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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Table 1. *Positional and displacement parameters for the atoms of a milbemycin β_3 intermediate, with e.s.d.'s in parentheses*

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $\frac{4}{3}\mathrm{Tr}\beta.\mathbf{G}$.

	x	*y*	*z*	*B*(Å²)
Si	0·34259 (5)	0·47351 (6)	0·32897 (2)	1·590 (9)
O(1)	−0·1684 (2)	0·5596 (2)	0·48172 (6)	2·81 (3)
O(2)	−0·0469 (2)	0·6594 (2)	0·53848 (5)	2·69 (3)
O(3)	0·2455 (2)	0·5358 (2)	0·37098 (5)	2·00 (3)
O(4)	0·3949 (1)	0·5044 (2)	0·53043 (5)	2·02 (3)
O(5)	0·1707 (1)	0·5159 (1)	0·51589 (5)	1·68 (2)
C(1)	−0·0557 (2)	0·5831 (2)	0·50605 (7)	1·79 (4)
C(2)	0·0586 (2)	0·5001 (2)	0·48508 (7)	1·71 (4)
C(3)	0·0892 (2)	0·5466 (2)	0·43468 (7)	1·90 (4)
C(4)	0·2159 (2)	0·4794 (2)	0·41618 (7)	1·74 (4)
C(5)	0·3305 (2)	0·5029 (2)	0·45097 (7)	1·76 (4)
C(6)	0·2944 (2)	0·4533 (2)	0·50041 (7)	1·78 (4)
C(7)	0·2864 (2)	0·3000 (2)	0·50408 (7)	2·17 (4)
C(8)	0·2771 (3)	0·2547 (2)	0·55535 (8)	2·69 (5)
C(9)	0·3897 (3)	0·3161 (3)	0·58489 (8)	2·69 (5)
C(10)	0·3824 (2)	0·4673 (3)	0·57986 (7)	2·23 (4)
C(11)	0·4933 (3)	0·5442 (3)	0·60485 (8)	3·43 (6)
C(12)	0·3811 (4)	0·2703 (3)	0·63660 (9)	4·08 (6)
C(13)	−0·2871 (3)	0·6358 (3)	0·4955 (1)	3·24 (5)
C(14)	0·5170 (2)	0·5439 (2)	0·33531 (7)	1·93 (4)
C(15)	0·5448 (3)	0·6523 (2)	0·36523 (8)	2·41 (5)
C(16)	0·6731 (3)	0·7071 (3)	0·36842 (9)	3·31 (5)
C(17)	0·7773 (3)	0·6550 (3)	0·34091 (9)	3·39 (5)
C(18)	0·7522 (3)	0·5488 (3)	0·3110 (1)	3·26 (5)
C(19)	0·6243 (2)	0·4938 (3)	0·30834 (8)	2·53 (4)
C(20)	0·3532 (2)	0·2860 (2)	0·33512 (7)	1·87 (4)
C(21)	0·4455 (2)	0·2328 (2)	0·36742 (8)	2·33 (4)
C(22)	0·4523 (3)	0·0959 (3)	0·37696 (9)	2·75 (5)
C(23)	0·3662 (2)	0·0092 (2)	0·35372 (9)	2·90 (5)
C(24)	0·2770 (3)	0·0579 (3)	0·32066 (9)	3·04 (5)
C(25)	0·2696 (3)	0·1951 (3)	0·31182 (8)	2·58 (5)
C(26)	0·2636 (2)	0·5368 (2)	0·27229 (7)	2·12 (4)
C(27)	0·3379 (3)	0·4814 (3)	0·22927 (8)	3·46 (5)
