Table 4. *Selected torsion angles* (°)

	(II*A*)	(II*B*)	Pyridinium ClO_4^-* (*A*)	(*B*)
N(1')—Ag(1)—N(1)—C(2)	−130·5 (3)	−106·1 (4)		
N(1')—Ag(1)—N(1)—C(9)	39·0 (4)	68·9 (4)		
Ag(1)—N(1)—C(2)—C(1)	−14·6 (6)	−6·2 (6)		
C(11)—C(1)—C(2)—N(1)	75·7 (6)	96·5 (6)	67·4 (4)	74·0 (4)
C(17)—C(1)—C(2)—N(1)	−159·9 (5)	−138·6 (5)	−167·1 (3)	−161·1 (3)
C(2)—C(1)—C(11)—C(12)	39·0 (7)	29·4 (7)	34·7 (4)	41·6 (4)
C(2)—C(1)—C(11)—C(16)	−146·1 (5)	−150·9 (5)	−148·9 (3)	−142·7 (3)
C(17)—C(1)—C(11)—C(12)	−86·6 (7)	−96·7 (6)	−91·9 (4)	−84·3 (4)
C(17)—C(1)—C(11)—C(16)	88·3 (7)	83·0 (7)	84·6 (4)	91·4 (4)

* Iwasaki & Yamazaki (1991).

2·97 (1) Å, respectively, which are shorter than the sum of the van der Waals radii (3·24 Å). This interaction is probably related to the unusually small N···Ag···N angle. The O(1)···Ag(1)···O(4), N(1*A*)—Ag(1)···O(1), N(1*A*)—Ag(1)···O(4), N(1*B*)—Ag(1)···O(1) and N(1*B*)—Ag(1)···O(4) coordination angles are 42·8 (3), 78·0 (2), 113·0 (3), 124·1 (2) and 94·9 (3)°, respectively.

For both ligands the configurations are R_C. In the reaction shown in the scheme the absolute configurations of the phenylethyl groups are also retained completely and the reaction mechanism of the ligand coupling *via* hypervalent σ-sulfurane is concluded (Oae, Takeda, Wakabayashi, Iwasaki, Yamazaki & Katsube, 1990).

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Structure of a Dihydrooxazole Oxa-Bridged Octalin*

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Abstract. $C_{26}H_{41}NO_7$, $M_r = 479·61$, triclinic, $P\bar{1}$, $a = 11·894$ (4), $b = 12·360$ (2), $c = 9·969$ (2) Å, $\alpha = 106·11$ (2), $\beta = 100·68$ (2), $\gamma = 99·02$ (2)°, $V = 1349·5$ (6) Å^3, $Z = 2$, $D_x = 1·180$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·079$ mm^{-1}, $F(000) = 520$, $T = 296$ K, $R = 0·041$, $wR = 0·047$ for 3196 observed unique reflections. A new approach to the total synthesis of the nargenicin macrolide system employing a [2,3] Wittig rearrangement to control the remote C16—C17 stereochemistry required characterization of a pivotal intermediate by X-ray single-crystal methods. The structure is characterized by the formation of strong intermolecular alcohol to dihydrooxazole-N hydrogen bonds, an essentially strain-free tricyclic oxa-bridged octalin nucleus, and low-energy conformations of the methoxymethyl ether side chains.

Introduction. The nargenicins [(1*a*), (1*b*)] comprise a new class of macrolide antibiotics which have attracted attention owing to their novel structure and activity against drug-resistant microorganisms (Celmer, Chmurny, Moppett, Ware, Watts & Whipple, 1980; Whaley, Chidester, Mizsak & Wnuk, 1980; Whaley & Coates, 1981). In 1988, we described the first synthesis of the naturally occurring 18-deoxynargenicin A_1 (1*b*), in which the nargenicin

* IUPAC name: (*E*)-4-{(1*S**,2*S**,3*R**,3′*R**,4*R**,4′*R**,5*R**,6*R**,7*R**)-3,5-bis(methoxymethoxy)-4-methyl-11-oxatricyclo[4.4.1.0^{2,7}]-undec-9-en-1-yl}-1-(4,4-dimethyl-4,5-dihydro-1,3-oxazol-2-yl)-2,4-dimethyl-3-buten-1-ol.

