and anion, including atomic labeling, are displayed in Fig. 1.

Related literature. A full discussion of the method of preparation is given by Booth & Collis (1988). Earlier work by Amer, Booth, Noori & Proença (1983) and Norell (1976) suggested that the product would be 2,4,6-tribenzyl-1,3,5-triazine and not the title molecule, whose structure was established as a result of this X-ray investigation. The isoquinoline nucleus resembles those in several reported structures, e.g. McClure & Schlemper (1978), Ljungström & Lindqvist (1978), and Ribár, Divjaković, Janić, Argay, Kálmán & Djurić (1974).

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Structure of Thiolactomycin δ

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Abstract. [4RS,5SR-(2E,6E)]-3-Hydroxy-2,4-dimethyl-5-(1-methyl-1-propenyl)-2-penten-5-thiolide,† $C_{11}H_{16}O_2S$, $M_r = 212\cdot31$, triclinic, $P\bar{1}$, $a = 8\cdot6196$ (6), $b = 10\cdot387$ (1), $c = 6\cdot9962$ (4) Å, $\alpha = 95\cdot97$ (1), $\beta = 99\cdot40$ (1), $\gamma = 107\cdot22$ (1)°, $V = 582\cdot5$ Å^3, $Z = 2$, $D_x = 1\cdot210$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1\cdot5418$ Å, $\mu = 22\cdot14$ cm^{-1}, $F(000) = 228$, $T = 298$ K, final $R = 0\cdot059$ for 1988 unique reflections $[F_o^2 > 2\sigma(F_o^2)]$. The title compound is a racemate and one of the homologues of thiolactomycin. The thiolactone ring adopts a sofa conformation, with S—C bond distances of $1\cdot750$ (4) and $1\cdot824$ (3) Å, and a C—S—C angle of $101\cdot8$ (2)°. The angle between the least-squares planes of the five ring atoms and the propenyl moiety is $144\cdot6°$. Intermolecular hydrogen bonding and van der Waals contacts are observed: O(6)···O(8) $(x, y, 1+z) = 2\cdot581$ (3) and C(7)···C(7) $(1-x, 1-y, 1-z) = 3\cdot633$ (6) Å.

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† Alternative name: [4RS,5SR-(2E,6E)]-3-hydroxy-2,4-dimethyl-5-(1-methyl-1-propenyl)-6-thiacyclohex-2-en-1-one.

Experimental. The title compound was isolated from the culture broth (Oishi, Noto, Sasaki, Suzuki, Hayashi. Okazaki, Ando & Sawada, 1982) by silica-gel column chromatography (eluent: benzene), prior to the elution of thiolactomycin (eluent: benzene/acetone = 95:5, v/v). Colorless prisms were grown from acetone solution (m.p. 403 K; found: C 62·4, H 7·8, S 15·0%. $C_{11}H_{16}O_2S$ requires C 62·2, H 7·6, S 15·1%). Crystal size 0·38 × 0·25 × 0·18 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu Kα radiation, graphite monochromator, θ–2θ scan with scan speed 2·75–5·49° min^{-1} in θ, scan width $(0\cdot8 + 0\cdot2\tan\theta)°$. Range of indices, $-10 \le h \le 10$, $-13 \le k \le 13$, $0 \le l \le 8$ $(2\theta < 150°)$. Lattice constants determined based on 25 2θ values $(21 < \theta < 35°)$. Variation of standard < 1·3%; 2382 unique reflections measured; 1988 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. No correction for absorption. Structure solved by direct methods with $MULTAN11/82$ (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). 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, and H atoms with fixed isotropic thermal parameters (B = 5·0 Å^2). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1·0$ for $|F_o| < 65·02$, $w = (65·02/F_o)^2$ for $|F_o| \geq 65·02$. Final $R = 0·059$, $wR = 0·053$, $S = 11·0$ for 192 variables, secondary-extinction factor (g) $6·2\,(2) \times 10^{-6}$ $[|F_o| = |F_c|/(1 + gIc)]$; $\Delta/\sigma < 0·36$, largest peak in final ΔF map $+0·29\,\mathrm{e\,Å^{-3}}$; atomic scattering factors from International Tables for X-ray Crystallography (1974); programs: Enraf–Nonius SDP (Frenz, 1984), ORTEPII (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 isotropic temperature factors are given in Table 1, bond distances and angles are listed in Table 2.*

Related literature. Thiolactomycin is a broad-spectrum antibiotic (Oishi, Noto, Sasaki, Suzuki, Hayashi, Okazaki, Ando & Sawada, 1982; Noto, Miyakawa, Oishi, Endo & Okazaki, 1982). For its molecular and crystal structures see Nawata, Sasaki, Oishi, Suzuki, Sawada, Ando & Iitaka (1989).

