

[P(2)—O(E21) = 1,554 (3) contre 1,487 (3) Å pour P(2)—O(E22) et P(2)—O(E23)].

(b) Une liaison P—O(L) dissymétrique 1,583 (3) et 1,623 (3) Å.

Ces constatations avaient déjà été observées dans $\text{K}_3\text{HP}_2\text{O}_7 \cdot 3\text{H}_2\text{O}$ (Dumas, Galigne & Falgueirettes, 1973); alors que dans les sels: $[\text{C}(\text{NH}_3)_3]_4\text{P}_2\text{O}_7 \cdot \text{H}_2\text{O}$ (Adams & Ramdas, 1977), $(\text{NH}_4)_2\text{MoO}_2\text{P}_2\text{O}_7$ (Averbuch-Pouchot, 1988) et $[\text{C}(\text{NH}_3)_3]_3\text{HP}_2\text{O}_7$ (Adams & Ramdas, 1976), la liaison P—O(L) est symétrique à cause respectivement de l'absence de proton et de sa délocalisation sur l'ensemble des atomes d'oxygène du groupement diphosphate.

La maille élémentaire renferme quatre molécules d'eau: chaque molécule est liée aux deux tétraèdres d'un même groupement $[\text{HP}_2\text{O}_7^{3-}]$ par deux liaisons hydrogène du type: O(w)—H(1w)⋯O(E23) et O(w)—H(2w)⋯O(E12). Les groupements organiques

$[(\text{CH}_2)_2(\text{NH}_3)_2]^{2+}$, situés dans le canal, sont placés dans deux sites cristallographiques indépendants: une molécule centrosymétrique, l'autre en position générale. Les molécules organiques se lient aux anions $[\text{HP}_2\text{O}_7^{3-}]$, des différentes chaînes, par des liaisons par pont hydrogène du type N—H⋯O, assurant la cohésion du réseau tridimensionnel.

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Structures of Two Neolignans

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Abstract. 11,12,13,14-Tetrahydro-12,13-dimethylcycloocta[1,2-*f*:3,4-*f'*]bis[1,3]benzodioxol-11-yl acetate (1), $\text{C}_{22}\text{H}_{22}\text{O}_6$, $M_r = 382.412$, orthorhombic, $P2_12_12_1$, $a = 9.393$ (1), $b = 11.395$ (3), $c = 17.888$ (5) Å, $V = 1914.6$ (7) Å³, $Z = 4$, $D_x = 1.327$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.68$ cm⁻¹, $F(000) = 808$, $T = 301$ K. Final R and wR for 1780 reflections with $F_o \geq 3\sigma(F_o)$ are 0.042 and 0.043. The dimethyl groups on the cyclooctadiene ring are equatorial and the eight-membered ring is in a twist-boat-chair conformation. The bond distances and angles are normal. 11,12,13,14-Tetrahydro-12,13-dimethyl-11,14-epoxycycloocta[1,2-*f*:3,4-*f'*]bis[1,3]benzodioxole (2), $\text{C}_{20}\text{H}_{18}\text{O}_5$, $M_r = 338.359$, orthorhombic, $P2_12_12_1$, $a = 6.659$ (2), $b = 11.358$ (2), $c = 21.307$ (4) Å, $V = 1611.5$ (6) Å³, $Z = 4$, $D_x = 1.394$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.74$ cm⁻¹, $F(000) = 712$, $T = 301$ K. Final R and wR for 1745 reflections with F_o

$\geq 3\sigma(F_o)$ are 0.032 and 0.036. The epoxide link distorts the eight-membered cyclooctadiene ring toward the twist-boat form. The two methyl groups are in the equatorial position.

Introduction. A number of dibenzocyclooctadiene-type neolignans have been isolated from plants and shown to have interesting and potentially useful biological activities [see Chattopadhyay & Rao (1987) for details and references]. A number of synthetic dibenzocyclooctadienes were prepared by Chattopadhyay & Rao (1987) as part of a study of structure *versus* biological activity. A crystal structure study was undertaken to establish unequivocally the stereochemistry of two of the derivatives.

