Related literature. Structural data of several cyclic sulfones have been published by Yasuoka, Kasai, Tanaka, Nagai & Tokura (1972), Yasuoka, Kai & Kasai (1975), Towns & Simonsen (1975), Ealick, van der Helm, Ramalingan, Thyvelikakath & Berlin (1977), Ealick, van der Helm & Baker (1979), Desiraju & Kamala (1983) and Castellano, De Simone, Zukerman-Schpector, Ferreira & Catani (1989).

This work has received partial support from CNPq, CAPES, FAPESP and FINEP. One of us (JZS) thanks CNPq for the award of a research grant.

References

CASTELLANO, E. E., DE SIMONE, C. A., ZUKERMAN-SCHPECTOR, J., FERREIRA, J. T. B. & CATANI, V. (1989). *Acta Cryst.* C**45**, 959–961.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

DESIRAJU, G. R. & KAMALA, R. (1983). *Acta Cryst.* C**39**, 358–360.

EALICK, S. E., VAN DER HELM, D. & BAKER, J. R. (1979). *Acta Cryst.* B**35**, 495–497.

EALICK, S. E., VAN DER HELM, D., RAMALINGAN, K., THYVELIKAKATH, G. X. & BERLIN, K. D. (1977). *J. Heterocycl. Chem.* **14**, 387–390.

FERREIRA, J. T. B. & CATANI, V. (1987). Int. Congr. Heterocycl. Chem., Hamburg, Federal Republic of Germany.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TOWNS, R. L. R. & SIMONSEN, S. H. (1975). *Cryst. Struct. Commun.* **4**, 469–472.

YASUOKA, N., KAI, Y. & KASAI, N. (1975). *Acta Cryst.* B**31**, 2729–2731.

YASUOKA, N., KASAI, N., TANAKA, M., NAGAI, T. & TOKURA, N. (1972). *Acta Cryst.* B**28**, 3393–3399.

Acta Cryst. (1989). C**45**, 1448–1450

Structures of Sulfur Analogues of Precocenes. IV. 7-Ethoxy-6-methoxy-2,2-dimethyl-2*H*-1-benzothiopyran 1,1-Dioxide

BY C. A. DE SIMONE, J. ZUKERMAN-SCHPECTOR AND E. E. CASTELLANO

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369, 13560 *São Carlos, SP, Brazil*

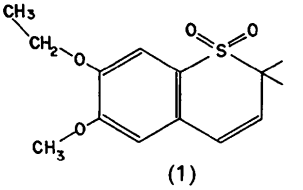
AND J. TERCIO B. FERREIRA AND V. CATANI

Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, 13560 *São Carlos, SP, Brazil*

(*Received* 23 *February* 1989; *accepted* 31 *March* 1989)

Abstract. $C_{14}H_{18}O_4S$, $M_r = 282{\cdot}36$, monoclinic, $P2_1/c$, $a = 11{\cdot}372\ (2)$, $b = 10{\cdot}544\ (3)$, $c = 12{\cdot}122\ (3)$ Å, $\beta = 106{\cdot}23\ (2)°$, $V = 1396\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}344$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}19$ mm^{-1}, $F(000) = 600$, $T = 298$ K, $R = 0{\cdot}042$ for 1265 observed reflections. The S atom is tetrahedral with a dihedral angle between planes C—S—C and O—S—O of 90·0 (1)°. The S—C(sp^2) and S—C(sp^3) bond distances are 1·758 (4) and 1·800 (3) Å, respectively.

Experimental. The synthesis of compound (1) has been reported previously (Ferreira & Catani, 1987). The data-collection and refinement parameters are summarized in Table 1. The structure was solved using standard direct methods and difference Fourier techniques. In final cycles of full-matrix least-squares refinement all non-H atoms anisotropic. H atoms included, as fixed contributors, at positions found in difference synthesis, all with a common isotropic temperature factor that refined to $U = 0{\cdot}077$ Å^2. Scattering factors for non-H atoms from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970), for H from Stewart, Davidson & Simpson (1965). Programs used: *SHELX*76 (Sheldrick, 1976), *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 11/780 computer of the Instituto de Física e Química de São Carlos.



