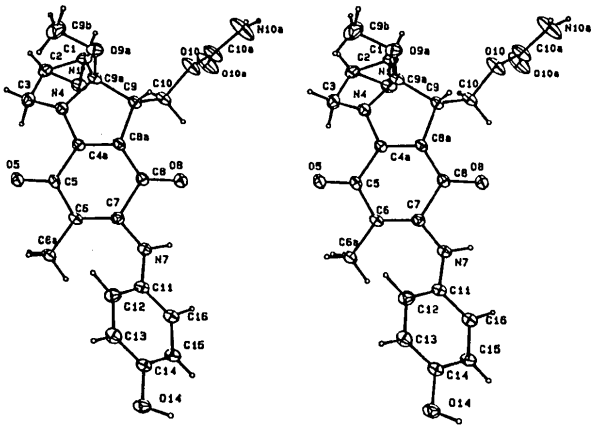

Fig. 1. *ORTEP*II (Johnson, 1976) drawing showing 30% ellipsoids.

Related literature. Mitomycin C is one of the successful potent anticancer antibiotics in clinical use today. In order to screen for derivatives more active than mitomycin C, several 7-*N*-alkylated derivatives of mitomycin C were synthesized (Imai, Ashizawa, Urakawa, Morimoto & Nakamura, 1980). M83, a 7-*N*-(*p*-hydroxylphenyl) derivative, showed more potent antitumor activity against the ascitic form of P-388 leukemia than mitomycin C (Kobayashi, Inaba, Tsukagoshi, Sakurai, Imai & Morimoto, 1981). The crystal structure of mitomycin C itself has been determined for the two different crystal forms (Arora, 1979; Ogawa, Nomura, Fujiwara & Tomita, 1979). The absolute configuration was determined by the Bijvoet method (Shirahata & Hirayama, 1983).

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Structure of 10,10-Dihydro-10-deoxo-10a-methyl-10a-aza-10a-homoerythronolide A

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Abstract. $C_{22}H_{43}NO_7$, $M_r = 433{\cdot}58$, monoclinic, $P2_1$, $a = 20{\cdot}427\ (5)$, $b = 6{\cdot}995\ (2)$, $c = 8{\cdot}372\ (2)$ Å, $\beta = 97{\cdot}60\ (1)°$, $V = 1185{\cdot}7$ Å^3, $Z = 2$, $D_m = 1{\cdot}22$ (by flotation), $D_x = 1{\cdot}218$ g cm^{-3}, λ(Mo $K\alpha$) = 1·5418 Å, $\mu = 6{\cdot}45$ cm^{-1}, $F(000) = 476$, $T = 293$ K, $R = 0{\cdot}039$ for 1709 reflections. The geometry and dimensions of the 15-membered aglycone ring are not significantly different from those found in the analogous azaerythronolide A derivatives. The presence of the methyl group linked to the N atom does not significantly influence the structure. The distances between O(61)···O(131^{i}) [(i) x, y, $1+z$] of 2·821 (5) and O(61)···O(141^{ii}) [(ii) x, $1+y$, $1+z$] of 2·861 (5) Å suggest the existence of intermolecular hydrogen bonding while the O(71)···N(11) distance of 2·816 (5) Å suggests intramolecular hydrogen bonding.

Experimental. As part of a wider investigation of a novel 15-membered macrolide antibiotic the title

