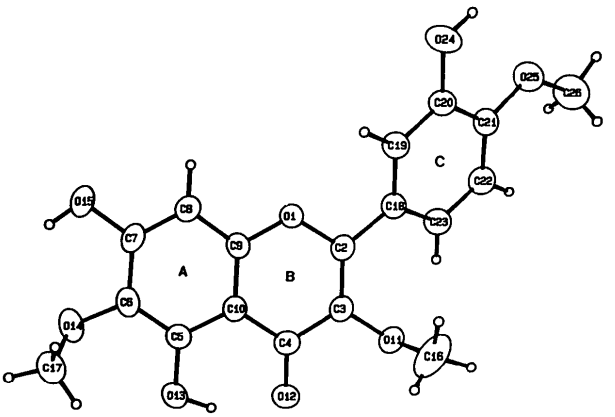

Fig. 1. Numbering scheme with thermal ellipsoids drawn at the 40% probability level. H atoms have arbitrary radius.

Related literature. Isolation of centaureidin from *Centaurea* species (Bohlmann & Zdero, 1967). Antitumor activity of centaureidin (Kupchan & Bauerschmidt, 1971). Crystal structure of the pharmacologically active 5,4′-dihydroxy-3,6,7,8-tetramethoxyflavone, calycopterin (Vijayalakshmi, Rajan, Srinivasan & Ramachandran Nair, 1986).

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Structure of the Guaianolide Dehydrocostus Lactone

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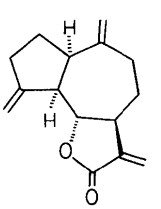
Abstract. Decahydro-3,6,9-tris(methylene)azuleno-[4,5-*b*]furan-2(3*H*)-one, $C_{15}H_{18}O_2$, $M_r = 230{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}810\ (1)$, $b = 11{\cdot}403\ (1)$, $c = 14{\cdot}240\ (1)$ Å, $V = 1268{\cdot}2\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}206$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 5{\cdot}87$ cm^{-1}, $F(000) = 496$, $T = 298$ K, $R = 0{\cdot}035$ for 1432 observations (of 1515 unique data). The title compound, which exhibits no molluscicidal acitivity, differs in conformation from its 7α-hydroxy analog, 7α-hydroxy-3-desoxyzaluzanin C, which is highly active [Fronczek, Vargas, Fischer & Hostettmann (1984). *J. Nat. Prod.* **47**, 1036–1039]. The conformation of the seven-membered ring is a distorted twist-chair, with the pseudodiad axis passing through C8, and asymmetry parameter $\Delta C_2 = 8{\cdot}2°$. The lactone ring is in the half-chair conformation with carbonyl carbon C12 on the local twofold axis, and $\Delta C_2 = 3{\cdot}0°$. The other five-membered ring has a distorted half-chair conformation with the axis passing through C4, and $\Delta C_2 = 7{\cdot}0°$. Crystals of the guaianolide dehydrocostus lactone were isolated from costus oil purchased from Pierre Chauvet S. A., France.

Experimental. Dehydrocostus lactone, (1), was obtained as colorless needles, data-collection crystal of dimensions 0·44 × 0·48 × 0·72 mm. Space group from absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd. Enraf–Nonius CAD-4 diffractometer with graphite monochromator; cell dimensions from setting angles of 25 reflections having $40 > \theta > 35°$. Data collection by ω–2θ scans designed for $I = 50\sigma(I)$ subject to max. scan time = 120 s. Scan rates varied 0·63 – 4·0° min^{-1}. Reflections having $4 < 2\theta < 150°$, $0 \le h \le 9$, $0 \le k \le 14$, $0 \le l \le 17$ were measured; corrected for background, Lorentz, polarization and absorption by ψ scans, minimum relative



(1)