Experimental. The cell constants and intensity measurements for both compounds were made with a Nicolet R3m diffractometer using graphite-monochromated molybdenum radiation. A sum-

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Table 1. Summary of data collection and refinement parameters

	(1)	(2)
Crystal size (mm)	0.32 × 0.20 × 0.10	0.22 × 0.24 × 0.36
No. of reflections used for cell constants	25	25
2θ range (°)	8–21	11–22
Scan type	θ/2θ	θ/2θ
Scan width (°)	2.0	2.0
Scan speed (° min ⁻¹)	4.88–29.30	4.88–29.30
Range for data collection 2θ (°)	3–55	3–55
h	0–13	0–9
k	0–15	0–15
l	0–24	0–28
Crystal decay (%)	3	4
Reflections measured		
total	2532	2200
unique	2483	2132
observed [<i>F</i> _o ≥ 3σ(<i>F</i> _o)]	1783	1886
<i>R</i> _{merge}	0.006	0.007
<i>R</i> _w , <i>wR</i>	0.042, 0.043	0.032, 0.036
No. of variables	341	298
GOF	1.11	1.40
Min./max. in final difference Fourier map (e Å ⁻³)	-0.21/0.18	-0.14/0.14
Max. shift/e.s.d.	0.24	0.18
<i>g</i> in <i>w</i> = (σ ² <i>F</i> + <i>gF</i> ²) ⁻¹	0.0012	0.00057

Table 2. Atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å² × 10³) for C₂₂H₂₂O₆

*U*_{eq} is defined as one third of the trace of the orthogonalized *U*_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
C(1)	1230 (6)	3042 (4)	-2771 (2)	74 (2)
C(2)	2190 (4)	3105 (3)	-1628 (2)	47 (1)
C(3)	2792 (4)	3437 (3)	-967 (2)	44 (1)
C(4)	3382 (4)	2554 (3)	-509 (2)	36 (1)
C(5)	4097 (4)	2946 (3)	204 (2)	40 (1)
C(6)	3078 (4)	3101 (3)	878 (2)	45 (1)
C(7)	2923 (4)	1980 (3)	1345 (2)	44 (1)
C(8)	2515 (4)	921 (3)	859 (2)	38 (1)
C(9)	3739 (4)	292 (3)	479 (2)	35 (1)
C(10)	4548 (4)	-505 (3)	898 (2)	43 (1)
C(11)	5574 (4)	-1137 (3)	544 (2)	42 (1)
C(12)	7374 (5)	-2331 (4)	239 (2)	61 (1)
C(13)	5830 (4)	-1029 (3)	-210 (2)	42 (1)
C(14)	5076 (4)	-240 (3)	-637 (2)	40 (1)
C(15)	4028 (4)	451 (3)	-284 (2)	35 (1)
C(16)	3321 (4)	1380 (3)	-736 (2)	37 (1)
C(17)	2696 (4)	1079 (3)	-1428 (2)	44 (1)
C(18)	2148 (4)	1947 (3)	-1857 (2)	44 (1)
C(19)	3579 (5)	4154 (4)	1348 (3)	65 (2)
C(20)	1824 (6)	2188 (5)	1972 (3)	81 (2)
C(21)	767 (4)	-570 (3)	1080 (2)	45 (1)
C(22)	93 (5)	-1301 (5)	1678 (2)	75 (2)
O(6)	419 (3)	-573 (2)	436 (1)	57 (1)
O(1)	1567 (4)	3806 (2)	-2166 (1)	66 (1)
O(2)	1472 (4)	1874 (3)	-2536 (1)	69 (1)
O(3)	6867 (3)	-1780 (2)	-430 (1)	60 (1)
O(4)	6484 (3)	-1957 (2)	846 (2)	68 (1)
O(5)	1822 (2)	78 (2)	1356 (1)	49 (1)

mary of the experimental details and refinement procedures is presented in Table 1. The two standard reflections for each compound were measured after every 98 reflections and used to derive an empirical decay correction curve to compensate for the slight decrease in their intensity.

