References

Belletti, D., Ugozzoli, F., Cantoni, A. & Pasquinelli, G. (1979). *Gestione on Line di Diffrattometro a Cristallo Singolo Siemens AED con Sistema General Automation Jumbo* 220. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma. Internal Reports 1,2,3–79.

Bocelli, G. & Grenier-Loustalot, M. F. (1983*a*). *Acta Cryst.* C**39**, 636–638.

Bocelli, G. & Grenier-Loustalot, M. F. (1983*b*). *Acta Cryst.* C**39**, 1659–1661.

Bocelli, G. & Grenier-Loustalot, M. F. (1984*a*). *Acta Cryst.* C**40**, 1397–1399.

Bocelli, G. & Grenier-Loustalot, M. F. (1984*b*). *Acta Cryst.* C**40**, 1400–1402.

Bocelli, G. & Grenier-Loustalot, M. F. (1984*c*). Unpublished results.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–589.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Pérez, S. & Brisse, F. (1976). *Acta Cryst.* B**32**, 1518–1521.

Pérez, S. & Brisse, F. (1977). *Acta Cryst.* B**33**, 3259–3262.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sutton, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions, Suppl.* 1956–1959. Spec. Publ. No. 18. London: The Chemical Society.

Acta Cryst. (1985). C**41**, 252–255

Structure of a 1/1 Molecular Complex of Tylolupenol A Acetate and Tylolupenol B Acetate, $C_{32}H_{52}O_2.C_{32}H_{52}O_2$

By Hiroshi Nakai, Motoo Shiro, Takehiko Tozyo and Hiroshi Ishii

Shionogi Research Laboratories, Shionogi & Co. Ltd, Fukushima-ku, Osaka 553, Japan

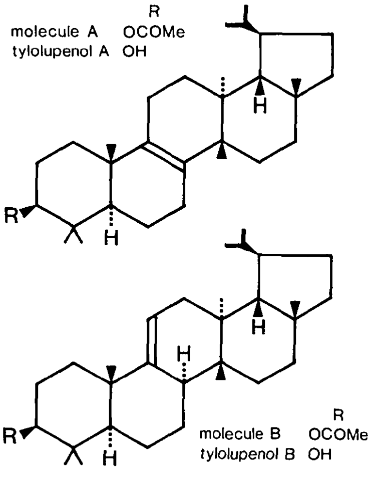
and Zhen-Wen Xu

Tianjin Institute of Medical and Pharmaceutical Science, 2 He Yan Road, He-Ping District, Tianjin, China

(*Received* 27 *August* 1984; *accepted* 1 *October* 1984)

Abstract. $M_r = 937\cdot5$, triclinic, $P1$, $a = 13\cdot562\,(2)$, $b = 14\cdot492\,(2)$, $c = 7\cdot691\,(2)$ Å, $\alpha = 93\cdot61\,(1)$, $\beta = 90\cdot77\,(1)$, $\gamma = 71\cdot31\,(1)°$, $V = 1429\cdot1\,(6)$ Å^3, $Z = 1$, $D_x = 1\cdot089$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot54178$ Å, $\mu = 0\cdot50$ mm^{-1}, $F(000) = 520$, $T = 295$ K, $R = 0\cdot044$ for 4883 reflections. The coexistence of the two isomeric acetate molecules in the present complex suggests the presence of the corresponding isomers in tylolupenol. The molecules have the migrated lupane skeleton. There are no unusual bond distances and angles, and no intermolecular contacts shorter than the sums of the van der Waals radii.

Introduction. Xu, Wang & Zhao (1983) isolated five crystalline substances from the EtOH extract of roots of *Tylophora kerrii* Craib. Infrared, ^{13}C-NMR and mass spectral evidence suggested one of them, tylolupenol, to be a 1:1 mixture of two triterpene alcohols, isomeric with respect to the position of the double bond, which could not be separated by silica-gel column chromatography. X-ray study of its acetate revealed a 1:1 molecular complex of *D:C*-friedolup-8-en-3β-ol acetate (molecule *A*) and *D:C*-friedolup-9(11)-en-3β-ol acetate (molecule *B*). Thus, tylolupenol crystals probably consist of the corresponding alcohols, tylolupenols A and B.



