WS is indebted to the SERC for a research studentship.

References

GILCHRIST, T. L. & ROBERTS, T. G. (1983). *J. Chem. Soc. Perkin Trans.* 1, p. 1283.

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

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

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

Acta Cryst. (1989). C**45**, 978–979

Structure of Thiolactomycin

BY Y. NAWATA,* H. SASAKI, H. OISHI, K. SUZUKI, M. SAWADA AND K. ANDO

Research Laboratories, Chugai Pharmaceutical Co. Ltd, Takada, Toshima, Tokyo 171, *Japan*

AND Y. IITAKA†

Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Bunkyo, Tokyo 117, *Japan*

(*Received* 11 *October* 1988; *accepted* 20 *December* 1988)

Abstract. [4*R*,(2*E*,5*E*)]-3-Hydroxy-2,4,6-trimethyl-2,5,7-octatriene-4-thiolide,‡ $C_{11}H_{14}O_2S$, $M_r = 210{\cdot}30$, hexagonal, $P6_5$, $a = b = 9{\cdot}8514$ (6), $c = 19{\cdot}954$ (1) Å, $V = 1677{\cdot}1$ Å^3, $Z = 6$, $D_x = 1{\cdot}249$ g cm^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}5418$ Å, $\mu = 23{\cdot}07$ cm^{-1}, $F(000) = 672$, $T = 298$ K, $R = 0{\cdot}028$ for 1021 unique reflections [$F_o^2 > 2\sigma(F_o^2)$]. The absolute configuration was determined by the Bijvoet method. The thiolactone ring is planar with S—C bond distances of 1·774 (3) and 1·856 (3) Å, and C—S—C angle of 93·3 (1)°. The angle between least-squares planes for the thiolactone and butadienyl groups is 101·7°.

Experimental. Colorless plates of thiolactomycin were grown from acetone solution. Crystal size 0·43 × 0·20 × 0·09 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed 1·65–4·12° min^{-1} in θ, scan width (0·5 + 0·2tanθ)°. Range of indices, $0 \le (h, k) \le 12$, $0 \le l \le 25$ ($2\theta < 150°$). Lattice constants determined based on 25 2θ values ($15 < 2\theta < 40°$). Variation of standard < 1·5%; 1219 unique reflections measured; 1021 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. Empirical corrections for absorption (North, Phillips & Mathews, 1968); min., max. transmission coefficients 0·79, 0·99. Structure solved by the heavy-atom method. Refined by full-matrix least squares. The locations of all the H atoms were found on difference-Fourier maps. Non-H atoms refined with anisotropic thermal parameters, but H atoms with fixed isotropic thermal parameters ($B = 5{\cdot}0$ Å^2). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1{\cdot}0$ for $|F_o| < 120{\cdot}8$, $w = (120{\cdot}8/F_o)^2$ for $|F_o| \ge 120{\cdot}8$. Final $R = 0{\cdot}028$, $wR = 0{\cdot}025$, $S = 2{\cdot}8$ for 184 variables, secondary-extinction factor (g) 1·59 (4) × 10^{-6} [$|F_o| = |F_c|/(1+gI_c)$]; $\Delta/\sigma < 0{\cdot}18$, largest peak in final ΔF map +0·14 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *UNICS* (1967), Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The absolute configuration of thiolactomycin 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. Thiolactomycin is a thiotetronic acid antibiotic (Oishi, Noto, Sasaki, Suzuki, Hayashi, Okazaki, Ando & Sawada, 1982), which possesses antibacterial activity (Noto, Miyakawa, Oishi, Endo & Okazaki, 1982). Physicochemical properties and struc-

* To whom all correspondence should be addressed.

† Present address: Medical Department, University of Teikyo, Otsuka 359, Hachioji, Tokyo 193-03, Japan.

‡ Alternative nomenclature: [5*R*-(1′*E*,3*E*)]-4-hydroxy-2,5-dimethyl-5-(2methyl-1,3-pentadienyl)-2(5*H*)-thiophenone.

§ 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 51691 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/89/060978-02$03.00

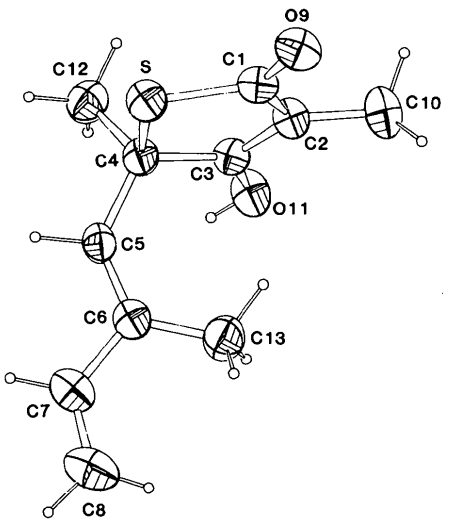

Fig. 1. A perspective view of the molecule with the numbering scheme.

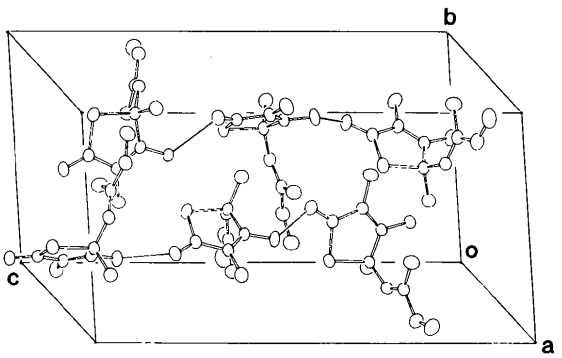

Fig. 2. Packing diagram for thiolactomycin. Hydrogen bonds are shown as narrow lines.

