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The Structure of the Five-Membered Cyclic Sultone 9,10-Dihydro-10a-methyl-3a,9-methano-3*H*,10a*H*-benzo[5,6]cyclohept[1,2-*d*][1,2]oxathiol-4(3a*H*)-one 2,2-Dioxide, $C_{14}H_{14}O_4S$

BY D. S. BROWN, H. HEANEY AND K. G. MASON

*Loughborough University of Technology, Loughborough, Leics, LE*11 3*TU, England*

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Abstract. $M_r = 278{\cdot}2$, monoclinic, $P2_1/c$, $a = 11{\cdot}672$ (2), $b = 7{\cdot}717$ (1), $c = 14{\cdot}489$ (2) Å, $\beta = 101{\cdot}7$ (1)°, $U = 1277{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}446$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}250$ mm^{-1}, $F(000) = 584$, $T = 293$ K, $R = 0{\cdot}074$ for 1467 observed reflections. The structure is the first example of a saturated five-membered cyclic sultone. It is compared with known structures of partly unsaturated sultone rings. Bond lengths of interest are: mean acyclic S—O 1·417 (4), cyclic S—O 1·559 (3) and S—C 1·772 (7) Å.

Introduction. Our preliminary studies of the acid-catalysed reactions of alkyl-substituted homologues of tetrafluoro(1-methoxy)benzobarrelene (1) showed that trifluoroacetic acid gave products derived from simple rearrangements (Hales, Heaney & Ley, 1974). On the other hand, reactions carried out in sulphuric acid (98%) frequently gave products, derived from subsequent rearrangements, with more complex structures (Heaney & Ley, 1974; Hales & Heaney, 1975). The reactions of 1-methoxy-2,6-dimethylbenzobarrelene (2) and its tetrafluorobenzo analogue are such cases. The reactions of these compounds, but not the tetrachloro analogue, gave polar products, the structures of which could not be fully elucidated by a consideration of the available spectral data (Brown, Heaney, Ley, Mason & Singh, 1978). In particular, the stereochemistry of the sultone ring could not be deduced with certainty. It has been established that the ketones (3) and (4) also give the sultone in sulphuric acid and the fact that the tetrachloro compounds fail to react in an analogous manner suggests that a 1,2-acyl shift in the ketone (4) is a key step involved in the formation of the sultone (5).

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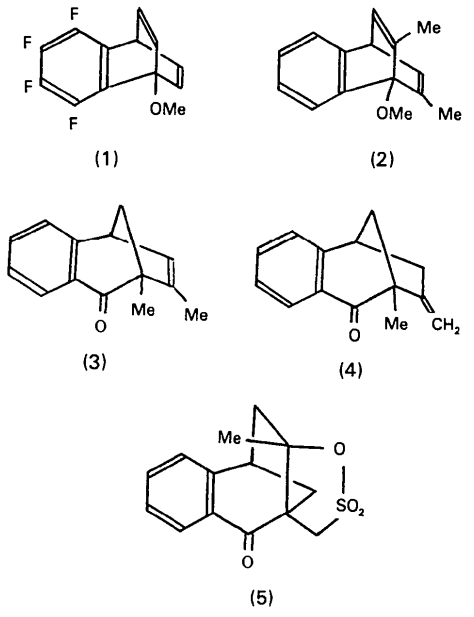

In order to confirm the molecular structure and stereochemistry of (5) the X-ray structure was determined.

Experimental. Acicular crystals grown from ethanol, crystal $0{\cdot}7 \times 0{\cdot}1 \times 0{\cdot}15$ mm; Hilger & Watts Y290 four-circle diffractometer, $\theta < 25°$; lattice parameters from least-squares fit of 13 reflections; 2416 reflections measured, 1467 with $I > 3\sigma(I)$; h 0–13, k 0–9, l −16–16; 1 standard reflection every 50 reflections, no significant change; no correction for absorption and extinction; structure solved by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971), refined by full-matrix least squares on F to $R = 0{\cdot}074$; non-hydrogen atoms anisotropic, hydrogen atoms in calculated positions, not refined; in final refinement cycles, $w = 14{\cdot}0/F^2$ for $F > 14$, otherwise $w = 1$; max. $\Delta/\sigma = 0{\cdot}008$, $\Delta\rho$ excursions $= \pm 0{\cdot}3$ e Å^{-3} (no structural significance); scattering factors for C, O and S from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); calculations carried out with *XRAY* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) implemented at the University of Manchester Regional Computer Centre.*

Discussion. Final atomic coordinates are listed in Table 1, and bond lengths and angles in Table 2. The molecular structure and atom numbering are shown in Fig. 1 and the unit-cell contents in Fig. 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39057 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters*

$B_{eq} = \frac{1}{3}\sum_i B_{ii}$.

