

Fig. 2. Crystal structure viewed along the c axis. The hydrogen bonds are indicated by broken lines. The ethanol molecule with the lower occupancy factors is omitted.

triazine rings, are disordered. The triazine and ethanol molecules are alternately connected to form a ribbon along the b axis by the two types of intermolecular hydrogen bond: $N(5)\cdots H-O(31)$ and $N(3)-H\cdots O(31)$. The distances of $N(5)\cdots O(31)$, $N(3)\cdots O(31)$, $N(5)\cdots O(31')$, and $N(3)\cdots O(31')$ are 2.90 (1),

2.86 (1), 2.78 (2), and 3.01 (3) Å, respectively. Except for the hydrogen bonds, there are no intermolecular distances less than the van der Waals distances. N(3) serves as a hydrogen-bond donor and H(3) is in the direction of the O atom of the ethanol [$N(3)-H\cdots O(31) = 157$ (3), $N(3)-H\cdots O(31') = 155$ (3)°], while N(5) serves as a hydrogen-bond acceptor. This hydrogen-bonding scheme suggests that the H atom could be partially bound to N(5). Although the mechanism of the photocoloration is still not clarified after the determination of the structure of the title compound, it could be proposed that the hydrogen-bonding scheme may inhibit the photocoloration for this inclusion compound.

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Structure of Naphtho[1,8-*de*][1,2]oxathiepin 3-Oxide

BY MARC-J. OLIVIER AND FRANÇOIS BRISSE

Département de Chimie, Université de Montréal, CP 6128, Succ. 'A', Montréal, Québec, Canada H3C 3J7

AND DAVID N. HARPP AND KOSTA STELIOU

Department of Chemistry, McGill University, 801 Sherbrooke Street West, Montreal, Quebec, Canada H3A 2K6

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Abstract. $C_{12}H_{10}O_2S$, $M_r = 218.28$, orthorhombic, $Pna2_1$, $a = 11.574$ (4), $b = 5.794$ (2), $c = 30.128$ (8) Å, $V = 2020.4$ Å³, $Z = 8$, $D_x = 1.435$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.28$ mm⁻¹, $F(000) = 912$, $T = 295$ K, $R = 0.035$ and $wR = 0.040$ for 1442 observed reflections. The two molecules in the asymmetric unit are almost centrosymmetrically related. The $-O-SO-$ group is incorporated in a seven-membered-ring system bridging the naphthalene moiety. The $S=O$ bonds are 1.463 (3) and

1.485 (3) Å while the two intracyclic $S-O$ bonds are 1.602 (3) and 1.610 (3) Å. For both molecules the three bond angles involving the S atom are in the range 98.7 (2) to 109.5 (2)°.

Introduction. The sulfinate functional group in a ring system was first reported by Baumann & Walter (1893) although to our knowledge its structure has never been verified. It was nearly 80 years before the next examples appeared by way of a thermolysis reaction (Dittmer,

Henion & Takashina, 1969), and in the work of Harpp & Gleason (1969) who first synthesized the parent five- and six-membered-ring alicyclic sultines. More recently, Harpp & Steliou (1987) have been successful in introducing this functional moiety into the benzylic position of a seven-membered-ring system. This paper reports the first structural study of a sultine in a seven-membered ring.

Experimental. The synthesis of the title compound is reported elsewhere (Steliou, 1978). Colorless crystal, $0.20 \times 0.20 \times 0.32$ mm, m.p. 406–407 K, density by flotation in ZnCl_2 aqueous solution. Nonius CAD-4 diffractometer, graphite-monochromatized $\text{Mo K}\alpha$, unit-cell dimensions with 25 reflections with $10 \leq 2\theta \leq 25^\circ$, data collection in ω - 2θ scan mode, scan range $\Delta\omega = (1.00 + 0.35\tan\theta)^\circ$, $2\theta_{\text{max}} = 50.0^\circ$ ($0 \leq h \leq 13$, $0 \leq k \leq 6$, $0 \leq l \leq 35$), orientation checked every 100 reflections, intensity monitored every hour using three standard reflections (largest fluctuation of the standard 3.6%), 1806 independent reflections, 1442 satisfying $I \geq 1.96\sigma(I)$ retained for structure determination and refinement. Lp correction, no absorption correction; direct methods (*MULTAN*).*

Anisotropic block-diagonal least-squares refinement based on F , quantity minimized $\sum w(|F_o| - |F_c|)^2$, H atoms located on difference Fourier syntheses, refined isotropically. Final $R = 0.035$, $wR = 0.040$, weights based on the counting statistics, $S = 1.50$. Maximum $\Delta/\sigma = 0.34$, mean $\Delta/\sigma = 0.05$, extreme values of the residual electron density on the final difference Fourier map $-0.22, +0.32 \text{ e } \text{\AA}^{-3}$. Scattering factors for S, O, C from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); real and imaginary components of the anomalous dispersion of S from Cromer & Liberman (1970) included in the least-squares calculations.

