

KRR is indebted to the SERC for a research studentship.

Table 1. Fractional atomic coordinates, mean isotropic thermal parameters, and their e.s.d.'s

$$\bar{U} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

| | x | y | z | $\bar{U}(\text{\AA}^2)$ |
|-------|-------------|-------------|-------------|-------------------------|
| N(1) | 0.2367 (2) | 0.0106 (1) | 0.0161 (2) | 0.0671 (10) |
| N(4) | 0.1729 (1) | -0.0739 (1) | 0.3457 (2) | 0.0632 (9) |
| C(2) | 0.2853 (2) | -0.0563 (1) | 0.0838 (2) | 0.0645 (11) |
| C(3) | 0.2796 (2) | -0.0768 (1) | 0.2659 (2) | 0.0571 (10) |
| C(5) | 0.0518 (2) | -0.0469 (1) | 0.2537 (3) | 0.0739 (12) |
| C(6) | -0.0158 (2) | 0.1067 (2) | 0.2828 (3) | 0.0770 (13) |
| C(7) | -0.0001 (2) | 0.1905 (2) | 0.2438 (3) | 0.0824 (14) |
| C(8) | 0.0963 (2) | 0.2144 (1) | 0.1350 (3) | 0.0795 (13) |
| C(9) | 0.1752 (2) | 0.1545 (1) | 0.0632 (3) | 0.0710 (12) |
| C(10) | 0.1612 (2) | 0.0694 (1) | 0.1028 (2) | 0.0578 (9) |
| C(11) | 0.0639 (2) | 0.0449 (1) | 0.2136 (2) | 0.0608 (10) |
| C(12) | 0.3503 (3) | -0.1193 (2) | -0.0261 (3) | 0.0985 (18) |
| C(13) | 0.4061 (2) | -0.1000 (1) | 0.3549 (2) | 0.0560 (10) |
| C(14) | 0.5278 (2) | -0.0698 (1) | 0.3026 (3) | 0.0751 (13) |
| C(15) | 0.6439 (2) | -0.0889 (2) | 0.3899 (4) | 0.0885 (16) |
| C(16) | 0.6411 (2) | -0.1387 (2) | 0.5279 (3) | 0.0844 (15) |
| C(17) | 0.5210 (2) | -0.1705 (2) | 0.5814 (3) | 0.0799 (13) |
| C(18) | 0.4035 (2) | -0.1508 (1) | 0.4964 (2) | 0.0664 (11) |

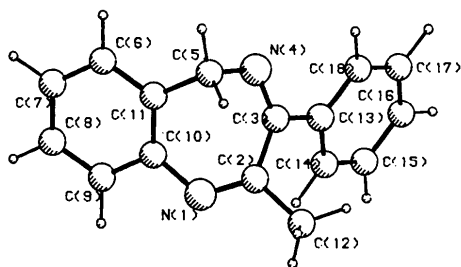


Fig. 1. A view of the molecule drawn with PLUTO (Motherwell & Clegg, 1978).

Table 2. Bond distances (\AA) and angles ($^\circ$) involving non-H atoms, with e.s.d.'s in parentheses