(1)

0108-2701/89/091448-03$03.00

Table 1. *Crystallographic summary for* (1)

(*a*) Data collection[i,ii]	
Mode	ω–2θ
Scan rate (° min^{-1})	2·6–6·7
θ range (°)	0–22
Range of *hkl*	$-11<h<11$, $0<k<11$, $0<l<12$
Total reflections measured	1990
Unique reflections	1703
R_{int}	0·014
Crystal dimensions (mm)	~0·23 × 0·28 × 0·30
(*b*) Structure refinement[iii]	
Reflections used [$I > 3\sigma(I)$][iv]	1265
No. of variables	173
R, wR	0·042, 0·045
Max. shift/e.s.d.	0·003
Max., min. density in final difference map (e Å^{-3})	0·21, −0·25
S	1·99

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $11 < \theta < 20°$. (ii) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator. Two standard reflections (804 and 404) measured every hour showed no significant variation. No correction for absorption. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = [\sigma^2(F_o) + 0{\cdot}0005F_o^2]$. (iv) Reflections 002 and $\bar{1}22$ were also excluded during refinement.

Table 2. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2)

	x	y	z	B_{iso}*
S	0·4236 (1)	0·6399 (1)	0·3170 (1)	2·40 (3)
O(1)	0·4365 (2)	0·7613 (2)	0·3737 (2)	3·65 (9)
O(2)	0·5115 (2)	0·5437 (2)	0·3684 (2)	3·41 (8)
O(3)	−0·0711 (2)	0·4496 (3)	0·2769 (2)	4·4 (1)
O(4)	0·1072 (2)	0·3619 (2)	0·4378 (2)	3·47 (9)
C(2)	0·4216 (3)	0·6631 (3)	0·1694 (3)	2·2 (1)
C(3)	0·3097 (3)	0·7413 (3)	0·1129 (3)	2·7 (1)
C(4)	0·2021 (4)	0·7245 (3)	0·1339 (3)	2·9 (1)
C(5)	0·0606 (3)	0·5895 (3)	0·2069 (3)	2·7 (1)
C(6)	0·0401 (3)	0·4990 (4)	0·2809 (3)	2·8 (1)
C(7)	0·1379 (3)	0·4506 (3)	0·3698 (3)	2·6 (1)
C(8)	0·2559 (3)	0·4932 (3)	0·3808 (3)	2·3 (1)
C(9)	0·2748 (3)	0·5827 (3)	0·3023 (3)	2·1 (1)
C(10)	0·1792 (3)	0·6329 (3)	0·2150 (3)	2·3 (1)
C(11)	0·5410 (3)	0·7311 (4)	0·1707 (3)	3·5 (1)
C(12)	0·4141 (3)	0·5345 (3)	0·1100 (3)	3·3 (1)
C(13)	0·1984 (3)	0·3250 (4)	0·5405 (3)	4·0 (1)
C(14)	0·1372 (4)	0·2350 (4)	0·6037 (3)	4·4 (2)
C(15)	−0·1700 (4)	0·4744 (5)	0·1769 (4)	5·2 (2)

*$B_{iso} = \frac{4}{3}\sum_i\sum_j B_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

Atomic coordinates are listed in Table 2,* bond lengths and bond angles in Table 3. Shortest intermolecular distances: O(1)···C(12)($1-x$, $0{\cdot}5+y$, $0{\cdot}5-z$) = 3·322 (5), O(2)···C(11)($1-x$, $0{\cdot}5+y$, $0{\cdot}5-z$) = 3·360 (5) and O(3)···C(4)($-x$, $0{\cdot}5+y$, $0{\cdot}5-z$) = 3·150 (5) Å. Fig. 1 is a perspective drawing of the molecule illustrating the atom labeling.

* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51859 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Interatomic bond distances* (Å) *and angles* (°)

S—O(1)	1·441 (3)	C(2)—C(12)	1·526 (5)
S—O(2)	1·438 (3)	C(3)—C(4)	1·329 (6)
S—C(2)	1·800 (3)	C(4)—C(10)	1·453 (5)
S—C(9)	1·758 (4)	C(5)—C(6)	1·373 (5)
O(3)—C(6)	1·356 (5)	C(5)—C(10)	1·401 (5)
O(3)—C(15)	1·429 (5)	C(6)—C(7)	1·410 (5)
O(4)—C(7)	1·355 (4)	C(7)—C(8)	1·386 (5)
O(4)—C(13)	1·434 (5)	C(8)—C(9)	1·399 (5)
C(2)—C(3)	1·510 (5)	C(9)—C(10)	1·392 (5)
C(2)—C(11)	1·532 (5)	C(13)—C(14)	1·507 (6)