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compound was prepared from the corresponding azaerythromycin A derivative by the removal of both sugar components, D-desosamine and L-cladinose, either by strong hydrolysis or by selective removal of both sugars and reductive methylation of the resulting aglycone. The crystals suitable for X-ray work were obtained from acetone. Crystal dimensions: 0·27 × 0·28 × 0·04 mm. Philips PW1100 diffractometer; graphite-monochromated Cu $K\alpha$ radiation, ω–2θ scan in the range $3 < \theta < 70°$ ($-24 \le h \le 24$, $0 \le k \le 8$, $0 \le l \le 10$), scan width 1·609°, scan speed 0·04° s^{-1}. Lattice parameters from least-squares refinement of 25 reflections with $11 < \theta < 20°$. Of 1847 independent reflections, 1709 with $I \ge 3\sigma(I)$ were used in the structure determination. Three standard reflections monitored every 2 h showed no significant variation. The data were corrected for Lorentz and polarization effects but not for absorption. Most of the non-H atoms were obtained with *SIR* (Cascarano, Giacovazzo, Burla, Nunzi, Polidori, Camalli, Spagna & Viterbo, 1985); remaining atoms based on Fourier method. Full-matrix least-squares refinement with anisotropic thermal parameters for all non-H atoms performed with *SHELX*76 (Sheldrick, 1976). The H-atom positions were obtained from a difference Fourier map and refined isotropically, except three H atoms on C(152) which were fixed. The refinement converged at $R = 0·039$, $\Sigma(w|F_o| - |F_c|)^2$, $w = 1·0$, $(\Delta/\sigma)_{max} = 0·6$, maximum and minimum peak heights in final ΔF map were +0·20 and −0·25 e $Å^{-3}$, respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). A perspective view of the structure drawn with the program *ORTEP* (Johnson, 1965) is shown in Fig. 1, packing of the molecules in the unit cell in Fig. 2. Atomic parameters are given in Table 1, bond lengths and angles in Table 2.*

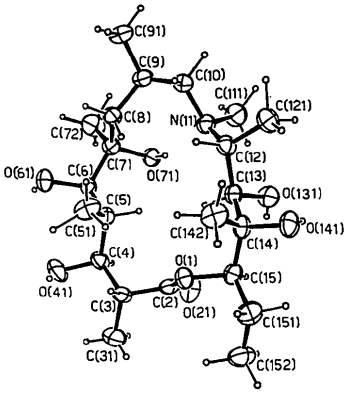

Fig. 1. A perspective view of the molecule with the atom-labelling scheme. Ellipsoids are drawn at the 50% probability level and H atoms are represented by spheres of arbitrary size.

