plane, passing through C(1) perpendicular to C(1)—C(7); and $\varphi_3 = 1{\cdot}0$ (4)°, the corresponding angle of rotation for ring C(7)–(12). A similar value for $\varphi_1 = 30{\cdot}4°$ has been obtained for 3′-iodobiphenyl-4-carboxylic acid (Sutherland & Mottram, 1972) with corresponding values of 0·7 and 0·4° for φ_2 and φ_3. φ_1 is significantly smaller than the values of 36·3 (6)° in 3′-fluorobiphenyl-4-carboxylic acid (Sutherland & Rawas, 1983) and 38·6° in 4-acetyl-3′-bromobiphenyl (Sutherland & Hoy, 1969*b*). These variations are probably due to intermolecular forces arising from the different packing arrangements in the compounds.

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Garuganin-I,* an Antibiotic, $C_{22}H_{24}O_5$†

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Abstract. $M_r = 368$, monoclinic, $P2_1/a$, $a = 9{\cdot}3993$ (5), $b = 15{\cdot}0431$ (8), $c = 13{\cdot}7949$ (8) Å, $\beta = 102{\cdot}577$ (4)°, $V = 1903{\cdot}7$ Å^3 at 296 K, $Z = 4$, $D_m = 1{\cdot}30$ (1), $D_x = 1{\cdot}285$ g cm^{-3}, $\lambda(\text{Mo } K\alpha_1) = 0{\cdot}70932$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}84$ cm^{-1}, $F(000) = 784$. The structure was refined to a final $R(F) = 0{\cdot}040$ for 1962 observed reflections. Most of the bond lengths and angles agree with standard values. The structure is stabilized by van der Waals forces.

Introduction. The title compound is an antibiotic obtained from Hindustan Antibiotics Ltd, India. A study of garuganin-I (GRGN), a member of a series of antibiotics, was undertaken to establish a probable structure–activity relationship in this class of antibiotic. Garuganin-I, a plant product, shows anti-asthmatic properties and has a 15-membered macrocyclic ring. Other antibiotics such as rifamycin B (Brufani, Fedeli, Giacomello & Vaciago, 1964) and erythromycin A (Harris, McGeachin & Mills, 1965) also have large rings, and the conformation of these rings is of interest in establishing the structure–activity relationships.

Experimental. Crystal size 0·3 × 0·25 × 0·25 mm, $\theta/2\theta$ scan with line-profile analysis (Grant & Gabe, 1978), $2\theta_{\max} = 50°$, Picker four-circle automatic diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, no absorption correction, data corrected for direct-beam polarization (Le Page, Gabe & Calvert, 1979) and Lorentz effects, unit-cell parameters determined from least-squares refinement of measured angle values for 48 reflections with $20 < \theta < 25°$. 3366 independent reflections for $2\theta < 50°$ with $-11 < h < 10$, $0 < k < 17$, $0 < l < 16$, 1962 with $I_{\text{net}} > 2{\cdot}5\sigma(I_{\text{net}})$, 1404 unobserved. Structure solved using *MULTAN* (Germain, Main & Woolfson, 1971), 446 E values $> 1{\cdot}5$ for phase generation. Full-matrix least-squares refinement on F; isotropic temperature factors gave $R(F) = 0{\cdot}14$; anisotropic refinement gave $R(F) = 0{\cdot}08$. Hydrogen positions (from ΔF synthesis) and geometry were refined with isotropic temperature factors; final $R(F) = 0{\cdot}040$, $R_w(F) = 0{\cdot}028$ with $w = 1/\sigma^2(F_o)$ based on counting statistics; $R(\text{all}) = 0{\cdot}086$, $R_w(\text{all}) = 0{\cdot}029$;

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[*] The IUPAC name is 4,6,12-trimethoxy-2-oxatricyclo[13.2.2.1^{3,7}]icosa-3,5,7(20),11,15,17,18-heptaen-10-one.