Experimental. The title compounds were obtained by esterification of tylolupenol, a crystalline substance extracted from the roots of *Tylophora kerrii* Craib.

Prismatic colorless crystals grown from methanol–chloroform solution. Crystal 0·3 × 0·3 × 0·3 mm, Rigaku AFC-5UD diffractometer, graphite-monochromatized Cu $K\alpha$. Cell dimensions from 2θ angles for 25 reflections ($33° < 2\theta < 45°$). Intensities

0108-2701/85/020252-04$01.50

measured up to $2\theta = 140°$ in h −16/16, k −17/17 and l 0/9. Three standard reflections monitored every 100 measurements showed no significant variation. 5269 unique reflections measured, 5005 intensities observed with $|F_o| > \sigma_1(F_o)$, where $\sigma_1(F_o)$ is the e.s.d. due to counting errors. No absorption corrections. Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms located on difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom set equal to the isotropic temperature factor of the bonded atom. $R = 0{\cdot}044$, $wR = 0{\cdot}062$, $S = 1{\cdot}07$ for 4883 observed reflections ($w \neq 0$).* No significant peaks in final difference map, highest peak 0·2 e Å^{-3}. $\sum w|\Delta F|^2$ minimized, $w = 1/\sigma^2(F_o)$ for $|F_c| \geq \sigma(F_o)$ and $|\Delta F| < 3\sigma(F_o)$, $w = 0$ otherwise, $\sigma(F_o) = [\sigma_1^2(F_o) + 0{\cdot}00227 \times |F_o|^2]^{1/2}$ (Grant, Killean & Lawrence, 1969). Ratio of final shift to e.s.d. less than 0·5. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1{\sim}4$) (*International Tables for X-ray Crystallography*, 1974). Calculations performed by a FACOM M-150F computer at Shionogi Research Laboratories.

Discussion. The atomic coordinates and equivalent isotropic temperature factors of the non-H atoms are listed in Table 1. Bond distances and angles, none of which were unusual, are given in Table 2. Perspective views with the atom-numbering systems are shown in Fig. 1. The absolute configurations were determined on the basis of the β configuration of the methyl group attached to C(10) in lupane.

The ring junctions *A*/*B*, *C*/*D* and *D*/*E* are respectively *trans, trans* and *cis* for molecules *A* and *B*. Rings *A*–*E* respectively adopt the chair, envelope, envelope, chair and envelope conformations in molecule *A* and the chair, skew boat, half-chair, chair and envelope in molecule *B*, though they are more or less distorted.

The crystal structure is presented in Fig. 2. There are no intermolecular contacts shorter than the sum of the van der Waals radii and no discrete units of molecules *A* and *B* bound by prominent intermolecular forces.

Since molecules *A* and *B* were obtained by esterification of tylolupenol, their corresponding alcohols probably coexist in tylolupenol. Tylolupenol was recently separated by automatic recycling HPLC by Kawanishi & Hashimoto (1984), who confirmed the structures of the alcohols with spectral evidence.

Biogenetical consideration revealed that tylolupenols A and B are members of the migrated lupane triterpenoid series. Other types of the migrated lupane series, cymbopogone and cymbopogonol (Yokoyama, Tsuyuki, Nakamura, Takahashi, Hanson & Matsushita, 1980), and neolupenol, tarolupenol and their corresponding acetates (Ageta, Shiojima, Masuda & Lin, 1981), have also been isolated from natural sources.