| | | | | | | | |
|------|-------|-----------|-----------|-------|-----------|-------|-----------|
| N(1) | C(2) | 1.282 (2) | C(7) | C(8) | 1.381 (3) | | |
| N(1) | C(10) | 1.402 (2) | C(8) | C(9) | 1.377 (3) | | |
| N(4) | C(3) | 1.269 (2) | C(9) | C(10) | 1.394 (3) | | |
| N(4) | C(5) | 1.468 (3) | C(10) | C(11) | 1.400 (3) | | |
| C(2) | C(3) | 1.507 (3) | C(13) | C(14) | 1.391 (3) | | |
| C(2) | C(12) | 1.498 (3) | C(13) | C(18) | 1.397 (3) | | |
| C(3) | C(13) | 1.485 (2) | C(14) | C(15) | 1.377 (3) | | |
| C(5) | C(11) | 1.496 (3) | C(15) | C(16) | 1.365 (4) | | |
| C(6) | C(7) | 1.374 (3) | C(16) | C(17) | 1.389 (3) | | |
| C(6) | C(11) | 1.393 (3) | C(17) | C(18) | 1.382 (3) | | |
| C(2) | N(1) | C(10) | 122.8 (2) | N(1) | C(10) | C(11) | 122.3 (2) |
| C(3) | N(4) | C(5) | 117.0 (2) | C(9) | C(10) | C(11) | 119.4 (2) |
| N(1) | C(2) | C(3) | 124.6 (2) | C(5) | C(11) | C(6) | 123.1 (2) |
| N(1) | C(2) | C(12) | 117.8 (2) | C(5) | C(11) | C(10) | 118.0 (2) |
| C(3) | C(2) | C(12) | 117.6 (2) | C(6) | C(11) | C(10) | 118.9 (2) |
| N(4) | C(3) | C(2) | 123.0 (2) | C(3) | C(13) | C(14) | 121.0 (2) |
| N(4) | C(3) | C(13) | 119.2 (2) | C(3) | C(13) | C(18) | 120.0 (2) |
| C(2) | C(3) | C(13) | 117.8 (1) | C(14) | C(13) | C(18) | 119.0 (2) |
| N(4) | C(5) | C(11) | 108.7 (2) | C(13) | C(14) | C(15) | 120.5 (2) |
| C(7) | C(6) | C(11) | 121.1 (2) | C(14) | C(15) | C(16) | 120.3 (2) |
| C(6) | C(7) | C(8) | 119.9 (2) | C(15) | C(16) | C(17) | 120.2 (2) |
| C(7) | C(8) | C(9) | 120.2 (2) | C(16) | C(17) | C(18) | 120.0 (2) |
| C(8) | C(9) | C(10) | 120.5 (2) | C(13) | C(18) | C(17) | 119.9 (2) |
| N(1) | C(10) | C(9) | 117.9 (2) | | | | |

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Structure of Ethyl 6-Methyl-6-phenyl-5,6-dihydro-4H-1,2-oxazine-3-carboxylate

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Abstract. $C_{14}H_{17}NO_3$, $M_r = 247.29$, monoclinic, $P2_1$, $a = 10.59$ (2), $b = 5.84$ (1), $c = 11.17$ (2) \AA , $\beta = 109.8$ (2) $^\circ$, $V = 650.7$ \AA^3 , $Z = 2$, $D_m = 1.26$, $D_x = 1.26$ g cm^{-3} , $\lambda(\text{Cu K}\alpha) = 1.5418$ \AA , $\mu = 6.40$ cm^{-1} , $F(000) = 264$, $T = ca$ 293 K, $R = 0.051$ for 930 unique observed reflections. The phenyl group occupies an

axial position and the methyl group an equatorial position in an essentially twist-chair conformation of the 1,2-oxazine ring in which the O(1)–N(2)–C(3)–C(4) torsion angle is 2.8 (6) $^\circ$ and the deviations of C(5) and C(6) from the least-squares plane through the dihydro-oxazine ring are -0.33 (1) and 0.34 (1) \AA ,

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Table 1. Fractional atomic coordinates, mean isotropic thermal parameters, and their e.s.d.'s

$$\bar{U} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

| | x | y | z | $\bar{U}(\text{\AA}^2)$ |
|-------|------------|--------------|------------|-------------------------|
| O(1) | 0.3772 (3) | 0.1758 | 0.6189 (3) | 0.0376 (15) |
| O(15) | 0.7473 (5) | -0.3046 (12) | 0.7189 (5) | 0.0752 (26) |
| O(16) | 0.7723 (4) | 0.0697 (10) | 0.7522 (4) | 0.0538 (20) |
| N(2) | 0.5158 (4) | 0.1292 (11) | 0.6611 (4) | 0.0348 (18) |
| C(3) | 0.5510 (5) | -0.0806 (13) | 0.6734 (5) | 0.0338 (21) |
| C(4) | 0.4653 (6) | -0.2906 (13) | 0.6442 (5) | 0.0434 (24) |
| C(5) | 0.3205 (6) | -0.2181 (13) | 0.5685 (5) | 0.0423 (23) |
| C(6) | 0.2864 (5) | -0.0040 (12) | 0.6296 (5) | 0.0347 (22) |
| C(7) | 0.2994 (5) | -0.0347 (12) | 0.7691 (5) | 0.0291 (19) |
| C(8) | 0.2438 (5) | -0.2251 (14) | 0.8064 (5) | 0.0463 (24) |
| C(9) | 0.2509 (6) | -0.2472 (14) | 0.9335 (6) | 0.0542 (27) |
| C(10) | 0.3116 (6) | -0.0820 (14) | 1.0222 (5) | 0.0462 (25) |
| C(11) | 0.3670 (5) | 0.1023 (13) | 0.9852 (5) | 0.0436 (24) |
| C(12) | 0.3611 (5) | 0.1287 (13) | 0.8587 (5) | 0.0383 (21) |
| C(13) | 0.1493 (5) | 0.0900 (13) | 0.5519 (6) | 0.0444 (25) |
| C(14) | 0.6988 (6) | -0.1156 (14) | 0.7166 (5) | 0.0421 (23) |
| C(17) | 0.9162 (6) | 0.0416 (16) | 0.7904 (8) | 0.0702 (35) |
| C(18) | 0.9795 (7) | 0.2612 (18) | 0.8289 (9) | 0.0865 (43) |