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Table 1. *Atomic parameters and their e.s.d.'s in parentheses*

B_{eq} (Å^2) is the arithmetic mean of the principal axes of the thermal ellipsoid.

	x	y	z	B_{eq}
C(1)	0·3983 (4)	0·52818 (19)	0·65577 (22)	4·3
O(2)	0·34103 (21)	0·46877 (13)	0·57915 (14)	5·8
C(3)	0·3800 (3)	0·38038 (18)	0·60328 (20)	4·2
C(4)	0·3028 (3)	0·31572 (19)	0·54262 (20)	4·1
C(5)	0·3375 (3)	0·22720 (20)	0·56276 (21)	4·1
C(6)	0·4474 (3)	0·20430 (18)	0·64310 (20)	3·9
C(7)	0·5254 (3)	0·26795 (18)	0·70542 (19)	3·7
C(8)	0·6396 (4)	0·24248 (21)	0·79628 (24)	4·5
C(9)	0·7582 (4)	0·31090 (21)	0·83116 (24)	4·3
C(10)	0·7251 (3)	0·39022 (21)	0·88930 (20)	4·4
C(11)	0·8237 (3)	0·46588 (22)	0·88738 (22)	4·5
C(12)	0·8162 (3)	0·54739 (21)	0·92347 (21)	4·7
C(13)	0·7068 (4)	0·58479 (24)	0·9757 (3)	5·4
C(14)	0·6189 (4)	0·66192 (23)	0·91673 (25)	5·4
C(15)	0·5320 (3)	0·62625 (18)	0·81950 (22)	4·3
C(16)	0·3993 (4)	0·58510 (22)	0·8165 (3)	5·2
C(17)	0·3324 (4)	0·53581 (23)	0·7346 (3)	5·2
C(18)	0·5226 (4)	0·57303 (21)	0·65377 (23)	4·5
C(19)	0·5898 (4)	0·62248 (20)	0·73573 (24)	4·5
C(20)	0·4887 (3)	0·35581 (20)	0·68270 (21)	4·4
O(4)	0·19584 (21)	0·34559 (12)	0·46515 (14)	5·3
C(21)	0·1078 (5)	0·2804 (3)	0·4058 (3)	5·9
O(6)	0·48388 (20)	0·11739 (12)	0·66812 (13)	5·1
C(22)	0·4141 (7)	0·0498 (3)	0·6028 (4)	7·4
O(10)	0·62784 (22)	0·38823 (14)	0·93503 (14)	6·3
O(12)	0·9126 (3)	0·61303 (14)	0·91382 (16)	6·1
C(23)	1·0227 (5)	0·5952 (3)	0·8585 (4)	6·7
H(5)	0·2835 (22)	0·1814 (14)	0·5206 (15)	3·3 (6)
H(81)	0·587 (3)	0·2327 (17)	0·8530 (18)	6·3 (8)
H(82)	0·688 (3)	0·1857 (16)	0·7826 (17)	5·3 (7)
H(91)	0·7968 (24)	0·3340 (15)	0·7762 (15)	4·1 (7)
H(92)	0·845 (3)	0·2804 (16)	0·8778 (17)	5·7 (7)
H(11)	0·9000 (24)	0·4537 (15)	0·8532 (16)	4·0 (7)
H(131)	0·641 (3)	0·5328 (18)	0·9869 (19)	6·7 (9)
H(132)	0·758 (3)	0·6084 (17)	1·0410 (19)	7·1 (8)
H(141)	0·696 (3)	0·7097 (17)	0·9033 (18)	6·0 (8)
H(142)	0·550 (3)	0·6885 (17)	0·9600 (18)	7·2 (8)
H(16)	0·354 (3)	0·5891 (18)	0·8772 (20)	7·4 (9)
H(17)	0·242 (3)	0·5033 (18)	0·7395 (20)	8·0 (10)
H(18)	0·5648 (23)	0·5679 (14)	0·6017 (15)	3·3 (6)
H(19)	0·678 (3)	0·6528 (16)	0·7366 (18)	5·8 (8)
H(20)	0·5414 (22)	0·4017 (14)	0·7227 (15)	3·9 (6)
H(211)	0·069 (3)	0·2408 (17)	0·4446 (17)	5·3 (8)
H(212)	0·031 (3)	0·3147 (20)	0·3620 (21)	8·6 (10)
H(213)	0·172 (3)	0·2363 (21)	0·3704 (21)	9·5 (11)
H(221)	0·461 (3)	−0·0091 (19)	0·6292 (20)	7·4 (9)
H(222)	0·421 (4)	0·0647 (22)	0·5371 (23)	8·2 (12)
H(223)	0·315 (3)	0·0521 (24)	0·590 (3)	10·4 (15)
H(231)	1·094 (3)	0·5441 (23)	0·8942 (24)	11·3 (13)
H(232)	0·974 (3)	0·5759 (21)	0·7901 (22)	9·6 (12)
H(233)	1·077 (4)	0·6541 (23)	0·8584 (24)	11·5 (13)

