illustrates the atom labeling and molecular conformation; Fig. 2 illustrates the unit-cell packing and hydrogen bonding.*

Related literature. The title compound has shown antitumor activity (Revankar *et al.*, 1990). The structure of 6-thioguanine (Bugg & Thewalt, 1970) and 6-thioguanosine (Thewalt & Bugg, 1972) have been reported. 6-Thioguanosine has an *anti* conformation. The *syn* conformation observed in (1) is characteristic of the 8-substituted guanosines such as 7-methyl-8-oxoguanosine (Larson, Cottam & Robins, 1989), 7-methyl-8-thioxoguanosine (Larson, Henry, Kini & Robins, 1990), 8-bromoguanosine (Tavale & Sobell, 1970), 8-chloroguanosine (Birnbaum, Lassota & Shugar, 1984) and 8-methylguanosine (Hamada, Honda, Fujii, Fujiwara & Tomita, 1985). Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52343 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ALTONA, C. & SUNDARALINGAM, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

BIRNBAUM, G. I., LASSOTA, P. & SHUGAR, D. (1984). *Biochemistry*, **23**, 5048–5053.

BUGG, C. E. & THEWALT, U. (1970). *J. Am. Chem. Soc.* **92**, 7441–7445.

CORDES, A. W. (1983). Personal communication.

FRENZ, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*, version 3.0. Enraf–Nonius, Delft, The Netherlands.

HAMADA, K., HONDA, I., FUJII, S., FUJIWARA, T. & TOMITA, K. (1985). *Acta Cryst.* C**41**, 1486–1488

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

LARSON, S. B. (1980). PhD Dissertation, Brigham Young University, Provo, Utah, USA.

LARSON, S. B., COTTAM, H. B. & ROBINS, R. K. (1989). *Acta Cryst.* C**45**, 1979–1983.

LARSON, S. B., HENRY, E. M., KINI, G. D. & ROBINS, R. K. (1990). *Acta Cryst.* C**46**, 506–508.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

REVANKAR, G. R., HANNA, N. B., IMAMURA, N., LEWIS, A. F., LARSON, S. B., FINCH, R. A., AVERY, T. L. & ROBINS, R. K. (1990). *J. Med. Chem.* In the press.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TAVALE, S. S. & SOBELL, H. M. (1970). *J. Mol. Biol.* **48**, 109–123.

THEWALT, U. & BUGG, C. E. (1972). *J. Am. Chem. Soc.* **94**, 8892–8898.

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Structure of Tetrahydrofuniculosin

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Abstract. *rel*-(2′*S*,5′*R*,6′*R*,1″*R*,3″*R*,5″*S*,2‴*R*,3‴*R*,4‴*S*,5‴*S*)-4-Hydroxy-1-methyl-3-[5′-methyl-6′-(1″,3″,5″-trimethylheptyl)tetrahydro-2′*H*-pyran-2′-yl]-5-(2‴,3‴,4‴,5‴-tetrahydroxy-1‴-cyclopentyl)-2-pyridone, $C_{27}H_{45}NO_7$, $M_r = 495{\cdot}66$, monoclinic, $P2_1$, $a = 19{\cdot}275\ (2)$, $b = 6{\cdot}968\ (1)$, $c = 10{\cdot}717\ (2)$ Å, $\beta = 104{\cdot}10\ (1)°$, $V = 1395{\cdot}9$ Å^3, $Z = 2$, $D_x = 1{\cdot}179$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6{\cdot}474$ cm^{-1}, $F(000) = 540$, $T = 298$ K, final $R = 0{\cdot}038$ for 1788 unique reflections [$F_o^2 > 1\sigma(F_o^2)$]. The cyclopentyl ring takes an envelope conformation and the four hydroxy groups are attached on the same side of the ring. Intra- and intermolecular hydrogen bonds are observed in this moiety.

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Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses*