C(28)	0·2738 (3)	0·6905 (3)	0·27281 (9)	3·70 (6)
C(29)	0·1139 (2)	0·5011 (3)	0·26928 (9)	3·25 (5)
H(2)	0·025 (2)	0·407 (2)	0·4855 (7)	0·9 (4)*
H(3A)	0·112 (2)	0·645 (2)	0·4336 (7)	2·0 (5)*
H(3B)	0·019 (2)	0·527 (2)	0·4155 (8)	2·6 (5)*
H(4)	0·198 (2)	0·378 (2)	0·4128 (7)	1·0 (4)*
H(5A)	0·407 (2)	0·454 (2)	0·4410 (7)	1·6 (4)*
H(5B)	0·349 (2)	0·596 (2)	0·4542 (8)	2·4 (5)*
H(7A)	0·367 (2)	0·267 (2)	0·4891 (7)	1·9 (5)*
H(7B)	0·213 (2)	0·268 (2)	0·4883 (8)	2·4 (5)*
H(8A)	0·285 (2)	0·164 (2)	0·5570 (8)	2·6 (5)*
H(8B)	0·183 (3)	0·283 (3)	0·5686 (8)	3·2 (6)*
H(9)	0·477 (3)	0·289 (3)	0·5737 (9)	3·4 (6)*
H(10)	0·297 (2)	0·498 (2)	0·5896 (7)	1·1 (4)*
H(11A)	0·485 (2)	0·536 (3)	0·6384 (9)	3·2 (6)*
H(11B)	0·493 (3)	0·644 (3)	0·5961 (9)	3·9 (7)*
H(11C)	0·580 (3)	0·501 (3)	0·596 (1)	5·4 (7)*
H(12A)	0·384 (3)	0·169 (3)	0·640 (1)	4·2 (6)*
H(12B)	0·297 (3)	0·308 (3)	0·650 (1)	4·8 (7)*
H(12C)	0·459 (3)	0·302 (3)	0·657 (1)	6·0 (8)*
H(13A)	−0·282 (3)	0·727 (3)	0·4861 (9)	3·7 (6)*
H(13B)	−0·294 (3)	0·628 (3)	0·5288 (9)	3·7 (6)*
H(13C)	−0·353 (3)	0·609 (3)	0·474 (1)	5·8 (8)*
H(15)	0·477 (2)	0·683 (2)	0·3853 (8)	2·3 (5)*
H(16)	0·691 (3)	0·777 (3)	0·3894 (9)	4·6 (7)*
H(17)	0·864 (3)	0·694 (3)	0·3438 (9)	4·5 (7)*
H(18)	0·823 (2)	0·516 (3)	0·2934 (8)	3·3 (5)*
H(19)	0·609 (2)	0·420 (2)	0·2892 (7)	2·0 (5)*
H(21)	0·505 (2)	0·291 (3)	0·3804 (9)	3·0 (6)*
H(22)	0·518 (3)	0·066 (3)	0·3991 (8)	2·8 (6)*
H(23)	0·380 (3)	−0·082 (3)	0·3605 (9)	3·6 (6)*
H(24)	0·221 (2)	0·002 (3)	0·3043 (9)	3·3 (5)*
H(25)	0·207 (2)	0·225 (3)	0·2906 (8)	3·0 (6)*
H(27A)	0·338 (3)	0·388 (3)	0·2268 (9)	4·6 (7)*
H(27B)	0·426 (2)	0·511 (3)	0·2295 (9)	3·7 (6)*
H(27C)	0·291 (2)	0·514 (3)	0·2007 (8)	3·0 (5)*
H(28A)	0·228 (3)	0·733 (4)	0·302 (1)	6·5 (9)*
H(28B)	0·365 (3)	0·720 (3)	0·278 (1)	4·8 (7)*
H(28C)	0·227 (3)	0·724 (3)	0·2436 (8)	3·6 (6)*
H(29A)	0·064 (2)	0·550 (3)	0·2431 (9)	3·9 (6)*
H(29B)	0·064 (3)	0·534 (3)	0·296 (1)	4·7 (7)*
H(29C)	0·097 (3)	0·404 (3)	0·265 (1)	5·1 (7)*