goodness of fit = 2·84. Final difference map had no peaks $> 0{\cdot}2\ \mathrm{e}\ \text{Å}^{-3}$; $(\Delta/\sigma)_{\max} = 0{\cdot}2$, $(\Delta/\sigma)_{\text{mean}} = 0{\cdot}1$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations performed using the NRC-PDP-8e system of programs (Larson & Gabe, 1978) adapted for the VAX computer.

Discussion. Atomic positions and equivalent isotropic temperature factors are listed in Table 1.* Bond lengths and angles are shown in Fig. 1, and a stereoview of the molecule in Fig. 2. Most of the lengths and angles agree with the standard values within experimental error. However, the bond length C(1)—C(18), 1·354 (3) Å, and angles C(20)—C(7)—C(6) and C(4)—C(3)—O(2) deviate significantly (0·026 Å and 3·3°) from the standard values. Further, the angles C(13)—C(12)—C(11) and C(12)—C(11)—C(10) are 128·9 and 128·4° respectively. Similar values for C—C—C angles have been observed in cleavamine methiodide (Camerman & Trotter, 1964) and retrorsine hydrobromide ethanol solvate (Stoeckli-Evans, 1979).

The phenyl group I [C(3) to C(7) and C(20)] is planar ($\chi^2 = 4{\cdot}5$) while phenyl group II [C(15) to C(19) and C(1)] shows significant non-planarity ($\chi^2 = 372$). Atoms O(2) and C(14) deviate from plane II by 0·218 (3) and 0·340 (3) Å and are on the same side of

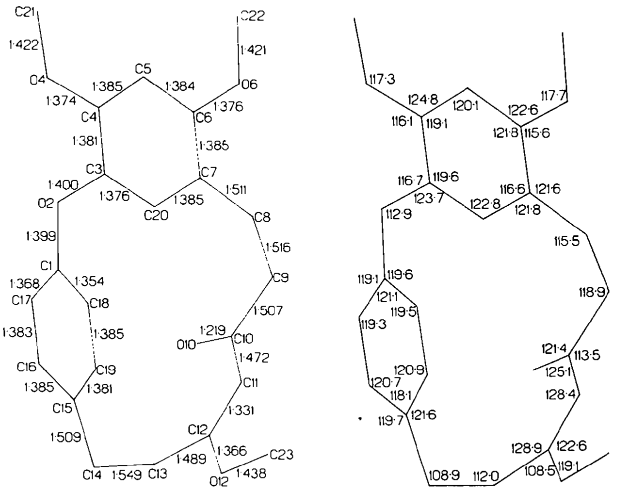

Fig. 1. Bond lengths (Å) and angles (°) observed in garuganin-I. E.s.d.'s range from 0·004 to 0·005 Å for C—C bonds and 0·003 to 0·004 Å for C—O bonds and close to 0·3° for all angles.

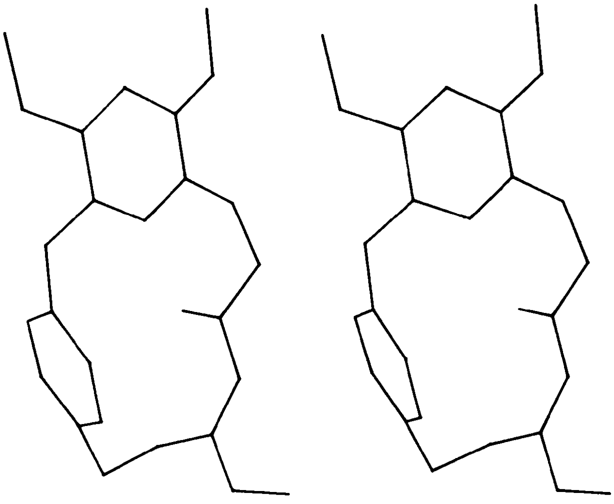

Fig. 2. Stereoview of the molecule.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39124 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Torsion angles of interest* (°)

