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Structure of an Iridoid-like Photolysis Product

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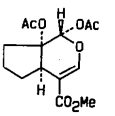
Abstract. Methyl 1α,7aα-diacetoxy-1,4aα,5,6,7,7a-hexahydrocyclopenta[*c*]pyran-4-carboxylate, $C_{14}H_{18}O_7$, $M_r = 298{\cdot}3$, triclinic, $P\bar{1}$, $a = 5{\cdot}615\ (2)$, $b = 8{\cdot}013\ (1)$, $c = 16{\cdot}673\ (4)$ Å, $\alpha = 89{\cdot}98\ (2)$, $\beta = 87{\cdot}90\ (2)$, $\gamma = 85{\cdot}07\ (2)°$, $V = 747$ Å^3, $Z = 2$, $D_x = 1{\cdot}326$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 1{\cdot}0$ cm^{-1}, $F(000) = 316$, $T = 298$ K, $R = 0{\cdot}050$ for 2069 observed reflections. The title compound is the product of a photolysis reaction. From spectroscopic data it was not clear whether the ring junction was *cis* (as established here) or *trans*. The six-membered ring adopts a half-chair conformation, whereas the five-membered ring does not correspond closely to any standard conformation. The acetyl groups lie equatorial [C(7a)] and axial [C(1)] respectively.

Experimental. (I): crystal $0{\cdot}3 \times 0{\cdot}3 \times 0{\cdot}4$ mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and and speed (Clegg, 1981). 2628 unique reflections, $2\theta_{max}$ 50°, $\pm h \pm k + l$, three check reflections with no intensity change. 2069 reflections with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*, Sheldrick, 1978). Index ranges $|h| \le 6$, $|k| \le 9$, $l \le 19$. Cell constants refined from $\pm 2\theta$ values of 20 reflections in the range 20–25°. Absorption and extinction corrections unnecessary.



(I)

Structure solution by multisolution direct methods. Refinement on F to $R = 0{\cdot}050$, $wR = 0{\cdot}069$; all non-H atoms anisotropic, H atoms included using a riding model [C–H 0·96 Å, $U(H) = 1{\cdot}2U_{eq}(C)$], 199 parameters, $S = 1{\cdot}89$, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}00064F^2$ which gave a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0{\cdot}067$, max. and min. height in final $\Delta\rho$ map 0·18 and −0·17 e Å^{-3} respectively. Atomic scattering factors from *SHELXTL*.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows the atom numbering.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43129 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Engalnd.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

	x	*y*	*z*	U_{eq}*
C(1)	6642 (4)	6525 (3)	8154 (1)	56 (1)
O(2)	8142 (3)	7810 (2)	7949 (1)	66 (1)
C(3)	7293 (4)	8910 (3)	7391 (2)	63 (1)
C(4)	5519 (4)	8695 (2)	6910 (1)	53 (1)
C(4a)	4328 (3)	7083 (2)	6891 (1)	49 (1)
C(5)	4555 (4)	6281 (3)	6059 (1)	62 (1)
C(6)	7133 (5)	5519 (3)	6001 (1)	73 (1)
C(7)	7697 (4)	4930 (3)	6843 (1)	53 (1)
C(7a)	5717 (3)	5770 (3)	7403 (1)	48 (1)
O(8)	3932 (2)	4614 (2)	7634 (1)	53 (1)
C(9)	4578 (4)	3223 (3)	8056 (1)	55 (1)
C(10)	2503 (4)	2242 (3)	8240 (2)	71 (1)
O(11)	6586 (3)	2850 (2)	8263 (1)	68 (1)
C(12)	4723 (4)	10123 (3)	6407 (1)	59 (1)
O(13)	2504 (3)	10031 (2)	6153 (1)	66 (1)
C(14)	1571 (5)	11392 (3)	5665 (2)	82 (1)
O(15)	5864 (3)	11281 (2)	6233 (1)	94 (1)
O(16)	4607 (3)	7231 (2)	8605 (1)	64 (1)
C(17)	4773 (5)	7311 (4)	9412 (1)	76 (1)
C(18)	2479 (5)	8060 (4)	9790 (2)	100 (1)
O(19)	6554 (4)	6870 (4)	9742 (1)	118 (1)

