Related literature. The title compound undergoes radical polymerization to yield a high molecular weight polymer (Saotome, Miyazawa & Endo, 1988), which serves as an efficient column packing for the chromatographic resolution of various aromatic racemates (Saotome, Miyazawa & Endo, 1989). The structures of other penicillin sulfoxides have been reported: phenoxymethylpenicillin sulfoxide (Cooper, DeMarco, Cheng & Jones, 1969), cloxacillin sulfoxide (Blanpain & Durant, 1976), and benzylpenicillin sulfoxide (Labischinski, Naumann, Barnickel, Dreissig, Gruszecki, Hofer & Bradaczek, 1987).

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Structure of (±)-3-Benzyloxy-2,3,3a,7a-tetrahydrobenzo[*b*]thiophen-5(4*H*)-one 1,1-Dioxide

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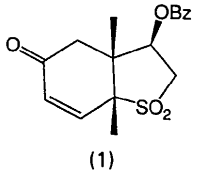
Abstracts. $C_{15}H_{16}O_4S$, (1), $M_r = 292{\cdot}35$, orthorhombic, *Pbca*, $a = 5{\cdot}9496\,(10)$, $b = 11{\cdot}483\,(3)$, $c = 40{\cdot}262\,(7)$ Å, $V = 2750{\cdot}7\,(10)$ Å^3, $Z = 8$, $D_x = 1{\cdot}41$ g cm^{-3}, $\mu = 2{\cdot}340$ cm^{-1}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $F(000) = 1232$, $T = 198$ K, $R = 0{\cdot}0451$ for 1703 reflections $[F_o \geq 4\sigma(F_o)]$. The two rings are *cis*-fused with the ring junction torsion angles being −38·2 (3)° for C3—C3a—C7a—S1 and −38·4 (4)° for C4—C3a—C7a—C7. The thiophene ring assumes the envelope conformation [C3a is 0·590 (3) Å from the plane through S1, C2, C3 and C7a] while the cyclohexanone moiety is in the sofa conformation [C3a is 0·530 (3) Å from the plane through C4, C5, C6, C7 and C7a].

Experimental. (1) was synthesized *via* an intermolecular Diels–Alder reaction of *trans*-1-methoxy-3-trimethylsiloxy-1,3-butadiene (Danishefski & Kitahara, 1974) and the benzyl protected allylic alcohol obtained from 2,5-dihydrothiophene 1,1-dioxide. Full synthetic details will be described elsewhere (Martin & Daniel, 1990). Crystals of (1) were obtained by slow evaporation from ethyl acetate. The data crystal was a colorless plate of approximate dimensions 0·09 × 0·49 × 0·51 mm. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and equipped with a Nicolet LT-2 low-temperature delivery system. Lattice parameters were obtained from the least-squares refinement of 40 reflections with $18{\cdot}2 < 2\theta < 24{\cdot}8°$. Data were collected using the ω-scan technique, with a 1·2° ω scan at 4–8° min^{-1} and 2θ range from 4·0–50·0° ($h = -7\rightarrow 7$, $k = 0\rightarrow 13$, $l = 0\rightarrow 47$). 5196 reflections were collected, of which 2431 were unique, with an $R_{\rm int} = 0{\cdot}0185$. Three reflections ($\bar{1},\bar{2},\overline{17}$; $\bar{1},3,\overline{15}$; 2,1,13) were remeasured every 97 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor ranged from 0·996–1·05. The data were also corrected for Lp effects but not absorption. Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (728 reflections). Data reduction and decay correction were performed using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick,



(1)