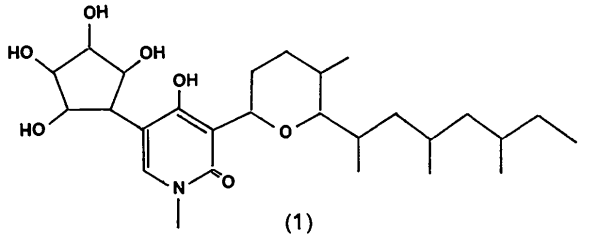
$B_{eq} = (4/3)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} (Å^2)
C(1)	0·8561 (2)	0·674	0·1043 (4)	4·5 (1)
O(2)	0·9194 (2)	0·7758 (6)	0·1658 (3)	6·25 (9)
C(3)	0·8560 (2)	0·6521 (8)	−0·0381 (4)	4·8 (1)
O(4)	0·8841 (2)	0·8197 (6)	−0·0833 (3)	6·10 (9)
C(5)	0·8963 (2)	0·4648 (8)	−0·0490 (3)	4·6 (1)
O(6)	0·9561 (2)	0·4927 (6)	−0·1008 (2)	5·44 (8)
C(7)	0·9180 (2)	0·3775 (8)	0·0889 (4)	4·3 (1)
O(8)	0·9885 (2)	0·4236 (6)	0·1574 (3)	5·24 (8)
C(9)	0·8599 (2)	0·4672 (8)	0·1493 (3)	4·0 (1)
C(10)	0·8702 (2)	0·4270 (7)	0·2914 (3)	3·13 (9)
C(11)	0·9203 (2)	0·5164 (7)	0·3821 (3)	3·37 (9)
N(12)	0·9308 (2)	0·4758 (5)	0·5104 (3)	3·05 (7)
C(13)	0·9874 (2)	0·5746 (7)	0·6049 (4)	3·9 (1)
C(14)	0·8910 (2)	0·3388 (7)	0·5553 (3)	3·39 (9)
O(15)	0·9028 (2)	0·3073 (5)	0·6738 (2)	4·18 (7)
C(16)	0·8379 (2)	0·2393 (6)	0·4618 (3)	2·97 (8)
C(17)	0·8291 (2)	0·2817 (7)	0·3339 (3)	3·34 (9)
O(18)	0·7806 (2)	0·1916 (5)	0·2394 (2)	4·98 (8)
C(19)	0·7991 (2)	0·0843 (7)	0·5150 (3)	3·38 (9)
C(20)	0·8454 (2)	−0·0903 (7)	0·5600 (4)	4·0 (1)
C(21)	0·8012 (2)	−0·2433 (7)	0·6065 (4)	4·4 (1)
C(22)	0·7353 (2)	−0·2948 (7)	0·5009 (4)	4·3 (1)
C(23)	0·6921 (3)	−0·4495 (9)	0·5490 (6)	6·7 (1)
C(24)	0·6925 (2)	−0·1145 (7)	0·4569 (4)	4·0 (1)
O(25)	0·7362 (1)	0·0298 (4)	0·4178 (2)	3·61 (6)
C(26)	0·6258 (2)	−0·1387 (8)	0·3469 (4)	4·5 (1)
C(27)	0·6437 (3)	−0·202 (1)	0·2227 (5)	6·7 (2)
C(28)	0·5806 (3)	0·0432 (9)	0·3278 (5)	5·9 (1)
C(29)	0·5086 (3)	0·0353 (9)	0·2294 (5)	6·1 (1)
C(30)	0·4706 (3)	0·229 (1)	0·2230 (7)	8·6 (2)
C(31)	0·4621 (2)	−0·1299 (9)	0·2550 (5)	5·5 (1)
C(32)	0·3897 (3)	−0·1606 (9)	0·1595 (5)	5·7 (1)
C(33)	0·3974 (3)	−0·186 (2)	0·0253 (6)	9·8 (2)
C(34)	0·3503 (3)	−0·329 (1)	0·2042 (5)	6·4 (1)
C(35)	0·2770 (3)	−0·362 (1)	0·1291 (7)	9·5 (2)