Table 3. Atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å² × 10³) for C₂₀H₁₈O₅

*U*_{eq} is defined as one third of the trace of the orthogonalized *U*_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
O(1)	10510 (2)	2972 (1)	1712 (1)	52 (1)
O(2)	10555 (3)	959 (1)	1854 (1)	59 (1)
O(3)	1728 (2)	-1334 (1)	4541 (1)	55 (1)
O(4)	4979 (2)	-1875 (1)	4306 (1)	57 (1)
O(5)	2888 (2)	3079 (1)	2936 (1)	46 (1)
C(1)	11670 (4)	1933 (2)	1611 (1)	57 (1)
C(2)	9089 (3)	2657 (2)	2149 (1)	40 (1)
C(3)	7749 (3)	3347 (2)	2453 (1)	40 (1)
C(4)	6395 (3)	2810 (2)	2867 (1)	36 (1)
C(5)	4787 (3)	3561 (2)	3144 (1)	41 (1)
C(6)	4688 (3)	3631 (2)	3866 (1)	44 (1)
C(7)	2467 (3)	3377 (2)	3992 (1)	47 (1)
C(8)	2077 (3)	2489 (2)	3464 (1)	43 (1)
C(9)	3036 (3)	1292 (2)	3585 (1)	39 (1)
C(10)	1829 (3)	541 (2)	3952 (1)	44 (1)
C(11)	2596 (3)	-490 (2)	4162 (1)	41 (1)
C(12)	3112 (4)	-2271 (2)	4555 (2)	69 (1)
C(13)	4539 (3)	-810 (2)	4021 (1)	42 (1)
C(14)	5742 (3)	-132 (2)	3655 (1)	41 (1)
C(15)	5007 (3)	947 (2)	3410 (1)	35 (1)
C(16)	6408 (3)	1577 (2)	2970 (1)	35 (1)
C(17)	7816 (3)	894 (2)	2632 (1)	39 (1)
C(18)	9116 (3)	1454 (2)	2234 (1)	40 (1)
C(19)	1943 (4)	3038 (3)	4655 (1)	66 (1)
C(20)	5479 (6)	4783 (2)	4127 (2)	72 (1)

Table 4. Bond lengths (Å) and bond angles (°) for C₂₂H₂₂O₆

C(1)—O(1)	1.425 (5)	C(1)—O(2)	1.414 (5)
C(2)—C(3)	1.364 (5)	C(2)—C(18)	1.382 (5)
C(2)—O(1)	1.380 (4)	C(3)—C(4)	1.411 (5)
C(4)—C(5)	1.509 (5)	C(4)—C(16)	1.399 (4)
C(5)—C(6)	1.549 (5)	C(6)—C(7)	1.534 (5)
C(6)—C(19)	1.538 (6)	C(7)—C(8)	1.537 (5)
C(7)—C(20)	1.543 (6)	C(8)—C(9)	1.515 (5)
C(8)—O(5)	1.462 (4)	C(9)—C(10)	1.401 (5)
C(9)—C(15)	1.402 (4)	C(10)—C(11)	1.360 (5)
C(11)—C(13)	1.375 (5)	C(11)—O(4)	1.377 (5)
C(12)—O(3)	1.433 (5)	C(12)—O(4)	1.435 (5)
C(13)—C(14)	1.376 (5)	C(13)—O(3)	1.354 (4)
C(14)—C(15)	1.410 (5)	C(15)—C(16)	1.489 (4)
C(16)—C(17)	1.413 (5)	C(17)—C(18)	1.354 (5)
C(18)—O(2)	1.373 (4)	C(21)—C(22)	1.496 (6)
C(21)—O(6)	1.197 (4)	C(21)—O(5)	1.331 (4)
O(1)—C(1)—O(2)	108.3 (3)	C(3)—C(2)—C(18)	122.2 (3)
C(3)—C(2)—O(1)	128.2 (3)	C(18)—C(2)—O(1)	109.5 (3)
C(2)—C(3)—C(4)	117.9 (3)	C(3)—C(4)—C(5)	117.1 (3)
C(3)—C(4)—C(16)	119.8 (3)	C(5)—C(4)—C(16)	123.1 (3)
C(4)—C(5)—C(6)	114.6 (3)	C(5)—C(6)—C(7)	112.8 (3)
C(5)—C(6)—C(19)	109.0 (3)	C(7)—C(6)—C(19)	112.4 (3)
C(6)—C(7)—C(8)	111.7 (3)	C(6)—C(7)—C(20)	109.4 (3)
C(8)—C(7)—C(20)	111.4 (3)	C(7)—C(8)—C(9)	115.9 (3)
C(7)—C(8)—O(5)	106.4 (3)	C(9)—C(8)—O(5)	107.5 (3)
C(8)—C(9)—C(10)	118.5 (3)	C(8)—C(9)—C(15)	121.4 (3)
C(10)—C(9)—C(15)	120.0 (3)	C(9)—C(10)—C(11)	118.6 (3)
C(10)—C(11)—C(13)	122.2 (3)	C(10)—C(11)—O(4)	128.1 (3)
C(13)—C(11)—O(4)	109.7 (3)	O(3)—C(12)—O(4)	107.9 (3)
C(11)—C(13)—C(14)	120.8 (3)	C(11)—C(13)—O(3)	110.7 (3)
C(14)—C(13)—O(3)	128.4 (3)	C(13)—C(14)—C(15)	118.4 (3)
C(9)—C(15)—C(16)	119.9 (3)	C(9)—C(15)—C(16)	122.3 (3)
C(14)—C(15)—C(16)	117.7 (3)	C(4)—C(16)—C(15)	120.3 (3)
C(4)—C(16)—C(17)	120.2 (3)	C(15)—C(16)—C(17)	119.3 (3)
C(16)—C(17)—C(18)	118.5 (3)	C(2)—C(18)—C(17)	121.3 (3)
C(2)—C(18)—O(2)	109.5 (3)	C(17)—C(18)—O(2)	129.2 (3)
C(22)—C(21)—O(5)	124.7 (4)	C(22)—C(21)—O(5)	111.0 (3)
O(6)—C(21)—O(5)	124.2 (3)	C(1)—O(1)—C(2)	105.6 (3)
C(1)—O(2)—C(18)	106.3 (3)	C(12)—O(3)—C(13)	105.9 (3)
C(11)—O(4)—C(12)	105.5 (3)	C(8)—O(5)—C(21)	118.0 (2)

