We thank the Department of Science and Technology and the University Grants Commission, Government of India, for financial assistance.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SE1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Construction of the Tetracyclic Skeleton of Leucothol A. 1,2,4a,5,6,7,8,9,10,10a,11,11a-Dodecahydro-11-hydroxy-11-methyl-7-phenylthiomethyl-5a*H*-5a,8-methanocyclohepta[*b*]naphthalen-10-one

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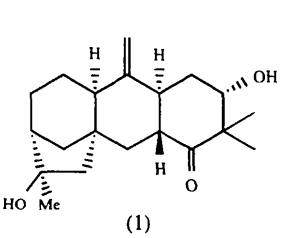
Abstract

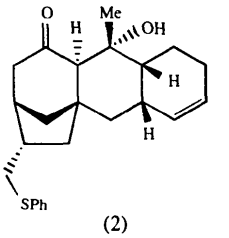
The title compound, $C_{24}H_{30}O_2S$, is a tetracyclic intermediate for the synthesis of leucothol A and the first example of an ethanoanthracene skeleton to be synthetized. The single-crystal X-ray structure of this compound is presented.

Comment

Leucothol A, (1), is a novel diterpene isolated from the leaves of *Leucothol grayana* (Hikino, Koriyama & Takemoto, 1973). Although structurally different members of this family, such as the grayanotoxins, have been synthetized previously, the ethanoanthracene skeleton has not. The significant central nervous system activity of closely related products and the novel structural elements contained within the leucothol A skeleton combine to make it a worthwhile synthetic objective. We have recently completed the first synthetic pathway leading to an ethanoanthracene product. The title compound, (2), is formed in a seven-step synthetic sequence that is initiated by a novel bridgehead connection. The details of the synthesis are published elsewhere (Kraus & Su, 1994).



(1)



(2)

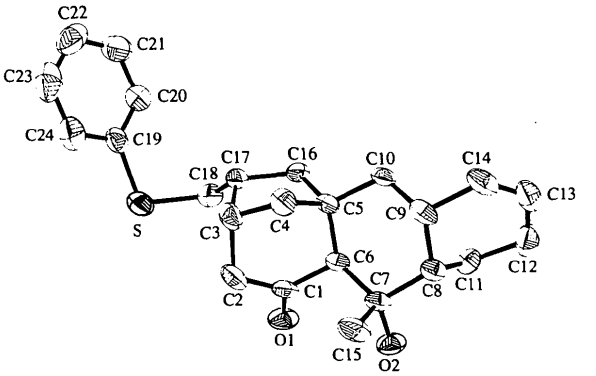


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

Crystal data

$C_{24}H_{30}O_2S$
M_r = 382.54
Monoclinic
$P2_1/c$
a = 5.976 (1) Å
b = 13.146 (1) Å
c = 25.883 (3) Å
β = 93.84 (1)°
V = 2028.8 (4) Å^3
Z = 4
D_x = 1.252 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 20.9–26.9°
μ = 1.529 mm^{-1}
T = 213 (1) K
Monoclinic
0.50 × 0.45 × 0.15 mm
Colorless

