Table 1. *Atomic coordinates* ($\times 10^4$) *and* U_{eq} ($Å^2 \times 10^4$)

	x	y	z	U_{eq}*
Cl	1826 (1)	10496 (2)	2470 (2)	880 (8)
O(1)	6378 (4)	5586 (4)	6373 (5)	720 (15)
O(2)	8547 (3)	6793 (4)	4431 (4)	600 (12)
C(1)	6278 (4)	8655 (6)	3514 (5)	476 (16)
C(2)	4636 (5)	8743 (6)	3653 (7)	542 (20)
C(3)	3895 (5)	10399 (6)	2335 (7)	573 (20)
C(4)	4758 (6)	11944 (7)	911 (8)	683 (24)
C(5)	6398 (6)	11876 (7)	800 (7)	715 (23)
C(6)	7167 (5)	10216 (6)	2101 (6)	575 (21)
C(7)	7034 (5)	6859 (6)	4940 (6)	527 (19)
C(8)	9426 (5)	5087 (6)	5715 (6)	591 (18)
C(9)	10305 (8)	5417 (10)	7306 (9)	810 (33)

* Hamilton (1959).

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

Cl–C(3)	1·736 (4)	C(2)–C(3)	1·382 (6)
O(1)–C(7)	1·201 (5)	C(3)–C(4)	1·372 (6)
O(2)–C(7)	1·339 (5)	C(4)–C(5)	1·376 (7)
O(2)–C(8)	1·459 (5)	C(5)–C(6)	1·392 (6)
C(1)–C(2)	1·382 (6)	C(8)–C(9)	1·522 (9)
C(1)–C(6)	1·385 (5)	C(8)–C(8′)	1·528 (7)
C(1)–C(7)	1·480 (5)		

C(7)–O(2)–C(8)	117·4 (3)	C(4)–C(5)–C(6)	121·3 (5)
C(6)–C(1)–C(7)	122·0 (4)	C(1)–C(6)–C(5)	119·8 (4)
C(2)–C(1)–C(7)	117·6 (4)	O(2)–C(7)–C(1)	112·0 (4)
C(2)–C(1)–C(6)	120·4 (4)	O(1)–C(7)–C(1)	124·8 (4)
C(1)–C(2)–C(3)	118·9 (4)	O(1)–C(7)–O(2)	123·2 (4)
Cl–C(3)–C(2)	119·0 (4)	C(2)–C(8)–C(8′)	103·2 (3)
C(2)–C(3)–C(4)	121·3 (5)	O(2)–C(8)–C(9)	109·3 (5)
Cl–C(3)–C(4)	119·7 (4)	C(9)–C(8)–C(8′)	112·6 (4)
C(3)–C(4)–C(5)	119·9 (5)		

C(2)–C(1)–C(7)–O(1)	−11·0 (7)	O(1)–C(7)–O(2)–C(8)	−0·8 (5)
C(2)–C(1)–C(7)–O(2)	168·0 (4)	C(7)–O(2)–C(8)–C(9)	−90·0 (5)
C(6)–C(1)–C(7)–O(1)	168·1 (5)	C(7)–O(2)–C(8)–C(8′)	149·9 (4)
C(6)–C(1)–C(7)–O(2)	−13·0 (6)	O(2)–C(8)–C(8′)–C(9′)	−62·3 (5)
C(1)–C(7)–O(2)–C(8)	−179·7 (4)	C(9)–C(8)–C(8′)–C(9′)	180·0

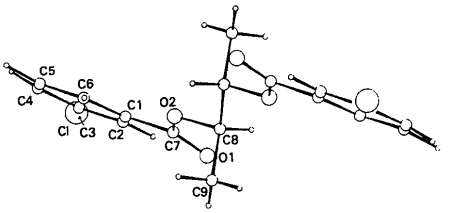

Fig. 1. Projection of the molecule viewed down **a**.

Related literature. Bocelli, Ugozzoli & Grenier-Loustalot (1985).