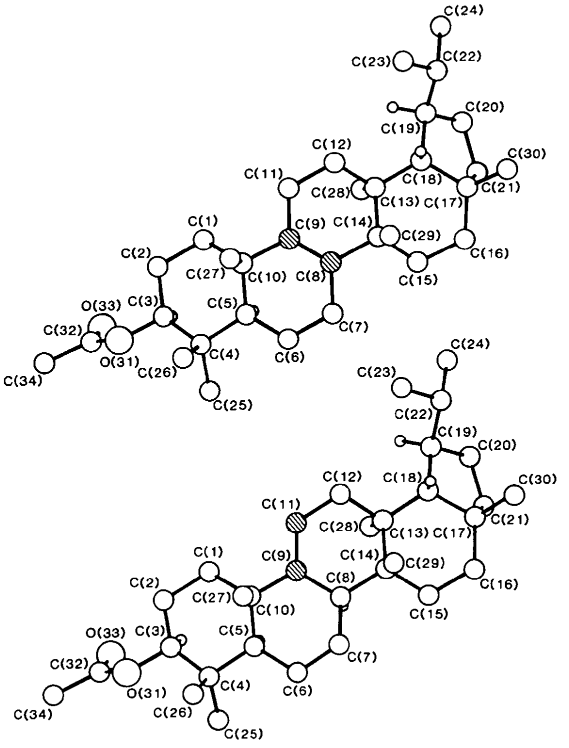


Fig. 1. Perspective views of the molecules *A* (upper) and *B* (lower). Hatched circles represent the position of the double bond. The H atoms attached to the asymmetric C atoms are also shown.

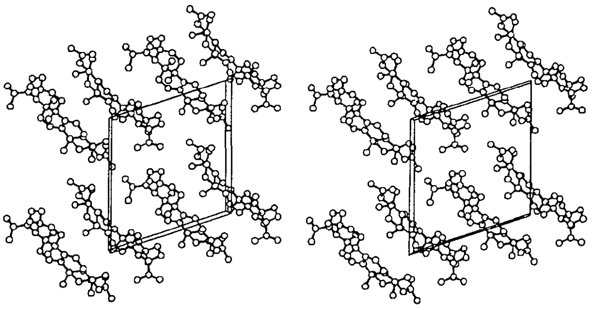


Fig. 2. Stereoview of the molecular packing. The view is approximately down the *c* axis. The molecule at the lower left is a molecule *A*, and the next one at the right-hand side is a molecule *B*.

