Acta Cryst. (1990). C46, 307–310

Structures of Garugamblin-I and Garugamblin-II: Two Natural Ansa Bridged Biaryl Ethers

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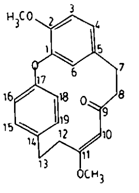
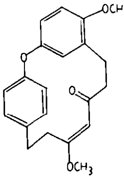
(*Received* 8 *March* 1989; *accepted* 31 *May* 1989)

Abstract. (I) Garugamblin-I; 4,12-dimethoxy-2-oxatricyclo[13.2.2.$1^{3,7}$]icosa-3,5,7(20),11,15,17(1),18-heptaen-10-one, $C_{21}H_{22}O_4$, $M_r = 338{\cdot}41$, monoclinic, $P2_1$, $a = 8{\cdot}416\,(1)$, $b = 10{\cdot}828\,(2)$, $c = 9{\cdot}657\,(1)$ Å, $\beta = 91{\cdot}36\,(1)°$, $V = 879{\cdot}8\,(7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, Cu $K\alpha$ ($\lambda = 1{\cdot}5418$ Å), $\mu = 6{\cdot}71$ cm^{-1}, $F(000) = 360$, m.p. 478–479 K $\{[\alpha]_D = +48°$ (0·2%) $CHCl_3\}$, $T = 295$ K, $R = 0{\cdot}044$, $wR = 0{\cdot}056$, for 1652 observed reflections $[I \geq 2{\cdot}5\sigma(I)]$. (II) Garugamblin-II; 15-methoxy-2,5,7-trioxatetracyclo[16.2.2.$0^{4,8}$.$1^{3,10}$]-tricosa-3,8,10(23),14,18,20(1),21-heptaen-13-one, $C_{21}H_{20}O_5$, $M_r = 352{\cdot}39$, monoclinic, $P2_1$, $a = 8{\cdot}403\,(2)$, $b = 10{\cdot}854\,(2)$, $c = 9{\cdot}662\,(2)$ Å, $\beta = 92{\cdot}08\,(2)°$, $V = 880{\cdot}7\,(7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}32$ g cm^{-3}, Cu $K\alpha$ ($\lambda = 1{\cdot}5418$ Å), $\mu = 7{\cdot}4$ cm^{-1}, $F(000) = 370$, m.p. 468–470 K $\{[\alpha]_D^{30°C} = +51°$ (0·23%) $CHCl_3\}$, $T = 295$ K, $R = 0{\cdot}039$, $wR = 0{\cdot}048$ for 1669 observed reflections $[I \geq 2{\cdot}5\sigma(I)]$. Garugamblin-I and -II are two naturally occurring ansa bridged biaryl ethers found in *Garuga gamblei* King. All four phenyl rings in (I) and (II) are distorted towards a boat conformation with ring *B* more puckered than ring *A* in both compounds. The dihedral angles between rings *A* and *B* are 79·6 (1) and 79·9 (1)° in (I) and (II) respectively. The ansa bridge makes angles with rings *A* and *B* of 57·7 (1), 57·6 (1)° in (I) and 53·8 (2), 54·0 (1)° in (II). The carbonyl group is perpendicular to ring *A* and parallel to ring *B* in both structures. These conformational features resemble those of active ansamycin antibiotics.

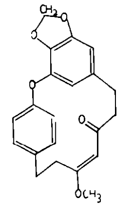
Introduction. Two novel macrocyclic biaryl ethers named garugamblin-I and -II have been isolated from the bark of *Garuga gamblei* King (Burs.). The extracts of *Garuga Pinnata* have found use in indigenous medicine to cure asthma, pulmonary infections and the opacity of conjuctiva. Garugamblin-I and -II are two more members of this class of compounds. Their structures have been deduced by NMR studies (Kalchhauser, Krishnamurthy, Talukdar & Schmid, 1988), but an unambiguous decision could not be made between the alternative structures (I), (I*a*) and (II), (II*a*) respectively. This paper describes X-ray crystallographic analyses, which establish the molecular structures as (I) and (II) respectively. The structural studies have been undertaken as a continuation of our work on natural products. Crystal structures of two related compounds, *viz.* garuganin-I (III) and garuganin-II (IV), have previously been reported (Pattabhi, Krishnaswamy & Gabe, 1984; Krishnaswamy, Pattabhi & Gabe, 1987).



