24. Crystal Structures of *rac*-Methyl-(3*R**, 3a*S**, 4*R**, 7*R**, 7a*R**)-7-Acetoxymethyl)-3-benzyl-4,5-dimethyl-1-oxo-3a,4,7,7a-tetrahydroisoindoline-7a-carboxylate[1]) and *rac*-(3*R**,3a*S**,4*R**,7*R**,7a*S**)-3-Benzyl-2-benzyloxycarbonyl-4,5-dimethyl-7,7a-(3′-oxo-2′-oxatrimethylene)-3a,4,7,7a-tetrahydroisoindolin-1-one[1])

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Summary

The X-ray structures of the compounds **4** and **2** have been determined by direct methods and refined by least squares techniques. Crystals of $C_{22}H_{27}NO_5$ and $C_{27}H_{27}NO_5$ are triclinic, space groups $P\bar{1}$ with lattice parameters $a = 13.652(5)$ Å, $b = 10.926(3)$ Å, $c = 7.755(2)$ Å, $\alpha = 111.554(4)$ Å, $\beta = 85.541(3)$ Å, $\gamma = 104.813(4)$ Å, and $a = 15.394(4)$ Å, $b = 9.674(3)$ Å, $c = 8.522(3)$ Å, $\alpha = 111.04(4)$ Å, $\beta = 93.65(4)$ Å, $\gamma = 95.01(4)$ Å, respectively.

Introduction. – The cytochalasans are secondary metabolites of microorganisms, and in most cases of moulds which exhibit unusual effects on mamalian cells [2]. At present 30 members of this family are known. Chemically they are characterized by a highly substituted tetrahydroisoindolinone group, to which is fused a macrocyclic ring. The latter is either carbocyclic, a lactone or a cyclic carbonate [2]. The unusual structures of the cytochalasans represent an exciting and difficult challenge for partial and total synthesis. Any approach for the construction of this kind of molecules involves two major synthetic problems, namely the formation of the bicyclic tetrahydroisoindolinone moiety and the attachment of the macrocyclic ring system.

Schmidlin & Tamm [3] have reported the synthesis of the tetrahydroisoindolinone moiety related to proxiphomin by an intramolecular *Diels-Alder* reaction to which structure **1a** ($C_{27}H_{27}NO_5$) was tentatively assigned mainly on the basis of the 400-MHz-^{1}H-NMR. spectrum. However, the chirality of the quaternary C-atom C(9) could not be determined unambigously. Therefore an X-ray analysis of this compounds was undertaken. It showed that the relative configuration at C(3), C(4), C(5) and C(8) of the synthetic compound was the same as in

[1]) For the nomenclature s. footnotes 3 and 7 in [1].

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Table 1. *Positional and thermal parameters of the nonhydrogen atoms of structure* **4**
The temperature factors are of the form $T = \exp\{-2\pi^2(U_{11}h^2a^{*2} + ... + 2U_{12}hka^*b^*)\}$.