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *for the non*-H *atoms*

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

	x	*y*	*z*	U_{eq}
S1	−0·18211 (13)	0·56522 (7)	0·64601 (2)	0·0294 (2)
C2	−0·1823 (6)	0·7098 (3)	0·62929 (8)	0·0304 (11)
C3	0·0371 (5)	0·7652 (3)	0·64112 (8)	0·0268 (9)
C3a	0·0944 (5)	0·7148 (3)	0·67519 (7)	0·0245 (9)
C4	−0·0495 (6)	0·7661 (3)	0·70286 (8)	0·0309 (11)
C5	−0·0270 (5)	0·7017 (3)	0·73513 (8)	0·0305 (10)
C6	0·0065 (5)	0·5753 (3)	0·73304 (8)	0·0358 (11)
C7	0·0420 (5)	0·5207 (3)	0·70452 (8)	0·0309 (10)
C7a	0·0646 (5)	0·5835 (3)	0·67238 (7)	0·0251 (10)
O8	−0·3785 (4)	0·5475 (2)	0·66641 (6)	0·0399 (8)
O9	−0·1367 (4)	0·4811 (2)	0·62026 (6)	0·0490 (9)
O10	−0·0457 (4)	0·7509 (2)	0·76195 (5)	0·0415 (8)
O11	0·2178 (3)	0·7334 (2)	0·61965 (5)	0·0284 (7)
C12	0·2273 (7)	0·7995 (3)	0·58951 (8)	0·0322 (11)
C13	0·3892 (5)	0·7412 (3)	0·56645 (7)	0·0270 (9)
C14	0·3805 (7)	0·6220 (3)	0·56194 (9)	0·0418 (13)
C15	0·5333 (8)	0·5671 (4)	0·54154 (10)	0·0548 (15)
C16	0·6943 (7)	0·6300 (4)	0·52514 (9)	0·0496 (15)
C17	0·7050 (6)	0·7481 (4)	0·52921 (8)	0·0432 (13)
C18	0·5538 (6)	0·8034 (3)	0·55001 (8)	0·0356 (11)

Table 2. *Bond lengths* (Å) *and angles* (°) *for the non*-H *atoms*

1	2	3	1–2	1–2–3
C2	S1	C7a	1·792 (3)	96·45 (15)
C2	S1	O8		110·14 (15)
C7a	S1	O8	1·824 (3)	109·67 (14)
C7a	S1	O9		110·18 (15)
O8	S1	O9	1·442 (2)	117·79 (15)
O9	S1	C2	1·442 (3)	110·53 (15)
C3	C2	S1	1·528 (5)	105·5 (2)
C3a	C3	O11	1·527 (4)	106·2 (2)
C3a	C3	C2		108·2 (3)
O11	C3	C2	1·427 (4)	110·4 (2)
C4	C3a	C7a	1·524 (5)	111·9 (3)
C4	C3a	C3		112·6 (3)
C7a	C3a	C3	1·522 (4)	106·4 (2)
C5	C4	C3a	1·501 (5)	113·1 (3)
C6	C5	O10	1·468 (5)	121·3 (3)
C6	C5	C4		116·7 (3)
O10	C5	C4	1·224 (4)	121·9 (3)
C7	C6	C5	1·325 (5)	122·6 (3)
C7a	C7	C6	1·488 (4)	122·6 (3)
S1	C7a	C3a		104·6 (2)
S1	C7a	C7		112·2 (2)
C3a	C7a	C7		115·2 (3)
C12	O11	C3	1·433 (4)	114·0 (2)
C13	C12	O11	1·496 (5)	108·3 (3)
C14	C13	C18	1·382 (5)	118·4 (3)
C14	C13	C12		120·0 (3)
C18	C13	C12	1·381 (5)	121·5 (3)
C15	C14	C13	1·378 (6)	120·5 (4)
C16	C15	C14	1·370 (6)	120·5 (4)
C17	C16	C15	1·367 (6)	119·9 (4)
C18	C17	C16	1·383 (5)	119·8 (4)
C13	C18	C17		120·9 (3)

1988). The structure was solved by direct methods and refined by full-matrix least squares (Sheldrick, 1988). In all, 245 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{\text{peak}} - I_{\text{background}}) \times$ (scan rate); where 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = (I_{\text{peak}} + I_{\text{background}})^{1/2} \times$ (scan rate). Final $R = 0{\cdot}0451$ for 1703 reflections, with $wR = 0{\cdot}0514$ ($R_{\text{all}} = 0{\cdot}0748$, $wR_{\text{all}} = 0{\cdot}0595$) and goodness of fit = 1·582. The maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were −0·33 and 0·23 e Å^{-3}, respectively. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55). The positional and thermal parameters for non-H atoms are listed in Table 1,[*] while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1 (*SHELXTL-Plus*; Sheldrick, 1988). Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The benzothiophene structure is a component of many known biologically active compounds. It is a component of Breynins A and B (Sakai, Ohkuma, Koshiyama, Naito & Kawaguchi, 1976; Nishiyama, Ikeda, Yoshida & Yamamura, 1989). Breynins A and B exhibit significant hypocholesterolemic activity in mammals (Koshiyama, Hatori, Ohkuma, Sakai, Imanishi, Ohbayashi & Kawaguchi, 1976). The structures of some similar

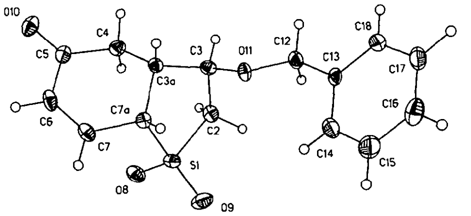

Fig. 1. View of (1) showing the atom-labeling scheme. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size.