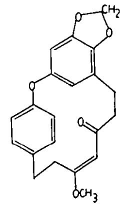
(I)



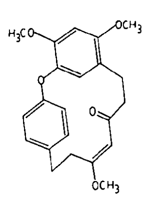
(I*a*)



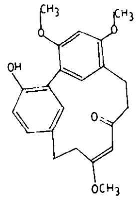
(II)



(II*a*)



(III)



(IV)

Experimental. (I) Transparent colourless crystals 0·2 × 0·15 × 0·13 mm from benzene. Intensity data on Enraf–Nonius CAD-4 automated diffractometer, $\omega/2\theta$ scan mode with $2 \leq 2\theta \leq 140°$, data corrected for direct beam polarization and Lorentz effects and for absorption (the mean transmission factor is 0·936

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† This article is contribution No. 743 from this Department.

0108-2701/90/020307-04$03.00

Table 1. *Positional parameters* (× 10^4) *and their e.s.d.'s given in parentheses*

	x	y	z	B_{eq} (Å^2)*
(I)				
O(1)	4708 (2)	005	6993 (1)	3·37 (4)
C(1)	3565 (3)	−0064 (3)	8011 (3)	2·68 (5)
C(2)	3855 (3)	−0920 (3)	9054 (3)	3·08 (5)
C(3)	2719 (4)	−1069 (3)	10069 (3)	3·55 (6)
C(4)	1306 (4)	−0396 (3)	10011 (3)	3·25 (6)
C(5)	1027 (3)	0468 (3)	8970 (3)	2·70 (5)
C(6)	2184 (3)	0634 (3)	7985 (3)	2·69 (5)
C(7)	−0485 (3)	1214 (3)	8876 (3)	3·47 (6)
C(8)	−1471 (3)	0968 (4)	7548 (3)	3·46 (6)
C(9)	−0977 (3)	1689 (3)	6269 (3)	2·99 (5)
O(9)	−0222 (3)	2644 (2)	6387 (2)	4·34 (5)
C(10)	−1547 (3)	1147 (3)	4963 (3)	3·28 (5)
C(11)	−1238 (3)	1543 (3)	3670 (3)	3·25 (6)
C(12)	−0252 (4)	2633 (3)	3263 (3)	3·53 (6)
C(13)	1295 (4)	2238 (3)	2537 (3)	3·25 (6)
C(14)	2419 (3)	1612 (3)	3537 (3)	2·94 (5)
C(15)	2280 (4)	0348 (3)	3819 (3)	3·13 (5)
C(16)	3099 (3)	−0182 (3)	4926 (3)	2·99 (5)
C(17)	4072 (3)	0546 (3)	5744 (3)	2·85 (5)
C(18)	4346 (3)	1779 (3)	5426 (3)	3·32 (6)
C(19)	3526 (4)	2296 (3)	4330 (3)	3·32 (6)
O(2)	5251 (3)	−1562 (3)	9019 (3)	4·60 (5)
C(20)	5361 (5)	−2640 (4)	9832 (5)	6·3 (1)
O(11)	−1799 (3)	0961 (3)	2514 (2)	4·56 (5)
C(21)	−2755 (5)	−0128 (5)	2662 (4)	5·8 (1)
(II)				
O(1)	4838 (2)	6050 (2)	1899 (2)	4·04 (4)
O(2)	5366 (2)	4352 (2)	4204 (2)	5·22 (4)
O(3)	3540 (3)	4022 (2)	5906 (2)	6·03 (5)
O(9)	−0131 (2)	8554 (2)	1378 (2)	4·41 (4)
O(11)	−1855 (2)	6913 (2)	−2504 (2)	4·79 (4)
C(1)	3735 (3)	5900 (2)	2913 (3)	3·20 (4)
C(2)	4038 (3)	5078 (3)	3964 (3)	3·61 (5)
C(3)	2948 (3)	4898 (3)	4973 (3)	4·09 (5)
C(4)	1512 (3)	5502 (3)	5004 (2)	3·89 (5)
C(5)	1208 (3)	6373 (2)	3952 (2)	3·12 (4)
C(6)	2317 (3)	6562 (2)	2936 (3)	3·07 (4)
C(7)	−0343 (3)	7088 (3)	3859 (2)	3·81 (5)
C(8)	−1320 (3)	6848 (3)	2523 (2)	3·47 (5)
C(9)	−0890 (3)	7594 (3)	1255 (2)	3·11 (4)
C(10)	−1519 (3)	7075 (3)	−0060 (2)	3·33 (5)
C(11)	−1256 (3)	7478 (3)	−1344 (3)	3·55 (5)
C(12)	−0280 (3)	8561 (3)	−1774 (3)	3·79 (5)
C(13)	1234 (3)	8174 (3)	−2520 (3)	3·82 (5)
C(14)	2401 (3)	7559 (2)	−1524 (3)	3·34 (4)
C(15)	3509 (3)	8259 (3)	−0750 (3)	3·89 (5)
C(16)	4379 (3)	7759 (3)	0355 (3)	3·83 (5)
C(17)	4144 (3)	6527 (3)	0666 (3)	3·40 (5)
C(18)	3161 (3)	5783 (2)	−0137 (3)	3·50 (5)
C(19)	2291 (3)	6302 (2)	−1234 (3)	3·55 (5)
C(20)	4866 (5)	349	5232 (4)	6·22 (8)
C(21)	−2810 (4)	5828 (4)	−2360 (3)	6·01 (8)