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
O1	0·0837 (2)	0·93054 (9)	0·91826 (9)	4·32 (2)
O2	0·2286 (2)	1·0930 (1)	0·9535 (1)	5·96 (3)
C1	−0·2560 (2)	0·7551 (1)	0·7845 (1)	4·03 (3)
C2	−0·2999 (3)	0·6599 (2)	0·8563 (2)	5·26 (4)
C3	−0·1302 (3)	0·5966 (2)	0·8712 (2)	5·98 (5)
C4	0·0059 (2)	0·6883 (1)	0·8559 (1)	4·09 (3)
C5	−0·0784 (2)	0·8000 (1)	0·8186 (1)	3·48 (3)
C6	−0·0911 (2)	0·8972 (1)	0·8915 (1)	3·44 (3)
C7	−0·1730 (2)	1·0105 (1)	0·8545 (1)	3·66 (3)
C8	−0·3646 (3)	1·0218 (2)	0·8722 (2)	5·27 (4)
C9	−0·4670 (3)	0·9114 (2)	0·8477 (2)	5·97 (5)
C10	−0·3967 (2)	0·8427 (2)	0·7662 (2)	4·80 (4)
C11	−0·0645 (2)	1·1041 (1)	0·8977 (1)	3·84 (3)
C12	0·0979 (2)	1·0483 (2)	0·9268 (1)	4·21 (3)
C13	−0·0958 (3)	1·2162 (2)	0·9116 (2)	5·11 (4)
C14	−0·4591 (4)	0·8538 (2)	0·6802 (2)	7·24 (6)
C15	0·1705 (3)	0·6721 (2)	0·8678 (2)	5·20 (4)

transmission 0·8644; 1515 unique data. Standard reflections 200, 040, 004, ± 1·8% random variation, no decay correction. Structure solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ with 1432 data for which $I > 1\sigma(I)$ (83 unobserved reflections), using Enraf–Nonius *SDP* (Frenz & Okaya, 1980). Non-H atoms anisotropic; H atoms located by ΔF synthesis and refined isotropically. Atomic scattering factors of Cromer & Waber (1974) and anomalous coefficients of Cromer (1974). Final $R = 0{\cdot}035$, $wR = 0{\cdot}044$, $S = 2{\cdot}732$ for 227 variables, extinction coefficient $g = 4{\cdot}6\,(4) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c, max. shift in final cycle $0{\cdot}19\sigma$, max. residual density 0·18, min. −0·16 e Å^{-3}. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances, bond angles and torsion angles in Table 2. Fig. 1 shows the atom-numbering scheme.

Related literature. Crystal structure of related compounds: 7α-hydroxy-3-desoxyzaluzanin C (Fronczek, Vargas, Fischer & Hostettmann, 1984) and solstitialin (Thiessen & Hope, 1970). Dehydrocostus lactone: isolation (Semmler & Feldstein, 1914), structure elucidation (Romanuk, Herout & Sorm, 1956), plant growth regulatory activity (Kalsi, Vij, Singh & Wadia, 1977) and conformational asymmetry parameters (Duax & Norton, 1975).

* Tables of H-atom parameters, distances and angles involving H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52080 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

O1—C6	1·468 (2)	C5—C6	1·522 (2)
O1—C12	1·353 (2)	C6—C7	1·535 (2)
O2—C12	1·203 (2)	C7—C8	1·523 (3)
C1—C2	1·530 (3)	C7—C11	1·496 (2)
C1—C5	1·557 (2)	C8—C9	1·531 (3)
C1—C10	1·508 (2)	C9—C10	1·504 (3)
C2—C3	1·525 (3)	C10—C14	1·323 (4)
C3—C4	1·507 (3)	C11—C12	1·478 (3)
C4—C5	1·529 (2)	C11—C13	1·317 (2)
C4—C15	1·310 (3)		
C6—O1—C12	110·8 (1)	C6—C7—C8	115·1 (1)
C2—C1—C5	103·0 (1)	C6—C7—C11	102·9 (1)
C2—C1—C10	115·0 (2)	C8—C7—C11	115·4 (1)
C5—C1—C10	119·0 (1)	C7—C8—C9	113·9 (2)
C1—C2—C3	103·5 (2)	C8—C9—C10	114·5 (2)
C2—C3—C4	105·3 (2)	C1—C10—C9	118·6 (2)
C3—C4—C5	109·0 (2)	C1—C10—C14	119·4 (2)
C3—C4—C15	125·1 (2)	C9—C10—C14	122·0 (2)
C5—C4—C15	125·8 (2)	C7—C11—C12	107·1 (1)
C1—C5—C4	102·6 (1)	C7—C11—C13	130·5 (2)
C1—C5—C6	113·2 (1)	C12—C11—C13	122·4 (2)
C4—C5—C6	113·5 (1)	O1—C12—O2	121·2 (2)
O1—C6—C5	107·7 (1)	O1—C12—C11	109·4 (1)
O1—C6—C7	105·0 (1)	O2—C12—C11	129·4 (2)
C5—C6—C7	113·9 (1)		