atom[a])	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	0.6367 (3)	1.0315 (4)	0.1067 (6)	0.0215 (2)	0.0308 (2)	0.0268 (2)	0.0160 (1)	0.0024 (1)	0.0027 (1)
N	0.5982 (3)	0.9087 (4)	−0.0137 (5)	0.0219 (1)	0.0309 (1)	0.0331 (1)	0.0132 (1)	−0.0050 (1)	0.0040 (1)
C(3)	0.6624 (3)	0.8141 (4)	−0.0601 (6)	0.0264 (2)	0.0319 (2)	0.0289 (2)	0.0102 (1)	−0.0045 (2)	0.0069 (1)
C(4)	0.7533 (3)	0.8901 (4)	0.0784 (6)	0.0250 (2)	0.0293 (2)	0.0342 (2)	0.0143 (1)	−0.0001 (2)	0.0052 (1)
C(5)	0.8564 (3)	0.8869 (5)	−0.0199 (7)	0.0267 (2)	0.0412 (2)	0.0480 (2)	0.0135 (2)	−0.0014 (2)	0.0124 (2)
C(6)	0.8697 (3)	0.9721 (5)	−0.1421 (6)	0.0230 (2)	0.0401 (2)	0.0428 (2)	0.0063 (2)	0.0024 (2)	0.0044 (2)
C(7)	0.8544 (3)	1.0945 (5)	−0.0633 (7)	0.0254 (2)	0.0426 (2)	0.0487 (2)	0.0229 (2)	0.0015 (2)	0.0021 (2)
C(8)	0.8268 (3)	1.1452 (4)	0.1379 (6)	0.0248 (2)	0.0309 (2)	0.0399 (2)	0.0112 (2)	−0.0010 (2)	0.0057 (1)
C(9)	0.7395 (3)	1.0372 (4)	0.1803 (6)	0.0237 (2)	0.0248 (2)	0.0322 (2)	0.0086 (1)	−0.0032 (1)	0.0020 (1)
C(10)	0.6048 (3)	0.6725 (4)	−0.0605 (6)	0.0372 (2)	0.0291 (2)	0.0336 (2)	0.0090 (2)	−0.0027 (2)	0.0039 (2)
C(11)	0.8679 (4)	0.7428 (5)	−0.1147 (8)	0.0423 (2)	0.0428 (2)	0.0869 (2)	0.0151 (2)	0.0128 (2)	0.0234 (2)
C(12)	0.8976 (4)	0.9146 (6)	−0.3442 (8)	0.0504 (2)	0.0545 (2)	0.0607 (2)	0.0126 (2)	0.0178 (2)	0.0125 (2)
C(13)	0.8046 (3)	1.2849 (5)	0.2039 (7)	0.0265 (2)	0.0365 (2)	0.0615 (2)	0.0213 (2)	0.0023 (2)	−0.0003 (2)
C(14)	0.7336 (3)	1.0662 (5)	0.3892 (6)	0.0333 (2)	0.0343 (2)	0.0350 (2)	0.0132 (2)	−0.0068 (2)	0.0075 (2)
C(15)	0.9057 (5)	1.5090 (6)	0.2728 (9)	0.0568 (2)	0.0408 (2)	0.1120 (2)	0.0247 (2)	−0.0034 (2)	0.0101 (2)
C(16)	1.0088 (5)	1.5990 (7)	0.2831 (10)	0.0671 (2)	0.0653 (2)	1.1125 (2)	0.0247 (2)	−0.0111 (2)	−0.0211 (2)
C(17)	0.6308 (4)	1.0058 (5)	0.6186 (6)	0.0693 (2)	0.0554 (2)	0.0260 (2)	0.0215 (2)	0.0037 (2)	0.0177 (2)
C(18)	0.5625 (4)	0.6686 (4)	0.1233 (6)	0.0529 (2)	0.0200 (2)	0.0328 (2)	0.0061 (1)	0.0010 (2)	−0.0007 (2)
C(19)	0.4676 (5)	0.6904 (5)	0.1767 (7)	0.0720 (2)	0.0351 (2)	0.0548 (2)	0.0182 (2)	0.0094 (2)	0.0105 (2)
C(20)	0.4290 (5)	0.6858 (6)	0.3478 (9)	0.1029 (2)	0.0450 (2)	0.0850 (2)	0.0252 (2)	0.0502 (2)	0.0241 (2)
C(21)	0.4839 (7)	0.6567 (6)	0.4617 (8)	0.1075 (2)	0.0349 (2)	0.0460 (2)	0.0140 (2)	0.0241 (2)	0.0139 (2)
C(22)	0.5773 (6)	0.6332 (6)	0.4087 (9)	0.1111 (2)	0.0397 (2)	0.0569 (2)	0.0201 (2)	−0.0164 (2)	−0.0004 (2)
C(23)	0.6190 (5)	0.6382 (5)	0.2399 (7)	0.0856 (2)	0.0337 (2)	0.0470 (2)	0.0188 (2)	−0.0210 (2)	−0.0086 (2)
O(1)	0.5976 (2)	1.1289 (3)	0.1565 (4)	0.0263 (2)	0.0324 (2)	0.0441 (2)	0.0089 (1)	−0.0076 (1)	0.0068 (1)
O(2)	0.9029 (3)	1.3792 (3)	0.2150 (5)	0.0363 (2)	0.0300 (1)	0.0835 (2)	0.0134 (1)	0.0035 (2)	0.0005 (1)
O(3)	0.8010 (3)	1.1334 (4)	0.4965 (5)	0.0548 (2)	0.0813 (2)	0.0415 (2)	0.0234 (2)	−0.0237 (2)	−0.0192 (2)
O(4)	0.6442 (2)	1.0013 (3)	0.4303 (4)	0.0366 (2)	0.0534 (2)	0.0306 (1)	0.0190 (1)	0.0012 (1)	0.0017 (1)
O(5)	0.8307 (4)	1.5489 (5)	0.3172 (11)	0.0835 (2)	0.0486 (2)	0.1003 (2)	0.0341 (2)	0.0025 (2)	0.0204 (2)