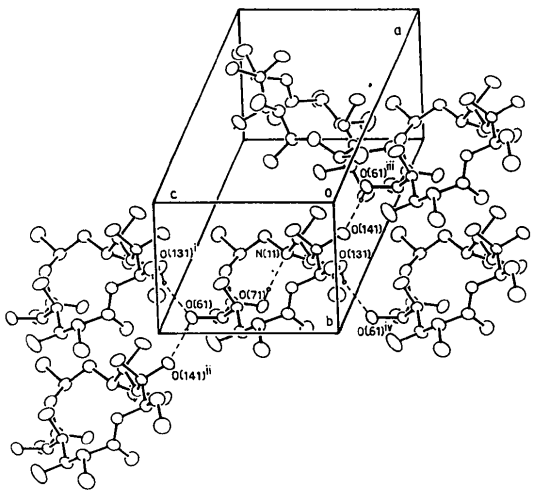

Fig. 2. Unit-cell packing diagram with intramolecular and intermolecular hydrogen bonds shown by broken lines. H atoms, except those participating in hydrogen bonding, have been omitted for clarity. Symmetry code: (i) $x, y, 1 + z$; (ii) $x, 1 + y, 1 + z$; (iii) $x, y - 1, z - 1$; (iv) $x, y, z - 1$.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* ($Å^2$) *for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\Sigma_i\Sigma_j b_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O(1)	1324 (1)	8453 (4)	3175 (3)	2·78 (8)
C(2)	1181 (2)	10297	3364 (5)	2·7 (1)
O(21)	1285 (2)	11535 (4)	2420 (4)	4·4 (1)
C(3)	863 (2)	10638 (6)	4868 (5)	2·8 (1)
C(31)	192 (2)	11580 (8)	4410 (6)	4·5 (2)
C(4)	1317 (2)	11867 (6)	6090 (5)	2·9 (1)
O(41)	978 (1)	12200 (6)	7442 (4)	4·8 (1)
C(5)	1996 (2)	10991 (6)	6613 (4)	2·4 (1)
C(51)	1934 (2)	9023 (7)	7399 (6)	3·6 (2)
C(6)	2440 (2)	12443 (6)	7659 (5)	2·5 (1)
O(61)	2315 (2)	12240 (5)	9314 (4)	3·09 (8)
C(7)	3189 (2)	12364 (6)	7494 (5)	2·7 (1)
O(71)	3234 (1)	12756 (5)	5820 (3)	3·13 (8)
C(72)	3543 (3)	13977 (8)	8477 (7)	4·3 (2)
C(8)	3515 (2)	10419 (7)	7940 (5)	2·9 (1)
C(9)	4217 (2)	10159 (6)	7470 (5)	2·8 (1)
C(91)	4690 (2)	9338 (8)	8877 (5)	4·0 (1)
C(10)	4202 (2)	8778 (7)	6055 (5)	3·1 (1)
N(11)	3763 (2)	9422 (5)	4603 (4)	2·70 (9)
C(111)	4149 (2)	10575 (8)	3581 (5)	4·0 (1)
C(12)	3398 (2)	7776 (6)	3765 (5)	2·8 (1)
C(121)	3837 (3)	6473 (8)	2857 (6)	4·3 (2)
C(13)	2765 (2)	8466 (6)	2726 (4)	2·4 (1)
O(131)	2948 (1)	9344 (5)	1296 (3)	3·58 (9)
C(14)	2241 (2)	6885 (6)	2195 (4)	2·6 (1)
O(141)	2428 (1)	5945 (4)	773 (3)	3·35 (9)
C(142)	2195 (2)	5345 (7)	3444 (5)	3·6 (1)
C(15)	1567 (2)	7854 (6)	1674 (4)	2·7 (1)
C(151)	1035 (2)	6576 (7)	766 (5)	3·8 (1)
C(152)	405 (2)	7673 (8)	218 (6)	4·5 (2)

Related literature. Syntheses and other structural investigations of a novel 15-membered-ring erythro-

* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52850 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

O(1)—C(2)	1·336 (3)	C(8)—C(9)	1·546 (6)
O(1)—C(15)	1·473 (5)	C(9)—C(91)	1·534 (6)
C(2)—O(21)	1·209 (4)	C(9)—C(10)	1·526 (6)
C(2)—C(3)	1·510 (6)	C(10)—N(11)	1·483 (5)
C(3)—C(31)	1·523 (6)	N(11)—C(111)	1·477 (6)
C(3)—C(4)	1·549 (6)	N(11)—C(12)	1·495 (5)
C(4)—O(41)	1·422 (5)	C(12)—C(121)	1·547 (7)
C(4)—C(5)	1·526 (5)	C(12)—C(13)	1·537 (5)
C(5)—C(51)	1·537 (6)	C(13)—O(131)	1·438 (5)
C(5)—C(6)	1·552 (6)	C(13)—C(14)	1·562 (5)
C(6)—O(61)	1·449 (5)	C(14)—O(141)	1·455 (5)
C(6)—C(7)	1·555 (6)	C(14)—C(142)	1·513 (6)
C(7)—O(71)	1·443 (5)	C(14)—C(15)	1·544 (6)
C(7)—C(72)	1·522 (7)	C(15)—C(151)	1·529 (6)
C(7)—C(8)	1·539 (6)	C(151)—C(152)	1·518 (6)