Discussion. The final atomic coordinates† are given in Table 1 while the atomic numbering is shown in Fig. 1. The bond distances and angles are compared in Table 2 to those of the only known related sultine (Haasnoot, Liskamp, van Dael, Noordik & Ottenheijm, 1983) and the naphthalene ring system (Bright, Maxwell & de

Boer, 1973). Selected torsion angles of the oxathiepin moiety are also listed in Table 2.

Table 1. Final atomic coordinates and their e.s.d.'s ($\times 10^5$ for S, $\times 10^4$ for O and C) and U_{eq} ($\text{\AA}^2 \times 10^4$)

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
S(1A)	29055 (10)	14541 (18)	71006	456
O(1A)	3228 (3)	2022 (5)	7565 (1)	525
O(2A)	1933 (2)	3232 (5)	6938 (1)	492
C(1A)	2450 (4)	6067 (7)	6387 (1)	416
C(2A)	1822 (4)	7803 (9)	6193 (2)	543
C(3A)	1875 (5)	8215 (9)	5735 (2)	677
C(4A)	2520 (5)	6820 (9)	5478 (2)	688
C(5A)	3831 (5)	3509 (10)	5379 (2)	631
C(6A)	4456 (5)	1806 (10)	5547 (2)	678
C(7A)	4508 (4)	1473 (9)	6009 (2)	577
C(8A)	3905 (3)	2865 (7)	6293 (1)	398
C(9A)	3178 (3)	4644 (8)	6124 (1)	404
C(10A)	3173 (4)	5012 (8)	5652 (1)	514
C(11A)	2261 (4)	5649 (8)	6873 (1)	459
C(12A)	4109 (3)	2496 (8)	6782 (1)	444
S(1B)	50427 (9)	-19695 (17)	78025 (4)	432
O(1B)	4853 (3)	-2638 (6)	7340 (1)	529
O(2B)	5972 (2)	-3684 (5)	8025 (1)	485
C(1B)	5322 (4)	-6422 (7)	8577 (2)	440
C(2B)	5866 (4)	-8103 (8)	8821 (2)	607
C(3B)	5660 (5)	-8442 (9)	9270 (2)	742
C(4B)	4940 (5)	-7020 (10)	9485 (2)	667
C(5B)	3643 (5)	-3657 (10)	9497 (1)	644
C(6B)	3090 (5)	-1910 (10)	9287 (2)	645
C(7B)	3180 (4)	-1721 (8)	8830 (2)	496
C(8B)	3844 (3)	-3183 (7)	8579 (1)	398
C(9B)	4507 (4)	-4942 (7)	8798 (1)	412
C(10B)	4357 (4)	-5215 (8)	9265 (1)	528
C(11B)	5659 (4)	-6096 (8)	8101 (2)	470
C(12B)	3762 (4)	-2975 (7)	8087 (1)	430

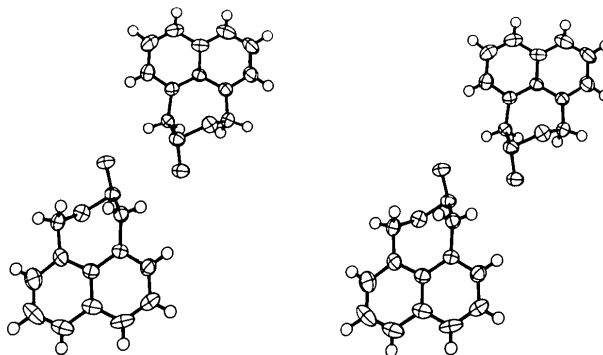
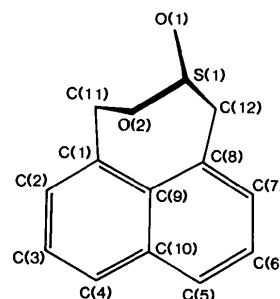


Fig. 1. Atomic numbering and stereopair of the two oxathiepin molecules in the asymmetric unit.