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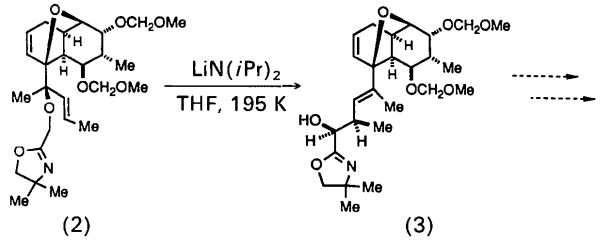
framework was constructed by the addition of an optically active C14—C19 (see scheme) fragment to a racemic precursor of the oxatricycloundecene nucleus (Plata & Kallmerten, 1988). This convergent strategy, while rapidly assembling the major structural features of the nargenicin skeleton, is neither stereoselective nor directly applicable to the syntheses of the 18-hydroxynargenicin congeners, leading us to consider a sterocontrolled linear approach to the development of the remote stereochemistry at C14 and C17 of the nargenicin macrolide system. Recently, we reported such an approach, wherein the [2,3] Wittig rearrangement (Wittman & Kallmerten, 1988) of tertiary allylic ether (2) (Rossano, Plata & Kallmerten, 1988) affords oxazoline (3), incorporating the key C16—C17 stereochemistry. An unambiguous assignment of the C16—C17 stereochemistry of (3) was of critical importance to our ongoing synthetic studies, prompting an investigation of the structural details of this compound by single-crystal X-ray analysis.



(2) (3)

(1*a*) R = OH, nargenicin A_1
(1*b*) R = H, 18-deoxynargenicin A_1

Experimental. Colorless transparent crystals were grown from ether at 253 K. Crystal size 0·40 × 0·33 × 0·90 mm. Rigaku AFC-5S four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. All data obtained at 296 K. Unit-cell dimensions from 25 reflections ($31{\cdot}48 < 2\theta < 37{\cdot}67°$). Intensities measured in the ω–2θ mode, $(\sin\theta)/\lambda_{max} = 0{\cdot}65$ Å^{-1} (h 0 to 15, k −16 to 16, l −12 to 12), scan speed 8·0° min^{-1} in ω. Of the 6521 reflections which were collected, 6220 were unique ($R_{int} = 0{\cdot}014$) and 3196 observed [$I > 3\sigma(I)$]. Weights were assigned as $w = 1/\sigma^2(F_o)$, where σ is the standard deviation of observed amplitudes based on counting statistics and the function minimized in least squares was $\sum w(|F_o| - |F_c|)^2$. No systematic changes in $1\bar{2}1$, $2\bar{2}1$, $0\bar{3}1$ measured every 100 reflections. Range of ψ-scan transmission factor (averged) 0·9915 to 1·0000. ψ correction not applied. Structure solved by *MITHRIL* (Gilmore, 1984). All of the 34 non-H atoms were located from the initial E maps. 37 of the 41 H atoms were located on difference Fourier maps. Only two H atoms were located on each of the methyl groups C3*D*, C5*D*, C4*A* and C1*A*′. Also, the bond lengths and angles to one H atom of each of the methyl groups C4*A*″ and C4*B*″ were unsatisfactory. Two H-atom positions for each of these six methyl groups were generated based on the best H-atom position found on the difference maps. Two cycles of least-squares refinement of the 34 non-H atoms with H-atom parameters fixed were followed by two cycles of least-squares isotropic refinement of all 41 H atoms with the non-H atoms fixed. The final cycles consisted of anisotropic full-matrix least-squares refinement of all 34 non-H atoms with isotropic refinement of all 41 H atoms (470 variables plus scale factor, 3196 observed reflections) which converged at $R = 0{\cdot}041$, $wR = 0{\cdot}047$, $S = 1{\cdot}58$. The maximum shift in the final cycle $(\Delta/\sigma)_{max} = 0{\cdot}06$. Largest peaks in the final difference map were +0·20 and −0·14 e Å^{-3}. A MicroVAXII-based *TEXSAN–TEXRAY* (Molecular Structure Corporation, 1985) system was used for diffractometer control and all calculations. Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964). Atomic scattering factors and the values for f' and f'' were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Hydrogen scattering factors are those of Stewart, Davidson & Simpson (1965).

Discussion. Final positional parameters for the non-H atoms with the e.s.d.'s and B_{eq} values are given in Table 1.* Table 2 contains intramolecular bond distances and angles for all the non-H atoms. Fig. 1 is an *ORTEP* (Johnson, 1976) drawing of a single molecule showing the atomic numbering scheme and illustrates the molecular configuration.