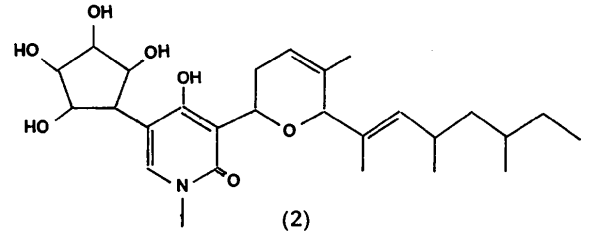
Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—O(2)	1·427 (5)	C(16)—C(19)	1·503 (6)
C(1)—C(3)	1·532 (6)	C(17)—O(18)	1·354 (5)
C(1)—C(9)	1·513 (5)	C(19)—C(20)	1·517 (6)
C(3)—O(4)	1·421 (7)	C(19)—O(25)	1·442 (4)
C(3)—C(5)	1·538 (8)	C(20)—C(21)	1·522 (7)
C(5)—O(6)	1·408 (5)	C(21)—C(22)	1·524 (5)
C(5)—C(7)	1·559 (6)	C(22)—C(23)	1·526 (8)
C(7)—O(8)	1·414 (5)	C(22)—C(24)	1·513 (6)
C(7)—C(9)	1·555 (6)	C(24)—O(25)	1·438 (6)
C(9)—C(10)	1·514 (5)	C(24)—C(26)	1·528 (5)
C(10)—C(11)	1·345 (5)	C(26)—C(27)	1·518 (8)
C(10)—C(17)	1·427 (6)	C(26)—C(28)	1·524 (7)
C(11)—N(12)	1·369 (4)	C(28)—C(29)	1·525 (6)
N(12)—C(13)	1·466 (5)	C(29)—C(30)	1·532 (9)
N(12)—C(14)	1·382 (6)	C(29)—C(31)	1·525 (8)
C(14)—O(15)	1·254 (4)	C(31)—C(32)	1·531 (6)
C(14)—C(16)	1·426 (5)	C(32)—C(33)	1·492 (9)
C(16)—C(17)	1·372 (5)	C(32)—C(34)	1·536 (9)
		C(34)—C(35)	1·464 (7)
O(2)—C(1)—C(3)	107·6 (4)	C(11)—N(12)—C(13)	119·9 (3)
O(2)—C(1)—C(9)	111·0 (3)	C(11)—N(12)—C(14)	122·3 (3)
C(3)—C(1)—C(9)	102·3 (3)	C(13)—N(12)—C(14)	117·8 (3)
C(1)—C(3)—O(4)	110·6 (4)	N(12)—C(14)—O(15)	119·7 (3)
C(1)—C(3)—C(5)	106·3 (3)	N(12)—C(14)—C(16)	117·1 (3)
O(4)—C(3)—C(5)	115·4 (4)	O(15)—C(14)—C(16)	123·2 (4)
C(3)—C(5)—O(6)	113·0 (4)	C(14)—C(16)—C(17)	119·3 (4)
C(3)—C(5)—C(7)	106·4 (4)	C(14)—C(16)—C(19)	115·0 (3)
O(6)—C(5)—C(7)	111·8 (3)	C(17)—C(16)—C(19)	125·6 (3)
C(5)—C(7)—O(8)	113·9 (4)	C(10)—C(17)—C(16)	121·9 (3)
C(5)—C(7)—C(9)	100·6 (3)	C(10)—C(17)—O(18)	115·2 (3)
O(8)—C(7)—C(9)	113·1 (3)	C(16)—C(17)—O(18)	122·8 (4)
C(1)—C(9)—C(7)	103·4 (4)	C(16)—C(19)—C(20)	112·8 (3)
C(1)—C(9)—C(10)	118·7 (4)	C(16)—C(19)—O(25)	108·9 (3)
C(7)—C(9)—C(10)	114·4 (3)	C(20)—C(19)—O(25)	110·1 (3)
C(9)—C(10)—C(11)	122·5 (4)	C(19)—C(20)—C(21)	109·4 (4)
C(9)—C(10)—C(17)	120·3 (3)	C(20)—C(21)—C(22)	110·7 (4)
C(11)—C(10)—C(17)	117·1 (3)	C(21)—C(22)—C(23)	109·9 (4)
C(10)—C(11)—N(12)	122·3 (4)	C(21)—C(22)—C(24)	109·1 (4)
C(23)—C(22)—C(24)	113·0 (4)	C(28)—C(29)—C(30)	110·1 (5)
C(22)—C(24)—O(25)	111·0 (3)	C(28)—C(29)—C(31)	112·3 (5)
C(22)—C(24)—C(26)	116·2 (4)	C(30)—C(29)—C(31)	112·2 (5)
O(25)—C(24)—C(26)	107·1 (3)	C(29)—C(31)—C(32)	117·7 (4)
C(19)—O(25)—C(24)	115·0 (3)	C(31)—C(32)—C(33)	111·9 (5)
C(24)—C(26)—C(27)	112·4 (4)	C(31)—C(32)—C(34)	109·7 (4)
C(24)—C(26)—C(28)	111·0 (4)	C(33)—C(32)—C(34)	112·5 (6)
C(27)—C(26)—C(28)	111·6 (4)	C(32)—C(34)—C(35)	115·7 (5)
C(26)—C(28)—C(29)	117·2 (5)		

Experimental. The title compound (1) is one of the catalytic hydrogenation products of antibiotic funiculosin (2) with H_2 and Pd—C in methanol at atmospheric pressure and ambient temperature. Colorless thin plates of tetrahydrofuniculosin were grown from acetone solution (m.p. 454–455 K, found: C 65·65; H 9·17; N 2·89%. $C_{27}H_{45}O_7N$ requires C 65·42; H 9·15; N 2·83%). Crystal size 0·25 × 0·25 × 0·05 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed 0·47–8·24° min^{-1} in θ, scan width $(0{\cdot}50 + 0{\cdot}14\tan\theta)°$. Range of indices, $-22 \le h \le 22$, $0 \le k \le 8$, $0 \le l \le 12$ ($2\theta < 130°$). Lattice constants determined based on 18 2θ values ($13 < \theta < 34°$). Variation of standard



(1)



(2)

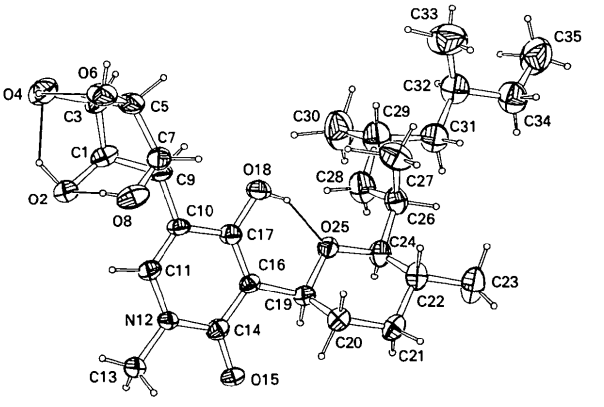


Fig. 1. A perspective view of the molecule with numbering scheme.