* The programs used here are modified versions of *NRC2*, data reduction, *NRC10*, bond distances and angles, *NRC22*, mean planes (Ahmed, Hall, Pippy & Huber, 1973), *FORDAP*, Fourier and Patterson maps (A. Zalkin), *NUCLS*, least-squares refinement (Doedens & Ibers, 1967); *MULTAN*, multiresolution program (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); *BMF*, best molecular fit (Nyburg, 1969); and *ORTEP*, stereodrawings (Johnson, 1965).

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44604 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

There are two distinct molecules, *A* and *B*, in the asymmetric unit. As can be observed in Fig. 1 the two molecules are almost identical and almost centrosymmetrically related. A close examination of the bond

Table 2. Comparison of the bond distances (Å), angles (°) and relevant torsion angles (°) of the two oxathiepin molecules in the asymmetric unit with literature analogs

	<i>A</i>	<i>B</i>	<i>A</i> *	<i>B</i> *
S(1)—O(1)	1.485 (3)	1.463 (3)	1.466 (7)	1.453 (7)
S(1)—O(2)	1.602 (3)	1.610 (3)	1.586 (7)	1.598 (6)
S(1)—C(12)	1.796 (4)	1.809 (5)	1.780 (7)	1.833 (9)
O(2)—C(11)	1.464 (5)	1.462 (5)	1.426 (11)	1.403 (8)
C(1)—C(11)	1.500 (6)	1.498 (7)		
C(8)—C(12)	1.507 (6)	1.490 (6)	†	
C(1)—C(2)	1.372 (7)	1.373 (7)	1.360 (1)	
C(1)—C(9)	1.420 (6)	1.438 (6)	1.437 (1)	
C(1)—C(11)	1.500 (6)	1.498 (7)	1.512 (1)	
C(2)—C(3)	1.401 (7)	1.387 (8)	1.388 (2)	
C(3)—C(4)	1.345 (8)	1.339 (8)	1.346 (2)	
C(4)—C(10)	1.394 (7)	1.410 (7)	1.416 (1)	
C(5)—C(6)	1.324 (8)	1.354 (8)	1.341 (2)	
C(5)—C(10)	1.419 (7)	1.409 (7)	1.415 (1)	
C(6)—C(7)	1.406 (7)	1.385 (7)	1.394 (2)	
C(7)—C(8)	1.367 (7)	1.371 (6)	1.365 (1)	
C(8)—C(9)	1.425 (6)	1.436 (6)	1.428 (1)	
C(8)—C(12)	1.507 (6)	1.490 (6)	1.516 (1)	
C(9)—C(10)	1.438 (6)	1.426 (6)	1.431 (1)	
O(1)—S(1)—O(2)	108.8 (2)	109.5 (2)	109.4	110.8
O(1)—S(1)—C(12)	103.5 (2)	104.1 (2)	106.4	103.1
O(2)—S(1)—C(12)	99.5 (2)	98.7 (2)	93.3	93.2
S(1)—O(2)—C(11)	118.3 (3)	119.3 (3)		
S(1)—C(12)—C(8)	116.7 (3)	116.4 (3)		
O(2)—C(11)—C(1)	108.8 (4)	109.6 (4)	†	
C(2)—C(1)—C(11)	117.2 (4)	118.9 (4)	116.2 (1)	
C(9)—C(1)—C(11)	122.5 (4)	122.6 (4)	124.7 (1)	
C(2)—C(1)—C(9)	120.2 (4)	118.4 (4)	119.1 (1)	
C(1)—C(2)—C(3)	121.4 (5)	122.9 (5)	123.2 (1)	
C(2)—C(3)—C(4)	119.2 (5)	119.4 (5)	119.8 (1)	
C(3)—C(4)—C(10)	122.4 (5)	121.8 (5)	120.5 (1)	
C(4)—C(10)—C(5)	122.3 (5)	121.5 (5)	119.5 (1)	
C(4)—C(10)—C(9)	119.0 (4)	119.2 (4)	120.2 (1)	
C(5)—C(10)—C(9)	118.7 (4)	119.3 (4)	120.3 (1)	
C(6)—C(5)—C(10)	121.9 (5)	121.6 (5)	120.1 (1)	
C(5)—C(6)—C(7)	120.3 (5)	119.2 (5)	120.2 (1)	
C(6)—C(7)—C(8)	121.1 (5)	122.8 (4)	122.7 (1)	
C(7)—C(8)—C(9)	120.3 (4)	119.0 (4)	118.9 (1)	
C(7)—C(8)—C(12)	116.6 (4)	117.6 (4)	116.2 (1)	
C(9)—C(8)—C(12)	123.0 (4)	123.3 (4)		
C(1)—C(9)—C(8)	124.8 (4)	124.1 (4)	125.2 (1)	
C(1)—C(9)—C(10)	117.6 (4)	118.1 (4)	117.2 (1)	
C(8)—C(9)—C(10)	117.6 (4)	117.8 (4)	117.6 (1)	
Seven-membered ring				
O(1)—S(1)—O(2)—C(11)	68.6 (3)	-68.3 (3)	-78.9	-74.6
O(1)—S(1)—C(12)—C(8)	-161.5 (3)	162.3 (3)	107.4	106.6
O(2)—S(1)—C(12)—C(8)	-49.4 (3)	49.6 (3)	-4.0	-5.6
C(12)—S(1)—O(2)—C(11)	-39.3 (3)	40.1 (3)	29.8	30.1
S(1)—O(2)—C(11)—C(1)	100.1 (4)	-99.7 (4)	-46.3	-46.9
C(9)—C(1)—C(11)—O(2)	-51.2 (5)	49.5 (5)		
C(11)—C(1)—C(9)—C(8)	-7.7 (7)	7.0 (7)		
C(9)—C(8)—C(12)—S(1)	69.0 (5)	-71.7 (5)		
C(12)—C(8)—C(9)—C(1)	-8.6 (7)	11.7 (6)		