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Stereochemical Studies of Oligomers. XVII.* Dibenzyl Phthalate

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Abstract. $C_{22}H_{18}O_4$, $M_r = 346·4$, orthorhombic, $P2_12_12_1$, $a = 15·731$ (3), $b = 10·932$ (3), $c = 10·646$ (2) Å, $V = 1830·8$ (7) Å^3, $Z = 4$, $D_x = 1·26$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6·63$ cm^{-1}, $F(000) = 728$, room temperature, $R = 0·063$ for 1301 observed reflections. The molecule contains five planar parts: the three aromatic rings and the two carboxylic groups. These last two planar fragments are tilted by 57·7 (2) and 115·4 (2)° with respect to the central aromatic ring respectively and form dihedral angles of 108·1 (3) and 85·0 (2)° with respect to their respective terminal rings. In their turn, the planes of these last

* Part XVI: Bocelli & Grenier-Loustalot (1986).

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rings form a dihedral angle of 116·3 (2)° to each other. Finally the dihedral angles formed by the central ring plane with the aromatic rings are 108·4 (3) and 38·8 (2)° respectively. The O(1) and O(3) atoms are *cis* with respect to the central ring.

Experimental. Crystals recrystallized from a 1:1 cyclohexane:methanol mixture, one crystal, cut from a larger one, approximately a prism 0·4 × 0·6 × 0·8 mm, used for data collection; Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer using Ni-filtered Cu $K\alpha$ radiation. All data measured using a local version (Belletti, Ugozzoli, Cantoni & Pasquinelli, 1979) of the Lehmann & Larsen (1974) method, $2 \le \theta \le 70°$, a standard reflection, measured every 50 counts, did not show any decrease in intensity, corrections for Lorentz and polarization effects, absorption ignored. A total of 1999 unique reflections collected, $0 \le h \le 18$, $0 \le k \le 12$, $0 \le l \le 12$, 1305 considered observed at $2\sigma(I)$ level, preliminary cell dimensions determined by least squares for angular values of 21 reflections accurately measured on the diffractometer, structure solved by direct methods and refined by block-matrix least squares with *SHELX*76 (Sheldrick, 1976), first with isotropic temperature factors and then with anisotropic ones, most H atoms located in a difference Fourier map, two put in their theoretical position and all refined isotropically. Four reflections, probably affected by extinction, omitted in last cycles, final $R = 0·063$ and $wR = 0·068$ with $w = 1·0/(\sigma^2F + 0·02136F^2)$, $\sum w(\Delta F)^2$ minimized, maximum peak in final ΔF map = 0·23 e Å^{-3}, $(\Delta/\sigma)_{max} = 0·506$, scattering factors of *SHELX*.

Table 1. *Atomic fractional coordinates* (×10⁴) *and* U_{eq} (Å² ×10⁴)

	x	*y*	*z*	U_{eq}*
O(1)	2165 (4)	5339 (5)	1059 (5)	845 (20)
O(2)	3085 (3)	3804 (4)	828 (5)	652 (15)
O(3)	2707 (3)	3389 (4)	3407 (4)	648 (14)
O(4)	3971 (3)	2013 (4)	−757 (5)	610 (14)
C(1)	3496 (4)	5444 (6)	2132 (7)	611 (21)
C(2)	3815 (4)	4851 (6)	3179 (6)	549 (19)
C(3)	4454 (5)	5393 (8)	3864 (9)	741 (28)
C(4)	4741 (5)	6536 (8)	3462 (12)	884 (36)
C(5)	4413 (7)	7123 (8)	2465 (12)	978 (43)
C(6)	3766 (6)	6583 (7)	1784 (8)	804 (32)
C(7)	2807 (5)	4872 (6)	1298 (6)	618 (20)
C(8)	2468 (6)	3109 (9)	115 (9)	846 (34)
C(9)	2806 (4)	1879 (7)	−55 (6)	638 (23)
C(10)	3145 (6)	1464 (10)	−1184 (8)	792 (31)
C(11)	3463 (5)	313 (8)	−1307 (8)	813 (29)
C(12)	3464 (5)	−452 (8)	−302 (8)	763 (28)
C(13)	3149 (6)	−94 (7)	796 (9)	791 (29)
C(14)	2815 (5)	1065 (8)	949 (7)	708 (26)
C(15)	3401 (4)	3677 (6)	3592 (5)	501 (17)
C(16)	3628 (4)	1868 (7)	4752 (9)	690 (26)
C(17)	4325 (3)	1248 (5)	5450 (4)	599 (14)
C(18)	4485 (4)	5 (6)	5263 (6)	730 (21)
C(19)	5111 (5)	−574 (6)	5904 (7)	928 (26)
C(20)	5618 (4)	19 (11)	6725 (7)	1068 (34)
C(21)	5481 (5)	1258 (8)	6898 (7)	948 (27)
C(22)	4863 (4)	1848 (7)	6266 (6)	756 (22)

* Hamilton (1959).

