Both structures solved by direct methods and refined by full-matrix least squares. $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(|F_o|)^2]$ and $g(F_o)^2$ is included to account for random instrument error (g estimated to be 0·00025). For (I), 262 parameters refined: atom coordinates and anisotropic temperature factors for all non-H atoms, H atoms from difference maps, coordinates refined if involved in a hydrogen bond, otherwise idealized (C—H distance set at 0·96 Å, CH angles set to trigonal or tetrahedral values) then allowed to ride on covalently bonded neighbors, thermal parameters constant. $(\Delta/\sigma)_{\max} = 0{\cdot}371$, $R = 0{\cdot}047$, $wR = 0{\cdot}061$, $S = 3{\cdot}1$. For II, 133 parameters refined: same as for (I), $(\Delta/\sigma)_{\max} = 0{\cdot}089$, $R = 0{\cdot}066$, $wR = 0{\cdot}069$, $S = 1{\cdot}7$. Final difference Fourier excursions 0·66 and −0·33 for (I); 0·33 and −0·24 for (II). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atom numbering for Tables 1 and 2, which list atom coordinates for (I) and (II), follows that shown in Figs. 1 and 2.* Bond lengths and angles for (I) and (II) are given in Tables 3 and 4, respectively.

Related literature. *cis*-Thymine glycol shows a density similar to that of molecule (I) (Flippen, 1973).

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* Lists of structure factors, bond lengths, bond angles, H-atom coordinates, H-bond parameters, torsion angles and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44131 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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O-Acetylisomoschatoline

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Abstract. $C_{20}H_{15}NO_5$, $M_r = 349{\cdot}35$, triclinic, $P\bar{1}$, $a = 7{\cdot}451\,(1)$, $b = 10{\cdot}458\,(3)$, $c = 10{\cdot}666\,(4)$ Å, $\alpha = 97{\cdot}55\,(2)$, $\beta = 95{\cdot}86\,(2)$, $\gamma = 100{\cdot}20\,(2)°$, $V = 804{\cdot}2\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}443$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 8{\cdot}26$ cm^{-1}, $F(000) = 364$, $T = 298$ K, $R = 0{\cdot}043$ for 1921 unique reflections. The oxoaporphine isomoschatoline (3-hydroxy-1,2-dimethoxy-7*H*-dibenzo[*de,g*]quinolin-7-one) was isolated from *Cleistopholis patens* and converted into the acetyl derivative to aid in assignment of substituents. The acetyl group is located at the C(3) position in the alkaloid. The alkaloid framework is planar, and the plane of the acetoxy moiety is almost perpendicular [96·5 (3)°] to the alkaloid molecular plane.

Experimental. The sample recrystallized from chloroform, space group *Pbca*, contained disordered molecule of solvent, decomposed during data collection, refined only to $R = 0{\cdot}133$. Good transparent light yellow crystal from ethanol, 0·30 × 0·35 × 0·40 mm; Nicolet $R3m/\mu$ update of $P2_1$ diffractometer; data collected in θ:2θ mode ($3 \le 2\theta \le 120°$, 4·00 to 29·3° min^{-1}), graphite-monochromated Cu $K\alpha$ radiation; lattice parameters from a least-squares refinement of 17 reflections ($24{\cdot}36 \le 2\theta \le 46{\cdot}82°$), angles measured by a centering routine; no systematic absences and statistics consistent with space group $P\bar{1}$, Laue symmetry $\bar{1}$; monitored reflections ($1\bar{1}\bar{2}$ and $2\bar{2}\bar{1}$) showed no significant changes in intensities; 2170 independent reflections measured ($0 \le h \le 9$, $-12 \le k \le 12$, $-12 \le l \le 12$), 1921 with $I \ge 2{\cdot}5\sigma(I)$ considered observed; Lorentz–polarization corrections, ψ-scan empirical absorption correction (transmission factors 0·921 to 0·952); structure solved by direct-methods techniques, block-cascade least-squares refinement with H atoms refined isotropically; final $R = 0{\cdot}0431$, $wR = 0{\cdot}0503$, 295 parameters refined, $S = 1{\cdot}48$, $(\Delta/\sigma)_{\max} = 0{\cdot}23$; largest peaks in the final difference Fourier map of 0·20 and −0·22 e Å^{-3}, $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}0017F_o^{\,2}]^{-1}$. All computer programs supplied by Nicolet Instrument Corporation (1986) for Desktop 30 Microeclipse and Nova 4/C configurations; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Table 1 lists atomic positional

