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Structure of a Possible Precursor to Harringtonolide

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Abstract. Methyl (1α,3α,$3a\alpha$,$10a\alpha$,$10b\alpha$)-1,2,3,3a,-6,9,10,10bα-octahydro-3-hydroxy-7-methoxy-1,5-dimethyl-4-oxocyclohept[*bc*]acenaphthalene-10a(4*H*)-carboxylate, $C_{21}H_{26}O_5$, $M_r = 358{\cdot}43$, triclinic, $P\bar{1}$, $a = 16{\cdot}215\,(2)$, $b = 11{\cdot}985\,(1)$, $c = 10{\cdot}480\,(2)$ Å, $\alpha = 69{\cdot}40\,(1)$, $\beta = 75{\cdot}28\,(1)$, $\gamma = 76{\cdot}11\,(2)°$, $V = 1818$ Å^3, $Z = 4$, $D_x = 1{\cdot}31$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}667$ mm^{-1}, $F(000) = 768$, $T = 298$ K, $R = 0{\cdot}040$ for 4553 observed reflections. The structure contains two independent molecules in the asymmetric unit which have essentially identical conformations. The cycloheptatriene ring adopts the expected boat conformation with C(6) at the 'prow'. The methoxy substituent on the cycloheptatriene ring is eclipsed with the C(7)—C(8) bond [C(11)—O(1)—C(7)—C(8) $-5{\cdot}8\,(3)°$]. This eclipsing results in close intramolecular contacts between the hydrogens attached to C(11) and the hydrogen on C(8). The structure is held together by weak intermolecular hydrogen bonds: O(2)···O(4) 2·890 (2), H(16)···O(4) 2·03 (4) Å, O(2)—H(16)···O(7) 153 (2)°; O(7)···O(6) 3·005 (2), H(26)···O(6) 2·15 (4), O(7)—H(26)···O(6) 151 (2)°. There also exist weak intramolecular hydrogen bonds within the β-hydroxy ketone: O(2)···O(4) 3·091 (2), H(16)···O(4) 2·52 (3) Å, O(2)—H(16)···O(4) 120 (3)°; O(7)···O(6) 3·089 (2), H(26)···O(6) 2·45 (3) Å, O(7)—H(26)···O(6) 125 (2)°.

Experimental. Colourless crystal $0{\cdot}28 \times 0{\cdot}20 \times 0{\cdot}24$ mm. Philips PW 1100/20 diffractometer, graphite monochromator. Lattice parameters from least-squares analysis of setting angles of 25 reflections $50 < 2\theta < 60°$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å. θ–2θ scans of width $(1{\cdot}0 + 0{\cdot}142\tan\theta)°$ in θ and rate $8°\ \mathrm{min}^{-1}$ in θ with 5 s backgrounds on each side of every scan. $2\theta_{\max} = 120°$ with $-18 \le h \le 18$, $-13 \le k \le 13$, $0 \le l \le 12$, 5170 unique reflections, 4553 with $I > 3\sigma(I)$ regarded as observed. Three check reflections measured every 120 min showed no significant decrease in intensity during data collection. Data corrected for absorption (maximum/minimum transmission 0·91/0·83). Structure solution by direct methods (*SHELXS*86, Sheldrick, 1985), ΔF synthesis and full-matrix least-squares refinement. Non-H atoms refined with anisotropic displacement factors, H atoms refined with isotropic thermal parameters. Refinement on F, 678 parameters, to $R = 0{\cdot}040$, $wR = 0{\cdot}058$, $S = 1{\cdot}94$, weighting scheme $w = [\sigma^2(F) + (0{\cdot}0005)F^2]^{-1}$, max. $\Delta/\sigma = 0{\cdot}03$, max. and min. heights in final $\Delta\rho$ map 0·2 and $-0{\cdot}2$ e Å^{-3}. Atomic

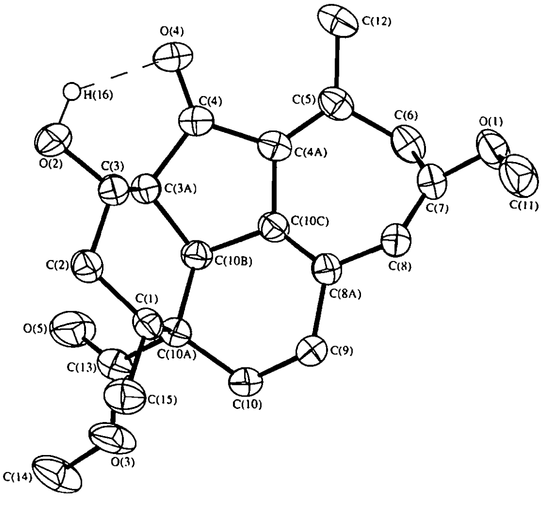

Fig. 1. Thermal-ellipsoid diagram of one of the independent molecules of (1) showing labelling of non-H atoms. Ellipsoids show 50% probability levels. The hydroxyl hydrogen is drawn as a small circle.