Data collection

Siemens *P4RA* diffractometer
Profile fitted $2\theta/\omega$ scans (Clegg, 1981)
Absorption correction: ψ scan (*SHELXTL-Plus*; Sheldrick, 1990)
T_{min} = 0.694, T_{max} = 0.945
5926 measured reflections
2736 independent reflections
2389 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0445
θ_{max} = 56.86°
$h = -6 \rightarrow 0$
$k = -14 \rightarrow 14$
$l = -28 \rightarrow 28$
3 standard reflections monitored every 97 reflections
intensity decay: 4.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.096
S = 1.080
2736 reflections
357 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.9025P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.403 e Å^{-3}
$\Delta\rho_{min}$ = −0.307 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0018 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S	0.16288 (13)	0.31442 (4)	0.29855 (2)	0.0589 (3)
C1	0.0904 (4)	0.35986 (14)	0.46657 (8)	0.0368 (5)
O1	−0.0751 (3)	0.41436 (11)	0.46247 (6)	0.0541 (5)
C2	0.2827 (4)	0.3757 (2)	0.43266 (10)	0.0471 (6)
C3	0.3921 (4)	0.2759 (2)	0.41721 (9)	0.0426 (5)
C4	0.4675 (4)	0.2183 (2)	0.46662 (9)	0.0415 (5)
C5	0.2419 (3)	0.18320 (13)	0.48588 (8)	0.0304 (5)
C6	0.1060 (3)	0.27392 (14)	0.50571 (7)	0.0285 (4)
C7	0.1736 (3)	0.31403 (14)	0.56165 (8)	0.0334 (5)
C15	0.3874 (4)	0.3782 (2)	0.56617 (9)	0.0462 (6)
O2	−0.0071 (3)	0.37624 (12)	0.57724 (6)	0.0461 (4)
C8	0.1944 (3)	0.22451 (15)	0.59982 (8)	0.0329 (5)
C9	0.3458 (3)	0.14004 (15)	0.58103 (9)	0.0377 (5)
C10	0.2612 (4)	0.10084 (15)	0.52745 (8)	0.0360 (5)
C11	−0.0282 (4)	0.1786 (2)	0.61416 (8)	0.0390 (5)
C12	0.0113 (5)	0.1046 (2)	0.65935 (9)	0.0531 (6)
C14	0.3615 (4)	0.0547 (2)	0.62013 (10)	0.0487 (6)
C13	0.2133 (5)	0.0399 (2)	0.65477 (9)	0.0525 (7)
C16	0.1232 (4)	0.1431 (2)	0.43505 (8)	0.0363 (5)
C17	0.2259 (4)	0.20086 (15)	0.38970 (8)	0.0382 (5)
C18	0.0498 (4)	0.2456 (2)	0.35134 (9)	0.0468 (6)
C19	0.3093 (4)	0.2189 (2)	0.26625 (8)	0.0439 (6)
C20	0.2230 (5)	0.1225 (2)	0.25692 (10)	0.0540 (6)
C21	0.3406 (6)	0.0529 (2)	0.22955 (11)	0.0684 (8)
C22	0.5446 (6)	0.0798 (3)	0.21097 (10)	0.0750 (10)
C23	0.6272 (6)	0.1755 (3)	0.21977 (12)	0.0744 (9)
C24	0.5130 (5)	0.2446 (2)	0.24764 (10)	0.0622 (7)

Table 2. *Selected geometric parameters* (Å, °)

S—C19	1.771 (2)	C8—C11	1.529 (3)
S—C18	1.807 (2)	C8—C9	1.532 (3)
C1—O1	1.220 (2)	C9—C14	1.510 (3)
C1—C2	1.506 (3)	C9—C10	1.533 (3)
C1—C6	1.516 (3)	C11—C12	1.528 (3)
C2—C3	1.531 (3)	C12—C13	1.487 (4)
C3—C4	1.528 (3)	C14—C13	1.316 (4)
C3—C17	1.540 (3)	C16—C17	1.558 (3)
C4—C5	1.539 (3)	C17—C18	1.516 (3)
C5—C10	1.525 (3)	C19—C24	1.381 (4)
C5—C16	1.545 (3)	C19—C20	1.383 (3)
C5—C6	1.550 (2)	C20—C21	1.378 (4)
C6—C7	1.568 (3)	C21—C22	1.386 (4)
C7—O2	1.434 (2)	C22—C23	1.365 (5)
C7—C15	1.529 (3)	C23—C24	1.370 (4)
C7—C8	1.536 (3)		
C19—S—C18	102.93 (10)	C11—C8—C9	109.4 (2)
O1—C1—C2	120.9 (2)	C11—C8—C7	115.1 (2)
O1—C1—C6	120.6 (2)	C9—C8—C7	112.0 (2)
C2—C1—C6	118.5 (2)	C14—C9—C8	109.7 (2)
C1—C2—C3	113.0 (2)	C14—C9—C10	111.0 (2)
C4—C3—C2	108.2 (2)	C8—C9—C10	111.4 (2)
C4—C3—C17	102.4 (2)	C5—C10—C9	113.8 (2)
C2—C3—C17	113.3 (2)	C12—C11—C8	110.2 (2)
C3—C4—C5	101.8 (2)	C13—C12—C11	112.8 (2)
C10—C5—C4	114.5 (2)	C13—C14—C9	123.5 (2)
C10—C5—C16	111.6 (2)	C14—C13—C12	123.8 (2)
C4—C5—C16	100.9 (2)	C5—C16—C17	107.1 (2)
C10—C5—C6	109.2 (2)	C18—C17—C3	116.9 (2)
C4—C5—C6	111.5 (2)	C18—C17—C16	113.0 (2)
C16—C5—C6	108.9 (2)	C3—C17—C16	103.8 (2)
C1—C6—C5	111.2 (2)	C17—C18—S	114.3 (2)
C1—C6—C7	111.53 (15)	C24—C19—C20	119.3 (2)
C5—C6—C7	117.1 (2)	C24—C19—S	117.8 (2)
O2—C7—C15	107.8 (2)	C20—C19—S	122.8 (2)
O2—C7—C8	106.6 (2)	C21—C20—C19	120.1 (3)
C15—C7—C8	110.0 (2)	C20—C21—C22	120.0 (3)
O2—C7—C6	107.4 (2)	C23—C22—C21	119.6 (3)
C15—C7—C6	114.7 (2)	C22—C23—C24	120.8 (3)
C8—C7—C6	109.92 (15)	C23—C24—C19	120.2 (3)

The crystal was attached to the tip of a glass fiber for data collection. The cell constants used for the data collection were determined from reflections found from a 360° rotation photograph. High-angle cell constants were determined from a subset of intense reflections in the 2θ range 35.0 to 50.0°. Lorentz and polarization corrections were applied, along with a correction based on the decay of the standard reflections. A series of azimuthal reflections were collected and used to generate a semi-empirical absorption correction.