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

	x	y	z	U_{eq}^*
C(1)	7986 (2)	2598 (1)	714 (1)	51 (1)
O(1)	8584 (2)	3435 (1)	−90 (1)	67 (1)
C(2)	7712 (2)	3171 (1)	1955 (1)	53 (1)
O(2)	8120 (2)	4500 (1)	2240 (1)	66 (1)
C(3)	7183 (2)	2384 (2)	2821 (1)	53 (1)
O(3)	6862 (2)	2936 (1)	4021 (1)	64 (1)
C(3*A*)	6887 (2)	1006 (1)	2518 (1)	52 (1)
C(4)	6354 (2)	169 (2)	3392 (2)	64 (1)
C(5)	6096 (3)	−1152 (2)	3023 (2)	74 (1)
N(6)	6276 (2)	−1722 (1)	1859 (2)	69 (1)
C(6*A*)	6802 (2)	−944 (1)	1024 (2)	54 (1)
C(7)	6981 (2)	−1614 (2)	−259 (2)	62 (1)
O(7)	6581 (2)	−2807 (1)	−535 (1)	88 (1)
C(7*A*)	7652 (2)	−778 (2)	−1188 (2)	57 (1)
C(8)	7893 (2)	−1415 (2)	−2384 (2)	73 (1)
C(9)	8531 (3)	−710 (2)	−3296 (2)	78 (1)
C(10)	8988 (3)	635 (2)	−3011 (2)	75 (1)
C(11)	8769 (2)	1283 (2)	−1844 (2)	65 (1)
C(11*A*)	8066 (2)	603 (2)	−898 (1)	52 (1)
C(11*B*)	7764 (2)	1256 (1)	364 (1)	48 (1)
C(11*C*)	7168 (2)	448 (1)	1289 (1)	49 (1)
C(12)	7459 (3)	4357 (2)	−427 (2)	74 (1)
C(13)	6687 (3)	5165 (2)	2642 (2)	81 (1)
C(14)	8351 (3)	3399 (2)	4923 (1)	61 (1)
O(14)	9859 (2)	3280 (1)	4739 (1)	82 (1)
C(15)	7785 (3)	4023 (2)	6105 (2)	85 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

C(1)—O(1)	1·355 (2)	C(1)—C(2)	1·430 (2)
C(1)—C(11*B*)	1·380 (2)	O(1)—C(12)	1·440 (2)
C(2)—O(2)	1·356 (2)	C(2)—C(3)	1·358 (2)
O(2)—C(13)	1·444 (3)	C(3)—O(3)	1·395 (2)
C(3)—C(3*A*)	1·408 (2)	O(3)—C(14)	1·364 (2)
C(3*A*)—C(4)	1·401 (2)	C(3*A*)—C(11*C*)	1·414 (2)
C(4)—C(5)	1·359 (2)	C(5)—N(6)	1·336 (2)
N(6)—C(6*A*)	1·326 (2)	C(6*A*)—C(7)	1·482 (2)
C(6*A*)—C(11*C*)	1·417 (2)	C(7)—O(7)	1·220 (2)
C(7)—C(7*A*)	1·474 (2)	C(7*A*)—C(8)	1·403 (2)
C(7*A*)—C(11*A*)	1·408 (2)	C(8)—C(9)	1·369 (3)
C(9)—C(10)	1·372 (3)	C(10)—C(11)	1·377 (3)
C(11)—C(11*A*)	1·401 (2)	C(11*A*)—C(11*B*)	1·485 (2)
C(11*B*)—C(11*C*)	1·435 (2)	C(14)—O(14)	1·185 (2)
C(14)—C(15)	1·477 (3)		