[a]) Numbering according to *Fig. 1.*

Table 2. *Positional and thermal parameters of the nonhydrogen atoms of structure* **2**.
The temperature factors are of the form $T = \exp\{-2\pi^2(U_{11}h^2a^{*2} + \ldots + 2U_{12}hka^*b^*)\}$.

atom[a])	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	0.5308 (3)	0.4147 (6)	0.3056 (6)	0.0393 (2)	0.0399 (2)	0.0321 (2)	0.0129 (2)	0.0046 (2)	0.0046 (2)
N	0.5559 (3)	0.2833 (4)	0.3210 (5)	0.0298 (2)	0.0347 (2)	0.0484 (2)	0.0190 (2)	0.0025 (2)	0.0015 (2)
C(3)	0.6536 (3)	0.2985 (6)	0.3895 (7)	0.0259 (2)	0.0441 (2)	0.0544 (2)	0.0271 (2)	0.0012 (2)	0.0005 (2)
C(4)	0.6813 (3)	0.4691 (6)	0.4460 (7)	0.0306 (2)	0.0410 (2)	0.0423 (2)	0.0187 (2)	0.0056 (2)	0.0011 (2)
C(5)	0.7786 (4)	0.5395 (6)	0.4730 (7)	0.0376 (2)	0.0484 (2)	0.0497 (2)	0.0229 (2)	0.0091 (2)	−0.0024 (2)
C(6)	0.7794 (4)	0.7056 (6)	0.5071 (7)	0.0524 (2)	0.0491 (2)	0.0467 (2)	0.0088 (2)	0.0141 (2)	−0.0098 (2)
C(7)	0.7082 (4)	0.7645 (6)	0.4685 (7)	0.0591 (2)	0.0433 (2)	0.0516 (2)	0.0115 (2)	0.0168 (2)	0.0001 (2)
C(8)	0.6154 (4)	0.6826 (6)	0.3834 (7)	0.0533 (2)	0.0307 (2)	0.0477 (2)	0.0127 (2)	0.0019 (2)	0.0034 (2)
C(9)	0.6171 (3)	0.5127 (5)	0.3236 (6)	0.0361 (2)	0.0357 (2)	0.0334 (2)	0.0125 (2)	0.0081 (2)	0.0036 (2)
C(10)	0.6661 (3)	0.2450 (6)	0.5398 (7)	0.0330 (2)	0.0658 (2)	0.0650 (2)	0.0427 (2)	0.0052 (2)	0.0043 (2)
C(11)	0.8299 (4)	0.4661 (7)	0.3341 (8)	0.0405 (2)	0.0602 (3)	0.0790 (3)	0.0197 (2)	0.0218 (2)	0.0060 (2)
C(12)	0.8683 (4)	0.8036 (8)	0.5939 (9)	0.0593 (3)	0.0652 (3)	0.0961 (3)	0.0166 (3)	0.0092 (3)	−0.0154 (3)
C(13)	0.5860 (4)	0.7009 (6)	0.2128 (8)	0.0881 (3)	0.0400 (2)	0.0673 (3)	0.0255 (2)	0.0104 (2)	0.0156 (2)
C(14)	0.6326 (4)	0.4692 (6)	0.1397 (7)	0.0488 (2)	0.0422 (2)	0.0425 (2)	0.0182 (2)	0.0069 (2)	−0.0003 (2)
C(15)	0.7636 (3)	0.2609 (6)	0.6144 (7)	0.0404 (2)	0.0547 (2)	0.0600 (2)	0.0387 (2)	0.0015 (2)	0.0021 (2)