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Table 1. *Non*-H *coordinates and isotropic thermal parameters for methyl* (1*α*,3*α*,3*aα*,10*aα*,10*bα*)-1,2,3,-3*a*,6,9,10,10*bα-octahydro*-3-*hydroxy*-7-*methoxy*-1,5-*dimethyl*-4-*oxocyclohept*[*bc*]*acenaphthalene*-10*a*(4*H*)-*carboxylate* (1)

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

	x	y	z	$U_{\rm eq}$(Å^2)
O(1)	0·94133 (9)	0·93942 (13)	0·17022 (17)	0·053 (2)
O(2)	0·45665 (10)	0·82307 (15)	0·46751 (16)	0·058 (2)
O(3)	0·54599 (10)	0·67451 (14)	−0·03466 (18)	0·059 (2)
O(4)	0·54631 (9)	1·04849 (13)	0·34353 (15)	0·051 (1)
O(5)	0·46821 (10)	0·82544 (16)	0·04012 (20)	0·069 (2)
C(1)	0·60202 (12)	0·64331 (16)	0·24453 (19)	0·035 (2)
C(2)	0·52409 (13)	0·68132 (18)	0·34602 (22)	0·039 (2)
C(3)	0·53177 (12)	0·79347 (17)	0·37281 (19)	0·037 (2)
C(3*A*)	0·54370 (11)	0·90063 (16)	0·23785 (19)	0·033 (2)
C(4)	0·58481 (12)	0·98762 (16)	0·26750 (19)	0·036 (2)
C(4*A*)	0·67623 (12)	0·98096 (15)	0·19500 (19)	0·034 (2)
C(5)	0·73238 (12)	1·05341 (16)	0·18345 (20)	0·038 (2)
C(6)	0·81555 (14)	1·04777 (18)	0·07937 (24)	0·044 (2)
C(7)	0·87084 (12)	0·92799 (17)	0·13181 (20)	0·040 (2)
C(8)	0·85081 (12)	0·82435 (18)	0·13585 (21)	0·039 (2)
C(8*A*)	0·77112 (12)	0·81470 (16)	0·10550 (19)	0·036 (2)
C(9)	0·77492 (13)	0·70830 (20)	0·05709 (25)	0·045 (2)
C(10)	0·69486 (13)	0·71410 (20)	0·00289 (22)	0·042 (2)
C(10*A*)	0·60961 (11)	0·74220 (16)	0·10051 (18)	0·033 (2)
C(10*B*)	0·60793 (11)	0·86665 (16)	0·11562 (19)	0·032 (2)
C(10*C*)	0·69411 (11)	0·88604 (15)	0·12994 (18)	0·032 (2)
C(11)	0·99613 (17)	0·83173 (24)	0·23208 (36)	0·062 (3)
C(12)	0·71878 (18)	1·13758 (24)	0·26593 (31)	0·057 (3)
C(13)	0·53318 (13)	0·75392 (17)	0·03430 (20)	0·038 (2)
C(14)	0·47824 (22)	0·67990 (28)	−0·10375 (27)	0·065 (3)
C(15)	0·60164 (17)	0·51717 (19)	0·24163 (27)	0·051 (2)
O(6)	0·98483 (9)	0·43145 (12)	0·15683 (15)	0·051 (1)
O(7)	1·10280 (11)	0·62613 (14)	0·02750 (16)	0·057 (2)
O(8)	1·20928 (8)	−0·07143 (11)	0·30456 (16)	0·049 (1)
O(9)	1·21822 (10)	0·56328 (15)	0·53974 (18)	0·061 (2)
O(10)	1·09459 (10)	0·61437 (16)	0·46355 (20)	0·068 (2)
C(1′)	1·26038 (12)	0·49359 (17)	0·26787 (20)	0·038 (2)
C(2′)	1·21072 (13)	0·58867 (18)	0·16154 (22)	0·041 (2)
C(3′)	1·14515 (12)	0·53583 (17)	0·13098 (20)	0·038 (2)
C(3*A*′)	1·07933 (11)	0·48493 (16)	0·26252 (19)	0·034 (2)
C(4′)	1·03917 (11)	0·39837 (17)	0·23068 (19)	0·036 (2)
C(4*A*′)	1·07945 (11)	0·27320 (16)	0·29879 (18)	0·033 (2)
C(5′)	1·05729 (11)	0·16952 (17)	0·30274 (20)	0·037 (2)
C(6′)	1·09369 (12)	0·05391 (18)	0·40240 (23)	0·042 (2)
C(7′)	1·18883 (12)	0·02581 (16)	0·35183 (20)	0·038 (2)
C(8′)	1·24364 (12)	0·09047 (16)	0·35774 (21)	0·038 (2)
C(8*A*′)	1·21670 (11)	0·20096 (16)	0·39606 (19)	0·035 (2)
C(9′)	1·28063 (14)	0·23120 (18)	0·45598 (24)	0·045 (2)
C(20)	1·24545 (14)	0·33886 (19)	0·51015 (22)	0·044 (2)
C(20*A*)	1·19918 (11)	0·44773 (16)	0·40888 (19)	0·034 (2)
C(20*B*)	1·12203 (11)	0·40624 (16)	0·38785 (19)	0·033 (2)
C(20*C*)	1·14323 (11)	0·28116 (15)	0·36911 (18)	0·032 (2)
C(21)	1·29780 (15)	−0·10249 (23)	0·24384 (32)	0·058 (3)
C(22)	1·00056 (17)	0·16073 (23)	0·21620 (29)	0·054 (2)
C(23)	1·16350 (12)	0·55025 (18)	0·47272 (20)	0·040 (2)
C(24)	1·19259 (22)	0·66321 (30)	0·59723 (39)	0·068 (3)
C(25)	1·33920 (16)	0·53698 (27)	0·27855 (30)	0·059 (3)

scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974, Vol. IV). Data reduction and refinement computations performed with *XTAL*2.6 (Hall & Stewart, 1989). Final parameters for the non-H atoms are given in Table 1. Interatomic distances and angles are given in Table 2* and selected contact distances in Table 3. Fig. 1 shows a view of one of the independent molecules of (1) with labelling.

Table 2. *Interatomic distances* (Å) *and angles* (°), *for methyl* (1*α*,3*α*,3*aα*,10*aα*,10*bα*)-1,2,3,3*a*,6,9,10,10*bα*-*octahydro*-3-*hydroxy*-7-*methoxy*-1,5-*dimethyl*-4-*oxo-cyclohept*[*bc*]*acenaphthalene*-10*a*(4*H*)-*carboxylate* (1)

Labels are for molecule 1; the distances and angles for molecule 2 are for the corresponding atoms in molecule 2.

	Molecule 1	Molecule 2
O(1)—C(7)	1·355 (3)	1·360 (3)
O(1)—C(11)	1·424 (3)	1·432 (3)
O(2)—C(3)	1·421 (2)	1.419 (2)
O(3)—C(13)	1·336 (3)	1·329 (3)
O(3)—C(14)	1·440 (5)	1·453 (5)
O(4)—C(4)	1·222 (3)	1·223 (3)
O(5)—C(13)	1·191 (2)	1·199 (2)
C(1)—C(2)	1·522 (3)	1·520 (3)
C(1)—C(10*A*)	1·554 (2)	1·554 (2)
C(1)—C(15)	1·524 (3)	1·533 (4)
C(2)—C(3)	1·505 (3)	1·508 (4)
C(3)—C(3*A*)	1·546 (2)	1·544 (2)
C(3*A*)—C(4)	1·515 (3)	1·513 (3)
C(3*A*)—C(10*B*)	1·542 (3)	1·540 (3)
C(4)—C(4*A*)	1·481 (2)	1·483 (2)
C(4*A*)—C(5)	1·359 (3)	1·358 (3)
C(4*A*)—C(10*C*)	1·459 (3)	1·451 (3)
C(5)—C(6)	1·508 (3)	1·504 (2)
C(5)—C(12)	1·491 (4)	1·489 (4)
C(6)—C(7)	1·498 (3)	1·495 (3)
C(7)—C(8)	1·342 (3)	1·337 (3)
C(8)—C(8*A*)	1·446 (3)	1·454 (3)
C(8*A*)—C(9)	1·513 (4)	1·507 (4)
C(8*A*)—C(10*C*)	1·355 (2)	1·361 (2)
C(9)—C(10)	1·522 (4)	1·520 (3)
C(10)—C(10*A*)	1·540 (3)	1·534 (3)
C(10*A*)—C(10*B*)	1·548 (3)	1·542 (3)
C(10*A*)—C(13)	1·524 (3)	1·527 (3)
C(10*B*)—C(10*C*)	1·522 (3)	1·526 (3)
C(2)—C(1)—C(10*A*)	110·0 (1)	111·2 (1)
C(2)—C(1)—C(15)	110·6 (2)	111·6 (2)
C(10*A*)—C(1)—C(15)	115·0 (2)	114·3 (2)
C(1)—C(2)—C(3)	111·8 (2)	110·7 (2)
O(2)—C(3)—C(2)	108·3 (2)	108·8 (2)
O(2)—C(3)—C(3*A*)	110·9 (1)	111·1 (1)
C(2)—C(3)—C(3*A*)	112·3 (2)	112·0 (2)
C(3)—C(3*A*)—C(4)	106·9 (2)	106·4 (2)