Comparison of this structure with the previously reported crystal structure of a propynyl adduct (Pfluger, Kallmerten & Plata, 1989) reveals the similarities in the low-strain oxatricycloundecene nucleus (Kallmerten, 1984) and the fact that both methoxymethyl ether side chains are in the same $+sc$, $+sc$ ($\theta = 73{\cdot}5$, $\varphi = 85{\cdot}3°$) conformation for the C3 group and the same $-sc$, $-sc$ ($\theta = -68{\cdot}2$, $\varphi = -76{\cdot}1°$) conformation for the C5 group (Jeffrey, Pople, Binkley & Vishveshwara, 1978). The major difference between these structures is that while calculated

* Lists of structure factors, anisotropic thermal parameters, positional parameters for H atoms and conformational angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54163 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and equivalent isotropic thermal parameters for the non-H atoms and their e.s.d.'s at 296 K*

$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma + 2U_{13}\cos\beta + 2U_{23}\cos\alpha)$.

	x	y	z	B_{eq} (Å^2)
O11	0·6939 (1)	0·0747 (1)	0·2572 (2)	4·30 (5)
O3*A*	0·9586 (1)	0·2416 (1)	0·1724 (2)	4·84 (6)
O3*C*	1·0822 (2)	0·2673 (2)	0·0238 (2)	6·77 (9)
O5*A*	0·7026 (1)	−0·0919 (1)	0·1044 (2)	5·34 (6)
O5*C*	0·5493 (2)	−0·2439 (2)	−0·1244 (2)	7·18 (8)
C1	0·8108 (2)	0·1273 (2)	0·3486 (2)	3·95 (8)
C2	0·8907 (2)	0·0784 (2)	0·2510 (2)	3·79 (8)
C3	0·8977 (2)	0·1224 (2)	0·1244 (3)	4·11 (8)
C3*B*	1·0743 (2)	0·2623 (3)	0·1592 (4)	5·7 (1)
C3*D*	1·0588 (6)	0·3691 (4)	−0·0005 (7)	9·3 (2)
C4	0·7790 (2)	0·1117 (2)	0·0266 (3)	4·8 (1)
C4*A*	0·7928 (4)	0·1213 (4)	−0·1196 (4)	8·8 (2)
C5	0·6883 (2)	0·0017 (2)	0·0078 (3)	4·38 (9)
C5*B*	0·5971 (3)	−0·1695 (3)	−0·1901 (4)	7·2 (1)
C5*D*	0·6171 (4)	−0·3238 (3)	−0·1058 (5)	7·1 (1)
C6	0·7019 (2)	−0·0272 (2)	0·1473 (2)	4·09 (8)
C7	0·8239 (2)	−0·0473 (2)	0·1936 (3)	4·25 (8)
C8	0·8380 (3)	−0·1067 (2)	0·3084 (3)	5·7 (1)
C9	0·8339 (2)	−0·0300 (3)	0·4512 (3)	6·0 (1)
C10	0·8237 (2)	0·0744 (3)	0·4719 (3)	5·1 (1)
O4′	0·6844 (2)	0·5968 (1)	0·5265 (2)	4·87 (6)
C1′	0·8288 (2)	0·2573 (2)	0·3998 (2)	3·99 (8)
C1*A*′	0·9465 (3)	0·3180 (3)	0·5027 (4)	5·7 (1)
C2′	0·7489 (2)	0·3115 (2)	0·3585 (3)	4·06 (8)
C3′	0·7600 (2)	0·4403 (2)	0·3979 (3)	4·31 (8)
C3*A*′	0·7690 (3)	0·4815 (3)	0·2707 (4)	6·3 (1)
C4′	0·6572 (2)	0·4753 (2)	0·4588 (3)	3·94 (8)
O1″	0·7250 (1)	0·4215 (1)	0·6700 (2)	5·11 (6)
N3″	0·5353 (2)	0·3509 (2)	0·5552 (2)	4·62 (7)
C2″	0·6327 (2)	0·4134 (2)	0·5629 (2)	4·01 (8)
C4″	0·5538 (2)	0·2956 (2)	0·6696 (3)	5·5 (1)
C4*A*″	0·5246 (4)	0·1652 (3)	0·5984 (6)	7·8 (2)
C4*B*″	0·4763 (5)	0·3298 (4)	0·7709 (6)	8·5 (2)
C5″	0·6841 (3)	0·3434 (4)	0·7409 (4)	6·9 (1)

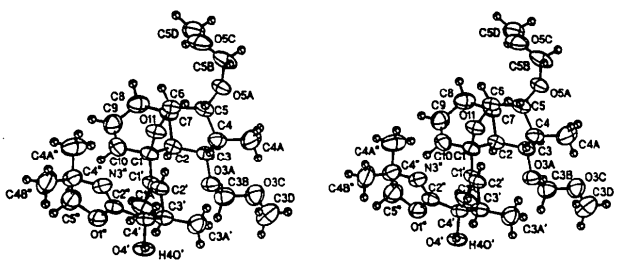


Fig. 1 Molecular structure and numbering scheme. Non-H-atom thermal ellipsoids are drawn at 50% probability.