Table 5. Bond lengths (Å) and bond angles (°) for $C_{20}H_{18}O_5$

O(1)—C(1)	1.426 (3)	O(1)—C(2)	1.375 (2)
O(2)—C(1)	1.430 (3)	O(2)—C(18)	1.375 (2)
O(3)—C(11)	1.380 (2)	O(3)—C(12)	1.408 (3)
O(4)—C(12)	1.425 (3)	O(4)—C(13)	1.384 (2)
O(5)—C(5)	1.448 (2)	O(5)—C(8)	1.416 (2)
C(2)—C(3)	1.353 (3)	C(2)—C(18)	1.378 (3)
C(3)—C(4)	1.400 (3)	C(4)—C(5)	1.491 (3)
C(4)—C(16)	1.417 (2)	C(5)—C(6)	1.541 (3)
C(6)—C(7)	1.531 (3)	C(6)—C(20)	1.516 (3)
C(7)—C(8)	1.534 (3)	C(7)—C(19)	1.506 (3)
C(8)—C(9)	1.524 (3)	C(9)—C(10)	1.408 (3)
C(9)—C(15)	1.420 (2)	C(10)—C(11)	1.353 (3)
C(11)—C(13)	1.377 (3)	C(13)—C(14)	1.358 (3)
C(14)—C(15)	1.419 (3)	C(15)—C(16)	1.504 (2)
C(16)—C(17)	1.413 (3)	C(17)—C(18)	1.368 (3)
C(1)—O(1)—C(2)	105.0 (1)	C(1)—O(2)—C(18)	105.0 (1)
C(11)—O(3)—C(12)	105.3 (2)	C(12)—O(4)—C(13)	104.8 (2)
C(5)—O(5)—C(8)	105.6 (1)	O(1)—C(1)—O(2)	107.7 (2)
O(1)—C(2)—C(3)	128.9 (2)	O(1)—C(2)—C(18)	109.8 (2)
C(3)—C(2)—C(18)	121.3 (2)	C(2)—C(3)—C(4)	118.3 (2)
C(3)—C(4)—C(5)	117.6 (2)	C(3)—C(4)—C(16)	121.5 (2)
C(5)—C(4)—C(16)	120.6 (2)	O(5)—C(5)—C(4)	106.8 (1)
O(5)—C(5)—C(6)	106.7 (1)	C(4)—C(5)—C(6)	117.1 (2)
C(5)—C(6)—C(7)	101.9 (2)	C(5)—C(6)—C(20)	113.3 (2)
C(7)—C(6)—C(20)	115.7 (2)	C(6)—C(7)—C(8)	99.1 (2)
C(6)—C(7)—C(19)	115.9 (2)	C(8)—C(7)—C(19)	118.8 (2)
O(5)—C(8)—C(7)	101.9 (1)	O(5)—C(8)—C(9)	113.5 (2)
C(7)—C(8)—C(9)	113.0 (2)	C(8)—C(9)—C(10)	113.3 (2)
C(8)—C(9)—C(15)	126.0 (2)	C(10)—C(9)—C(15)	120.4 (2)
C(9)—C(10)—C(11)	119.5 (2)	O(3)—C(11)—C(10)	129.5 (2)
O(3)—C(11)—C(13)	109.7 (2)	C(10)—C(11)—C(13)	120.7 (2)
O(3)—C(12)—O(4)	108.9 (2)	O(4)—C(13)—C(11)	109.5 (2)
O(4)—C(13)—C(14)	128.5 (2)	C(11)—C(13)—C(14)	122.0 (2)
C(13)—C(14)—C(15)	119.8 (2)	C(9)—C(15)—C(14)	117.4 (2)
C(9)—C(15)—C(16)	127.3 (2)	C(14)—C(15)—C(16)	115.2 (2)
C(4)—C(16)—C(15)	124.2 (2)	C(4)—C(16)—C(17)	117.9 (2)
C(15)—C(16)—C(17)	117.9 (2)	C(16)—C(17)—C(18)	118.7 (2)
O(2)—C(18)—C(2)	109.7 (2)	O(2)—C(18)—C(17)	128.1 (2)
C(2)—C(18)—C(17)	122.2 (2)		