C(3)—C(3*A*)—C(10*B*)	114·3 (1)	113·1 (2)
C(4)—C(3*A*)—C(10*B*)	104·4 (1)	104·4 (1)
O(4)—C(4)—C(3*A*)	123·1 (2)	123·3 (2)
O(4)—C(4)—C(4*A*)	128·0 (2)	128·3 (2)
C(3*A*)—C(4)—C(4*A*)	108·9 (2)	108·4 (2)
C(4)—C(4*A*)—C(5)	127·1 (2)	126·6 (2)
C(4)—C(4*A*)—C(10*C*)	107·2 (2)	107·5 (2)
C(5)—C(4*A*)—C(10*C*)	125·7 (2)	125·8 (2)
C(4*A*)—C(5)—C(6)	117·4 (2)	117·1 (2)
C(4*A*)—C(5)—C(12)	125·4 (2)	125·5 (2)
C(6)—C(5)—C(12)	117·2 (2)	117·4 (2)
C(5)—C(6)—C(7)	108·2 (1)	108·9 (1)
O(1)—C(7)—C(6)	112·0 (2)	111·5 (2)
O(1)—C(7)—C(8)	126·1 (2)	127·0 (2)
C(6)—C(7)—C(8)	121·9 (2)	121·5 (2)
C(7)—C(8)—C(8*A*)	124·3 (2)	124·0 (2)
C(8)—C(8*A*)—C(9)	115·7 (2)	115·7 (2)
C(8)—C(8*A*)—C(10*C*)	124·6 (2)	124·3 (2)
C(9)—C(8*A*)—C(10*C*)	119·4 (2)	119·7 (2)
C(8*A*)—C(9)—C(10)	113·5 (2)	113·7 (2)
C(9)—C(10)—C(10*A*)	113·3 (2)	113·3 (2)
C(1)—C(10*A*)—C(10)	111·8 (1)	111·8 (1)
C(1)—C(10*A*)—C(10*B*)	110·7 (2)	110·6 (2)
C(1)—C(10*A*)—C(13)	109·8 (2)	109·6 (2)
C(10)—C(10*A*)—C(10*B*)	107·0 (2)	106·8 (2)
C(10)—C(10*A*)—C(13)	109·8 (2)	110·0 (2)
C(10*B*)—C(10*A*)—C(13)	107·6 (1)	108·0 (1)
C(3*A*)—C(10*B*)—C(10*A*)	117·6 (1)	117·6 (1)
C(3*A*)—C(10*B*)—C(10*C*)	103·5 (2)	102·8 (2)
C(10*A*)—C(10*B*)—C(10*C*)	114·5 (1)	114·1 (1)
C(4*A*)—C(10*C*)—C(8*A*)	126·7 (2)	127·2 (2)
C(4*A*)—C(10*C*)—C(10*B*)	107·7 (1)	107·9 (1)
C(8*A*)—C(10*C*)—C(10*B*)	125·0 (2)	124·3 (2)

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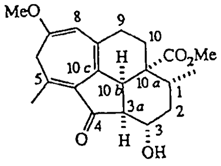
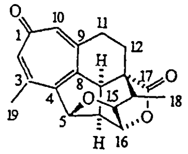
* Fig. 1 showing the second independent molecule, and lists of H coordinates, anisotropic thermal parameters, additional bond distances, angles and dihedral angles, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54231 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The title compound (1) is a possible precursor to the natural product Harringtonolide (2) (Buta, Flippen & Lusby, 1978). (1) is formed *via* an intramolecular aldol reaction of the keto aldehyde (3), giving (1) as a 6:1 mixture with the alternative epimer (4) (Rogers, 1990).