Table 2. *Bond distances* (Å) *and bond angles* (°) *for the non-H atoms and their e.s.d.'s*

O11—C1	1·451 (2)	C6—C7	1·519 (3)
O3*A*—C3*B*	1·387 (3)	C9—C10	1·315 (4)
O5*A*—C5	1·426 (3)	C1′—C2′	1·315 (3)
O5*C*—C5*D*	1·398 (4)	C3′—C4′	1·532 (3)
C1—C1′	1·512 (3)	O1″—C5″	1·424 (4)
C3—C4	1·522 (3)	C4″—C4*A*″	1·523 (4)
C5—C6	1·515 (3)	O3*A*—C3	1·439 (3)
C8—C9	1·490 (4)	O3*C*—C3*D*	1·404 (5)
C1′—C1*A*′	1·512 (3)	O5*C*—C5*B*	1·382 (4)
C3′—C3*A*′	1·507 (4)	C1—C10	1·514 (3)
O1″—C2″	1·352 (3)	C2—C7	1·525 (3)
N3″—C4″	1·484 (3)	C4—C5	1·541 (3)
C4″—C5″	1·527 (4)	C7—C8	1·519 (4)
O11—C6	1·451 (2)	O4′—C4′	1·422 (2)
O3*C*—C3*B*	1·388 (3)	C2′—C3′	1·508 (3)
O5*A*—C5*B*	1·412 (3)	C4′—C2″	1·493 (3)
C1—C2	1·553 (3)	N3″—C2″	1·265 (3)
C2—C3	1·517 (3)	C4″—C4*B*″	1·509 (4)
C4—C4*A*	1·531 (4)		

C1—O11—C6	107·6 (1)	C3—O3*A*—C3*B*	115·9 (2)
C3*B*—O3*C*—C3*D*	113·4 (3)	C5—O5*A*—C5*B*	114·8 (2)
C5*B*—O5*C*—C5*D*	112·8 (3)	O11—C1—C2	102·7 (2)
O11—C1—C10	107·0 (2)	O11—C1—C1′	110·5 (2)
C2—C1—C10	107·9 (2)	C2—C1—C1′	115·6 (2)
C10—C1—C1′	112·4 (2)	C1—C2—C3	117·2 (2)
C1—C2—C7	98·2 (2)	C3—C2—C7	108·6 (2)
O3*A*—C3—C2	111·0 (2)	O3*A*—C3—C4	107·9 (2)
C2—C3—C4	114·3 (2)	O3*A*—C3*B*—O3*C*	112·7 (2)
C3—C4—C4*A*	110·9 (3)	C3—C4—C5	113·7 (2)
C4*A*—C4—C5	110·9 (2)	O5*A*—C5—C4	109·7 (2)
O5*A*—C5—C6	110·3 (2)	C4—C5—C6	110·4 (2)
O5*A*—C5*B*—O5*C*	113·8 (3)	O11—C6—C5	106·8 (2)
O11—C6—C7	106·0 (2)	C5—C6—C7	110·6 (2)
C2—C7—C6	98·1 (2)	C2—C7—C8	111·9 (2)
C6—C7—C8	115·1 (2)	C7—C8—C9	112·0 (2)
C8—C9—C10	122·1 (3)	C1—C10—C9	120·9 (3)
C1—C1′—C1*A*′	113·5 (2)	C1—C1′—C2′	122·8 (2)
C1*A*′—C1′—C2′	123·7 (2)	C1′—C2′—C3′	126·9 (2)
C2′—C3′—C3*A*′	111·1 (2)	C2′—C3′—C4′	110·8 (2)
C3*A*′—C3′—C4′	110·9 (2)	O4′—C4′—C3′	109·1 (2)
O4′—C4′—C2″	110·7 (2)	C3′—C4′—C2″	110·5 (2)
C2″—O1″—C5″	105·6 (2)	C2″—N3″—C4″	107·6 (2)
C4′—C2″—O1″	116·2 (2)	C4′—C2″—N3″	125·6 (2)
O1″—C2″—N3″	118·1 (2)	N3″—C4″—C4*A*″	108·3 (3)
N3″—C4″—C4*B*″	109·8 (3)	N3″—C4″—C5″	102·3 (2)
C4*A*″—C4″—C4*B*″	110·7 (3)	C4*A*″—C4″—C5″	112·4 (3)
C4*B*″—C4″—C5″	112·9 (3)	O1″—C5″—C4″	106·2 (3)