The structures were solved and refined using the *SHELXTL* system (Sheldrick, 1986) on a Data General Eclipse Model 30 computer. Function minimized $\sum w(F_o - F_c)^2$. The scattering factors used in the *SHELXTL* programs are the analytical form given in *International Tables for X-ray Crystallography* (1974). The final positional parameters are given in Tables 2 and 3. Bond distances and angles are given in Tables 4 and 5.* The two molecules and the atomic numbering are shown in Figs. 1 and 2.

Discussion. Both molecules consist of substituted cyclooctadiene rings. The two double bonds require that the torsion angles at C(4)—C(16) and C(15)—C(9) be approximately 0°. Selected torsion angles for (1) and (2) are given in Table 6 together with values calculated from Spencer & Flippen-Anderson (1981) and the theoretical values of Anet

* Tables of anisotropic thermal parameters, H-atom parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52242 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

& Yavari (1978). Compound (1) is very close to the twist-boat-chair (TBC) or C_2 form described by Anet & Yavari (1978) and Allinger, Viskoch, Burkert & Yuh (1976). The formation of the epoxide in (2) tends to change the conformation slightly toward the twist-boat (TB) or C_1 form. The torsion angles found in the epoxide reported by Spencer & Flippen-Anderson (1981) are in good agreement with our results. The five-membered epoxide ring in (2) can be viewed as a distorted envelope with C(8) the flap or a distorted half-chair with the twofold axis passing through C(5) and the C(7)—C(8) bond.

The determination of the relative positions of the methyl groups was one reason for our study. As seen in Figs. 1 and 2, the methyl groups are approximately *cis* to each other. The C(19)—C(6)—C(7)—C(20) torsion angles are 59.6 and -73.3° for (1) and (2) respectively. Furthermore in (1) C(20) is approximately *cis* to O(5) of the acetate [C(20)—C(7)—C(8)—O(5) is -33.7°].

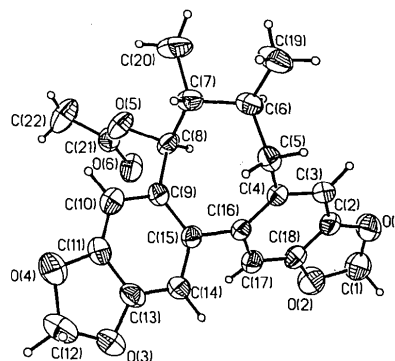


Fig. 1. A view of the 11,12,13,14-tetrahydro-12,13-dimethylcycloocta[1,2-*f*:3,4-*f'*]bis[1,3]benzodioxol-11-yl acetate molecule showing the atomic numbering and thermal ellipsoids (50% probability).