O(1)—S—O(2)	117·0 (2)	C(6)—C(5)—C(10)	121·1 (3)
O(1)—S—C(2)	109·0 (2)	O(3)—C(6)—C(5)	124·8 (3)
O(1)—S—C(9)	108·7 (2)	O(3)—C(6)—C(7)	114·6 (3)
O(2)—S—C(2)	109·9 (2)	C(5)—C(6)—C(7)	120·6 (3)
O(2)—S—C(9)	109·6 (2)	O(4)—C(7)—C(6)	115·6 (3)
C(2)—S—C(9)	101·6 (2)	O(4)—C(7)—C(8)	124·9 (3)
C(6)—O(3)—C(15)	117·7 (3)	C(6)—C(7)—C(8)	119·5 (3)
C(7)—O(4)—C(13)	117·9 (3)	C(7)—C(8)—C(9)	118·8 (3)
S—C(2)—C(3)	107·5 (2)	S—C(9)—C(8)	119·3 (3)
S—C(2)—C(11)	106·8 (2)	S—C(9)—C(10)	118·1 (3)
S—C(2)—C(12)	109·4 (2)	C(8)—C(9)—C(10)	122·5 (3)
C(3)—C(2)—C(11)	112·4 (3)	C(4)—C(10)—C(5)	121·4 (3)
C(3)—C(2)—C(12)	110·0 (3)	C(4)—C(10)—C(9)	121·2 (3)
C(11)—C(2)—C(12)	110·7 (3)	C(5)—C(10)—C(9)	117·4 (3)
C(2)—C(3)—C(4)	123·0 (3)	O(4)—C(13)—C(14)	106·7 (3)
C(3)—C(4)—C(10)	124·5 (3)		

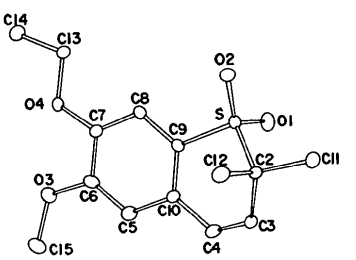


Fig. 1. Perspective view of the molecule showing the atom labeling.

Related literature. Structural data of several cyclic sulfones have been published by Yasuoka, Kasai, Tanaka, Nagai & Tokura (1972), Yasuoka, Kai & Kasai (1975), Towns & Simonsen (1975), Ealick, van der Helm, Ramalingan, Thyvelikakath & Berlin (1977), Ealick, van der Helm & Baker (1979), Desiraju & Kamala (1983) and Castellano, De Simone, Zukerman-Schpector, Ferreira & Catani (1989).

This work has received partial support from CNPq, CAPES, FAPESP and FINEP. One of us (JZS) thanks CNPq for the award of a research grant.

References

CASTELLANO, E. E., DE SIMONE, C. A., ZUKERMAN-SCHPECTOR, J., FERREIRA, J. T. B. & CATANI, V. (1989). *Acta Cryst.* C**45**, 959–961.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

DESIRAJU, G. R. & KAMALA, R. (1983). *Acta Cryst.* C**39**, 358–360.
EALICK, S. E., VAN DER HELM, D. & BAKER, J.R. (1979). *Acta Cryst.* B**35**, 495–497.
EALICK, S. E., VAN DER HELM, D., RAMALINGAN, K., THYVELIKAKATH, G. X. & BERLIN, K. D. (1977). *J. Heterocycl. Chem.* **14**, 387–390.
FERREIRA, J. T. B. & CATANI, V. (1987). Int. Congr. Heterocycl. Chem., Hamburg, Federal Republic of Germany.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
TOWNS, R. L. R. & SIMONSEN, S.H. (1975). *Cryst. Struct. Commun.* **4**, 469–472.
YASUOKA, N., KAI, Y. & KASAI, N. (1975). *Acta Cryst.* B**31**, 2729–2731.
YASUOKA, N., KASAI, N., TANAKA, M., NAGAI, T. & TOKURA, N. (1972). *Acta Cryst.* B**28**, 3393–3399.