*$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab\cos\gamma B(1,2) + ac\cos\beta B(1,3) + bc\cos\alpha B(2,3)]$.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

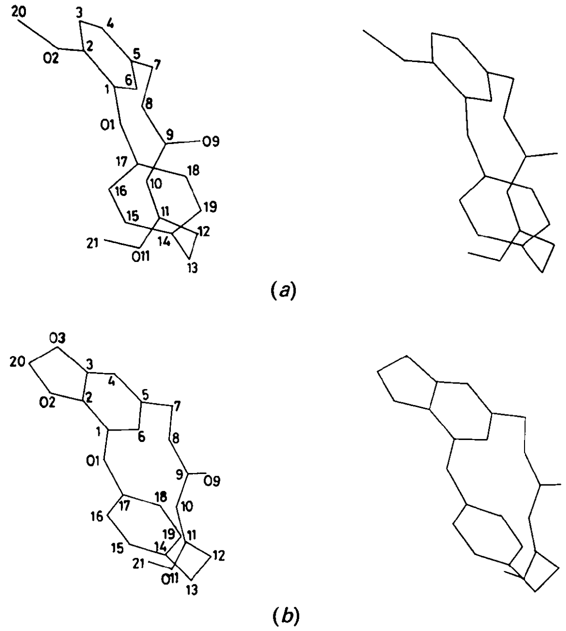
	(I)	(II)
O(1)—C(1)	1·397 (3)	1·383 (3)
O(1)—C(17)	1·412 (3)	1·406 (3)
C(1)—C(2)	1·387 (4)	1·369 (4)
C(1)—C(6)	1·386 (4)	1·392 (3)
C(2)—C(3)	1·394 (4)	1·375 (4)
C(2)—O(2)	1·366 (4)	1·379 (3)
C(3)—C(4)	1·395 (4)	1·375 (4)
C(4)—C(5)	1·390 (4)	1·404 (4)
C(5)—C(6)	1·388 (4)	1·393 (3)
C(5)—C(7)	1·507 (4)	1·517 (4)
C(7)—C(8)	1·534 (4)	1·527 (3)
C(8)—C(9)	1·527 (4)	1·523 (4)
C(9)—O(9)	1·217 (4)	1·225 (3)
C(9)—C(10)	1·462 (4)	1·471 (3)
C(10)—C(11)	1·351 (4)	1·342 (3)
C(11)—C(12)	1·500 (5)	1·501 (4)
C(11)—O(11)	1·357 (4)	1·359 (3)
C(12)—C(13)	1·553 (4)	1·543 (4)
C(13)—C(14)	1·498 (4)	1·505 (4)
C(14)—C(15)	1·401 (4)	1·397 (4)
C(14)—C(19)	1·403 (4)	1·397 (4)
C(15)—C(16)	1·383 (4)	1·383 (4)
C(16)—C(17)	1·373 (4)	1·387 (4)
C(17)—C(18)	1·391 (5)	1·375 (4)
C(18)—C(19)	1·369 (4)	1·385 (4)
O(2)—C(20)	1·410 (5)	1·438 (4)
O(11)—C(21)	1·436 (6)	1·435 (5)
C(3)—O(3)	—	1·390 (4)
O(3)—C(20)	—	1·432 (4)
C(1)—O(1)—C(17)	112·5 (2)	112·0 (2)
O(1)—C(1)—C(2)	117·4 (2)	119·4 (2)
O(1)—C(1)—C(6)	121·9 (2)	123·2 (2)
C(2)—C(1)—C(6)	120·7 (2)	117·5 (2)