* U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

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Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–O(2)	1·419 (3)	C(1)–C(7a)	1·518 (3)
C(1)–O(16)	1·421 (2)	O(2)–C(3)	1·352 (3)
C(3)–C(4)	1·324 (3)	C(4)–C(4a)	1·505 (3)
C(4)–C(12)	1·467 (3)	C(4a)–C(5)	1·526 (3)
C(4a)–C(7a)	1·533 (3)	C(5)–C(6)	1·522 (4)
C(6)–C(7)	1·517 (3)	C(7)–C(7a)	1·535 (3)
C(7a)–O(8)	1·464 (2)	O(8)–C(9)	1·348 (2)
C(9)–C(10)	1·484 (3)	C(9)–O(11)	1·205 (3)
C(12)–O(13)	1·339 (3)	C(12)–O(15)	1·202 (3)
O(13)–C(14)	1·435 (3)	O(16)–C(17)	1·353 (3)
C(17)–C(18)	1·492 (4)	C(17)–O(19)	1·186 (3)
O(2)–C(1)–C(7a)	110·5 (2)	O(2)–C(1)–O(16)	109·1 (2)
C(7a)–C(1)–O(16)	106·7 (2)	C(1)–O(2)–C(3)	115·4 (2)
O(2)–C(3)–C(4)	125·8 (2)	C(3)–C(4)–C(4a)	122·0 (2)
C(3)–C(4)–C(12)	116·7 (2)	C(4a)–C(4)–C(12)	121·3 (2)
C(4)–C(4a)–C(5)	111·6 (2)	C(4)–C(4a)–C(7a)	109·3 (2)
C(5)–C(4a)–C(7a)	102·3 (2)	C(4a)–C(5)–C(6)	103·7 (2)
C(5)–C(6)–C(7)	105·3 (2)	C(6)–C(7)–C(7a)	106·4 (2)
C(1)–C(7a)–C(4a)	112·3 (2)	C(1)–C(7a)–C(7)	113·7 (2)
C(4a)–C(7a)–C(7)	105·8 (2)	C(1)–C(7a)–O(8)	109·2 (2)
C(4a)–C(7a)–O(8)	103·6 (1)	C(7)–C(7a)–O(8)	111·7 (2)
C(7a)–O(8)–C(9)	120·0 (1)	O(8)–C(9)–C(10)	111·4 (2)
O(8)–C(9)–O(11)	123·7 (2)	C(10)–C(9)–O(11)	124·8 (2)
C(4)–C(12)–O(13)	112·6 (2)	C(4)–C(12)–O(15)	125·8 (2)
O(13)–C(12)–O(15)	121·7 (2)	C(12)–O(13)–C(14)	116·2 (2)
C(1)–O(16)–C(17)	117·5 (2)	O(16)–C(17)–C(18)	110·4 (2)
O(16)–C(17)–O(19)	122·6 (2)	C(18)–C(17)–O(19)	127·0 (2)

Related literature. For the preparation of the compound see Zimmermann (1980). For the structure of the iridoid compound loganin see Jones, Sheldrick, Glüsenkamp & Tietze (1980).

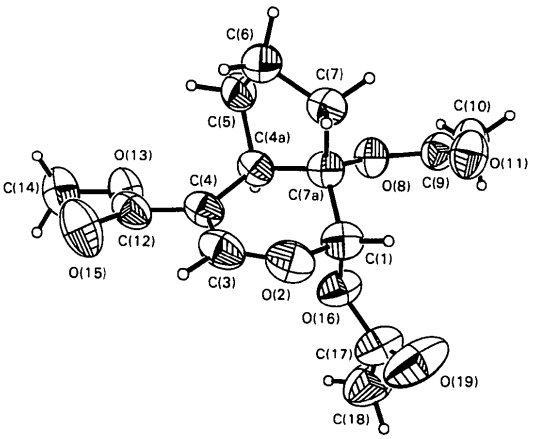

Fig. 1. The asymmetric unit of the title compound, showing the atom-numbering scheme.

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Structure of a Cycloheptenone Derivative

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Abstract. Racemic (1*R*,7*S*,9*R*)-2-methyl-6-oxobicyclo-[5.3.0]dec-2-en-9-yl methanesulfonate, $C_{12}H_{18}O_4S$, $M_r = 258·3$, orthorhombic, *Pbca*, $a = 8·745$ (2), $b = 33·451$ (5), $c = 8·845$ (2) Å, $V = 2588$ Å^3, $Z = 8$, $D_x = 1·326$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·24$ mm^{-1}, $F(000) = 1104$, $T = 298$ K, $R = 0·049$ for 1387 unique observed reflections. The seven-membered ring adopts a boat and the five-membered ring a half-chair conformation; the ring junction is *cis* and the mesyloxy group axial. It had not proved possible to assign the conformation unambiguously from NMR data because of the difficulty in applying correlations to relatively flexible five- and seven-membered rings.

Experimental. (I): crystal 0·3 × 0·3 × 0·4 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 2064 reflections, $2\theta_{max}$ 45°, $+h+k+l$ and some $-h+k+l$, three check reflections with no intensity change. 1680 unique

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