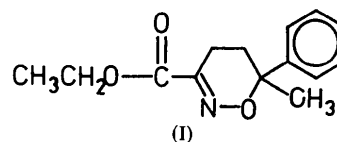
Table 2. Bond distances (Å) and bond angles (°) involving non-H atoms, with e.s.d.'s in parentheses

| | | | | | | | |
|-------|-------|-----------|-----------|-------|------------|-------|-----------|
| C(3) | C(4) | 1.495 (8) | C(8) | C(9) | 1.402 (8) | | |
| C(3) | C(14) | 1.488 (8) | C(9) | C(10) | 1.377 (9) | | |
| C(3) | N(2) | 1.276 (8) | C(10) | C(11) | 1.357 (9) | | |
| C(4) | C(5) | 1.539 (8) | C(11) | C(12) | 1.402 (8) | | |
| C(5) | C(6) | 1.526 (8) | C(14) | O(15) | 1.215 (8) | | |
| C(6) | C(7) | 1.528 (7) | C(14) | O(16) | 1.315 (8) | | |
| C(6) | C(13) | 1.520 (7) | C(17) | C(18) | 1.444 (11) | | |
| C(6) | O(1) | 1.456 (6) | C(17) | O(16) | 1.446 (7) | | |
| C(7) | C(8) | 1.388 (8) | O(1) | N(2) | 1.409 (5) | | |
| C(7) | C(12) | 1.378 (7) | | | | | |
| C(4) | C(3) | C(14) | 116.9 (5) | C(8) | C(7) | C(12) | 118.9 (5) |
| C(4) | C(3) | N(2) | 129.2 (5) | C(7) | C(8) | C(9) | 119.7 (6) |
| C(14) | C(3) | N(2) | 113.8 (5) | C(8) | C(9) | C(10) | 121.1 (6) |
| C(3) | C(4) | C(5) | 108.2 (5) | C(9) | C(10) | C(11) | 118.8 (5) |
| C(4) | C(5) | C(6) | 108.8 (4) | C(10) | C(11) | C(12) | 121.2 (6) |
| C(5) | C(6) | C(7) | 114.0 (5) | C(7) | C(12) | C(11) | 120.3 (5) |
| C(5) | C(6) | C(13) | 111.5 (5) | C(3) | C(14) | O(15) | 121.6 (6) |
| C(5) | C(6) | O(1) | 107.0 (4) | C(3) | C(14) | O(16) | 115.8 (5) |
| C(7) | C(6) | C(13) | 111.3 (4) | O(15) | C(14) | O(16) | 122.6 (5) |
| C(7) | C(6) | O(1) | 109.5 (4) | C(18) | C(17) | O(16) | 108.8 (6) |
| C(13) | C(6) | O(1) | 102.8 (4) | C(6) | O(1) | N(2) | 118.4 (4) |
| C(6) | C(7) | C(8) | 120.0 (5) | C(14) | O(16) | C(17) | 116.9 (5) |
| C(6) | C(7) | C(12) | 121.1 (5) | C(3) | N(2) | O(1) | 117.1 (4) |

respectively (*cf.* cyclohexene). The N—O bond distance of 1.409 (5) Å is in agreement with standard N—O bond lengths. No structures have previously been reported for the dihydrooxazine ring system.