O(1)—C(1)—C(2)	117·0 (1)	O(1)—C(1)—C(11*B*)	120·9 (1)
C(2)—C(1)—C(11*B*)	122·0 (1)	C(1)—O(1)—C(12)	118·3 (1)
C(1)—C(2)—O(2)	117·4 (1)	C(1)—C(2)—C(3)	119·8 (1)
O(2)—C(2)—C(3)	122·7 (1)	C(2)—O(2)—C(13)	117·7 (1)
C(2)—C(3)—O(3)	120·1 (1)	C(2)—C(3)—C(3*A*)	121·2 (1)
O(3)—C(3)—C(3*A*)	118·7 (1)	C(3)—O(3)—C(14)	117·4 (1)
C(3)—C(3*A*)—C(4)	122·5 (1)	C(3)—C(3*A*)—C(11*C*)	118·6 (1)
C(4)—C(3*A*)—C(11*C*)	118·8 (1)	C(3*A*)—C(4)—C(5)	118·8 (2)
C(4)—C(5)—N(6)	124·3 (2)	C(5)—N(6)—C(6*A*)	117·6 (1)
N(6)—C(6*A*)—C(7)	116·0 (1)	N(6)—C(6*A*)—C(11*C*)	124·1 (1)
C(7)—C(6*A*)—C(11*C*)	120·0 (1)	C(6*A*)—C(7)—O(7)	121·4 (2)
C(6*A*)—C(7)—C(7*A*)	117·3 (1)	O(7)—C(7)—C(7*A*)	121·3 (2)
C(7)—C(7*A*)—C(8)	117·2 (1)	C(7)—C(7*A*)—C(11*A*)	122·1 (1)
C(8)—C(7*A*)—C(11*A*)	120·7 (2)	C(7*A*)—C(8)—C(9)	120·9 (2)
C(8)—C(9)—C(10)	118·9 (2)	C(9)—C(10)—C(11)	121·3 (2)
C(10)—C(11)—C(11*A*)	121·6 (2)	C(7*A*)—C(11*A*)—C(11)	116·6 (1)
C(7*A*)—C(11*A*)—C(11*B*)	119·7 (1)	C(11)—C(11*A*)—C(11*B*)	123·7 (1)
C(1)—C(11*B*)—C(11*A*)	124·6 (1)	C(1)—C(11*B*)—C(11*C*)	116·9 (1)
C(11*A*)—C(11*B*)—C(11*C*)	118·5 (1)	C(3*A*)—C(11*C*)—C(6*A*)	116·3 (1)
C(3*A*)—C(11*C*)—C(11*B*)	121·4 (1)	C(6*A*)—C(11*C*)—C(11*B*)	122·3 (1)
O(3)—C(14)—O(14)	122·4 (1)	O(3)—C(14)—C(15)	110·4 (2)
O(14)—C(14)—C(15)	127·2 (2)		

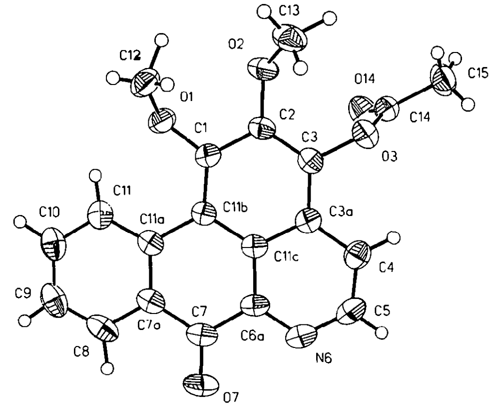

Fig. 1. Drawing of the title compound. Thermal ellipsoids are drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.

parameters, while Table 2 gives interatomic distances and valence angles.* A view of the molecule is given in Fig. 1.

Related literature. Isolation of isomoschatoline is described by Atti, Ammar, Phoebe, Schiff & Slatkin (1982). A general review of oxoaporphines (Shamma & Castenson, 1973) is available. The oxoaporphine can be compared with structures of aporphines (Kunitomo, Satoh, Watanabe, Matsui, Inoue & Ishida, 1982; West, Chackalamannil, Miller, Dalton, Zacharais & Glusker, 1980; Zabel, Watson, Phoebe, Knapp, Schiff & Slatkin, 1982).

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44084 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 11-Methylenelynestrenol

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Abstract. 11-Methylene-19-nor-17α-pregn-4-en-20-yn-17β-ol, $C_{21}H_{28}O$, $M_r = 296·45$, monoclinic, $P2_1$, $a = 7·143\ (1)$, $b = 11·537\ (2)$, $c = 10·353\ (1)$ Å, $\beta = 94·44\ (1)°$, $V = 850·6\ (2)$ Å^3, $Z = 2$, $D_x = 1·157$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·6 cm^{-1}, $F(000) = 324$, $T = 293$ K, $R = 0·045$ for 1832 observed reflections. The structure is isomorphous with that of 13-ethyl-11-methylene-28-norlynestrenol [van Soest, van Dijck & Zeelen (1980). *Recl Trav. Chim. Pays Bas*, **99**, 323–325]; a least-squares fit of C(1)—C(22) of both molecules gave an r.m.s. deviation of the fitted atoms of 0·05 Å. All intermolecular contacts are at normal van der Waals separations.