The space group $P2_1/c$ was chosen based on systematic absences and intensity statistics. Direct methods (Sheldrick, 1990) were used to generate an E map from which all non-H atoms were correctly placed. All non-H atoms were refined with anisotropic displacement parameters. H atoms were refined as riding isotropic atoms.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

We thank the National Institutes of Health for generous support of this research.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1094). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Bromo-2-(2-nitroethenyl)furan

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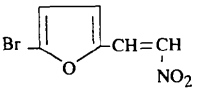
Abstract

The five-membered ring of the title compound, $C_6H_4BrNO_3$, has normal geometry and there are no unusual intramolecular distances or angles. The O atoms of the nitro group interact with the C—H groups of a neighboring molecule with H···O distances of 2.46 (1) and 2.56 (1) Å.

Comment

The structure determination of 5-bromo-2-(2-nitroethenyl)furan, (I), was undertaken as part of a molecular-modeling investigation. The crystals were obtained by slow recrystallization from ethanol. Details of the synthetic work will be published elsewhere (Castañedo & Estrada, 1995).



(I)

The structure and properties of the title compound are of great interest because of its proven pharmacological activity (Castañeda, 1993). A summary of the bond distances and angles, and intermolecular contacts shorter than the sum of the van der Waals radii (C 1.70, O 1.40 Å; Pauling, 1960), are listed in Table 2.

The displacement ellipsoids with the atomic numbering (*SHELXTL-Plus*; Sheldrick, 1991) are shown in Fig. 1. As shown in Fig. 2, the O atoms of the nitro group interact with the C—H groups of a neighboring molecule [C(4)—H(4)···O(2) and C(6)—H(6)···O(3) with H···O distances of 2.56 (1) and 2.46 (1) Å, respectively].

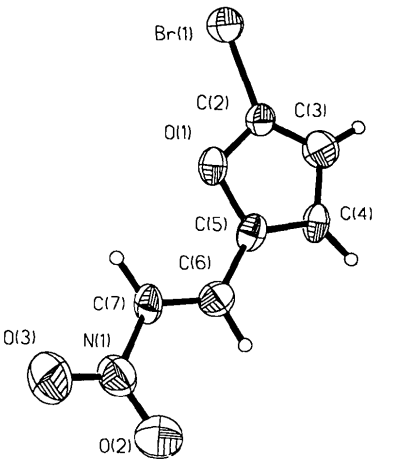


Fig. 1. Displacement ellipsoid plot (*SHELXTL-Plus*; Sheldrick, 1991) of the title compound. Ellipsoids are scaled to enclose 50% probability and H atoms are represented as spheres of arbitrary radii.

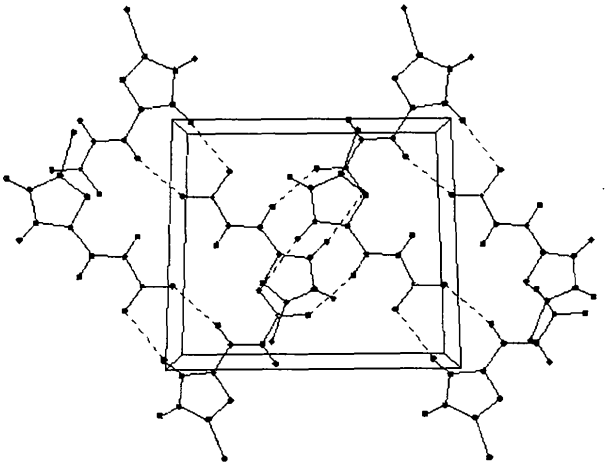


Fig. 2. The packing of the molecules in the unit cell. Hydrogen bonds are represented by dashed lines.

Experimental

Crystal data

$C_6H_4BrNO_3$
M_r = 218.01
Monoclinic
$P2_1/c$
a = 7.370 (1) Å
b = 10.919 (2) Å
c = 9.887 (1) Å
β = 108.45 (1)°

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 23 reflections
θ = 2.86–25°
μ = 5.3 mm^{-1}
T = 293 K
Prismatic