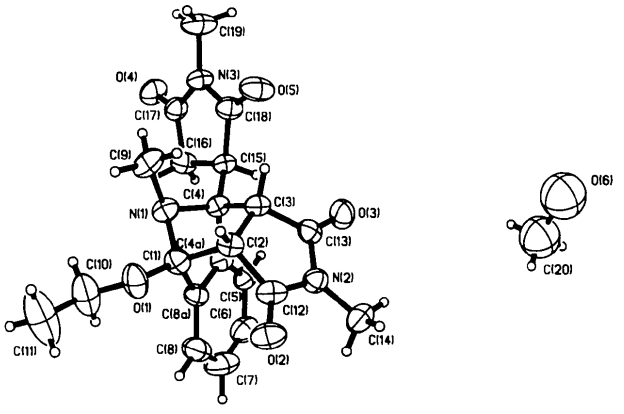

Fig. 1. General view (*SHELXTL-Plus* graphic) of the molecule, showing the atom-numbering scheme.

methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å). After locating the atoms of the molecule $C_{21}H_{23}N_3O_5$ the ΔF synthesis still showed rather high peaks. The two highest peaks were refined as O and C respectively with an occupancy factor 0·5. Refinement on F with 2392 reflections and 271 refined parameters; $w = 1{\cdot}00/[\sigma^2(F) + 0{\cdot}0005F^2]$; $S = 2{\cdot}51$, $R = 0{\cdot}092$, $wR = 0{\cdot}088$. The weakly diffracting crystal and the incompletely described disorder around the solvent molecule could be a reason for the rather high R value. $(\Delta/\sigma)_{max} = 0{\cdot}38$, no extinction correction; largest peak in final ΔF map $\pm 1{\cdot}0$ (2) e Å^{-3} near the probably disordered methanol molecule and $\pm 0{\cdot}2$ (2) e Å^{-3} in remaining parts of the unit cell, atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984). The molecule and the numbering scheme are shown in Fig. 1. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

Related literature. For preparation and spectroscopic data see Kreher & Hennige (1973) and Jelitto (1988).

*Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51673 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of Some Lanosterol Derivatives

BY E. E. CASTELLANO AND J. ZUKERMAN-SCHPECTOR*

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 *São Carlos, SP, Brazil*

AND VERA L. G. REHDER AND A. J. MARSAIOLI

Instituto de Química, Universidade Estadual de Campinas, Caixa Postal 6154, 13081 *Campinas, SP, Brazil*

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Abstract. (I) $C_{32}H_{54}O_4$: 3β-acetoxy-8,9-secolanostane-8,9-dione, $M_r = 502{\cdot}78$, monoclinic, $P2_1$, $a = 11{\cdot}456$ (3), $b = 7{\cdot}400$ (1), $c = 17{\cdot}843$ (5) Å, $\beta = 95{\cdot}69$ (2)°, $V = 1505$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}109$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}04$ mm^{-1}, $F(000) = 556$, $T = 296$ K, $R = 0{\cdot}089$ for 1061 independent reflections [$I > 3\sigma(I)$]. The ten-membered ring is in a crown conformation, the

* Permanent address: Laboratório de Cristalografia e Físico-Química dos Materiais, CCEN, Universidade Federal de Alagoas, 57000 – Maceió, AL, Brazil.

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Table 1. *Data-collection and refinement parameters*