C(1)—C(2)—C(3)	118·4 (3)	120·5 (2)
C(1)—C(2)—O(2)	117·3 (3)	128·7 (2)
C(3)—C(2)—O(2)	124·3 (3)	110·8 (2)
C(2)—C(3)—C(4)	120·6 (3)	123·7 (3)
C(3)—C(4)—C(5)	120·7 (3)	116·2 (3)
C(4)—C(5)—C(6)	118·3 (3)	120·0 (2)
C(4)—C(5)—C(7)	122·1 (2)	121·4 (2)
C(6)—C(5)—C(7)	119·6 (3)	118·5 (2)
C(1)—C(6)—C(5)	121·2 (3)	122·0 (2)
C(5)—C(7)—C(8)	113·3 (3)	113·4 (2)
C(7)—C(8)—C(9)	115·8 (3)	116·9 (2)
C(8)—C(9)—O(9)	120·7 (3)	120·8 (2)
C(8)—C(9)—C(10)	113·7 (3)	113·7 (2)
O(9)—C(9)—C(10)	125·6 (3)	125·4 (2)
C(9)—C(10)—C(11)	127·1 (3)	127·4 (2)
C(10)—C(11—C(12)	127·7 (3)	128·5 (2)
C(10)—C(11)—O(11)	122·8 (3)	123·2 (2)
C(12)—C(11)—O(11)	109·5 (3)	108·4 (2)
C(11)—C(12)—C(13)	112·1 (3)	112·6 (2)
C(12)—C(13)—C(14)	110·9 (2)	110·6 (2)
C(13)—C(14)—C(15)	120·8 (3)	120·5 (2)
C(13)—C(14)—C(19)	121·0 (3)	121·0 (2)
C(15)—C(14)—C(19)	117·8 (3)	118·1 (2)
C(14)—C(15)—C(16)	120·9 (3)	121·5 (3)
C(15)—C(16)—C(17)	119·1 (3)	118·1 (2)
O(1)—C(17)—C(16)	118·9 (3)	118·7 (2)
O(1)—C(17)—C(18)	119·3 (2)	119·0 (2)
C(16)—C(17)—C(18)	121·6 (3)	122·1 (2)
C(17)—C(18)—C(19)	118·7 (3)	118·9 (2)
C(14)—C(19)—C(18)	121·5 (3)	120·9 (2)
C(2)—O(2)—C(20)	116·9 (3)	103·4 (2)
C(11)—O(11)—C(21)	118·9 (3)	118·8 (2)
C(3)—O(3)—C(20)	—	104·2 (2)
O(3)—C(3)—C(2)	—	109·0 (2)
O(3)—C(3)—C(4)	—	127·3 (2)
O(2)—C(20)—O(3)	—	107·7 (2)