* Haasnoot, Liskamp, van Dael, Noordik & Ottenheijm (1983).

Only the relevant quantities are listed.

† Bright, Maxwell & de Boer (1973).

Table 2 (cont.)

	<i>A</i>	<i>B</i>
Naphthalene ring		
C(2)—C(1)—C(9)—C(8)	177.1 (7)	-177.3 (7)
C(2)—C(1)—C(9)—C(10)	-3.5 (7)	3.5 (7)
C(9)—C(1)—C(2)—C(3)	0.3 (7)	0.3 (7)
C(1)—C(2)—C(3)—C(4)	2.7 (7)	-3.0 (7)
C(2)—C(3)—C(4)—C(10)	-2.2 (7)	1.7 (7)
C(3)—C(4)—C(10)—C(5)	178.5 (7)	-176.9 (7)
C(3)—C(4)—C(10)—C(9)	-1.2 (7)	2.2 (7)
C(6)—C(5)—C(10)—C(4)	179.5 (7)	179.5 (7)
C(6)—C(5)—C(10)—C(9)	-0.8 (7)	0.3 (7)
C(10)—C(5)—C(6)—C(7)	-1.6 (7)	3.4 (7)
C(5)—C(6)—C(7)—C(8)	1.0 (7)	-2.5 (7)
C(6)—C(7)—C(8)—C(9)	2.1 (7)	-2.1 (7)
C(7)—C(8)—C(9)—C(1)	175.0 (7)	-173.5 (7)
C(7)—C(8)—C(9)—C(10)	-4.4 (7)	5.7 (7)
C(1)—C(9)—C(10)—C(4)	4.0 (7)	-4.7 (7)
C(1)—C(9)—C(10)—C(5)	-175.7 (7)	174.4 (7)
C(8)—C(9)—C(10)—C(4)	-176.5 (7)	176.0 (7)
C(8)—C(9)—C(10)—C(5)	3.8 (7)	-4.8 (7)

distances and angles of the two molecules revealed significantly different *exo* S—O bond lengths, short S...O contacts and dihedral angles between the two rings of the naphthalene moiety differing by as much as 4°. There are also two independent but very similar molecules per asymmetric unit in the only other crystallographic study of a sultine (Haasnoot *et al.*, 1983).

The present molecule consists of a seven-membered sultine ring system bridging the naphthalene moiety. The S(1)—O(1) bonds are significantly different but fall within the range of S—O distances. Other bond distances in the two oxathiepin rings compare well. The bond angles in these rings vary from 99.5 (3) at S(1) to 124.8 (3)° in *A* and from 98.7 (2) at S(1) to 124.0 (3)° in *B*. The angles at S(1) indicate a pyramidal arrangement of S and its neighbors, with S(1) atoms at 0.667 (1) and 0.662 (1) Å above the planes O(1), O(2) and C(12) in *A* and *B* respectively.