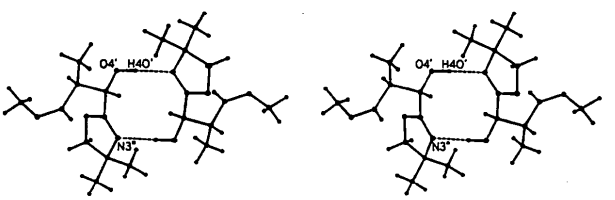


Fig. 2. Diagram which illustrates the strong intermolecular hydrogen bonding across the center of inversion. The octalin core has been deleted for clarity.

intermolecular packing contacts of the propynyl adduct revealed only weak C—H···O interactions the N atom in the dihydrooxazole allows the formation of a strong O4′—H4O′···N3″ hydrogen bond. Calculated intermolecular packing contacts showed this hydrogen bond to be the only intermolecular contact approximately equal to or less than the sum of the van der Waals radii: H4O′···N3″ = 1·91 (3) Å and ∠O4′—H4O′···N3″ = 174 (2)°. Fig. 2 is a partial packing diagram illustrating the hydrogen bond and its symmetry-related mate.

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Structure of 8-Amino-3,4-dihydro-2*H*,6*H*-pyrimido[2,1-*b*][1,3]thiazin-6-one

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Abstract. $C_7H_9N_3OS$, $M_r = 183{\cdot}23$, monoclinic, $P2_1/c$, $a = 7{\cdot}152\ (2)$, $b = 15{\cdot}407\ (3)$, $c = 7{\cdot}493\ (2)$ Å, $\beta = 101{\cdot}36\ (3)°$, $V = 809{\cdot}5\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}50$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 3{\cdot}0$ cm^{-1}, $F(000) = 384$, $T = 293$ K, $R = 0{\cdot}040$ for 1500 unique reflections with $I \geq 3\sigma(I)$. The structure consists of statistically disordered molecules with occupancy factors of 0·815 and 0·185. The disorder arises from two alternative half-chair conformations of the thiazine ring. The crystal packing is determined by intermolecular hydrogen-bonding interactions.

Introduction. The title compound was obtained from the reaction of 6-amino-2-thiouracil with 1,3-dibromopropane, and is a useful intermediate in the synthesis of thiazinopurine and triazolothiazinopyrimidine derivatives (Pecorari, Rinaldi & Costi, 1989). Their biological activity, on which little information is available, is one area of our current interest.

Experimental. Colourless prismatic crystals obtained at room temperature from ethanol–ether solution, dimensions 0·40 × 0·28 × 0·12 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Measurements carried out at room temperature; 25 ($11 \leq \theta \leq 18°$) reflections for cell-parameter determination; $\omega/2\theta$ scan, scan width $(1{\cdot}05 + 0{\cdot}35 \tan\theta)°$; two standard reflections ($\bar{6}\bar{1}0$ and $\bar{3}\bar{6}0$) measured at 1 h intervals showed no significant intensity decay; 2088 reflections measured in the range $2 \leq \theta \leq 28°$, $-9 \leq h \leq 9$, $0 \leq k \leq 20$, $0 \leq l \leq 9$, 1934 unique ($R_{int} = 0{\cdot}031$), 1500 with $I \geq 3\sigma(I)$ used for structure determination; intensities corrected for Lorentz–polarization effects and for absorption based on empirical ψ scan [0·909 ≤ T factor ≤ 0·999]; space group from systematic absences. Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986), and refined by full-matrix least squares with $\sum w\Delta F^2$ being minimized (*SHELX*76; Sheldrick, 1976). After isotropic refinement of all non-H atoms, an unrealistically high thermal parameter for the C(2) atom and a close residual in the ΔF map showed evidence of disorder, due to two positions in the crystal. The model was therefore adjusted to include two sites for this atom, for its H atoms and for those bonded to adjacent C atoms; preliminary least-squares refinement of their occupancy factors led to the values of 0·815 and 0·185, which were subsequently held fixed. All heavy atoms except C(2*b*) were then refined anisotropically, and H atoms with full or major occupancy (previously located in ΔF maps) isotropically; the H atoms at minor sites were added in calculated positions (C—H = 1·08 Å) with B values equal to those of the major sites; 146 parameters refined. $R = 0{\cdot}040$, $wR = 0{\cdot}047$, $S = 1{\cdot}44$,

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