	(I)	(II)	(III)	(IV)
Approximate crystal size (mm)	0·28 × 0·28 × 0·11	0·55 × 0·30 × 0·10	0·38 × 0·35 × 0·18	0·33 × 0·25 × 0·19
No. of reflections for orientation matrix	25	17	19	18
Range of θ (°)	$8 < \theta < 19$	$9 < \theta < 16$	$9 < \theta < 19$	$8 < \theta < 13$
Collection range (°)	$0 < \theta < 22$	$0 < \theta < 23$	$0 < \theta < 22$	$0 < \theta < 23$
Range of *hkl*	$-12 \le h \le 12, k \le 7, l \le 18$	$-1 \le h \le 7, k \le 19, l \le 25$	$-24 \le h < 24, k < 11, l < 12$	$-15 < h < 15, k < 6, l < 18$
No. of standard reflections, intensity variation (%)	1, 3·1	1, 2·8	1, 3·5	1, 3·7
Total data collected	2142	3235	2063	2122
Independent reflections	2015	2789	2005	1998
R_{int}	0·015	0·010	0·026	0·021
Data with $I > 3\sigma(I)$	1061	1057	1415	634
R	0·089	0·086	0·084	0·070
wR	0·089	0·090	0·091	0·069
Weight	Unit	$1/[\sigma^2(F) + 0.001F^2]$	$1/[\sigma^2(F) + 0.002F^2]$	$1/[\sigma^2(F) + 0.001F^2]$
No. of parameters refined	146	146	154	150
$(\Delta/\sigma)_{max}$	0·05	0·01	0·03	0·02
$\Delta\rho$(max., min.) (e Å^{-3})	0·36, −0·35	0·40, −0·36	0·43, −0·41	0·22, −0·28

Table 2. *Fractional atomic coordinates and isotropic temperature factors* (Å^2) *for* (I)

	x	y	z	B_{iso}*
C(1)	0·960 (1)	0·223 (2)	0·2167 (8)	3·8 (3)
C(2)	0·930 (1)	0·213 (2)	0·2993 (8)	4·3 (4)
C(3)	0·850 (1)	0·366 (2)	0·3149 (7)	3·3 (3)
C(4)	0·731 (1)	0·362 (2)	0·2654 (7)	3·5 (3)
C(5)	0·761 (1)	0·363 (2)	0·1808 (7)	3·3 (3)
C(6)	0·649 (1)	0·373 (2)	0·1248 (7)	3·5 (3)
C(7)	0·670 (1)	0·504 (2)	0·0591 (7)	3·5 (3)
C(8)	0·606 (1)	0·462 (2)	−0·0177 (7)	3·6 (3)
C(9)	0·888 (1)	0·262 (2)	0·0799 (7)	3·0 (3)
C(10)	0·848 (1)	0·208 (2)	0·1613 (7)	3·1 (3)
C(11)	0·832 (1)	0·152 (2)	0·0136 (7)	3·2 (3)
C(12)	0·846 (1)	0·241 (2)	−0·0637 (7)	2·8 (3)
C(13)	0·732 (1)	0·295 (2)	−0·1115 (7)	2·8 (3)
C(14)	0·678 (1)	0·485 (2)	−0·0873 (7)	3·4 (3)
C(15)	0·591 (1)	0·532 (2)	−0·1561 (8)	4·3 (4)
C(16)	0·647 (1)	0·452 (3)	−0·2274 (8)	5·1 (4)
C(17)	0·760 (1)	0·344 (2)	−0·1926 (7)	3·3 (3)
C(18)	0·797 (1)	0·018 (2)	0·1618 (8)	4·0 (3)
C(19)	0·642 (1)	0·137 (2)	−0·1162 (8)	3·8 (3)
C(20)	0·797 (1)	0·190 (2)	−0·2422 (8)	3·6 (3)
C(21)	0·907 (1)	0·261 (2)	−0·2787 (9)	5·0 (4)
C(22)	0·701 (1)	0·119 (2)	−0·3054 (9)	4·9 (4)
C(23)	0·750 (2)	−0·061 (3)	−0·3366 (9)	6·4 (5)
C(24)	0·652 (2)	−0·144 (3)	−0·396 (1)	6·9 (5)
C(25)	0·645 (2)	−0·043 (3)	−0·471 (1)	6·5 (4)
C(26)	0·757 (2)	−0·085 (3)	−0·513 (1)	8·6 (6)
C(27)	0·537 (2)	−0·098 (4)	−0·529 (1)	10·5 (7)
C(28)	0·648 (1)	0·217 (2)	0·2847 (8)	4·1 (4)
C(29)	0·668 (1)	0·550 (3)	0·2780 (9)	5·1 (4)
C(30)	0·758 (1)	0·637 (2)	−0·0731 (8)	4·4 (4)
C(31)	0·893 (1)	0·454 (2)	0·4452 (8)	4·3 (4)
C(32)	0·859 (1)	0·422 (2)	0·5246 (8)	4·5 (4)
O(1)	0·970 (1)	0·547 (2)	0·4290 (7)	7·2 (3)
O(2)	0·5094 (8)	0·399 (1)	−0·0223 (5)	3·8 (2)
O(3)	0·9580 (8)	0·380 (2)	0·0762 (5)	4·6 (2)
O(4)	0·8257 (7)	0·354 (2)	0·3944 (5)	3·8 (2)