C(16)	0.7990 (4)	0.3648 (7)	0.7793 (8)	0.0551 (3)	0.0876 (3)	0.0609 (3)	0.0382 (2)	0.0026 (2)	0.0038 (2)
C(17)	0.8887 (5)	0.3833 (9)	0.8471 (9)	0.0731 (3)	0.0988 (3)	0.0794 (3)	0.0369 (3)	−0.0117 (3)	−0.0028 (3)
C(18)	0.9434 (5)	0.2983 (8)	0.7498 (10)	0.0577 (3)	0.0955 (3)	0.1076 (3)	0.0550 (3)	−0.0073 (3)	0.0034 (3)
C(19)	0.9094 (4)	0.1963 (8)	0.5884 (10)	0.0574 (3)	0.0758 (3)	0.1128 (3)	0.0468 (3)	0.0205 (3)	0.0197 (3)
C(20)	0.8187 (4)	0.1753 (7)	0.5196 (8)	0.0464 (3)	0.0596 (3)	0.0863 (3)	0.0383 (2)	0.0106 (2)	0.0125 (2)
C(2A)	0.5043 (3)	0.1418 (5)	0.2490 (6)	0.0409 (2)	0.0419 (2)	0.0478 (2)	0.0183 (2)	0.0107 (2)	0.0028 (2)
C(2B)	0.3604 (3)	0.0067 (5)	0.1277 (7)	0.0364 (2)	0.0354 (2)	0.0685 (2)	0.0120 (2)	−0.0035 (2)	−0.0058 (2)
C(21)	0.2718 (3)	0.0340 (5)	0.0516 (6)	0.0381 (2)	0.0410 (2)	0.0449 (2)	0.0077 (2)	0.0088 (2)	0.0049 (2)
C(22)	0.2001 (4)	−0.0800 (6)	0.0061 (7)	0.0439 (2)	0.0497 (2)	0.0708 (2)	0.0140 (2)	0.0032 (2)	−0.0019 (2)
C(23)	0.1178 (4)	−0.0641 (7)	−0.0694 (8)	0.0475 (2)	0.0655 (2)	0.0850 (3)	0.0061 (2)	0.0054 (2)	0.0035 (2)
C(24)	0.1056 (4)	0.0632 (7)	−0.1010 (8)	0.0535 (3)	0.0715 (3)	0.0901 (3)	0.0144 (2)	−0.0058 (2)	0.0168 (2)
C(25)	0.1763 (4)	0.1766 (7)	−0.0553 (8)	0.0660 (3)	0.0673 (3)	0.0896 (3)	0.0348 (2)	−0.0044 (2)	0.0111 (2)
C(26)	0.2593 (4)	0.1628 (6)	0.0222 (7)	0.0539 (2)	0.0561 (2)	0.0691 (2)	0.0270 (2)	0.0046 (2)	0.0048 (2)
O(1)	0.4569 (2)	0.4434 (4)	0.2804 (4)	0.0374 (2)	0.0480 (2)	0.0593 (2)	0.0219 (2)	0.0050 (2)	0.0115 (1)
O(2)	0.6193 (3)	0.5808 (4)	0.0845 (5)	0.0985 (2)	0.0535 (2)	0.0447 (2)	0.0289 (2)	0.0122 (2)	0.0087 (2)
O(3)	0.6504 (3)	0.3528 (4)	0.0492 (5)	0.0749 (2)	0.0453 (2)	0.0452 (2)	0.0072 (2)	0.0200 (2)	0.0144 (2)
O(4)	0.5350 (2)	0.0288 (4)	0.2289 (5)	0.0461 (2)	0.0361 (2)	0.1025 (2)	0.0254 (2)	0.0012 (2)	0.0068 (2)
O(5)	0.4188 (2)	0.1498 (3)	0.2065 (4)	0.0295 (2)	0.0369 (2)	0.0618 (2)	0.0075 (1)	0.0027 (1)	0.0033 (1)

[a]) Numbering according to *Fig. 2*.