C(3)–O(2)–C(1)–C(18)	97·4 (3)	C(12)–C(11)–C(10)–O(10)	−8·6 (3)
O(2)–C(1)–C(18)–C(19)	−171·4 (3)	C(12)–C(11)–C(10)–C(9)	173·6 (3)
C(1)–C(18)–C(19)–C(15)	0·3 (3)	C(11)–C(10)–C(9)–C(8)	−158·8 (3)
C(18)–C(19)–C(15)–C(14)	166·1 (3)	C(10)–C(9)–C(8)–C(7)	78·4 (3)
C(19)–C(15)–C(14)–C(13)	−88·4 (3)	C(9)–C(8)–C(7)–C(20)	−28·6 (4)
C(15)–C(14)–C(13)–C(12)	63·7 (3)	C(8)–C(7)–C(20)–C(3)	−176·6 (3)
C(14)–C(13)–C(12)–C(11)	−116·7 (3)	C(7)–C(20)–C(3)–O(2)	179·9 (2)
C(14)–C(13)–C(12)–O(12)	61·3 (3)	C(20)–C(3)–O(2)–C(1)	−14·2 (3)
C(13)–C(12)–C(11)–C(10)	0·1 (3)		

the plane. The angle between these planes is 95·3 (3)°. The least-squares plane through C(12), C(11), C(10) and O(10) also shows deviation from planarity (χ^2 = 244); C(9) and C(13) are 0·105 (2) and 0·135 (3) Å from this plane whereas O(12) is essentially on the plane [deviation 0·005 (3) Å].

The torsion angles around the macrocylic ring are given in Table 2. Some of the unusual geometry observed in the present study may be related to the electronic properties of the C(9)···C(13) segment. However, further analysis will only become possible when more structures in this class of antibiotics are known. The structure is stabilized by van der Waals forces.

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Structure of 4-(*p*-Chlorobenzoyl)pyridine,* $C_{12}H_8ClNO$

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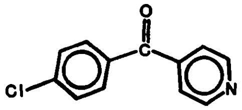
Abstract. $M_r = 217{\cdot}66$, monoclinic, $P2_1$, $a = 21{\cdot}585\ (4)$, $b = 5{\cdot}762\ (1)$, $c = 3{\cdot}843\ (1)$ Å, $\beta = 90{\cdot}27\ (2)°$, $V = 477{\cdot}99$ Å^3, $Z = 2$, $D_x = 1{\cdot}512$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3{\cdot}6$ cm^{-1}, $F(000) = 224$, $T = 110$ K. Final $R = 0{\cdot}0472$ for 1853 observed reflections. The molecular structure shows a twist in the essentially planar aromatic rings giving a dihedral angle of 52·2°; the repulsions between the non-bonded atoms are thus minimized. The carbonyl portion of the molecule exhibits a high degree of planarity and an increase in the endocyclic angle opposite the chlorine substituent is observed.

Introduction. The title compound (I) is one of a series of substituted benzophenones being studied in our laboratory (Syed, Stevens & Klein, 1984; Syed & Stevens, 1984). Structure determination was undertaken as a continuation of a program to determine the geometry of compounds containing carbonyl groups. The effect of an electronegative group substituted for an H atom on a phenyl ring has been observed.



(I)

Experimental. Sample recrystallized from equal volumes acetone and tetrahydrofuran, approximate dimensions 0·28 × 0·13 × 0·40 mm, Enraf–Nonius CAD-4 diffractometer, graphite-crystal monochromator, Mo $K\alpha$ radiation, temperature maintained at constant 110 K, lattice parameters from 25 reflections with $20 < 2\theta < 44°$, systematic absences: $0k0$, $k = 2n + 1$, absorption as a function of ψ observed to be insignificant and no correction applied, reflections measured within ranges $2 \leq 2\theta < 70°$, $-34 \leq h \leq 34$, $0 \leq k \leq 9$, $0 \leq l \leq 6$, ω:2θ scans; intensities of three standard reflections decreased by 1·2% and were used to correct data; 2315 total reflections measured, 1853

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* *p*-Chlorophenyl 4-pyridyl ketone.

† On leave from Tata Iron & Steel Company, Jamshedpur, India.

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