---

* Lists of structure factors, anisotropic temperature factors of the non-H atoms, and coordinates of the H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39798 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2 $\times 10^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	Molecule *A*				Molecule *B*			
	x	y	z	B_{eq}	x	y	z	B_{eq}
C(1)	6676	9347	1644	460 (7)	8923 (3)	12259 (3)	3847 (5)	517 (8)
C(2)	7445 (3)	8376 (3)	937 (5)	517 (8)	8072 (3)	13175 (3)	4572 (5)	571 (9)
C(3)	8522 (3)	8230 (3)	1647 (5)	463 (7)	8227 (3)	14080 (3)	3931 (5)	514 (8)
C(4)	8592 (3)	8263 (3)	3635 (5)	409 (6)	8217 (3)	14111 (3)	1952 (5)	505 (8)
C(5)	7755 (3)	9232 (3)	4311 (4)	369 (6)	9046 (3)	13148 (3)	1236 (5)	434 (7)
C(6)	7765 (3)	9417 (3)	6286 (5)	472 (7)	9162 (4)	13098 (3)	−759 (5)	642 (11)
C(7)	7114 (4)	10431 (3)	6847 (5)	734 (11)	9843 (4)	12163 (3)	−1546 (5)	735 (12)
C(8)	6184 (3)	10907 (3)	5807 (4)	387 (6)	10511 (3)	11446 (3)	−347 (4)	425 (7)
C(9)	5929 (3)	10428 (3)	4347 (4)	372 (6)	9950 (3)	11344 (3)	1216 (4)	379 (6)
C(10)	6619 (3)	9411 (3)	3655 (4)	368 (6)	8979 (3)	12167 (3)	1850 (5)	414 (6)
C(11)	4945 (3)	10850 (3)	3430 (5)	501 (8)	10268 (3)	10489 (3)	2080 (5)	483 (7)
C(12)	4141 (3)	11736 (3)	4272 (5)	460 (7)	11126 (3)	9593 (3)	1421 (5)	423 (7)
C(13)	4629 (3)	12424 (3)	5289 (4)	355 (6)	11897 (3)	9816 (3)	184 (4)	371 (6)
C(14)	5374 (3)	11816 (3)	6666 (4)	367 (6)	11236 (3)	10485 (3)	−1221 (4)	412 (7)
C(15)	5884 (3)	12502 (3)	7644 (6)	602 (9)	12009 (4)	10690 (3)	−2492 (5)	564 (10)
C(16)	5082 (4)	13329 (3)	8682 (7)	829 (13)	12706 (4)	9751 (3)	−3431 (5)	682 (12)
C(17)	4116 (3)	13894 (3)	7671 (6)	584 (9)	13248 (4)	8911 (3)	−2293 (5)	592 (10)
C(18)	3743 (3)	13271 (3)	6232 (5)	390 (6)	12598 (3)	8825 (3)	−688 (4)	413 (7)
C(19)	3073 (3)	14033 (3)	4985 (5)	513 (8)	13427 (3)	8192 (3)	551 (5)	445 (7)
C(20)	3391 (4)	14965 (3)	5347 (8)	866 (16)	14491 (3)	8260 (3)	−54 (6)	671 (11)
C(21)	4255 (4)	14727 (3)	6687 (8)	828 (14)	14268 (4)	8974 (4)	−1445 (6)	704 (12)
C(22)	1888 (3)	14261 (3)	5123 (6)	652 (10)	13447 (3)	7121 (3)	656 (5)	514 (8)
C(23)	1551 (4)	13360 (4)	4832 (10)	987 (19)	12442 (4)	7055 (3)	1391 (6)	660 (11)
C(24)	1313 (4)	15013 (5)	3866 (9)	1043 (18)	14343 (4)	6555 (3)	1784 (6)	686 (11)
C(25)	9683 (3)	8307 (3)	4157 (6)	532 (8)	8551 (4)	14988 (3)	1446 (6)	682 (11)
C(26)	8482 (3)	7338 (3)	4353 (5)	490 (7)	7101 (3)	14276 (3)	1253 (6)	663 (10)
C(27)	6107 (3)	8685 (3)	4279 (5)	494 (8)	8030 (3)	11895 (3)	1134 (6)	612 (10)
C(28)	5238 (3)	12783 (3)	3971 (5)	518 (8)	12499 (3)	10366 (3)	1314 (5)	473 (7)
C(29)	4786 (3)	11426 (3)	7984 (5)	583 (9)	10540 (4)	9978 (3)	−2255 (5)	561 (9)
C(30)	3231 (4)	14319 (4)	9050 (6)	842 (14)	13507 (4)	7970 (4)	−3487 (6)	797 (14)
O(31)	9197 (3)	7282 (2)	955 (4)	557 (6)	7404 (3)	14925 (3)	4667 (5)	681 (7)
C(32)	9743 (3)	7257 (3)	−492 (5)	452 (7)	7613 (4)	15402 (3)	6082 (5)	600 (10)
O(33)	9780 (3)	7966 (3)	−1152 (4)	665 (7)	8463 (3)	15226 (3)	6695 (5)	807 (10)
C(34)	10313 (3)	6220 (3)	−1117 (6)	645 (10)	6660 (5)	16184 (4)	6782 (8)	947 (17)

Table 2. *Bond lengths* (Å) *and angles* (°) *with estimated standard deviations in parentheses*