Table 3. *Bond distances and selected bond angles of structure* **4**

atoms[a])	distance [Å]	atoms[a])	distance [Å]	atoms[a])	angle [°]
C(1)–N	1.321 (5)	C(15)–C(16)	1.485 (9)	C(9)–C(1)–N	109.8 (4)
N–C(3)	1.452 (6)	C(14)–O(3)	1.185 (5)	C(1)–N–C(3)	116.1 (4)
C(3)–C(4)	1.552 (6)	C(14)–O(4)	1.326 (5)	N–C(3)–C(4)	103.9 (3)
C(4)–C(5)	1.549 (6)	O(4)–C(17)	1.441 (6)	C(3)–C(4)–C(9)	105.9 (4)
C(5)–C(6)	1.527 (8)	C(10)–C(18)	1.507 (7)	C(4)–C(9)–C(1)	103.6 (3)
C(6)–C(7)	1.314 (7)	C(18)–C(19)	1.382 (8)	C(3)–C(4)–C(5)	112.0 (3)
C(7)–C(8)	1.504 (6)	C(19)–C(20)	1.403 (12)	C(5)–C(4)–C(9)	113.0 (3)
C(8)–C(9)	1.558 (6)	C(20)–C(21)	1.369 (12)	C(4)–C(9)–C(8)	111.9 (3)
C(1)–C(9)	1.533 (7)	C(21)–C(22)	1.367 (12)	C(7)–C(8)–C(9)	110.4 (3)
C(4)–C(9)	1.563 (6)	C(22)–C(23)	1.400 (9)	C(6)–C(7)–C(8)	120.3 (5)
C(1)–O(1)	1.234 (6)	C(18)–C(23)	1.408 (9)	C(5)–C(6)–C(7)	116.5 (4)
C(3)–C(10)	1.545 (6)			C(4)–C(5)–C(6)	108.4 (4)
C(5)–C(11)	1.517 (7)			C(1)–C(9)–C(14)	109.6 (4)
C(6)–C(12)	1.518 (7)			C(8)–C(9)–C(14)	110.6 (3)
C(8)–C(13)	1.524 (7)			O(1)–C(1)–C(9)	123.7 (3)
C(9)–C(14)	1.532 (7)			O(4)–C(14)–C(9)	110.3 (3)
C(13)–O(2)	1.455 (5)			C(9)–C(8)–C(13)	112.7 (4)
O(2)–C(15)	1.312 (7)			C(6)–C(5)–C(11)	116.2 (4)
C(15)–O(5)	1.191 (9)			C(5)–C(6)–C(12)	120.4 (4)
				N–C(3)–C(10)	112.7 (3)
				N–C(1)–O(1)	126.5 (4)

[a]) Numbering according to *Fig. 1.*

Table 4. *Bond distances and selected bond angles of structure* **2**

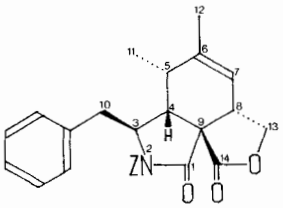
atoms[a])	distance [Å]	atoms[a])	distance [Å]	atoms[a])	angle [°]
C(1)–N	1.405 (7)	C(14)–O(3)	1.197 (6)	C(9)–C(1)–N	105.3 (4)
N–C(3)	1.502 (6)	C(10)–C(15)	1.511 (7)	C(1)–N–C(3)	113.2 (4)
C(3)–C(4)	1.544 (7)	C(15)–C(16)	1.393 (7)	N–C(3)–C(4)	100.6 (4)
C(4)–C(5)	1.537 (7)	C(16)–C(17)	1.384 (10)	C(3)–C(4)–C(9)	104.3 (4)
C(5)–C(6)	1.526 (9)	C(17)–C(18)	1.384 (11)	C(4)–C(9)–C(1)	101.9 (5)
C(6)–C(7)	1.330 (9)	C(18)–C(19)	1.363 (9)	C(3)–C(4)–C(5)	121.9 (4)
C(7)–C(8)	1.508 (7)	C(19)–C(20)	1.397 (9)	C(5)–C(4)–C(9)	113.6 (5)
C(8)–C(9)	1.540 (7)	C(15)–C(20)	1.381 (8)	C(4)–C(9)–C(8)	113.6 (4)
C(1)–C(9)	1.518 (7)	N–C(2A)	1.394 (6)	C(7)–C(8)–C(9)	108.9 (5)
C(4)–C(9)	1.523 (8)	C(2A)–O(4)	1.195 (7)	C(6)–C(7)–C(8)	127.2 (5)
C(1)–O(1)	1.201 (6)	C(2A)–O(5)	1.328 (6)	C(5)–C(6)–C(7)	124.1 (5)
C(3)–C(10)	1.537 (10)	O(5)–C(2B)	1.452 (5)	C(4)–C(5)–C(6)	107.4 (5)
C(5)–C(11)	1.518 (9)	C(2B)–C(21)	1.507 (7)	C(1)–C(9)–C(14)	104.2 (4)
C(6)–C(12)	1.511 (8)	C(21)–C(22)	1.391 (7)	C(8)–C(9)–C(14)	103.6 (5)
C(8)–C(13)	1.533 (9)	C(22)–C(23)	1.379 (8)	O(1)–C(1)–C(9)	126.8 (5)
C(9)–C(14)	1.531 (8)	C(23)–C(24)	1.374 (11)	O(3)–C(14)–C(9)	127.8 (6)
C(13)–O(2)	1.472 (7)	C(24)–C(25)	1.377 (9)	C(9)–C(8)–C(13)	101.8 (4)
O(2)–C(14)	1.343 (8)	C(25)–C(26)	1.388 (9)	C(6)–C(5)–C(11)	111.8 (6)
		C(21)–C(26)	1.379 (9)	C(5)–C(6)–C(12)	115.0 (5)
				N–C(3)–C(10)	109.3 (5)
				N–C(1)–O(1)	127.8 (5)