* $B_{iso} = \frac{4}{3}\sum_i\sum_j B_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

Table 3. *Fractional atomic coordinates and isotropic temperature factors* (Å^2) *for* (II)

	x	y	z	B_{iso}*
C(1)	0·528 (2)	−0·0498 (6)	0·0966 (5)	4·3 (3)
C(2)	0·544 (2)	0·0322 (6)	0·0825 (5)	4·4 (3)
C(3)	0·662 (2)	0·0425 (6)	0·0294 (5)	5·2 (3)
C(4)	0·874 (2)	0·0150 (7)	0·0333 (5)	5·1 (3)
C(5)	0·847 (2)	−0·0660 (6)	0·0528 (5)	3·3 (3)
C(6)	1·025 (2)	−0·1132 (6)	0·0615 (5)	4·1 (3)
C(7)	0·938 (2)	−0·1887 (6)	0·0774 (4)	3·7 (3)
C(8)	1·065 (2)	−0·2442 (7)	0·1035 (5)	4·1 (3)
C(9)	0·738 (2)	−0·1693 (5)	0·1049 (5)	3·2 (3)
C(10)	0·727 (2)	−0·0852 (5)	0·1055 (5)	3·2 (3)
C(11)	0·704 (2)	−0·2066 (6)	0·1641 (5)	4·7 (3)
C(12)	0·689 (2)	−0·2911 (6)	0·1684 (4)	3·5 (3)
C(13)	0·886 (2)	−0·3292 (6)	0·1716 (5)	3·9 (3)
C(14)	1·008 (2)	−0·3196 (6)	0·1148 (5)	4·4 (3)
C(15)	1·170 (2)	−0·3735 (6)	0·1261 (5)	4·7 (3)
C(16)	1·075 (2)	−0·4399 (6)	0·1557 (5)	5·5 (4)
C(17)	0·876 (2)	0·4150 (6)	0·1761 (5)	3·7 (3)
C(18)	0·812 (2)	−0·0555 (6)	0·1618 (5)	4·8 (3)
C(19)	1·005 (2)	−0·2931 (6)	0·2223 (5)	5·4 (3)
C(20)	0·832 (2)	−0·4499 (7)	0·2354 (5)	5·4 (3)
C(21)	0·636 (2)	−0·4268 (6)	0·2591 (5)	5·7 (4)
C(22)	0·838 (2)	−0·5385 (7)	0·2267 (5)	5·8 (4)
C(23)	0·682 (2)	−0·5645 (6)	0·1876 (5)	4·8 (3)
C(24)	0·696 (3)	−0·6552 (7)	0·1854 (6)	6·8 (4)
C(25)	0·549 (2)	0·6835 (8)	0·1468 (6)	6·4 (4)
C(26)	0·348 (3)	0·6707 (9)	0·1652 (7)	9·8 (5)
C(27)	0·584 (3)	−0·7688 (9)	0·1500 (7)	10·3 (5)
C(28)	0·966 (2)	0·0194 (6)	−0·0218 (5)	5·1 (3)
C(29)	0·990 (2)	0·0642 (7)	0·0760 (5)	5·9 (4)
C(30)	0·890 (2)	−0·3472 (6)	0·0636 (5)	4·1 (3)
C(31)	0·525 (3)	0·1465 (8)	−0·0177 (6)	6·7 (4)
C(32)	0·564 (2)	0·2302 (7)	−0·0256 (5)	6·5 (4)
O(1)	0·669 (1)	0·1214 (4)	0·0166 (3)	5·4 (2)
O(2)	1·223 (1)	−0·2210 (4)	0·1186 (3)	5·5 (2)
O(3)	0·594 (1)	−0·1989 (3)	0·0650 (3)	4·1 (2)
O(4)	0·420 (2)	0·1101 (6)	−0·0429 (4)	9·3 (3)