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* 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 51748 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
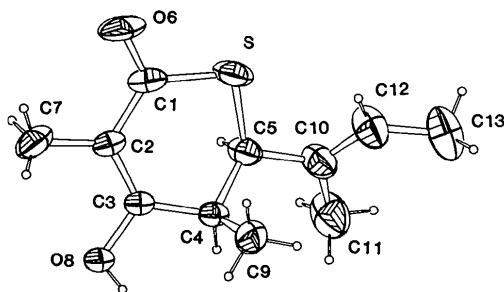

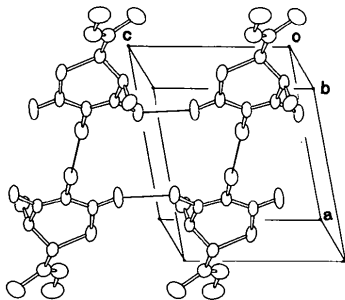Fig. 1. A perspective view of the molecule with the numbering scheme.

Fig. 2. Packing diagram for thiolactomycin δ. Hydrogen bonds and van der Waals contacts 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	$B_{eq}(\text{Å}^2)$
S	−0·0269 (1)	0·7546 (1)	0·5270 (1)	5·67 (2)
C(1)	0·1394 (4)	0·6897 (3)	0·5611 (4)	5·06 (8)
C(2)	0·2074 (4)	0·6456 (3)	0·4021 (4)	4·35 (6)
C(3)	0·1556 (3)	0·6640 (3)	0·2169 (3)	3·85 (6)
C(4)	0·0300 (3)	0·7309 (3)	0·1494 (4)	3·98 (6)
C(5)	−0·1093 (3)	0·7017 (3)	0·2638 (4)	4·47 (7)
O(6)	0·2006 (3)	0·6892 (3)	0·7338 (3)	7·73 (8)
C(7)	0·3475 (4)	0·5876 (4)	0·4505 (5)	6·12 (8)
O(8)	0·2332 (2)	0·6244 (2)	0·0810 (3)	5·20 (4)
C(9)	0·1138 (4)	0·8816 (3)	0·1505 (5)	5·55 (8)
C(10)	−0·2428 (4)	0·7633 (3)	0·1946 (5)	5·22 (8)
C(11)	−0·3412 (5)	0·6994 (5)	−0·0092 (6)	7·9 (1)
C(12)	−0·2721 (4)	0·8618 (4)	0·2996 (6)	6·43 (9)
C(13)	−0·4011 (5)	0·9284 (4)	0·2439 (8)	8·3 (1)

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

S—C(1)	1·750 (4)	C(3)—O(8)	1·346 (4)
S—C(5)	1·824 (3)	C(4)—C(5)	1·524 (4)
C(1)—C(2)	1·434 (4)	C(4)—C(9)	1·517 (4)
C(1)—O(6)	1·238 (3)	C(5)—C(10)	1·513 (5)
C(2)—C(3)	1·352 (4)	C(10)—C(11)	1·506 (5)
C(2)—C(7)	1·509 (5)	C(10)—C(12)	1·307 (5)
C(3)—C(4)	1·492 (4)	C(12)—C(13)	1·495 (6)

C(1)—S—C(5)	101·8 (2)	C(3)—C(4)—C(5)	111·6 (2)
S—C(1)—C(2)	123·0 (2)	C(3)—C(4)—C(9)	110·4 (2)
S—C(1)—O(6)	115·7 (3)	C(5)—C(4)—C(9)	114·0 (3)
C(2)—C(1)—O(6)	121·3 (4)	S—C(5)—C(4)	111·0 (2)
C(1)—C(2)—C(3)	121·1 (3)	S—C(5)—C(10)	111·2 (2)
C(1)—C(2)—C(7)	117·8 (3)	C(4)—C(5)—C(10)	113·3 (3)
C(3)—C(2)—C(7)	120·9 (3)	C(5)—C(10)—C(11)	113·4 (3)
C(2)—C(3)—C(4)	127·2 (3)	C(5)—C(10)—C(12)	124·1 (3)
C(2)—C(3)—O(8)	116·0 (3)	C(11)—C(10)—C(12)	122·6 (4)
C(4)—C(3)—O(8)	116·6 (2)	C(10)—C(12)—C(13)	127·8 (3)

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