[a]) Numbering according to *Fig. 2.*

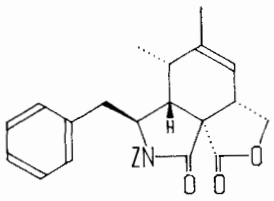
proxiphomin, but at C(9) the configuration was different. Thus the product of the *Diels-Alder* reaction possesses the structure *rac*-(3*R**, 3a*S**, 4*R**, 7*R**, 7a*S**)-3-benzyl-2-benzyloxycarbonyl-4, 5-dimethyl-7, 7a-(3′-oxo-2′-oxatrimethylene)-3a, 4, 7, 7a-tetrahydroisoindolin-1-one[1]) (**2**; $C_{27}H_{27}NO_5$).

Schmidlin et al. [1] were able to convert the lactone **2** *via* the hydroxy ester **3** into a lactone which was anticipated to have structure **1b** with all chiral centres corresponding to the configuration of the natural cytochalasans (s. *Scheme*).

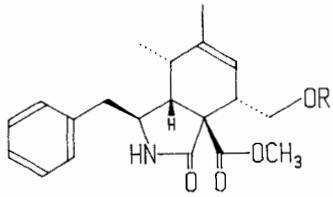
Scheme



1a Z = $C_6H_5CH_2OCO$
1b Z = H



2 Z = $C_6H_5CH_2OCO$



3 R = H
4 R = Ac

In order to prove this assumption the *O*-acetyl derivative **4** of the hydroxy acid **3** was subjected to an X-ray analysis as well, proving its structure to be *rac*-methyl (3*R**, 3a*S**, 4*R**, 7*R**, 7a*R**)-7-acetoxymethyl-3-benzyl-4, 5-dimethyl-1-oxo-3a, 4, 7, 7a-tetrahydroisoindoline-7a-carboxylate[1]) (**4**).