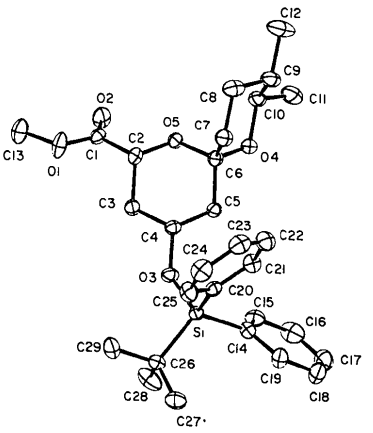

Fig. 1. Perspective drawing of the molecular structure of a milbemycin β_3 intermediate. In this and the following drawing the shapes of the ellipsoids correspond to 50% probability contours of atomic displacement, and the H atoms have been omitted for the sake of clarity.

Table 2. *Selected bond lengths* (Å) *and angles* (°) *for a milbemycin β_3 intermediate*

Si–O(3)	1·650 (1)	Si–C(14)	1·872 (2)
Si–C(20)	1·879 (2)	Si–C(26)	1·893 (2)
O(1)–C(1)	1·332 (3)	O(1)–C(13)	1·453 (3)
O(2)–C(1)	1·195 (2)	O(3)–C(4)	1·428 (2)
O(4)–C(6)	1·405 (2)	O(4)–C(10)	1·453 (2)
O(5)–C(2)	1·420 (2)	O(5)–C(6)	1·443 (2)
C(1)–C(2)	1·522 (3)	C(2)–C(3)	1·530 (3)
C(3)–C(4)	1·515 (3)	C(4)–C(5)	1·521 (3)
C(5)–C(6)	1·526 (3)	C(6)–C(7)	1·533 (3)
C(7)–C(8)	1·523 (3)	C(8)–C(9)	1·522 (4)
C(9)–C(10)	1·515 (4)	C(9)–C(12)	1·535 (3)
C(10)–C(11)	1·514 (4)		
C(14)–C(15)	1·400 (3)	C(14)–C(19)	1·400 (3)
C(15)–C(16)	1·386 (4)	C(16)–C(17)	1·393 (4)
C(17)–C(18)	1·378 (4)	C(18)–C(19)	1·382 (3)
C(20)–C(21)	1·397 (3)	C(20)–C(25)	1·393 (3)
C(21)–C(22)	1·392 (3)	C(22)–C(23)	1·380 (4)
C(23)–C(24)	1·376 (4)	C(24)–C(25)	1·391 (3)
C(26)–C(27)	1·526 (3)	C(26)–C(28)	1·535 (4)
C(26)–C(29)	1·527 (3)		

O(3)–Si–C(14)	109·10 (8)	O(3)–Si–C(20)	109·85 (9)
O(3)–Si–C(26)	104·17 (9)	C(14)–Si–C(20)	108·18 (9)
C(14)–Si–C(26)	109·73 (10)	C(20)–Si–C(26)	115·65 (10)
C(1)–O(1)–C(13)	116·55 (18)	Si–O(3)–C(4)	128·13 (13)
C(6)–O(4)–C(10)	115·47 (16)	C(2)–O(5)–C(6)	115·39 (14)
O(1)–C(1)–O(2)	124·78 (20)	O(1)–C(1)–C(2)	109·01 (17)
O(2)–C(1)–C(2)	126·19 (20)		
O(5)–C(2)–C(1)	106·34 (15)	O(5)–C(2)–C(3)	112·65 (16)
C(1)–C(2)–C(3)	110·26 (17)	C(2)–C(3)–C(4)	110·62 (18)
O(3)–C(4)–C(3)	107·83 (17)	O(3)–C(4)–C(5)	111·49 (16)
C(3)–C(4)–C(5)	109·03 (17)	C(4)–C(5)–C(6)	111·68 (17)
O(4)–C(6)–O(5)	105·13 (15)	O(4)–C(6)–C(5)	105·76 (16)
O(4)–C(6)–C(7)	110·92 (18)	O(5)–C(6)–C(5)	109·68 (16)
O(5)–C(6)–C(7)	111·49 (18)	C(5)–C(6)–C(7)	113·39 (18)
C(6)–C(7)–C(8)	111·30 (19)	C(7)–C(8)–C(9)	111·13 (21)
C(8)–C(9)–C(10)	108·27 (20)	C(8)–C(9)–C(12)	111·34 (25)
C(10)–C(9)–C(12)	112·47 (22)		
O(4)–C(10)–C(9)	109·85 (18)	O(4)–C(10)–C(11)	105·03 (19)
C(9)–C(10)–C(11)	115·12 (23)		
Si–C(14)–C(15)	121·91 (17)	Si–C(14)–C(19)	120·96 (16)
C(15)–C(14)–C(19)	117·06 (21)	C(14)–C(15)–C(16)	121·62 (23)
C(15)–C(16)–C(17)	119·72 (24)	C(16)–C(17)–C(18)	119·73 (24)
C(17)–C(18)–C(19)	120·20 (26)	C(14)–C(19)–C(18)	121·67 (23)
Si–C(20)–C(21)	118·34 (17)	Si–C(20)–C(25)	124·74 (17)
C(21)–C(20)–C(25)	116·80 (21)	C(20)–C(21)–C(22)	122·06 (24)
C(21)–C(22)–C(23)	119·39 (25)	C(22)–C(23)–C(24)	120·00 (24)
C(23)–C(24)–C(25)	120·17 (24)	C(20)–C(25)–C(24)	121·53 (24)
Si–C(26)–C(27)	110·89 (17)	Si–C(26)–C(28)	107·29 (17)
Si–C(26)–C(29)	111·76 (17)	C(27)–C(26)–C(28)	109·71 (24)
C(27)–C(26)–C(29)	109·81 (22)	C(28)–C(26)–C(29)	107·27 (24)