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(1)–C(2)	1·530 (4)	1·532 (6)	C(6)–C(7)	1·491 (7)	1·471 (8)	C(13)–C(18)	1·563 (6)	1·561 (6)	C(18)–C(19)	1·559 (6)	1·560 (6)
C(1)–C(10)	1·545 (4)	1·534 (6)	C(7)–C(8)	1·480 (7)	1·499 (7)	C(13)–C(28)	1·531 (6)	1·539 (6)	C(19)–C(20)	1·553 (8)	1·557 (6)
C(2)–C(3)	1·507 (6)	1·506 (6)	C(8)–C(9)	1·381 (6)	1·471 (6)	C(14)–C(15)	1·538 (6)	1·551 (7)	C(19)–C(22)	1·537 (6)	1·551 (6)
C(3)–C(4)	1·529 (6)	1·525 (6)	C(8)–C(14)	1·537 (6)	1·543 (6)	C(14)–C(29)	1·538 (6)	1·554 (7)	C(20)–C(21)	1·519 (9)	1·496 (7)
C(3)–O(31)	1·457 (6)	1·456 (6)	C(9)–C(10)	1·537 (6)	1·527 (6)	C(15)–C(16)	1·525 (7)	1·529 (8)	C(22)–C(23)	1·519 (9)	1·516 (7)
C(4)–C(5)	1·560 (6)	1·559 (6)	C(9)–C(11)	1·469 (6)	1·384 (6)	C(16)–C(17)	1·535 (7)	1·527 (8)	C(22)–C(24)	1·516 (9)	1·525 (7)
C(4)–C(25)	1·548 (6)	1·552 (7)	C(10)–C(27)	1·536 (6)	1·547 (6)	C(17)–C(18)	1·565 (6)	1·561 (7)	O(31)–C(32)	1·337 (6)	1·329 (7)
C(4)–C(26)	1·532 (6)	1·549 (6)	C(11)–C(12)	1·508 (6)	1·504 (6)	C(17)–C(21)	1·527 (8)	1·548 (8)	C(32)–O(33)	1·190 (6)	1·195 (7)
C(5)–C(6)	1·526 (6)	1·539 (7)	C(12)–C(13)	1·535 (6)	1·544 (6)	C(17)–C(30)	1·557 (7)	1·540 (8)	C(32)–C(34)	1·504 (6)	1·499 (9)
C(5)–C(10)	1·560 (6)	1·556 (6)	C(13)–C(14)	1·561 (6)	1·568 (6)						