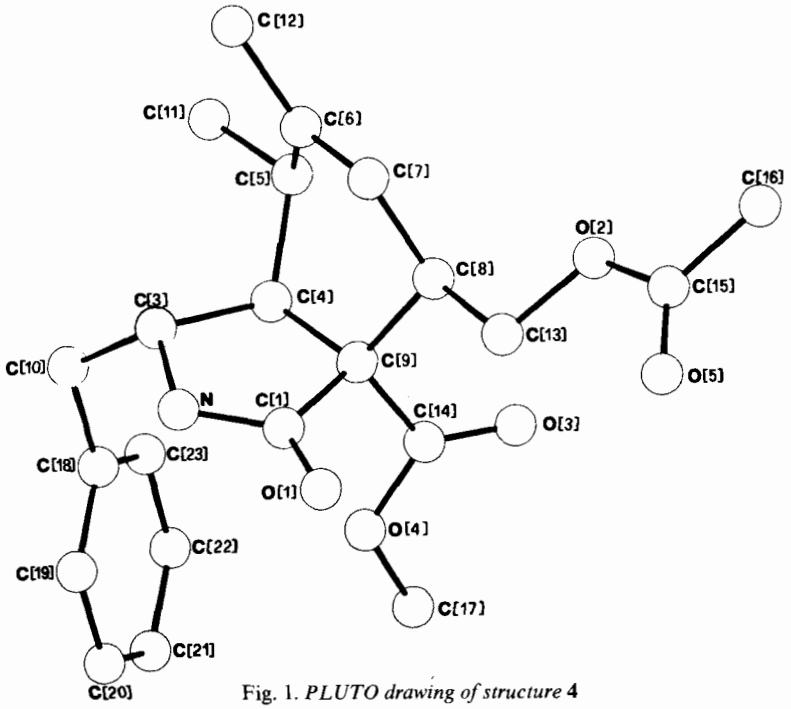


Fig. 1. *PLUTO drawing of structure* **4**

Crystallographic Part. – Crystallographic data of structure **4** ($C_{22}H_{27}NO_5$) and structure **2** ($C_{27}H_{27}NO_5$) are: triclinic, space group $P\bar{1}$, $a = 13.652(5)$ Å, $b = 10.926(3)$ Å, $c = 7.755(2)$ Å, $\alpha = 111.554(4)$ Å, $\beta = 85.541(3)$ Å, $\gamma = 104.813(4)$ Å, and $a = 15.394(4)$ Å, $b = 9.674(3)$ Å, $c = 8.522(3)$ Å, $\alpha = 111.04(4)$ Å, $\beta = 93.65(4)$ Å, $\gamma = 95.01(4)$ Å, respectively. Intensities of 3644 independent reflexions