* $B_{iso} = \frac{4}{3}\sum_i\sum_j B_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

six-membered ring in a chair conformation and the five-membered ring in a twist conformation. (II) $C_{32}H_{54}O_4$: 3β-acetoxy-9α-hydroxy-8,9-seco-7,9-cyclo-7α-lanostan-8-one, $M_r = 502{\cdot}78$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}040$ (2), $b = 18{\cdot}179$ (3), $c = 23{\cdot}497$ (5) Å, $V = 3007$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}111$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}04$ mm^{-1}, $F(000) = 1112$, $T = 296$ K, $R = 0{\cdot}086$ for 1057 independent reflections [$I > 3\sigma(I)$]. The seven-membered ring is in a chair conformation. (III) $C_{32}H_{50}O_6$: 3β-acetoxy-8β-hydroxy-8α,11α-epoxy-8,9-seco-6,11-cyclo-6β-lanostane-7,9-dione, $M_r = 530{\cdot}75$, monoclinic, $C2$, $a = 24{\cdot}79$ (1), $b = 10{\cdot}713$ (3), $c = 11{\cdot}670$ (3) Å, $\beta = 96{\cdot}07$ (6)°, $V = 3082$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}144$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}04$ mm^{-1}, $F(000) = 1160$, $T = 296$ K, $R = 0{\cdot}084$ for 1415 independent reflections [$I > 3\sigma(I)$]. The six-membered ring containing the epoxy bridge function is in a chair conformation and the corresponding fused five-membered ring is in an envelope conformation. There is an intermolecular hydrogen bond O(4)···O(2) ($\bar{x}$,y,2−z) = 2·757 (9) Å. (IV) $C_{32}H_{52}O_5$: 3β-acetoxy-

7α-hydroxy-8,9-seco-7,11-cyclo-11β-lanostane-8,9-dione, $M_r = 516{\cdot}77$, monoclinic, $P2_1$, $a = 14{\cdot}682\ (3)$, $b = 6{\cdot}145\ (2)$, $c = 16{\cdot}637\ (5)$ Å, $\beta = 98{\cdot}12\ (2)°$, $V = 1486\ (1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}16$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}04$ mm^{-1}, $F(000) = 568$, $T = 296$ K, $R = 0{\cdot}070$ for 634 independent reflections [$I > 3\sigma(I)$]. The three six-membered rings are in chair conformations and the five-membered ring is in an envelope conformation.

Experimental. The synthesis of compounds (I), (II), (III) and (IV) has been reported elsewhere (Rehder,

Table 4. *Fractional atomic coordinates and isotropic temperature factors* (Å^2) *for* (III)