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

O(1)–C(7)	1·160 (10)	C(8)–C(9)	1·457 (12)
O(2)–C(7)	1·343 (8)	C(9)–C(10)	1·391 (11)
O(2)–C(8)	1·448 (11)	C(9)–C(14)	1·391 (11)
O(3)–C(15)	1·153 (8)	C(10)–C(11)	1·360 (13)
O(4)–C(15)	1·367 (8)	C(11)–C(12)	1·358 (12)
O(4)–C(16)	1·439 (9)	C(12)–C(13)	1·329 (13)
C(1)–C(2)	1·384 (10)	C(13)–C(14)	1·381 (12)
C(1)–C(6)	1·367 (10)	C(16)–C(17)	1·488 (9)
C(1)–C(7)	1·534 (10)	C(17)–C(18)	1·396 (9)
C(2)–C(3)	1·376 (11)	C(17)–C(22)	1·379 (8)
C(2)–C(15)	1·505 (9)	C(18)–C(19)	1·355 (10)
C(3)–C(4)	1·396 (13)	C(19)–C(20)	1·349 (11)
C(4)–C(5)	1·343 (14)	C(20)–C(21)	1·384 (14)
C(5)–C(6)	1·382 (13)	C(21)–C(22)	1·347 (10)

C(7)–O(2)–C(8)	115·7 (6)	C(10)–C(9)–C(14)	116·8 (7)
C(15)–O(4)–C(16)	114·4 (5)	C(9)–C(10)–C(11)	121·7 (8)
C(6)–C(1)–C(7)	115·7 (6)	C(10)–C(11)–C(12)	119·6 (8)
C(2)–C(1)–C(7)	122·1 (6)	C(11)–C(12)–C(13)	120·8 (8)
C(2)–C(1)–C(6)	122·2 (7)	C(12)–C(13)–C(14)	121·1 (9)
C(1)–C(2)–C(15)	118·6 (6)	C(9)–C(14)–C(13)	120·0 (7)
C(1)–C(2)–C(3)	119·4 (7)	O(4)–C(15)–C(2)	109·7 (5)
C(3)–C(2)–C(15)	121·9 (6)	O(3)–C(15)–C(2)	126·4 (6)
C(2)–C(3)–C(4)	117·3 (8)	O(3)–C(15)–O(4)	123·9 (6)
C(3)–C(4)–C(5)	123·1 (9)	O(4)–C(16)–C(17)	107·2 (5)
C(4)–C(5)–C(6)	119·6 (9)	C(16)–C(17)–C(22)	123·4 (6)
C(1)–C(6)–C(5)	118·4 (8)	C(16)–C(17)–C(18)	120·3 (5)
O(2)–C(7)–C(1)	109·9 (6)	C(18)–C(17)–C(22)	116·2 (5)
O(1)–C(7)–C(1)	124·2 (7)	C(17)–C(18)–C(19)	120·9 (6)
O(1)–C(7)–O(2)	125·8 (7)	C(18)–C(19)–C(20)	122·1 (8)
O(2)–C(8)–C(9)	107·7 (7)	C(19)–C(20)–C(21)	117·7 (7)
C(8)–C(9)–C(14)	119·9 (7)	C(20)–C(21)–C(22)	121·0 (7)
C(8)–C(9)–C(10)	123·2 (8)	C(17)–C(22)–C(21)	122·0 (7)

C(2)–C(1)–C(7)–C(1)	124·8 (8)	C(1)–C(2)–C(15)–O(4)	155·1 (6)
C(2)–C(1)–C(7)–O(2)	−59·4 (8)	C(1)–C(2)–C(15)–O(3)	−25·9 (10)
C(6)–C(1)–C(7)–O(1)	−54·4 (10)	C(3)–C(2)–C(15)–O(4)	−29·9 (9)
C(6)–C(1)–C(7)–O(2)	121·5 (7)	C(3)–C(2)–C(15)–O(3)	149·1 (7)
C(1)–C(7)–O(2)–C(8)	174·5 (6)	C(2)–C(15)–O(4)–C(16)	176·9 (5)
O(1)–C(7)–O(2)–C(8)	−9·7 (11)	O(3)–C(15)–O(4)–C(16)	−2·1 (9)
C(7)–O(2)–C(8)–C(9)	−166·6 (6)	C(15)–O(4)–C(16)–C(17)	−179·3 (5)
O(2)–C(8)–C(9)–C(10)	−104·6 (9)	O(4)–C(16)–C(17)–C(18)	−132·6 (6)
O(2)–C(8)–C(9)–C(14)	74·1 (9)	O(4)–C(16)–C(17)–C(22)	45·0 (8)