with a maximum and minimum of 0·992 and 0·894), $0 \leq h \leq 10$, $0 \leq k \leq 13$, $-11 \leq l \leq 11$; 1872 measured reflections with 1766 unique and 1652 observed with $I \geq 2{\cdot}5\sigma(I)$; cell constants from 24 reflections $20 \leq 2\theta \leq 68°$, three standard reflections monitored in every 100 reflections showed no significant variation in intensity, scan width using the relation ($A + B\tan\theta$), where A and B are 0·7 and 0·14 respectively, aperture width using ($4{\cdot}0 + 2\tan\theta$), and maximum time spent on any reflection is 30 s and the background count was half the scan time, structure solution by direct methods, H atoms from $\Delta\rho$ map, full-matrix least-squares refinement on F_o, non-H atoms anisotropic and H atoms isotropic to a final $R = 0{\cdot}044$ and $wR = 0{\cdot}056$ with individual weights based on counting statistics where $w = 4(F_o)^2/\sigma^2(F_o)^2$, $(\Delta/\sigma)_{max} = 0{\cdot}08$, $S = 2{\cdot}67$ for 313 parameters, final $\Delta\rho$ map had no peaks $\geq \pm 0{\cdot}19$ e Å^{-3}.

(II) As for (I), but with specific values described below. Transparent colourless needles 0·3 × 0·15 × 0·13 mm from benzene, $\omega/2\theta$ scan mode with $2 \leq 2\theta \leq 120°$, absorption correction with an average transmission factor of 0·977 and maximum and minimum values of 0·995 and 0·960), $0 \le h \le 10$, $0 \le k \le 13$, $-11 \le l \le 11$; 1899 reflections measured, 1764 unique and 1669 observed with $I \ge 2{\cdot}5\sigma(I)$, cell constants from 25 reflections $14 \le 2\theta \le 50°$, three standard reflections monitored in every 100 reflections showed no significant variation in intensity. Non-H atoms anisotropic and H atoms isotropic gave a final $R = 0{\cdot}039$ and $wR = 0{\cdot}048$ with individual weights based on counting statistics where $w = 4(F_o)^2/\sigma^2(F_o)^2$, $(\Delta/\sigma)_{\max} = 0{\cdot}06$, $S = 2{\cdot}29$ for 234 parameters, final $\Delta\rho$ map had no peaks $\ge \pm 0{\cdot}30$ e Å^{-3}. All calculations on VAX 11/730 computing system using *SDP* package (Frenz, 1978).

Discussion. Atomic positions with equivalent isotropic temperature factors for the non-H atoms of both structures are given in Table 1.* Fig. 1 shows stereoviews of the two structures (Motherwell & Clegg, 1978). Fig. 2 shows the thermal ellipsoids drawn at the 50% probability level (Johnson, 1965). The bond lengths and angles for both (I) and (II) are given in Table 2 and are comparable with each other and with those observed in garuganin-I (Pattabhi, Krishnaswamy & Gabe, 1984). The diphenyl ether group is characterized by the torsion angles about the ether bonds, *i.e.* C(1)—O(1)—C(17)—C(18) 102·7 (3) and C(6)—C(1)—O(1)—C(17) −22·7 (3)° in (I) and C(1)—O(1)—C(17)—C(16) 103·6 (3) and C(6)—C(1)—O(1)—C(17) −23·7 (3)° in (II). The 'skewed' conformation defined by values of *ca* 90 and 0° for these angles is found to be preferred (Cody, 1974). The bond angles at the ether oxygen O(1), of 112·5 (2) and 112·0 (2)° for (I) and (II), deviate considerably from expected values, but are in good agreement with the value observed in garuganin-I (Pattabhi, Krishnaswamy & Gabe, 1984).

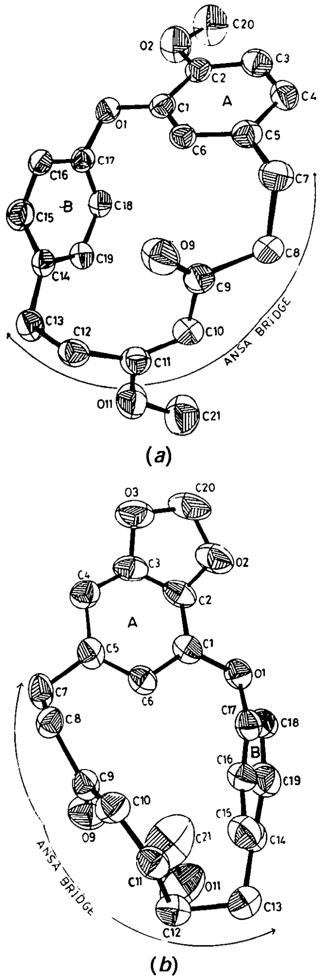
In both structures the aromatic rings *A* and *B* are deformed towards a boat conformation, with the deformation in *B* being more significant than in *A* (Table 3). Similar deformations are observed in garuganin-I (Pattabhi, Krishnaswamy & Gabe, 1984), [3.3]paracyclophane (Gantzel & Trueblood, 1965) and [2.2]paracyclophane (Coulter & Trueblood, 1963).



(*a*)

(*b*)

Fig. 1. Stereoviews of (*a*) the molecule GRGNI and (*b*) the molecule GRGNII.



(*a*)

(*b*)

Fig. 2. Thermal ellipsoids drawn at the 50% probability level of (*a*) GRGNI and (*b*) GRGNII.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, torsion angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52283 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Torsion angle* (°) *comparison*

The atom numbering is according to garugambline-I.