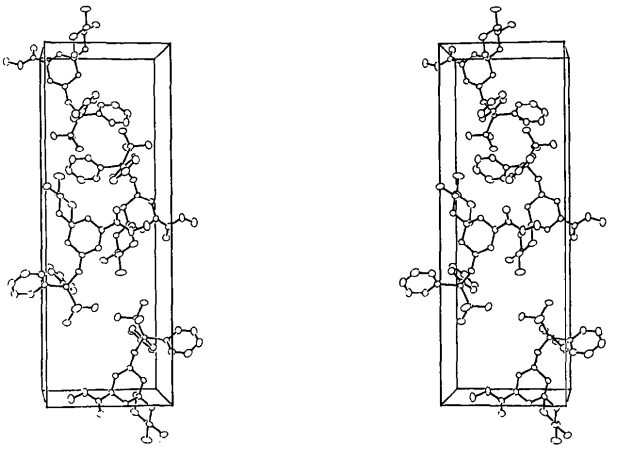

Fig. 2. Stereoscopic view of the unit cell of a milbemycin β_3 intermediate. The *a* axis points from right to left, the *c* axis points downwards, and the *b* axis points into the plane of the paper.

Related literature. This compound, which was prepared from (*S*)-citronellene by Attwood *et al.* (1985), is a key intermediate in the total synthesis of the spiroketal macrocyclic lactone milbemycin β_3. Takiguchi, Mishima, Okuda, Terao, Aoki & Fukuda (1980) have reported the structure of this natural product, and Williams, Barner, Nishitani & Phillips (1982) have established its absolute stereochemistry by synthesis.

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N,N'-Bis(*p*-chlorobenzylidene)-1,3-propanediamine

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Abstract. $C_{17}H_{16}Cl_2N_2$, $M_r = 319{\cdot}23$, monoclinic, $P2_1$, $a = 7{\cdot}371$ (3), $b = 30{\cdot}406$ (19), $c = 7{\cdot}268$ (4) Å, $\beta = 96{\cdot}23$ (4)°, $V = 1619$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}31$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 3{\cdot}97$ cm^{-1}, $F(000) = 664$, $T = 291$ K, $R = 0{\cdot}067$ for 1205 observed reflections. The two independent molecules in the asymmetric unit are similar and have a *trans* configuration around the imine bonds. In each molecule, the two Cl—C_6H_5—C=N—C moieties are planar (maximum deviation from mean planes <0·05 Å) and perpendicular to each other (in both molecules the angle between the mean planes is 86°).

Experimental. Parallelepipedal crystal, 0·25 × 0·2 × 0·4 mm. Lattice parameters refined using 15 reflections in range $5 < 2\theta < 15°$. Syntex $P2_1$, graphite-monochromatized Mo $K\alpha$ radiation. 2436 $hk\pm l$ independent reflections with $\sin\theta/\lambda < 0{\cdot}561$ Å^{-1}; $0 \le h \le 8$, $0 \le k \le 33$, $-7 \le l \le 7$, 1205 with $I \ge 2{\cdot}5\sigma(I)$. Standard reflection (131) checked every 50 reflections: no significant deviation. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Least-squares refinement with *SHELX*76 (Sheldrick, 1976), *F* magnitudes; isotropic then anisotropic temperature factors gave $R = 0{\cdot}067$, $wR = 0{\cdot}062$ including H atoms at calculated positions. $w = 1/(\sigma^2 + 0{\cdot}00039F^2)$. Final max. shift to e.s.d. = 0·38. $S = 1{\cdot}85$. Max. and min. heights in final difference Fourier synthesis = 0·28 and −0·32 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The atomic parameters are given in Table 1,* mean values of selected bond lengths and angles in Table 2. Fig. 1 is a view of one of the two independent molecules (*A*), showing the numbering of the atoms; Fig. 2 shows the packing in the unit cell (program *PLUTO*; Motherwell & Clegg, 1978).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a full list of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42637 (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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