	x	y	z	B_{iso}*
C(1)	0·1388 (4)	0·107 (1)	1·0934 (9)	3·8 (2)
C(2)	0·1197 (4)	0·197 (1)	1·1827 (9)	4·1 (2)
C(3)	0·0898 (4)	0·311 (1)	1·1286 (8)	3·5 (2)
C(4)	0·1200 (4)	0·387 (1)	1·0437 (8)	2·8 (2)
C(5)	0·1381 (3)	0·289 (1)	0·0597 (7)	2·6 (2)
C(6)	0·1695 (4)	0·3285 (9)	0·8601 (8)	2·6 (2)
C(7)	0·1373 (4)	0·384 (1)	0·7523 (8)	3·0 (2)
C(8)	0·1301 (3)	0·281 (1)	0·6593 (8)	2·9 (2)
C(9)	0·1847 (4)	0·112 (1)	0·9081 (8)	3·2 (2)
C(10)	0·1712 (4)	0·176 (1)	1·0143 (8)	3·2 (2)
C(11)	0·1855 (3)	0·2024 (9)	0·8093 (7)	2·4 (2)
C(12)	0·2368 (3)	0·1961 (9)	0·7421 (8)	2·6 (2)
C(13)	0·2316 (3)	0·2864 (9)	0·6400 (7)	2·1 (2)
C(14)	0·1718 (3)	0·2719 (9)	0·5756 (7)	2·4 (2)
C(15)	0·1747 (4)	0·366 (1)	0·4814 (8)	2·8 (2)
C(16)	0·2301 (4)	0·3448 (9)	0·4422 (8)	3·1 (2)
C(17)	0·2641 (4)	0·2628 (9)	0·5362 (8)	2·6 (2)
C(18)	0·2275 (5)	0·209 (1)	1·076 (1)	4·8 (3)
C(19)	0·2453 (4)	0·420 (1)	0·6815 (8)	3·1 (2)
C(20)	0·3252 (4)	0·297 (1)	0·5465 (8)	3·1 (2)
C(21)	0·3567 (4)	0·223 (1)	0·643 (1)	4·2 (2)
C(22)	0·3472 (4)	0·268 (1)	0·4309 (9)	3·9 (2)
C(23)	0·4062 (4)	0·314 (1)	0·4239 (9)	4·2 (2)
C(24)	0·4199 (5)	0·304 (1)	0·294 (1)	5·1 (3)
C(25)	0·4770 (5)	0·341 (1)	0·281 (1)	5·5 (3)
C(26)	0·4924 (7)	0·475 (2)	0·317 (2)	9·7 (5)
C(27)	0·4887 (8)	0·310 (2)	0·162 (2)	10·5 (5)
C(28)	0·0791 (4)	0·477 (1)	0·9802 (9)	4·0 (2)
C(29)	0·1648 (4)	0·467 (1)	1·1096 (9)	3·6 (2)
C(30)	0·1617 (4)	0·142 (1)	0·5161 (8)	3·1 (2)
C(31)	0·0313 (4)	0·380 (1)	1·2689 (8)	2·9 (2)
C(32)	0·0253 (4)	0·473 (1)	1·359 (1)	4·6 (3)
O(1)	0·0766 (3)	0·3961 (7)	1·2186 (5)	3·6 (1)
O(2)	−0·0010 (3)	0·2974 (8)	1·2415 (6)	4·5 (2)
O(3)	0·1174 (3)	0·4870 (7)	0·7394 (6)	4·1 (2)
O(4)	0·0777 (2)	0·2796 (7)	0·6024 (5)	3·5 (1)
O(5)	0·1376 (2)	0·1713 (6)	0·7337 (5)	2·8 (1)
O(6)	0·1951 (3)	−0·0017 (8)	0·9016 (6)	4·9 (2)

* $B_{iso} = \frac{4}{3}\sum_i\sum_j B_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

Table 5. *Fractional atomic coordinates and isotropic temperature factors* (Å^2) *for* (IV)

	x	y	z	B_{iso}*
C(1)	0·808 (1)	0·190 (3)	0·326 (1)	3·4 (5)
C(2)	0·767 (1)	0·089 (4)	0·403 (1)	4·1 (5)
C(3)	0·664 (1)	0·107 (3)	0·382 (1)	2·9 (4)
C(4)	0·620 (1)	−0·015 (3)	0·3046 (9)	1·6 (4)
C(5)	0·667 (1)	0·073 (4)	0·234 (1)	2·9 (4)
C(6)	0·629 (1)	−0·022 (4)	0·155 (1)	3·8 (5)
C(7)	0·661 (1)	0·098 (4)	0·081 (1)	3·0 (4)
C(8)	0·622 (1)	0·021 (3)	−0·008 (1)	3·3 (5)
C(9)	0·802 (1)	0·183 (3)	0·1783 (9)	2·4 (4)
C(10)	0·772 (1)	0·068 (4)	0·250 (1)	3·6 (5)
C(11)	0·769 (1)	0·107 (3)	0·0903 (9)	2·4 (4)
C(12)	0·813 (1)	0·198 (3)	0·021 (1)	2·9 (4)
C(13)	0·774 (1)	0·060 (4)	−0·057 (1)	3·1 (4)
C(14)	0·6661 (9)	0·104 (3)	−0·0706 (8)	1·2 (4)
C(15)	0·647 (1)	−0·020 (3)	−0·158 (1)	3·0 (4)
C(16)	0·727 (1)	0·049 (3)	−0·199 (1)	3·7 (5)
C(17)	0·802 (1)	0·139 (3)	−0·1381 (9)	2·5 (4)
C(18)	0·821 (1)	−0·162 (4)	0·250 (1)	4·2 (5)
C(19)	0·797 (1)	−0·174 (4)	−0·0462 (9)	2·3 (4)
C(20)	0·903 (1)	0·070 (4)	−0·155 (1)	4·0 (5)
C(21)	0·980 (1)	0·188 (4)	−0·099 (1)	6·0 (6)
C(22)	0·911 (1)	0·147 (4)	−0·241 (1)	4·9 (5)
C(23)	0·905 (1)	0·386 (4)	−0·266 (1)	5·3 (5)
C(24)	0·918 (1)	0·400 (5)	−0·356 (1)	6·7 (7)
C(25)	0·896 (2)	0·611 (7)	−0·390 (2)	12 (1)
C(26)	0·794 (2)	0·676 (5)	−0·408 (1)	8·5 (7)
C(27)	0·930 (2)	0·625 (5)	−0·477 (2)	10·9 (9)
C(28)	0·630 (1)	−0·270 (4)	0·313 (1)	5·0 (5)
C(29)	0·516 (1)	0·034 (4)	0·296 (1)	4·0 (5)
C(30)	0·642 (1)	0·355 (4)	−0·0896 (9)	3·0 (4)
C(31)	0·613 (1)	0·113 (5)	0·512 (1)	5·2 (5)
C(32)	0·579 (1)	−0·010 (4)	0·584 (1)	6·8 (6)
O(1)	0·6285 (8)	−0·003 (2)	0·4469 (7)	4·7 (3)
O(2)	0·632 (1)	0·302 (4)	0·5194 (9)	8·4 (5)
O(3)	0·6354 (7)	0·326 (2)	0·0849 (6)	3·6 (3)
O(4)	0·5561 (7)	−0·103 (2)	−0·0120 (6)	3·6 (3)
O(5)	0·8564 (7)	0·336 (2)	0·1825 (6)	3·9 (3)