C(2)—O(1)—C(15)	118·6 (3)	C(8)—C(9)—C(91)	111·1 (3)
O(1)—C(2)—O(21)	123·6 (4)	C(8)—C(9)—C(10)	110·4 (3)
O(1)—C(2)—C(3)	112·1 (3)	C(91)—C(9)—C(10)	107·7 (4)
O(21)—C(2)—C(3)	124·3 (3)	C(9)—C(10)—N(11)	113·0 (3)
C(2)—C(3)—C(31)	109·3 (3)	C(10)—N(11)—C(111)	109·2 (3)
C(2)—C(3)—C(4)	110·6 (3)	C(10)—N(11)—C(12)	111·1 (3)
C(31)—C(3)—C(4)	111·5 (4)	C(111)—N(11)—C(12)	114·8 (3)
C(3)—C(4)—O(41)	107·7 (3)	N(11)—C(12)—C(121)	113·5 (3)
C(3)—C(4)—C(5)	114·2 (3)	N(11)—C(12)—C(13)	110·7 (3)
O(41)—C(4)—C(5)	111·0 (3)	C(121)—C(12)—C(13)	113·9 (3)
C(4)—C(5)—C(51)	111·0 (3)	C(12)—C(13)—O(131)	108·3 (3)
C(4)—C(5)—C(6)	109·9 (3)	C(12)—C(13)—C(14)	115·6 (3)
C(51)—C(5)—C(6)	114·8 (3)	O(131)—C(13)—C(14)	108·0 (3)
C(5)—C(6)—O(61)	108·1 (3)	C(13)—C(14)—O(141)	107·9 (3)
C(5)—C(6)—C(7)	115·8 (3)	C(13)—C(14)—C(142)	114·2 (3)
O(61)—C(6)—C(7)	112·4 (3)	C(13)—C(14)—C(15)	108·7 (3)
C(6)—C(7)—O(71)	105·6 (3)	C(142)—C(14)—O(141)	106·9 (3)
C(6)—C(7)—C(72)	109·2 (4)	C(142)—C(14)—C(15)	111·9 (3)
C(6)—C(7)—C(8)	114·2 (4)	O(141)—C(14)—C(15)	106·8 (3)
O(71)—C(7)—C(72)	107·3 (4)	O(1)—C(15)—C(14)	105·8 (3)
O(71)—C(7)—C(8)	108·8 (3)	O(1)—C(15)—C(151)	107·0 (3)
C(72)—C(7)—C(8)	111·4 (3)	C(14)—C(15)—C(151)	115·6 (4)
C(7)—C(8)—C(9)	115·4 (4)	C(15)—C(151)—C(152)	111·9 (4)

mycin A and its analogous compounds such as 10,10-dihydro-10-deoxo-11-azaerythronolide A hydroiodide (Djokić, Kobrehel, Lazarevski, Lopotar, Tamburašev, Kamenar, Nagl & Vicković, 1986) and 10,10-dihydro-10-deoxo-11-methyl-11-azaerythromycin A (Djokić, Kobrehel, Lopotar, Kamenar, Nagl & Mrvoš, 1988) have been reported.

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Structure of Ratibinolide, a Sesquiterpene Lactone*

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Abstract. 5a,7a-Dimethyl-3-methyleneperhydrocyclopropa[2,3]indeno[4,5-*b*]furan-2,6-dione, $C_{15}H_{18}O_3$, $M_r = 246{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}779$ (4), $b = 11{\cdot}052$ (6), $c = 17{\cdot}760$ (9) Å, $V = 1331$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}23$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}079$ mm^{-1}, $F(000) = 528$, $T = 293$ K, $R = 0{\cdot}038$ for 1073 observed reflections. The six-membered ring adopts a chair conformation with the methyl group at C(10) in the axial position. The five-membered rings [C(1)—C(2)—C(4)—C(5)—C(10) and O(2)—C(6)—C(7)—C(11)—C(12)] are in a conformation intermediate between half-chair and α-envelope, and in a half-chair conformation, respectively. The Δ and φ values [Altona, Geise & Romers (1968). *Tetrahedron*, **24**, 13–32] are −15·7 (3), −39·7 (3)° and −4·1 (3), −37·8 (3)°, respectively. A C—H···O intermolecular contact is present, C(6)···O(1)(1 + x, y, z) 3·412 (4) Å. The packing in the crystal is entirely due to van der Waals forces.

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