The naphthalene part of the molecule is not planar and shows some distortions which are attributed to the presence of the seven-membered ring at C(1) and C(8). These differences in bond distances and angles are quite similar to those observed in 1,8-dimethylnaphthalene (Bright *et al.*, 1973). These authors have shown, by strain-energy-minimization calculations, that the repulsive energy between the methyl groups is reduced mainly by the enlargement of the bond angles at C(1), C(8) and C(9). We do observe the same effects in both *A* and *B* molecules. We also observe that C(1) and C(8) deviate the most from their corresponding naphthalene mean planes. These atoms are at 0.049 (4) and -0.052 (4) Å respectively from the mean plane (molecule *A*) and at -0.060 (4) and 0.064 (4) Å (molecule *B*). This is in agreement with similar observations reported in 1,4,5,8-tetrachloronaphthalene (Gafner & Herbstein, 1962) and 3-bromo-1,8-dimethylnaphthalene (Jameson & Penfold, 1965), where the substituents are significantly above and below the naphthalene mean plane.

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Structural Comparison of 1-Bromo- and 1-Fluoroestradiol with D-Estradiol

BY MARK D. HYLARIDES,*† EILEEN N. DUESLER,‡ FRED A. METTLER* AND ALBERTO A. LEON*

Department of Radiology and Department of Chemistry, University of New Mexico, Albuquerque, New Mexico 87131

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Abstract. 1-Bromoestra-1,3,5(10)-trien-3,17 β -diol, $C_{18}H_{23}BrO_2$, $M_r = 351.3$, orthorhombic, $P2_12_12_1$, $a = 9.3864$ (12), $b = 12.927$ (2), $c = 12.948$ (2) Å, $V = 1571.1$ (4) Å³, $Z = 4$, $D_x = 1.484$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 259$ mm⁻¹, $F(000) = 728$, $T = 298$ K, $R = 0.0514$ for 2083 observed reflections. 1-Fluoroestra-1,3,5(10)-trien-3,17 β -diol.0.5H₂O, $C_{18}H_{23}FO_2 \cdot 0.5H_2O$, $M_r = 299.4$, orthorhombic, $P2_12_12_1$, $a = 12.046$ (4), $b = 19.358$ (5), $c = 6.656$ (2) Å, $V = 1552.1$ (7) Å³, $Z = 4$, $D_x = 1.28$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 86$ mm⁻¹, $F(000) = 644$, $T = 293$ K, $R = 0.0776$ for 2781 observed reflections. Except for the carbon-halogen bond, bond angles and bond lengths of 1-bromoestradiol are within 7 e.s.d.'s of estradiol. Bond angles and bond lengths of 1-fluoroestradiol are within 11 and 7 e.s.d.'s of estradiol, respectively. The molecular conformations are nearly identical to that of estradiol. Like estradiol, 1-fluoroestradiol is present as a hemihydrate and extensive hydrogen bonding is observed. 1-Bromoestradiol is not

hydrated and hydrogen bonding is limited to hydroxyl groups along the c axis.

Introduction. The radiohalogenated analogs of 1-bromo- and 1-fluoroestradiol, (1) and (2), respectively, were being considered by our group as receptor-mediated radiopharmaceuticals for potential use in nuclear medicine for breast cancer studies. Their observed *in vitro* binding affinities to estrogen receptors, however, were substantially lower than that of estradiol. The structural characteristics of these compounds relative to estradiol became important for elucidation of the relationship between molecular conformations and the observed *in vitro* binding affinities. This paper describes the molecular and crystal structures of the 1-halogenated compounds and their structural similarities to estradiol.

Experimental. The title compounds were prepared in our laboratory (Hylarides, Leon, Mettler & Wilbur, 1984; Hylarides, Leon & Mettler, unpublished) as colorless transparent prisms by slow crystallization from ethanol/H₂O. Cell dimensions were determined from 25 diffractometer-measured reflections in the 2θ range 10 to 40° for (1) and 7 to 28° for (2) (Syntex

* Department of Radiology.

† Present address: NeoRx Corporation, 410 W. Harrison, Seattle, Washington 98119, USA.

‡ Department of Chemistry.