(1)



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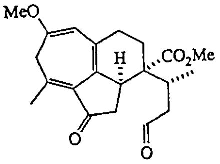

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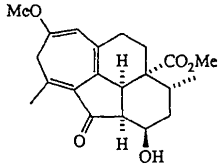

(4)

Table 3. *Selected contact distances* (Å) *for methyl* ($1\alpha,3\alpha,3a\alpha,10a\alpha,10b\alpha$)-1,2,3,3*a*,6,9,10,10*bα*-*octahydro*-3-*hydroxy*-7-*methoxy*-1,5-*dimethyl*-4-*oxocyclohept*[*bc*]*acenaphthalene*-10*a*(4*H*)-*carboxylate* (1)

H(16)···O(4)*	2·03 (4)	H(26)···O(6)†	2·15 (4)
O(2)···O(4)*	2·890 (3)	O(7)···O(6)†	3·005 (3)
H(16)···O(4)	2·52 (3)	H(26)···O(6)	2·45 (3)
O(2)···O(4)	3·091 (2)	O(7)···O(6)	3·089 (2)
H(11*b*)···H(8)	2·26 (4)	H(11*c*)···H(8)	2·37 (4)
H(21*b*)···H(8′)	2·40 (4)	H(21*c*)···H(8′)	2·35 (5)

* Generated from the coordinate list by the operation ($-x$, $2-y$, $-z$).

† Generated from the coordinate list by the operation ($2-x$, $-y$, $-z$).

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Two Polymorphs of 5-Carbamoyl-4-methyl-6-phenyl-1,2,3-triazine

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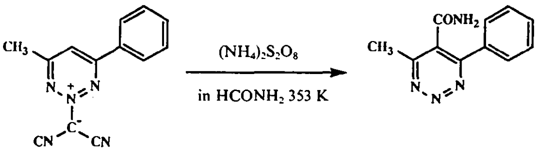
Abstract. $C_{11}H_{10}N_4O$, $M_r = 214{\cdot}23$, crystallizes in two different forms: (I) monoclinic, $P2_1/n$, $a = 14{\cdot}815\,(1)$, $b = 9{\cdot}477\,(1)$, $c = 7{\cdot}869\,(3)$ Å, $\beta = 98{\cdot}85\,(1)°$, $V = 1091{\cdot}7\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}303$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha_1) = 1{\cdot}54050$ Å, $\mu = 0{\cdot}692$ mm^{-1}, $F(000) = 448$, $T = 295$ K, final $R = 0{\cdot}048$ for 1512 reflections; (II) orthorhombic, $P2_12_12_1$, $a = 9{\cdot}299\,(1)$, $b = 14{\cdot}874\,(1)$, $c = 7{\cdot}557\,(4)$ Å, $V = 1045{\cdot}2\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}361$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha_1) = 1{\cdot}54050$ Å, $\mu = 0{\cdot}723$ mm^{-1}, $F(000) = 448$, $T = 295$ K, final $R = 0{\cdot}050$ for 760 reflections. Bond distances and angles are quite similar in the two structures.

Experimental. The title compound was prepared by the radical substitution reaction of 1,2,3-triazinium dicyanomethylide (III) with ammonium persulfate and formamide at 353 K (Minisci, Fontana & Vismara, 1990). The crystals, colorless prism (0·45 × 0·25 × 0·50 mm) (I) and clear needle (0·10 × 0·03 × 0·50 mm) (II) were recrystallized from methanol. Details of data collection and refinement are listed in Table 1. Intensity data were collected with a Rigaku AFC-5 four-circle diffractometer used in the ω–2θ scan mode, ω scan width $(1{\cdot}3 + 0{\cdot}41\tan\theta)°$ and scan speed 16° min^{-1}. Intensity variation was less than 3% for both crystals. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using program package *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985) version of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson,



(III) (I), (II)

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