)°. The C_{sp^2}—C_{sp^3} bond lengths involving C7 are asymmetric with C7—C6 1·501 (2) and C7—C11 1·538 (2) Å. The bond length C7—C8 is elongated [1·352 (3) Å] for a C—C double bond. The enone system is also non-planar [max. deviation $-0{\cdot}404$ (2) Å for C10].

Experimental. The ketoalcohol (1), which possesses the bicyclo[5.3.1]undecane ring system that is a key subunit found in the Taxane diterpenes (Martin, White & Wagner, 1982), was prepared by the chemoselective hydride ($NaBH_4$–$CeCl_3$) reduction of the relevant enedione (Martin, White, Wagner, Guinn, Tanaka, Assercq, Gluchowski, Austin & Dantanarayana, 1989). Crystals of (1) were obtained by slow evaporation from an ethyl acetate–hexane solution. The data crystal was a colorless needle approximately 0·5 mm long. The data were collected on a Syntex $P2_1$ diffractometer using a graphite monochromator and equipped with a Syntex LT-1 low-temperature delivery system (163 K). Lattice parameters were obtained from the least-squares refinement of 15 reflections with $24{\cdot}0 < 2\theta < 34{\cdot}9°$. The data were collected using the ω-scan technique (10 923 reflections, of which 5170 were unique, $R_{\rm int} = 0{\cdot}0329$), with a 2θ range from 4·0–60·0°, and a 1·0° ω scan at 5–10° min^{-1}. $h = 0 \rightarrow 10$, $k = -27 \rightarrow 27$, $l = -17 \rightarrow 17$. Four reflections (060, $\bar{1}\bar{1}\bar{1}$, 200, 006) were remeasured every 196 reflections to monitor instrument and crystal stability (maximum correction on I was <2%). The data were also corrected for Lp effects but not for absorption. Decay correction was applied according to Henslee & Davis (1975), while the data reduction was performed as described by Riley & Davis (1976). Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (1416 reflections). The structure was solved by direct methods (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by full-matrix least squares (Sheldrick, 1976). In all, 330 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = \{0{\cdot}5kI^{-1/2} \times [(\sigma I)^2 + (0{\cdot}02I)^2]^{1/2}\}$. The intensity, I, is given by



(1)

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$U_{\rm eq} = (1/3)\textstyle\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å^2)
C1	0·0662 (2)	0·37956 (8)	0·32601 (12)	0·0164 (5)
C2	–0·1067 (2)	0·34693 (9)	0·36805 (14)	0·0196 (5)
C3	–0·1219 (3)	0·32260 (10)	0·48321 (15)	0·0257 (6)
C4	–0·0334 (3)	0·25442 (10)	0·5220 (2)	0·0296 (6)
C5	0·1608 (3)	0·25314 (11)	0·5809 (2)	0·0308 (7)
C6	0·3259 (3)	0·26494 (10)	0·5136 (2)	0·0277 (6)
C7	0·3020 (2)	0·33259 (9)	0·45776 (13)	0·0216 (5)
C8	0·3215 (2)	0·39165 (9)	0·51337 (13)	0·0220 (5)
C9	0·2462 (2)	0·45502 (9)	0·46563 (13)	0·0206 (5)
C10	0·1126 (3)	0·45146 (9)	0·36766 (14)	0·0195 (5)
C11	0·2423 (2)	0·33440 (9)	0·33830 (13)	0·0180 (5)
C12	0·2032 (3)	0·26404 (10)	0·2875 (2)	0·0255 (6)
C13	0·4022 (3)	0·36617 (11)	0·2814 (2)	0·0260 (6)
O14	–0·0752 (2)	0·37429 (7)	0·56067 (10)	0·0291 (5)
C15	0·4002 (4)	0·39782 (13)	0·6264 (2)	0·0329 (7)
O16	0·2790 (2)	0·51110 (6)	0·50791 (10)	0·0284 (4)
O17	0·0243 (2)	0·38443 (6)	0·20953 (9)	0·0207 (4)
C18	–0·1127 (3)	0·42479 (9)	0·16516 (14)	0·0228 (5)
O19	–0·2159 (2)	0·45927 (7)	0·21243 (10)	0·0294 (4)
C20	–0·1213 (3)	0·42043 (9)	0·04733 (14)	0·0271 (6)
C21	0·0295 (4)	0·40064 (10)	–0·0052 (2)	0·0343 (7)
C22	0·0110 (5)	0·39691 (12)	–0·1159 (2)	0·0479 (9)
C23	–0·1555 (5)	0·41165 (12)	–0·1716 (2)	0·0576 (11)
C24	–0·3046 (5)	0·43192 (12)	–0·1196 (2)	0·0571 (10)
C25	–0·2887 (4)	0·43702 (11)	–0·0100 (2)	0·0405 (8)