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[*] Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving the H atoms, torsion angles, least-squares planes and H-atom parameters and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53768 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

compounds have already been reported (Lynch, Daniel, Martin & Davis, 1989, 1990).

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Structure of a Novel Bisindole Derivative

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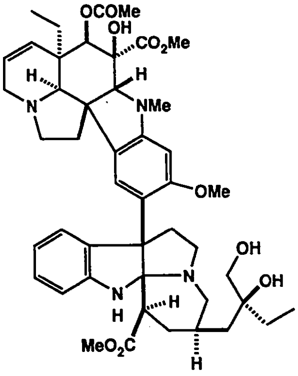
Abstract. (3a*R*-{3aα,4β,5aβ,9[1*R*,3*R*(2*S*),5*R*,7a*S*,12a*S*],10b*R*,13aα})-Methyl-4-(acetyloxy)-3a-ethyl-3a,4,5,5a,6,11,12,13a-octahydro-9-[1,2,3,4,6,7,7a,12-octahydro-3-(2-hydroxy-2-hydroxymethylbutyl)-1-(methoxycarbonyl)indolizino[1,2-*b*]indol-7a-yl]-5-hydroxy-8-methoxy-6-methyl-1*H*-indolizino[8,1-*c*,*d*]-carbazole-5-carboxylate methanol hydrate solvate, $C_{46}H_{60}N_4O_{10}.CH_3OH.H_2O$, $M_r = 879{\cdot}06$, monoclinic, $P2_1$, $a = 8{\cdot}2878\,(8)$, $b = 31{\cdot}018\,(4)$, $c = 9{\cdot}2267\,(7)$ Å, $\beta = 105{\cdot}211\,(7)°$, $V = 2288{\cdot}8\,(4)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, $\mu = 0{\cdot}8580$ cm^{-1}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $F(000) = 944$, $T = 198$ K, $R = 0{\cdot}0382$ for 2931 reflections $[F_o \geq 4\sigma(F_o)]$. A very short intramolecular hydrogen-bonding interaction is observed between the hydroxyl group and N9 of the vindoline portion of the molecule. The relevant parameters are: O3—H3O···N9, O···N 2·598 (4), H···N 1·68 (5) Å, O—H···N 164 (5)°. Molecules related by the 2_1 screw operation are hydrogen bonded into columns parallel to **b**. The relevant hydrogen-bonding parameters are: O18′—H18O′···O3 ($-x$, $0{\cdot}5+y$, $1+z$), O···O 2·925 (4), H···O 2·36 (6) Å, O—H···O 119 (5)°; O18′—H18O′···O23 ($-x$, $0{\cdot}5+y$, $1-z$), O···O 3·089 (4), H···O 2·17 (6) Å, O—H···O 168 (5)°; O19′—H19O′···O3 ($-x$, $0{\cdot}5+y$, $1-z$), O···O 2·801 (4), H···O 1·88 (6) Å, O—H···O 169 (5)°. The hydrogen bonding at H18O′ is bifurcated and the O3···H···O23 angle is 70 (2)°. The solvent methanol molecule (C1*A*, O1*A*) and water molecule (O1*B*) are also involved in hydrogen-bonding interactions with the following parameters: O1*A*—H1*A*···O19′ ($1+x$, y, z), O···O 2·711 (5), H···O 1·77 (9) Å, O—H···O 156 (7)°; O1*B*—H1*B*···O18′, O···O 2·932 (5), H···O 1·86 (12) Å, O—H···O 161 (9)°; O1*B*—H2*B*···O1*A*, O···O 2·777 (6), H···O 1·98 (7) Å, O—H···178 (7)°.

Experimental. (1) was obtained in two steps from the product of a novel coupling reaction between the *Aspidosperma* alkaloid vindoline and a substituted indolizino[8,7-*b*]indole derivative (Magnus, Ladow, Elliot & Kim, 1989; Magnus, Stamford & Ladlow, 1990). The absolute configuration of (1) was assigned



(1)

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