* $B_{iso} = \frac{4}{3}\sum_i\sum_j B_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

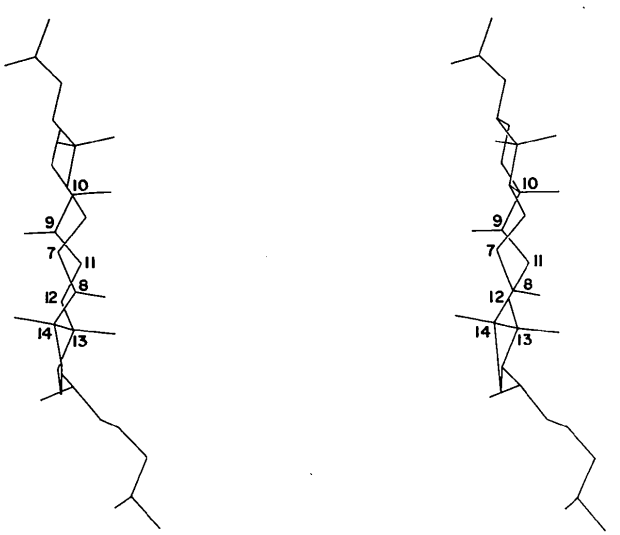


Fig. 1. Stereoscopic drawing of (I).

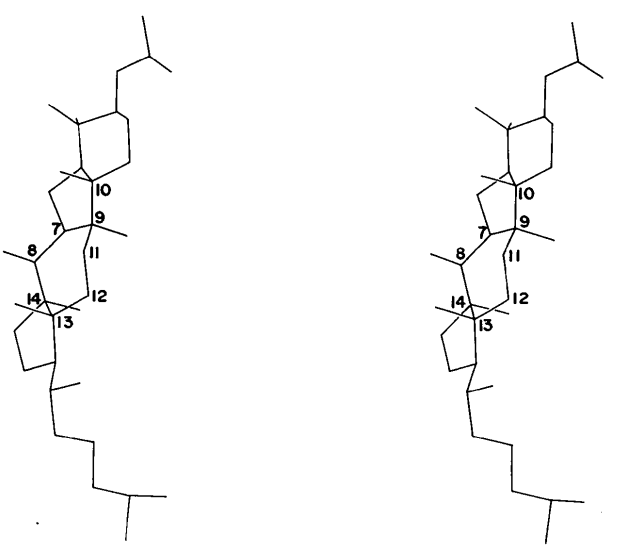


Fig. 2. Stereoscopic drawing of (II).