	(I)	(II)	Garuganin[a]
C(1)—C(2)—O(2)—C(20)	−163·5 (3)	−167·0 (3)	−184·4 (3)
C(6)—C(1)—O(1)—C(17)	−22·7 (3)	−23·7 (3)	14·2 (3)
C(12)—C(13)—C(14)—C(19)	−88·4 (3)	−87·7 (3)	−82·6 (3)
C(12)—C(11)—O(11)—C(21)	178·9 (3)	178·7 (3)	−175·1 (3)
C(10)—C(11)—O(11)—C(21)	−0·4 (5)	−0·3 (4)	−3·0 (4)
C(7)—C(8)—C(9)—O(9)	−21·9 (4)	−19·7 (4)	−23·3 (4)
C(9)—C(8)—C(7)—C(5)	−83·4 (4)	−83·8 (3)	−78·4 (3)
C(8)—C(7)—C(5)—C(6)	62·3 (3)	59·6 (4)	28·6 (4)
Ring *A*			
C(1)—C(2)—C(3)—C(4)	1·8 (5)	0·3 (4)	−0·5 (4)
C(2)—C(3)—C(4)—C(5)	−2·3 (5)	−2·0 (4)	0·4 (4)
C(3)—C(4)—C(5)—C(6)	0·5 (5)	1·6 (4)	0·1 (4)
C(4)—C(5)—C(6)—C(1)	1·7 (4)	0·6 (4)	−0·6 (4)
C(5)—C(6)—C(1)—C(2)	−2·3 (4)	−2·3 (4)	0·5 (4)
C(6)—C(1)—C(2)—C(3)	0·5 (4)	1·8 (4)	−0·0 (4)
Ring *B*			
C(14)—C(15)—C(16)—C(17)	−0·7 (4)	1·1 (4)	−0·3 (3)
C(15)—C(16)—C(17)—C(18)	−4·9 (4)	4·2 (4)	−4·6 (3)
C(16)—C(17)—C(18)—C(19)	5·1 (4)	−4·7 (4)	4·6 (3)
C(17)—C(18)—C(19)—C(14)	0·4 (4)	−0·2 (4)	0·3 (3)
C(18)—C(19)—C(14)—C(15)	−5·8 (4)	5·2 (4)	−5·0 (4)
C(19)—C(14)—C(15)—C(16)	5·9 (4)	−5·6 (4)	4·9 (3)

Reference: (*a*) Pattabhi, Krishnaswamy & Gabe (1984).

The two aromatic groups *A* and *B* make dihedral angles of 79·6 (1) and 79·9 (1)° in (I) and (II) while the ansa bridge (see Fig. 2) plane makes angles of 57·7 (1), 53·8 (2), and 57·6 (1), 54·0 (1)° with rings *A*, *B* in (I) and (II) respectively. The corresponding values for garuganin-I are 95·3, 45·6, 57·2° (Pattabhi, Krishnaswamy & Gabe, 1984). The carbonyl group is perpendicular to ring *A* [87·9 (1)° in both structures] and approximately parallel to ring *B* [37·4 (2), 36·2 (1)° in (I) and (II)].

Table 2 shows that the bond angles C(12)—C(11)—O(11) and C(8)—C(9)—C(10) in both structures are significantly compressed, while the related angles C(12)—C(11)—C(10), C(11)—C(10)—C(9) are significantly expanded from the values expected at a C(sp^2) centre. This tendency has been observed in garuganin-I (Pattabhi, Krishnaswamy & Gabe, 1984); in vinyl esters (Rappoport & Avramovitch, 1982), methyl (*E*)- and (*Z*)-β-chloro-α-cyano-*p*-nitrocinnamates (Strauss & Rappoport, 1982) and in *trans*-3-*p*-tolylthiocinnamic acid (Stephens, 1970). The bond C(9)—C(10) [1·462 (4) and 1·471 (3) Å in (I) and (II)] exhibits partial double bond character. Some relevant torsion angles observed in the present study have been compared (Table 3) with those in garuganin-I (Pattabhi, Krishnaswamy & Gabe, 1984), and are indicative of the conformational similarity of all three compounds. Indeed, the structural features of these macrocyclic diphenyl ethers, in comparison with ansamycin antibiotics, suggest that (I) and (II) may show antibiotic activity like garuganin-I (III). Biochemical studies to verify this possibility are being carried out.

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Structure of Quercetin Dihydrate

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(*Received* 17 *April* 1989; *accepted* 1 *June* 1989)

Abstract. 2-(3,4-Dihydroxyphenyl)-3,5,7-trihydroxy-4*H*-1-benzopyran-4-one, $C_{15}H_{10}O_7.2H_2O$, $M_r = 338·27$, triclinic, $P\bar{1}$, $a = 14·976$ (5), $b = 13·031$ (5), $c = 3·7198$ (4) Å, $\alpha = 85·69$ (1), $\beta = 83·97$ (2), $\gamma = 71·99$ (3)°, $V = 685·8$ (3) Å^3, $Z = 2$, $D_m = 1·680$ (7), $D_x = 1·638$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu =$

0108-2701/90/020310-04$03.00