Experimental. Crystal (0·9 × 0·5 × 0·15 mm) obtained from the Scientific Development Group of Organon, Oss, The Netherlands. Lattice parameters refined by *SET*4 method (de Boer & Duisenberg, 1984) from eight reflections in the 2θ range 30–35°. 2354 integrated intensities measured up to $2\theta_{\max} = 60°$, $h,k,\pm l$ (max. range 9, 15, 14); Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo $K\alpha$ radiation, ω–2θ scan mode, $\Delta\omega = (0·70 + 0·35\tan\theta)°$. Standard reflections showed intensity variations less than 1%; Lp corrections, no correction for absorption. 1833 reflections with $I \geq 2·5\sigma(I)$ were considered observed. Initial phases calculated with the coordinates of 13-ethyl-11-methylene-18-norlynestrenol (van Soest, van Dijck & Zeelen, 1980) and refined by tangent recycling with the *SHELXS*86 program (Sheldrick, 1986). H atoms were placed on calculated positions riding on their bonded atoms, except the hydroxyl-group H atom and the H atom bonded to the ethynyl group, which were located on a difference map. 211 parameters were refined on F with full-matrix least squares using *SHELX*76 (Sheldrick, 1976); reflection $\bar{1}\bar{1}1$ showed severe extinction and was excluded. All non-H atoms were refined anisotropically and for H atoms an overall isotropic thermal parameter was varied [$U = 0·067\ (2)$ Å^2]; convergence at $R = 0·047$ and $wR = 0·038$, where $w = 1/\sigma^2(F)$ and $S = 0·4$; $\Delta/\sigma = 0·02\ (2)$ (av.) and 0·1 (max.) for non-H atom parameters and $\Delta/\sigma = 0·1\ (2)$ (av.) and 0·4 (max.) for H-atom parameters; final electron density within $\Delta\rho = \pm 0·2$ e Å^{-3}. Scattering factors from *SHELX*76. Final atomic parameters are given in Table 1, and bond lengths and angles in Table 2.* Fig. 1 shows the

Table 1. *Positional and equivalent isotropic thermal parameters* (Å^2) *for non-*H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta)/3\sin^2\beta.$$

	x	y	z	U_{eq}
O(17)	0·2018 (3)	0·6283 (3)	0·3277 (2)	0·0631 (6)
C(1)	0·8711 (4)	0·8643 (4)	0·8872 (2)	0·048 (1)
C(2)	0·9476 (4)	0·9010 (4)	1·0230 (3)	0·062 (1)
C(3)	0·9116 (4)	1·0302 (4)	1·0402 (3)	0·070 (1)
C(4)	0·7146 (4)	1·0601 (4)	0·9918 (3)	0·057 (1)
C(5)	0·5964 (4)	0·9920 (4)	0·9210 (2)	0·041 (1)
C(6)	0·3942 (4)	1·0259 (4)	0·8904 (2)	0·046 (1)
C(7)	0·3298 (4)	1·0063 (3)	0·7481 (2)	0·043 (1)
C(8)	0·3629 (3)	0·8801 (3)	0·7098 (2)	0·0363 (8)
C(9)	0·5755 (3)	0·8505 (*)	0·7324 (2)	0·0327 (8)
C(10)	0·6551 (3)	0·8747 (3)	0·8734 (2)	0·0378 (8)
C(11)	0·6062 (4)	0·7277 (3)	0·6851 (2)	0·038 (1)
C(12)	0·5442 (4)	0·7083 (4)	0·5437 (2)	0·043 (1)
C(13)	0·3345 (4)	0·7343 (3)	0·5223 (2)	0·037 (1)
C(14)	0·3005 (3)	0·8584 (3)	0·5675 (2)	0·0363 (8)
C(15)	0·0947 (4)	0·8838 (4)	0·5193 (3)	0·056 (1)
C(16)	0·0662 (4)	0·8137 (4)	0·3919 (3)	0·057 (1)
C(17)	0·2468 (4)	0·7416 (4)	0·3808 (2)	0·046 (1)
C(18)	0·2234 (4)	0·6435 (4)	0·5948 (3)	0·055 (1)
C(20)	0·3741 (5)	0·8005 (4)	0·2962 (3)	0·047 (1)
C(21)	0·4727 (5)	0·8463 (4)	0·2266 (3)	0·066 (1)
C(22)	0·6693 (4)	0·6395 (4)	0·7587 (3)	0·057 (1)

* Kept fixed during the refinement.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44116 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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