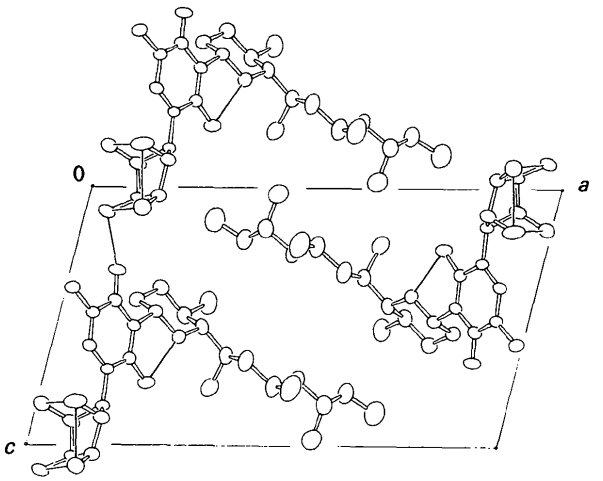


Fig. 2. The crystal structure projected along the *b* axis. Hydrogen bonds are shown as narrow lines.

$<0.7\%$; 2580 reflections measured; 1788 observed reflections with $F_o^2 > \sigma(F_o^2)$. Systematic absences $0k0$, k odd. No corrections for absorption. Structure solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971). Refined by full-matrix least squares. The locations of 45 H atoms were found on difference Fourier maps. Non-H atoms refined with anisotropic thermal parameters, H atoms with fixed isotropic thermal parameters ($B = 5.0$ Å^2). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1.0$ for $|F_o| < 81.84$, $w = (81.84/F_o)^2$ for $|F_o| \geq 81.84$. Final $R = 0.038$, $wR = 0.036$, $S = 2.40$ for 497 variables, secondary-extinction factor $g = 1.89\,(6) \times 10^{-6}$ [$|F_o| = |F_c|/(1 + gIc)$]; $\Delta/\sigma < 0.23$, largest peak in final ΔF map $+0.14$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The structure of the title compound is shown in Fig. 1, crystal packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond distances and angles are listed in Table 2.*

Related literature. Funiculosin is an antiviral and antifungal antibiotic (Ando, Suzuki, Saeki, Tamura & Arima, 1969), and its structure elucidation and antifungal activity have been reported (Ando, Matsuura, Nawata, Endo, Sasaki, Okutomi, Saeki & Tamura, 1978).

* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles, least-squares planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52364 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ANDO, K., MATSUURA, I., NAWATA, Y., ENDO, H., SASAKI, H., OKUTOMI, T., SAEKI, T. & TAMURA, G. (1978). *J. Antibiot.* **31**, 533–538.

ANDO, K., SUZUKI, S., SAEKI, T., TAMURA, G. & ARIMA, K. (1969). *J. Antibiot.* **22**, 189–194.

FRENZ, B. A. (1984). *Enraf–Nonius Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributors Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1971). *Acta Cryst.* A**27**, 368–376.

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5-Hydroxyflavone*

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Abstract. $C_{15}H_{10}O_3$, $M_r = 238.24$, monoclinic, $P2_1/c$, $a = 4.746$ (1), $b = 18.758$ (3), $c = 12.944$ (2) Å, $\beta = 95.38$ (2)°, $V = 1147.2$ (5) Å^3, $Z = 4$, $D_x = 1.384$ g cm^{-3}, Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å, $\mu = 8.0$ cm^{-1}, $F(000) = 496$, $T = 293$ K. Final $R = 0.054$ for 1003 observed reflections. The 5-hydroxy group forms a cyclic intramolecular hydrogen bond with the carbonyl group. The heterocyclic ring is coplanar with the benzene ring. The phenyl ring is slightly twisted, and makes a dihedral angle of 5.7 (7)° with the γ-benzopyrone portion of the molecule. The torsion angle for C(2′)—C(1′)—C(2)—O(1) is -5.8 (4)°.

* Flavone is 2-phenyl-4*H*-benzopyran-4-one.

Experimental. The title compound (Fig. 1) was purchased from the Indofine Chemical Company.

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