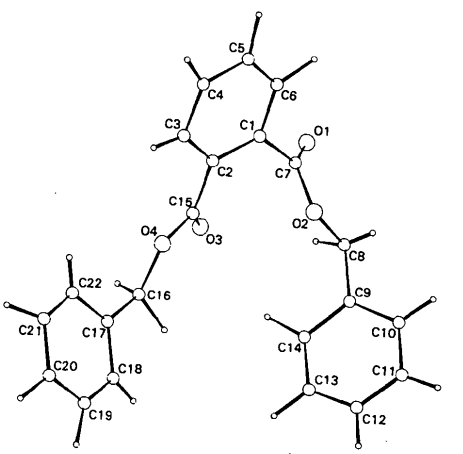

Fig. 1. Projection of the molecule with arbitrary numbering scheme.

All calculations performed on a Gould Sel 32/77 computer.*

Table 1 gives atom parameters and Table 2 bond lengths, angles and selected torsion angles. Fig. 1 shows the molecule and numbering scheme.

Related literature. Bocelli & Grenier-Loustalot (1986).

* Lists of structure-factor amplitudes, thermal parameters, H coordinates and bond lengths involving H have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42611 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Milbemycin β_3 Intermediate

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Abstract. (2*S*,4*R*,6*S*,8*R*,9*S*)-Methyl 8,9-dimethyl-4-[(*tert*-butyl)diphenylsilyloxy]-1,7-dioxaspiro[5,5]-undecane-2-carboxylate. $C_{29}H_{40}O_5Si$, $M_r = 496{\cdot}7$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}901$ (1), $b = 9{\cdot}964$ (2), $c = 28{\cdot}306$ (3) Å, $V = 2792{\cdot}5$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}182$ g cm^{-3}, Mo $K\alpha$ (graphite monochromator), $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}134$ cm^{-1}, $F(000) = 1072$, $T = 163$ (2) K, $R = 0{\cdot}030$ for 476 variables and 2726 unique reflections having $I > 3\sigma(I)$. The structure confirms the expected stereochemistry at C(2) and C(4) of this product, which is an intermediate on the route to the total synthesis of milbemycin β_3. There are no unusual bond distances or angles.

Experimental. A large, rather flat crystal (Attwood, Barrett, Carr, Finch & Richardson, 1985) grown from an ethyl ether/hexane solution was cut to give an approximate cube, 0·4 mm on a side. Data collected on an Enraf–Nonius CAD-4F diffractometer using ω scans; 22 reflections having $12{\cdot}0 \le \theta \le 14{\cdot}5°$ used to determine lattice parameters; empirical absorption correction with factors 0·942–0·998; $\theta \le 27{\cdot}5°$; octant of data with $0 \le h \le 12$, $0 \le k \le 12$, $0 \le l \le 36$; average intensity loss for 3 control reflections of 2·3% overall (corrected); 3613 unique reflections measured; 2726 data having $I > 3\sigma(I)$ used for refinement; structure solved using direct methods; absolute chirality assigned on basis of synthetic pathway; H atoms located in difference Fourier maps and refined isotropically; 476 variables; refinement on F magnitudes; R, $wR = 0{\cdot}030$, 0·037; error in an observation of unit weight = 1·29; $w = 4I/[\sigma^2(I)]$ where $[\sigma^2(I)]$ includes a term $(0{\cdot}04I)^2$; max. Δ/σ in last cycle of 0·11; largest peak in final difference Fourier synthesis has height 0·20 e Å^{-3} (top 10 peaks can be identified with bonding electron density); computer programs from the *Enraf–Nonius Structure Determination Package* (1982); scattering factors from Cromer & Waber (1974).

The molecular structure and the atom-numbering scheme are shown in Fig. 1; the crystal packing is displayed in Fig. 2. Atomic coordinates are listed in Table 1, and bond lengths and angles are given in Table 2.†

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† The refined atomic displacement parameters and the observed and calculated structure-factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42542 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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