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(2)–C(1)–C(10)	111·5 (2)	113·3 (4)	C(1)–C(10)–C(5)	106·4 (3)	107·3 (3)	C(16)–C(17)–C(18)	115·1 (4)	114·8 (4)
C(1)–C(2)–C(3)	111·3 (3)	110·7 (4)	C(1)–C(10)–C(9)	110·7 (3)	110·8 (4)	C(16)–C(17)–C(21)	114·2 (4)	114·5 (5)
C(2)–C(3)–C(4)	114·8 (4)	114·0 (4)	C(1)–C(10)–C(27)	110·2 (3)	109·1 (4)	C(16)–C(17)–C(30)	106·0 (4)	106·5 (4)
C(2)–C(3)–O(31)	107·9 (4)	108·3 (4)	C(5)–C(10)–C(9)	109·3 (3)	108·7 (3)	C(18)–C(17)–C(21)	103·5 (4)	102·5 (4)
C(4)–C(3)–O(31)	108·4 (4)	108·9 (4)	C(5)–C(10)–C(27)	114·1 (4)	114·2 (4)	C(18)–C(17)–C(30)	108·7 (4)	110·0 (4)
C(3)–C(4)–C(5)	106·2 (3)	106·4 (3)	C(9)–C(10)–C(27)	106·1 (3)	106·8 (4)	C(21)–C(17)–C(30)	109·1 (4)	108·5 (4)
C(3)–C(4)–C(25)	108·3 (4)	109·1 (4)	C(9)–C(11)–C(12)	118·4 (4)	123·2 (4)	C(13)–C(18)–C(17)	115·2 (4)	115·2 (4)
C(3)–C(4)–C(26)	111·3 (4)	110·3 (4)	C(11)–C(12)–C(13)	112·6 (4)	113·3 (4)	C(13)–C(18)–C(19)	113·9 (3)	114·1 (4)
C(5)–C(4)–C(25)	108·9 (4)	108·7 (4)	C(12)–C(13)–C(14)	107·3 (3)	107·3 (3)	C(17)–C(18)–C(19)	104·5 (3)	103·9 (4)
C(5)–C(4)–C(26)	114·8 (4)	114·9 (4)	C(12)–C(13)–C(18)	108·8 (3)	107·7 (3)	C(18)–C(19)–C(20)	105·9 (4)	105·7 (3)
C(25)–C(4)–C(26)	107·2 (4)	107·2 (4)	C(12)–C(13)–C(28)	107·0 (3)	106·2 (3)	C(18)–C(19)–C(22)	116·0 (4)	116·0 (4)
C(4)–C(5)–C(6)	113·2 (4)	112·4 (4)	C(14)–C(13)–C(18)	109·8 (3)	110·6 (3)	C(20)–C(19)–C(22)	111·5 (4)	112·3 (4)
C(4)–C(5)–C(10)	116·9 (4)	117·9 (4)	C(14)–C(13)–C(28)	110·9 (3)	110·6 (3)	C(19)–C(20)–C(21)	107·1 (5)	107·2 (4)
C(6)–C(5)–C(10)	110·5 (3)	110·6 (4)	C(18)–C(13)–C(28)	113·0 (4)	114·0 (4)	C(17)–C(21)–C(20)	105·7 (5)	104·7 (4)
C(5)–C(6)–C(7)	111·6 (4)	115·3 (4)	C(8)–C(14)–C(13)	111·5 (3)	110·5 (3)	C(19)–C(22)–C(23)	113·0 (4)	112·3 (4)
C(6)–C(7)–C(8)	118·3 (4)	117·6 (4)	C(8)–C(14)–C(15)	112·1 (4)	110·8 (4)	C(19)–C(22)–C(24)	111·7 (4)	111·1 (4)
C(7)–C(8)–C(9)	120·9 (4)	113·1 (4)	C(8)–C(14)–C(29)	105·3 (3)	107·5 (4)	C(23)–C(22)–C(24)	109·2 (5)	108·3 (4)
C(7)–C(8)–C(14)	116·9 (4)	116·2 (4)	C(13)–C(14)–C(15)	107·0 (3)	107·4 (4)	C(3)–O(31)–C(32)	117·7 (4)	118·3 (4)
C(9)–C(8)–C(14)	120·3 (4)	115·6 (4)	C(13)–C(14)–C(29)	112·0 (4)	111·3 (4)	O(31)–C(32)–O(33)	123·8 (4)	123·4 (5)
C(8)–C(9)–C(10)	122·4 (4)	120·0 (4)	C(15)–C(14)–C(29)	109·0 (4)	109·4 (4)	O(31)–C(32)–C(34)	110·6 (4)	111·7 (5)
C(8)–C(9)–C(11)	121·1 (4)	120·6 (4)	C(14)–C(15)–C(16)	111·4 (4)	111·7 (4)	O(33)–C(32)–C(34)	125·6 (4)	124·9 (5)
C(10)–C(9)–C(11)	116·5 (4)	119·4 (4)	C(15)–C(16)–C(17)	115·8 (4)	116·6 (5)			

References

AGETA, H., SHIOJIMA, K., MASUDA, K. & LIN, T. (1981). *Tetrahedron Lett.* **22**, 2289–2290.

GRANT, D. F., KILLEAN, R. C. G. & LAWRENCE, J. L. (1969). *Acta Cryst.* B**25**, 374–376.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 99. Birmingham: Kynoch Press.