Marsaioli, Castellano & Zukerman-Schpector, 1988). The different data-collection and refinement parameters are summarized in Table 1. Enraf–Nonius CAD-4 diffractometer, ω–2θ scan mode. The structures were solved using standard direct methods and difference Fourier techniques. No corrections for absorption. Full-matrix least-squares refinement on F, all atoms isotropic and H atoms included in the model, as fixed contributors to the structure, were those found in difference Fourier maps; an overall thermal parameter for these was refined and converged to: $U_{(I)} = 0{\cdot}02$, $U_{(II)} = 0{\cdot}05$, $U_{(III)} = 0{\cdot}05$ and $U_{(IV)} = 0{\cdot}05$ Å^2. Scattering factors for non-H atoms from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for H atoms from Stewart, Davidson & Simpson (1965); programs used: *SHELX*76 (Sheldrick, 1976), *ORTEP* (Johnson, 1965).

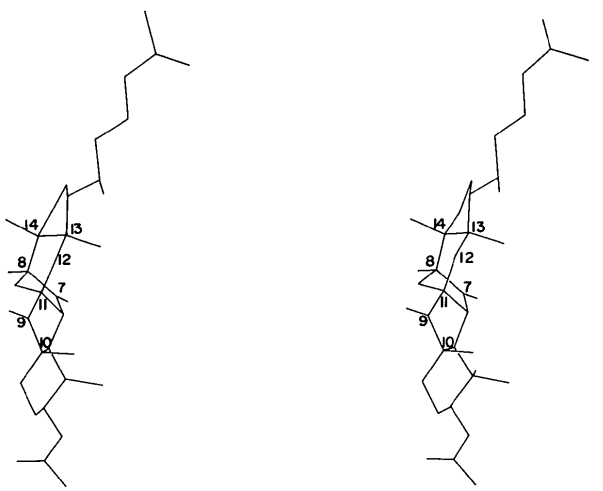


Fig. 3. Stereoscopic drawing of (III).

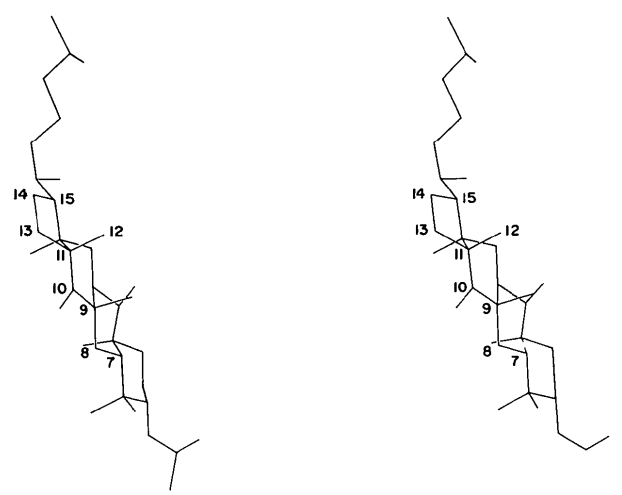
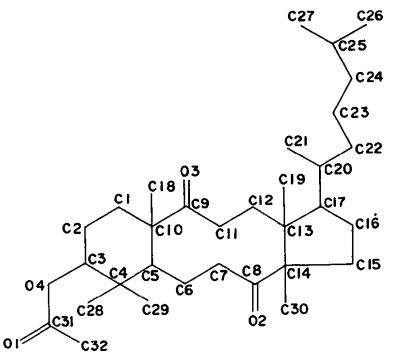
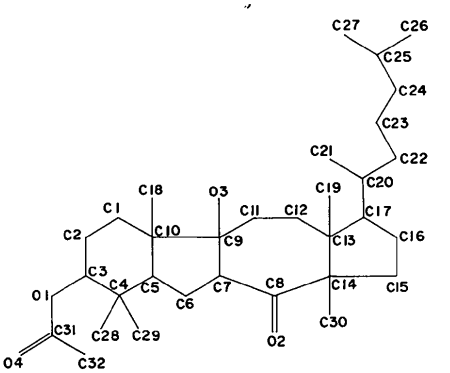


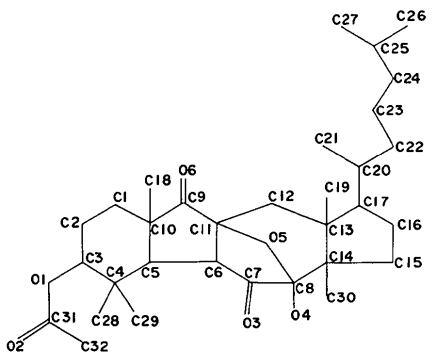
Fig. 4. Stereoscopic drawing of (IV).