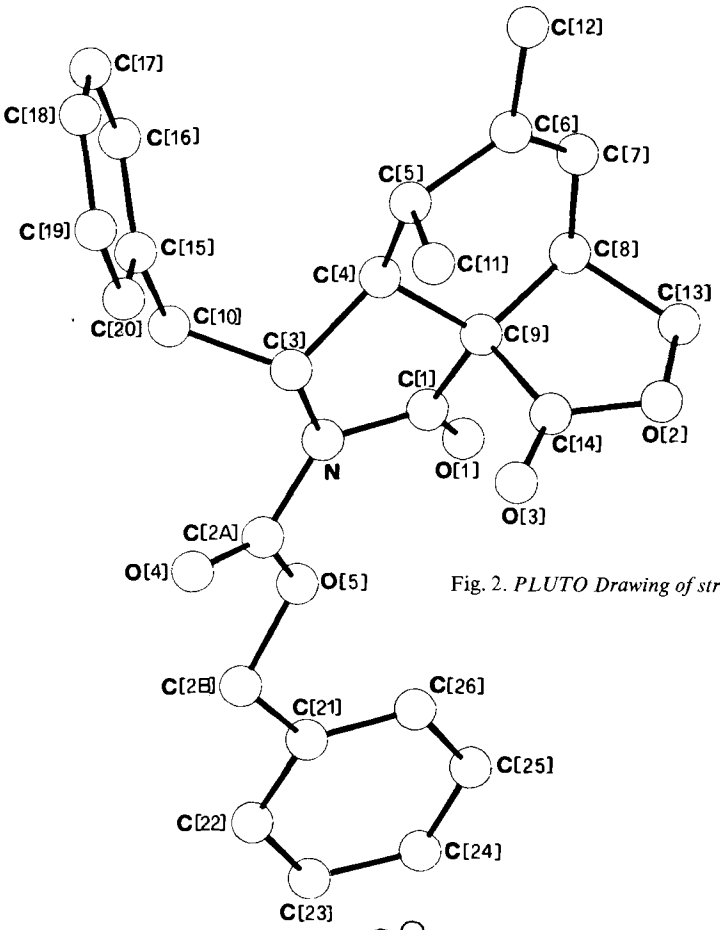


Fig. 2. *PLUTO Drawing of structure* **2**

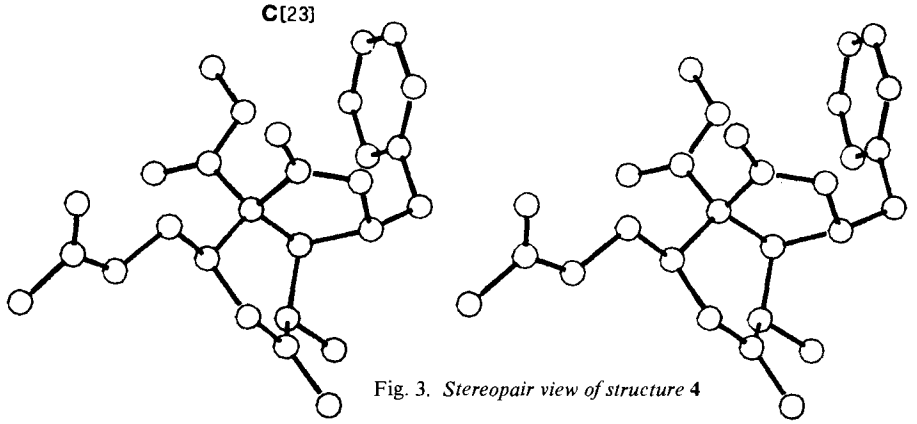


Fig. 3. *Stereopair view of structure* **4**

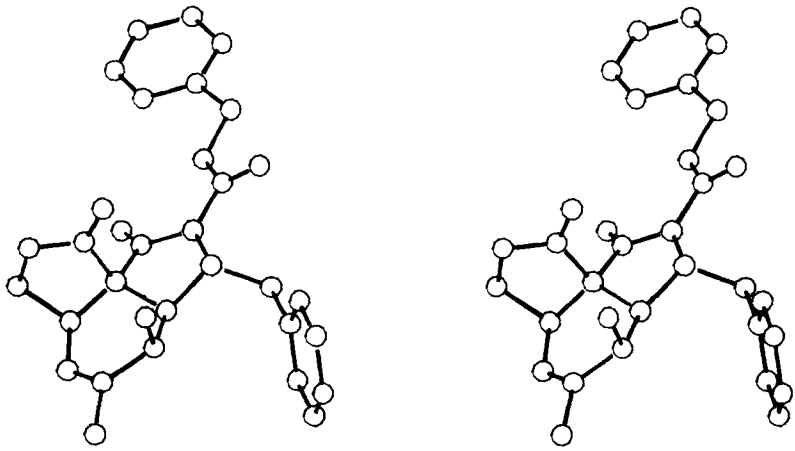


Fig. 4. *Stereopair view of structure* **2**

for structure **4** and 2812 independent reflexions for structure **2** were collected ($\theta/2\theta$ scan) in the range $2<\theta<26°$ with a *Philips*-PW-1100 diffractometer equipped with a fine focus molybdenum tube and a graphite monochromator (MoK_α, $\lambda=0.71069$ Å). No corrections for absorption were applied. The structures were solved by direct methods using the programs MULTAN 78 and SHELX-76 [4]. Scattering factors for neutral atoms including anomalous dispersion were taken from *Cromer et al.* [5]. The structures yielded 2012 and 2081 reflexions, respectively, with $(F_o)>2\sigma(F_o)$ which were used in the anisotropic refinements. All protons were localized in both cases. Final difference *Fourier* maps showed no electron-density peaks higher than 0.5 e/Å^3. The final R-indices were 0.067 and 0.062 ($R=\sum\|F_0|-|F_c\|/|F_0|$). Atomic positional and thermal parameters of structure **4** and **2** are given in *Table 1* and *Table 2*, while interatomic distances and selected bond angles are listed in *Table 3* and *Table 4*.

Discussion. – *Figure 1* shows a PLUTO[2]) drawing of the *O*-acetyl derivative **4**. As *Schmidlin et al.* report [1] **4** is obtained in its desired configuration from the lactone **2**. The PLUTO plot of **2** is given in *Figure 2*. *Figures 3* and *4* are stereopair views of structure **4** and **2**, respectively, illustrating the different configurations of the molecules in which bond distances and bond angles of the macrocyclic rings are practical the same.

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