KAWANISHI, K. & HASHIMOTO, Y. (1984). Private communication.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

XU, Z.-W., WANG, Q. & ZHAO, J.-J. (1983). *Zhongcaoyao*, **14**, 49–51.

YOKOYAMA, Y., TSUYUKI, T., NAKAMURA, N., TAKAHASHI, T., HANSON, S. W. & MATSUSHITA, K. (1980). *Tetrahedron Lett.* **21**, 3701–3702.

Acta Cryst. (1985). C**41**, 255–257

Elloramycin, $C_{32}H_{36}O_{15}$

BY PETER G. JONES AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, *D*-3400 *Göttingen, Federal Republic of Germany*

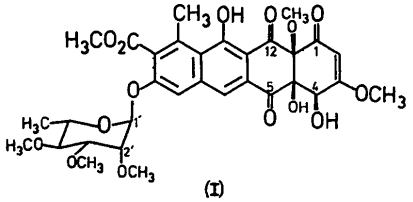
AND JÜRGEN ROHR AND AXEL ZEECK

Institut für Organische Chemie der Universität, Tammannstrasse 2, *D*-3400 *Göttingen, Federal Republic of Germany*

(*Received* 20 *August* 1984; *accepted* 2 *October* 1984)

Abstract. $C_{32}H_{36}O_{15}.xH_2O$ (x *ca* 0·5), M_r(for $x = 0·5$) = 669·64, monoclinic, $P2_1$, $a = 10·801$ (2), $b = 9·277$ (2), $c = 16·660$ (3) Å, $\beta = 105·71$ (2)°, $V = 1607·0$ (6) Å^3, $Z = 2$, $D_x = 1·38$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1·0$ cm^{-1}, $F(000) = 706$, $T = 293$ K. $R = 0·041$ for 3566 observed reflections. The structural features determined from chemical and spectroscopic studies are confirmed and extended. The molecule is an anthracycline analogue containing a permethylated α-L-rhamnose moiety. All ring systems show the expected conformations, the non-aromatic fused rings both being half-chairs. The extended structure, including the partly occupied water, is linked by hydrogen bonds.

Introduction. Elloramycin (I), a new antibiotic produced by *Streptomyces olivaceus* (strain Tü 2353), was detected by chemical screening and characterized by chemical and spectroscopic methods (Drautz, Reuschenbach, Zähner, Rohr & Zeeck, 1984). Elloramycin is an anthracycline-like antibiotic with weak antibacterial and antitumour activities; the aglycone resembles tetracenomycin C (Weber, Zähner, Siebers, Schröder & Zeeck, 1979), and the sugar moiety consists of permethylated α-L-rhamnose. We describe below the crystal-structure analysis of elloramycin by which the total structure of the antibiotic, including the relative stereochemistry, is proved.



(I)

Large, air-stable yellow blocks and plates were obtained by diffusion of n-pentane into an acetone solution at room temperature.

Experimental. Crystal 0·6 × 0·55 × 0·4 mm. 5109 profile-fitted intensities (Clegg, 1981) recorded on a Stoe–Siemens four-circle diffractometer. Monochromated Mo $K\alpha$ radiation, $2\theta_{\max}$ 55°, h $-14\rightarrow13$, k $0\rightarrow12$, l $0\rightarrow21$ and some Friedel opposites with negative k. Three check reflections, no intensity change. $R_{\rm int}$ 0·023 for 3913 unique reflections (Friedel opposites merged), 3566 with $F > 4\sigma(F)$ used for all calculations (program systems *SHELXTL* and *SHELX*84, written by GMS). Cell constants refined from 2θ values of 40 reflections in range 20–24°. No correction for absorption.

Structure solution by random-start multisolution direct methods. Refinement on $|F|$ to R 0·041, wR 0·043 [all non-H atoms anisotropic; H atoms (except OH) included using a riding model with C—H 0·96 Å,

0108-2701/85/020255-03$01.50