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

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

	x	y	z	B_{eq}(Å^2)
S	0·54723 (9)	0·19872 (8)	0·387	4·46 (2)
C(1)	0·6413 (3)	0·4063 (3)	0·3886 (2)	3·80 (6)
C(2)	0·6627 (3)	0·4706 (3)	0·3221 (1)	3·73 (6)
C(3)	0·6129 (3)	0·3609 (3)	0·2744 (1)	3·49 (6)
C(4)	0·5361 (3)	0·1902 (3)	0·2939 (1)	3·61 (6)
C(5)	0·6121 (3)	0·0992 (3)	0·2696 (2)	3·91 (7)
C(6)	0·7609 (3)	0·1460 (3)	0·2544 (2)	3·95 (7)
C(7)	0·7986 (4)	0·0235 (3)	0·2361 (2)	5·12 (8)
C(8)	0·9397 (4)	0·0445 (4)	0·2242 (2)	6·51 (9)
O(9)	0·6811 (2)	0·4798 (2)	0·4413 (1)	4·81 (5)
C(10)	0·7396 (4)	0·6431 (3)	0·3107 (2)	5·36 (9)
O(11)	0·6254 (2)	0·3981 (2)	0·20933 (9)	4·58 (5)
C(12)	0·3633 (4)	0·1078 (4)	0·2727 (2)	4·97 (8)
C(13)	0·8917 (4)	0·3113 (4)	0·2540 (2)	5·43 (9)

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

S–C(1)	1·774 (3)	C(3)–O(11)	1·338 (3)
S–C(4)	1·856 (3)	C(4)–C(5)	1·507 (5)
C(1)–C(2)	1·439 (4)	C(4)–C(12)	1·534 (4)
C(1)–O(9)	1·226 (3)	C(5)–C(6)	1·333 (4)
C(2)–C(3)	1·336 (4)	C(6)–C(7)	1·475 (5)
C(2)–C(10)	1·492 (4)	C(6)–C(13)	1·488 (3)
C(3)–C(4)	1·510 (4)	C(7)–C(8)	1·321 (5)

C(1)–S–C(4)	93·3 (1)	S–C(4)–C(5)	108·2 (2)
S–C(1)–C(2)	111·5 (2)	S–C(4)–C(12)	108·9 (2)
S–C(1)–O(9)	121·7 (2)	C(3)–C(4)–C(5)	117·3 (2)
C(2)–C(1)–O(9)	126·8 (3)	C(3)–C(4)–C(12)	108·9 (3)
C(1)–C(2)–C(3)	112·7 (2)	C(5)–C(4)–C(12)	110·3 (2)
C(1)–C(2)–C(10)	121·4 (3)	C(4)–C(5)–C(6)	130·9 (2)
C(3)–C(2)–C(10)	125·8 (3)	C(5)–C(6)–C(7)	117·2 (2)
C(2)–C(3)–C(4)	119·6 (2)	C(5)–C(6)–C(13)	125·5 (3)
C(2)–C(3)–O(11)	121·6 (2)	C(7)–C(6)–C(13)	117·4 (3)
C(4)–C(3)–O(11)	118·8 (2)	C(6)–C(7)–C(8)	126·3 (3)
S–C(4)–C(3)	102·8 (2)		

ture elucidation were reported previsouly (Sasaki, Oishi, Hayashi, Matsuura, Ando & Sawada, 1982: in which the absolute configuration of thiolactomycin was depicted incorrectly). The syntheses of racemic thiolactomycin (Wang & Salvino, 1984) and thiolactomycin analogues (Chambers, Thomas & Williams, 1987) were reported. Recently, (4*S*)-thiolactomycin has been successfully synthesized (Chambers & Thomas, 1989).

References

CHAMBERS, M. S. & THOMAS, E. J. (1989). *J. Chem. Soc. Chem. Commun.* pp. 23–24.

CHAMBERS, M. S., THOMAS, E. J. & WILLIAMS, D. J. (1987). *J. Chem. Soc. Chem. Commun.* pp. 1228–1230.

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 distributor Kluwer Academic Publishers, Dordrecht.)

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

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

NOTO, T., MIYAKAWA, S., OISHI, H., ENDO, H. & OKAZAKI, H. (1982). *J. Antibiot.* **35**, 401–410.

OISHI, H., NOTO, T., SASAKI, H., SUZUKI, K., HAYASHI, T., OKAZAKI, H., ANDO, K. & SAWADA, M. (1982). *J. Antibiot.* **35**, 391–395.

SASAKI, H., OISHI, H., HAYASHI, T., MATSUURA, I., ANDO, K. & SAWADA, M. (1982). *J. Antibiot.* **35**, 396–400.

UNICS (1967). *The Universal Crystallographic Computing System.* Japanese Crystallographic Association, Tokyo, Japan.

WANG, C.-L. & SALVINO, J. M. (1984). *Tetrahedron Lett.* **25**, 5243–5246.