Acta Cryst. (1989). C**45**, 1450–1451

Structure of *rac-D*-Homo-8α-estrone

BY W. L. DUAX, J. F. GRIFFIN AND P. D. STRONG

Medical Foundation of Buffalo, Inc., 73 High Street, Buffalo, NY 14203, *USA*

AND S. N. ANANCHENKO AND V. V. EGOROVA

Shemyakin Institute of Bioorganic Chemistry, USSR Academy of Sciences, Moscow, USSR

(*Received* 22 *December* 1988; *accepted* 28 *March* 1989)

Abstract. *rac-D*-Homo-3-hydroxy-8α-estra-1,3,5(10)-trien-17a-one, $C_{19}H_{24}O_2$, $M_r = 284{\cdot}4$, monoclinic, $P2_1/c$, $a = 7{\cdot}597$ (1), $b = 13{\cdot}814$ (1), $c = 14{\cdot}399$ (1) Å, $\beta = 92{\cdot}05°$, $V = 1510{\cdot}2$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}251$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}627$ mm^{-1}, $F(000) = 616$, $T = 298$ K, $R = 0{\cdot}045$ for 2413 reflections with $F^2 > 2{\cdot}0\sigma(F^2)$. The title compound is a synthetic estrogen whose anomalous activity required determination of the configuration at C(8) relative to the configuration at C(13) and the effect of configuration on the conformation of the compound.

Experimental. The title compound was synthesized by the methods reported in Sozokina, Barkova, Zakharychev, Chigir, Ananchenko & Torgov (1973). Crystallization from 90% ethanol, single crystal 0·24 × 0·28 × 0·32 mm. The unit-cell parameters were refined from least-squares analysis of 2θ values for 25 reflections with $50 < 2\theta < 54°$. Intensities for 2519 unique reflections having $2\theta < 114°$ ($-8 \le h \le 8$, $0 \le k \le 16$, $-16 \le l \le 16$) measured on a Syntex $P3$ diffractometer, using a θ–2θ scan mode, Ni-filtered Cu radiation, no monochromator, scan speed from 2·5 to 25° min^{-1} in 2θ, scan width (1·1 + 1·1tanθ)°. Four standard reflections (008, 080, 500, $\bar{3}44$) were measured every 100 reflections and varied in intensity by ≤ 5% during the data collection.

Direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least squares on F using the 2413 reflections for which $I > 2\sigma(I)$. The H-atom positions were located in a difference map and refined with assigned isotropic temperature parameters of the parent atom. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final $R =$

Table 1. *Fractional positional parameters* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10) *for non-*H *atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C(1)	−438 (2)	2872 (1)	9764 (1)	35 (1)
C(2)	−1843 (2)	2534 (1)	10260 (1)	37 (1)
C(3)	−3426 (2)	3036 (1)	10200 (1)	34 (1)
C(4)	−3556 (2)	3872 (1)	9680 (1)	33 (1)
C(5)	−2140 (2)	4217 (1)	9185 (1)	31 (1)
C(6)	−2316 (2)	5147 (1)	8647 (1)	38 (1)
C(7)	−1012 (2)	5231 (1)	7872 (1)	35 (1)
C(8)	849 (2)	5033 (1)	8272 (1)	28 (1)
C(9)	991 (2)	3994 (1)	8637 (1)	30 (1)
C(10)	−555 (2)	3699 (1)	9214 (1)	30 (1)
C(11)	1211 (2)	3269 (1)	7836 (1)	42 (1)
C(12)	2808 (3)	3514 (1)	7261 (1)	43 (1)
C(13)	2714 (2)	4536 (1)	6849 (1)	32 (1)
C(14)	2402 (2)	5273 (1)	7645 (1)	27 (1)
C(15)	2427 (2)	6316 (1)	7298 (1)	34 (1)
C(16)	4221 (2)	6558 (1)	6914 (1)	38 (1)
C(17)	4764 (3)	5836 (1)	6175 (1)	42 (1)
C(17a)	4476 (2)	4798 (1)	6435 (1)	35 (1)
C(18)	1357 (3)	4585 (2)	6039 (1)	46 (1)
O(3)	−4885 (2)	2735 (1)	10652 (1)	48 (1)
O(17a)	5597 (2)	4189 (1)	6287 (1)	46 (1)

0108-2701/89/091450-02$03.00