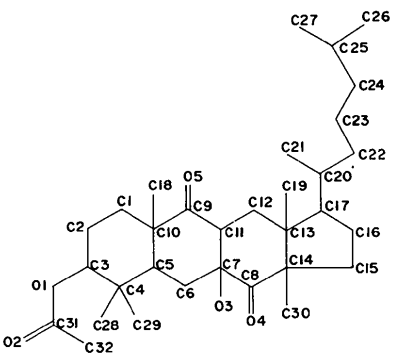
(I)



(II)



(III)



(IV)

Final atomic coordinates for non-H atoms are given in Tables 2, 3, 4 and 5 for (I), (II), (III) and (IV), respectively.* No anomalous bond distances or angles were found. Figs. 1, 2, 3 and 4 are stereoscopic drawings of compounds (I), (II), (III) and (IV), respectively.

Related literature. All the crystals were of similar poor diffracting quality and did not allow measurements of data with resolution good enough to perform anisotropic refinements for accurate calculation of distances and angles. Nevertheless, the main aim of the present study could still be achieved, namely the unambiguous determination of the four intermediate conformations; this knowledge is fundamental for proposing possible fragmentation reaction pathways (Sharpless, 1981; Snatzke & Fehlhaber, 1963; Prelog, 1965; Webster, Entenios & Silverstein, 1987).

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* Lists of structure factors, H-atom coordinates and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51654 (56 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structural Studies on Abscisic-Acid-Synthesis Intermediates: (1) 5,6-Dihydro-4-methyl-6-(7,9,9-trimethyl-1,4-dioxaspiro[4.5]dec-7-en-8-yl)-2*H*-pyran-2-one and (2) 5,6-Dihydro-4-methyl-6-(2′,2′,6′-trimethylspiro[1,3-dioxolane-2,4′-[7]oxabicyclo[4.1.0]hept]-1′-yl)-2*H*-pyran-2-one

BY E. E. CASTELLANO AND J. ZUKERMAN-SCHPECTOR*

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 *São Carlos, SP, Brazil*

AND MAURICIO G. CONSTANTINO AND PELLEGRINO LOSCO

Departamento de Química de Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto, Universidade de São Paulo, Av. Bandeirantes 3.900, 14049 *Ribeirão Preto, SP, Brazil*

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Abstract. (1) $C_{17}H_{24}O_4$, $M_r = 292{\cdot}38$, triclinic, $P\bar{1}$, $a = 6{\cdot}811$ (1), $b = 9{\cdot}564$ (2), $c = 13{\cdot}052$ (2) Å, $\alpha = 98{\cdot}66$ (1), $\beta = 98{\cdot}67$ (2), $\gamma = 107{\cdot}07$ (2)°, $V = 786{\cdot}2$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}234$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}05$ mm^{-1}, $F(000) = 316$, $T = 296$ K, final $R = 0{\cdot}056$ for 1463 observed reflections. The pyran ring is in a half-chair conformation. (2) $C_{17}H_{24}O_5$, $M_r = 308{\cdot}38$, triclinic, $P\bar{1}$, $a = 6{\cdot}401$ (3), $b = 9{\cdot}649$ (2), $c = 13{\cdot}417$ (4) Å, $\alpha = 96{\cdot}44$ (2), $\beta = 98{\cdot}73$ (3), $\gamma = 103{\cdot}77$ (2)°, $V = 786$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}303$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}06$ mm^{-1}, $F(000) = 332$, $T = 296$ K, final $R = 0{\cdot}055$ for 1311 observed reflections. The pyran and the epoxy O atoms are in a *cis* configuration.

* Permanent address: Laboratório de Cristalografia e Físico-Química dos Materiais, CCEN, Universidade Federal de Alagoas, 57000 Maceió, AL, Brazil.

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