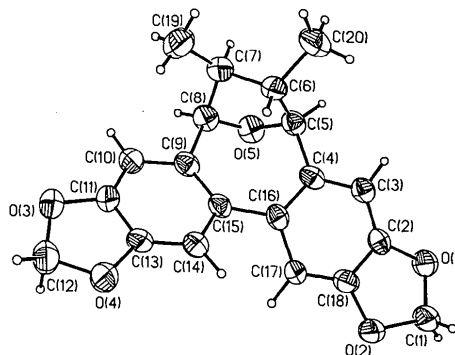


Fig. 2. A view of the 11,12,13,14-tetrahydro-12,13-dimethyl-11,14-epoxycycloocta[1,2-*f*:3,4-*f'*]bis[1,3]benzodioxole molecule showing the atomic numbering and thermal ellipsoids (50% probability).

Table 6. Selected *cis*-torsion angles ($^{\circ}$) for the cyclo-octadiene rings in three compounds compared to the theoretical values

Bond	(1)*	(2)*	SP†	TBC‡	TB‡
C(4)—C(5)	-96.2	64.1	69.0	-94	5
C(5)—C(6)	90.8	-130.1	-125.3	85	-82
C(6)—C(7)	-52.7	34.9	29.7	-59	59
C(7)—C(8)	84.3	72.8	77.4	85	52
C(8)—C(9)	-103.0	-89.1	-89.4	-94	-92
C(9)—C(15)	10.3	-12.5	-20.1	6	2
C(15)—C(16)	54.9	28.3	42.5	54	42
C(16)—C(4)	-2.4	-7.2	-18.6	6	4

* E.s.d.'s $\sim 0.2^{\circ}$.

† Values from Spencer & Flippen-Anderson (1981).

‡ Theoretical values for the twist-boat-chair, TBC, and twist-boat, TB, from Anet & Yavari (1978).

The dioxole rings are very close to the ideal cyclopentene envelope conformation given by Bucourt (1974). The slight distortions are probably related to the shorter C—O distances compared to C—C and a resultant flattening of the ring.

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Structure of Limonin

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Abstract. Limonoic acid 3,19:16,17-dilactone, $C_{26}H_{30}O_8$, $M_r = 470.52$, orthorhombic, $P2_12_12_1$, $a = 17.715$ (9), $b = 14.520$ (8), $c = 8.869$ (2) Å, $V = 2281$ (2) Å³, $Z = 4$, $D_x = 1.37$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.109$ mm⁻¹, $F(000) = 1000$, $T = 290$ K, $R = 0.065$ for 1929 observed reflections. The molecular structure of limonin, the bitter principle of citrus fruits, was determined by X-ray diffraction methods. The structure was essentially the same as epilimonol iodoacetate, the heavy-atom derivative of limonin. A structure energy calculation (MMP2) for limonin yields the same structure as that of the X-ray analysis.

Introduction. Limonin, the bitter principle of citrus fruits, has been focused upon for the biosynthesis of limonoid in citrus fruits (Hasegawa, Herman, Orme & Ou, 1986), the removal of bitterness from

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grapefruit (Show & Buslig, 1986), antifeedant activity against insects (Alford, Cullen, Storch & Bentley, 1987; Hassanali, Bentley, Ole Sitayo, Njoroge & Yatagai, 1986; Nakatani, Takao, Iwashita, Naoki & Hase, 1987, 1988), and inhibition of insect ecdysis (Kubo & Klocke, 1986).

After extensive chemical investigations (Arigoni *et al.*, 1960; Barton, Pradhan, Sternhell & Templeton, 1961), Arnott, Davie, Robertson, Sim & Watson (1961) determined the three-dimensional molecular structure of the heavy-atom derivative of limonin, epilimonol iodoacetate, by X-ray crystal analysis.

Since 1982 we have attempted the isolation of limonoids from *Evodia glauca* Miq. (Nakatani *et al.*, 1987, 1988) and produced a well shaped single crystal which was used for the X-ray analysis. An essentially identical structure to epilimonol iodoacetate was obtained. H atoms were found at all the