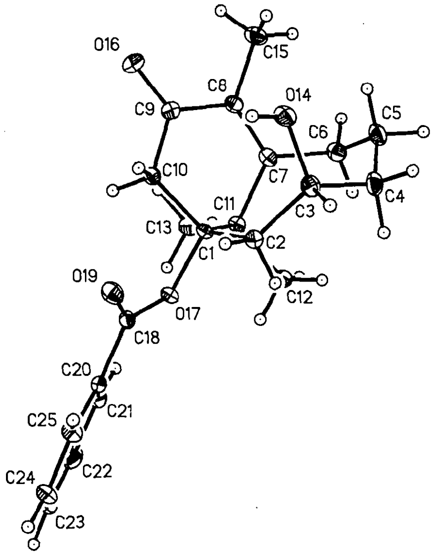


Fig. 1. View of (1) with the atom labeling scheme, showing the crown conformation of the cyclooctane portion of the [5.3.1] ring system. Ellipsoids are scaled to the 50% probability level. H atoms are of arbitrary size.

Table 2. *Bond lengths* (Å) *and angles* (°) *for the non*-H *atoms of* (1)

1	2	3	1—2	1—2—3
C2	C1	C10	1·537 (3)	114·93 (14)
C2	C1	C11		114·46 (14)
C10	C1	C11	1·526 (2)	109·68 (14)
C10	C1	O17		107·74 (13)
C11	C1	O17	1·543 (2)	103·36 (13)
O17	C1	C2	1·484 (2)	105·72 (13)
C3	C2	C1	1·548 (3)	125·52 (15)
C4	C3	O14	1·538 (3)	109·02 (15)
C4	C3	C2		120·3 (2)
O14	C3	C2	1·427 (2)	113·6 (2)
C5	C4	C3	1·528 (3)	120·5 (2)
C6	C5	C4	1·541 (3)	116·5 (2)
C7	C6	C5	1·501 (3)	109·3 (2)
C8	C7	C11	1·352 (3)	120·1 (2)
C8	C7	C6		120·1 (2)
C11	C7	C6	1·538 (2)	119·66 (15)
C9	C8	C15	1·461 (2)	115·1 (2)
C9	C8	C7		119·2 (2)
C15	C8	C7	1·499 (3)	125·5 (2)
C10	C9	O16	1·505 (2)	119·0 (2)
C10	C9	C8		119·36 (15)
O16	C9	C8	1·233 (2)	121·5 (2)
C1	C10	C9		115·66 (14)
C12	C11	C13	1·533 (3)	106·3 (2)
C12	C11	C1		110·50 (14)
C12	C11	C7		114·75 (14)
C13	C11	C1	1·543 (3)	111·45 (14)
C13	C11	C7		108·25 (14)
C1	C11	C7		105·63 (13)
C18	O17	C1	1·347 (2)	122·04 (13)
O19	C18	C20	1·202 (2)	123·9 (2)
O19	C18	O17		125·7 (2)
C20	C18	O17	1·492 (3)	110·4 (2)
C21	C20	C25	1·379 (3)	119·9 (2)
C21	C20	C18		122·3 (2)
C25	C20	C18	1·391 (3)	117·8 (2)
C22	C21	C20	1·399 (3)	119·3 (2)
C23	C22	C21	1·369 (5)	120·6 (3)
C24	C23	C22	1·368 (5)	120·2 (2)
C25	C24	C23	1·387 (3)	120·3 (3)
C20	C25	C24		119·8 (3)

$(I_{\text{peak}} - I_{\text{background}}) \times$ (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\text{peak}} + I_{\text{background}})^{1/2} \times$ (scan rate)]. The final $R = 0{\cdot}0632$ for 3754 reflections, with $wR = 0{\cdot}0445$ ($R_{\text{all}} = 0{\cdot}0957$, $wR_{\text{all}} = 0{\cdot}0474$) and a goodness of fit = 1·937. The maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were $-0{\cdot}31$ and 0·40 e Å^{-3}, respectively. 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 least-squares-planes program was supplied by Cordes (1983). 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. Distortions in ring systems induced by the presence of a bridgehead C as the terminus of a double bond are discussed by Wiseman (1967).

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* Tables of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving H atoms, torsion angles, selected least-squares-planes data, structure factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52701 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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