C12—O1—C6—C7	17·1 (2)	C1—C5—C6—C7	−64·5 (2)
C6—O1—C12—C11	−5·1 (2)	O1—C6—C7—C11	−21·4 (2)
C5—C1—C2—C3	−41·0 (2)	C5—C6—C7—C8	94·6 (2)
C2—C1—C5—C4	35·4 (2)	C6—C7—C8—C9	−46·8 (3)
C10—C1—C5—C6	41·3 (2)	C6—C7—C11—C12	18·8 (2)
C2—C1—C10—C14	−120·1 (2)	C7—C8—C9—C10	−32·9 (3)
C5—C1—C10—C9	−65·4 (2)	C8—C9—C10—C1	84·7 (2)
C1—C2—C3—C4	30·4 (2)	C7—C11—C12—O1	−9·4 (2)
C2—C3—C4—C5	−8·0 (2)	C13—C11—C12—O2	−10·9 (3)
C3—C4—C5—C1	−17·0 (2)		

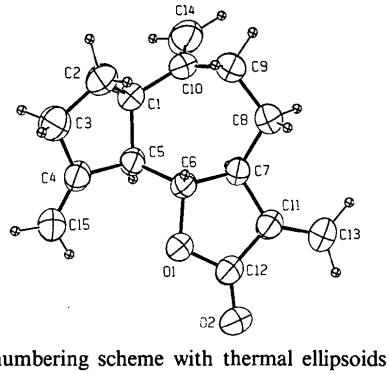

Fig. 1. The numbering scheme with thermal ellipsoids drawn at the 50% probability level. H atoms have arbitrary radius.

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1-Chloro-3-ethynyl-2,4-dimethoxybenzene

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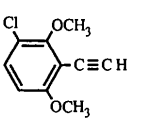
Abstract. $C_{10}H_9ClO_2$, $M_r = 196{\cdot}6$, monoclinic, $P2_1/c$, $a = 10{\cdot}8146\ (6)$, $b = 8{\cdot}8883\ (8)$, $c = 10{\cdot}4110\ (9)$ Å, $\beta = 103{\cdot}101\ (6)°$, $V = 974{\cdot}7\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}340$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 32{\cdot}27$ cm^{-1}, $F(000) = 408$, $T = 296$ K, $R = 0{\cdot}061$ for 1405 observations (of 2004 unique data). The molecule contains two methoxy groups; one is nearly coplanar with the benzenoid ring, with C—C—O—C torsion angle $-5{\cdot}2\ (5)°$, and the other, which resides between the chloro and the ethynyl groups, is nearly orthogonal, with the corresponding torsion angle 86·5 (4)°. The coplanar methoxy has an angle about O of 118·3 (2)° and the orthogonal, 114·7 (1)°. The O—CH_3 distance in the coplanar methoxy is 1·426 (2) Å compared to 1·439 (2) Å in the orthogonal. The six-membered ring is planar, with maximum deviation 0·008 (3) Å. The C—Cl distance is 1·734 (2) Å, and the triple-bond distance is 1·179 (3) Å. The ethynyl group forms a nearly linear C—H···O contact with the O atom of the orthogonal methoxy on a glide-related molecule, having C···O distance 3·293 (3) Å and angle at H of 167 (3)°.

Experimental. Colorless plates of (1), m.p. 365·5–365·9 K, were isolated by recrystallization in hexane from the crude reaction product of 2,6-dimethoxyacetophenone and phosphorus pentachloride in benzene at room temperature. Crystal size 0·08 × 0·30 × 0·38 mm, space group from systematic absences $h0l$ with l odd and $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$.



(1)

Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 1·10–3·30° min^{-1}. One quadrant of data having $2 < \theta < 75°$, $0 \le h \le 13$, $0 \le k \le 11$, $-13 \le l \le 13$ measured. Data corrected for background, Lorentz and polarization effects. Since the crystal sublimed during data collection, the standard reflections 300, 060, 002 decreased by 16·74%, and linear decay correction was applied. Absorption corrections were based on ψ scans, with min. relative transmission coefficient 62·95%. 2486 total data were collected, and redundant data merged, $R_{int} = 0{\cdot}016$, to yield 2004 unique data, 1405 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using Enraf-Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by ΔF map. Methyl H atoms fixed with C—H 0·95 Å and $B_{iso} = 1{\cdot}3B_{eq}$ for the methyl C atom. Other H atoms were refined isotropically. Final $R = 0{\cdot}061$ (0·090 for all data), $wR = 0{\cdot}072$, $S = 3{\cdot}007$ for 131 variables. Max. shift $< 0{\cdot}01\sigma$ in the final cycle, max. residual density 0·42, min. −0·31 e Å^{-3}, extinction coefficient $g = 3{\cdot}6\ (5) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Coordinates† are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1.

† Tables of H-atom coordinates, least-squares planes, torsion angles, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52060 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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