Experimental. Compound (I) prepared by cycloaddition of ethyl 2-nitrosopropenoate with α -methylstyrene (Gilchrist & Roberts, 1983). Crystals obtained by slow evaporation of a solution in ethyl ethanoate. Density measured by flotation in mixture of hexane and tetrachloromethane. A *ca* 0.3 × 0.2 × 0.2 mm cuboidal crystal mounted on glass fibre for X-ray analysis. Intensities of 2267 reflections were measured by the SERC Microdensitometer Service, Daresbury Laboratory, from Weissenberg photographs of layers

h0l to *h4l* and scaled initially on exposure times; equivalents merged (R_{int} 0.036) giving 930 unique observed data used for structure solution and refinement; absorption correction not applied. Unit-cell dimensions from precession photographs. Solved by direct methods with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least squares with unit weights on *F* values with *SHELX76* (Sheldrick, 1976). Non-H atoms refined anisotropically, H atoms calculated geometrically and allowed to 'ride' on associated heavy atoms with two common isotropic temperature factors for methyl and other hydrogens respectively; interlayer scale factors also refined for a total of 175 variables. $R = 0.051$. Extinction correction of form $F^* = F(1 - 0.001XF^2/\sin\theta)$ where $X = 0.136$ applied. Final $(\Delta/\sigma)_{\text{max}} < 0.38$ (methyl rotor), < 0.08 elsewhere, $\Delta\rho_{\text{max}} = 0.18$ and $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$ on final difference map. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom parameters are listed in Table 1 and bond lengths and angles in Table 2. The molecule and numbering scheme are shown in Fig. 1.



Related literature. A search of the Cambridge Crystallographic Database (version of June 1988, 67051 entries) for compounds containing the basic ring connectivity of the 1,2-oxazine system gave five 'hits' but none of these have the double-bond pattern of the system reported here (*i.e.* they are not dihydrooxazines). The structure described here is therefore unique in this respect.

† Lists of structure factors, H-atom coordinates and isotropic temperature factors, and anisotropic temperature factors for non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51698 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

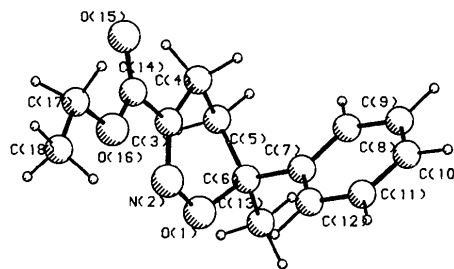


Fig. 1. A view of the molecule drawn with *PLUTO* (Motherwell & Clegg, 1978).

WS is indebted to the SERC for a research studentship.

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Structure of Thiolactomycin

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Abstract. [4*R*,(2*E*,5*E*)]-3-Hydroxy-2,4,6-trimethyl-2,5,7-octatriene-4-thiolide,‡ C₁₁H₁₄O₂S, *M_r* = 210.30, hexagonal, *P*6₅, *a* = *b* = 9.8514 (6), *c* = 19.954 (1) Å, *V* = 1677.1 Å³, *Z* = 6, *D_x* = 1.249 g cm⁻³, λ(Cu *K*α) = 1.5418 Å, μ = 23.07 cm⁻¹, *F*(000) = 672, *T* = 298 K, *R* = 0.028 for 1021 unique reflections [*F_o*² > 2σ(*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*α 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 ≤ (*h*, *k*) ≤ 12, 0 ≤ *l* ≤ 25 (2θ < 150°). Lattice constants determined based on 25 2θ values (15 < 2θ < 40°). Variation of standard < 1.5%; 1219 unique reflections measured; 1021 observed reflections with *F_o*² > 2σ(*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* = 5.0 Å²). ∑*w*(|*F_o*| – |*F_c*|)² minimized; *w* = 1.0 for |*F_o*| < 120.8, *w* = (120.8/*F_o*)² for |*F_o*| ≥ 120.8. Final *R* = 0.028, *wR* = 0.025, *S* = 2.8 for 184 variables, secondary-extinction factor (*g*) 1.59 (4) × 10⁻⁶ [|*F_o*| = |*F_c*|/(1+*gI_c*)]; Δ/σ < 0.18, largest peak in final Δ*F* 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), *ORTEPII* (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-

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‡ 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.