	x	y	z	B_{eq}(Å^2)
C(1)	10936 (5)	8468 (9)	8274 (5)	6·4 (4)
C(2)	9809 (5)	8977 (9)	7869 (4)	6·0 (3)
C(3)	8911 (5)	8689 (7)	8335 (4)	4·6 (3)
C(4)	9153 (4)	7917 (6)	9224 (4)	4·2 (2)
C(5)	10297 (5)	7385 (8)	9628 (4)	5·3 (3)
C(6)	11187 (5)	7685 (8)	9140 (5)	5·8 (3)
C(7)	8198 (4)	7533 (7)	9743 (3)	4·2 (2)
C(8)	6960 (4)	8016 (6)	9195 (3)	3·8 (2)
C(9)	6590 (4)	6723 (6)	8359 (3)	3·6 (2)
C(10)	6941 (4)	7592 (7)	7509 (3)	4·5 (2)
C(11)	7653 (5)	9177 (7)	7910 (4)	4·7 (3)
C(12)	7035 (5)	9754 (7)	8693 (4)	4·6 (3)
C(13)	6979 (5)	4860 (7)	8544 (4)	5·0 (3)
C(14)	6074 (4)	7806 (7)	9825 (3)	4·5 (2)
S(1)	4746 (1)	7435 (2)	9009 (1)	4·2 (1)
O(1)	5287 (3)	6685 (5)	8194 (2)	4·9 (2)
O(2)	8359 (3)	6954 (6)	10522 (3)	5·4 (2)
O(3)	4037 (3)	6153 (6)	9310 (3)	6·2 (2)
O(4)	4167 (3)	9017 (5)	8708 (3)	6·0 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

		C(8)–C(9)	1·56 (1)
C(1)–C(2)	1·38 (1)	C(8)–C(14)	1·52 (1)
C(1)–C(6)	1·37 (1)	C(9)–C(10)	1·53 (1)
C(2)–C(3)	1·38 (1)	C(9)–C(13)	1·52 (1)
C(3)–C(4)	1·39 (1)	C(9)–O(1)	1·49 (1)
C(3)–C(11)	1·52 (1)	C(10)–C(11)	1·53 (1)
C(4)–C(5)	1·41 (1)	C(12)–C(11)	1·53 (1)
C(4)–C(7)	1·50 (1)	C(14)–S(1)	1·772 (7)
C(5)–C(6)	1·39 (1)	S(1)–O(1)	1·559 (3)
C(7)–C(8)	1·55 (1)	S(1)–O(3)	1·414 (6)
C(7)–O(2)	1·19 (1)	S(1)–O(4)	1·421 (7)
C(8)–C(12)	1·54 (1)		

C(2)–C(1)–C(6)	121·5 (8)	C(8)–C(9)–C(10)	105·5 (6)
C(1)–C(2)–C(3)	119·9 (9)	C(8)–C(9)–C(13)	116·3 (6)
C(2)–C(3)–C(4)	119·3 (8)	C(8)–C(9)–O(1)	104·4 (6)
C(3)–C(4)–C(5)	120·5 (8)	C(10)–C(9)–C(13)	116·1 (7)
C(4)–C(5)–C(6)	119·0 (8)	C(10)–C(9)–O(1)	108·1 (5)
C(5)–C(6)–C(1)	119·8 (7)	C(13)–C(9)–O(1)	105·6 (6)
C(4)–C(3)–C(11)	118·6 (8)	C(9)–C(10)–C(11)	105·1 (6)
C(2)–C(3)–C(11)	122·1 (8)	C(3)–C(11)–C(10)	111·2 (7)
C(3)–C(4)–C(7)	121·1 (7)	C(3)–C(11)–C(12)	109·5 (7)
C(5)–C(4)–C(7)	118·3 (7)	C(10)–C(11)–C(12)	102·3 (7)
C(4)–C(7)–C(8)	114·0 (6)	C(8)–C(12)–C(11)	100·2 (6)
C(4)–C(7)–O(2)	124·0 (7)	C(8)–C(14)–S(1)	103·1 (6)
C(8)–C(7)–O(2)	121·9 (8)	C(14)–S(1)–O(1)	97·6 (3)
C(7)–C(8)–C(9)	109·1 (6)	C(14)–S(1)–O(3)	113·2 (4)
C(7)–C(8)–C(12)	108·4 (6)	C(14)–S(1)–O(4)	111·3 (4)
C(7)–C(8)–C(14)	110·1 (6)	O(1)–S(1)–O(3)	109·0 (3)
C(9)–C(8)–C(12)	102·9 (6)	O(1)–S(1)–O(4)	109·1 (3)
C(9)–C(8)–C(14)	106·4 (6)	O(3)–S(1)–O(4)	115·1 (4)
C(12)–C(8)–C(14)	119·2 (6)	C(9)–O(1)–S(1)	115·4 (3)

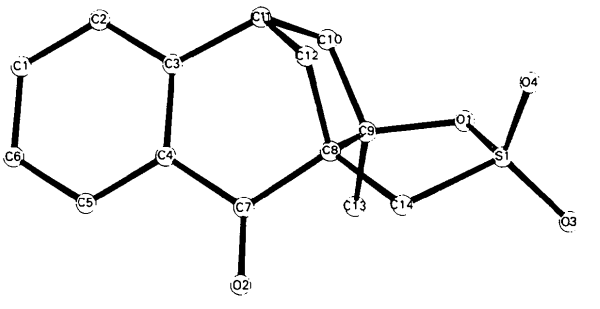

Fig. 1. *ORTEP* plot (Johnson, 1965) of the title compound with atom labelling.

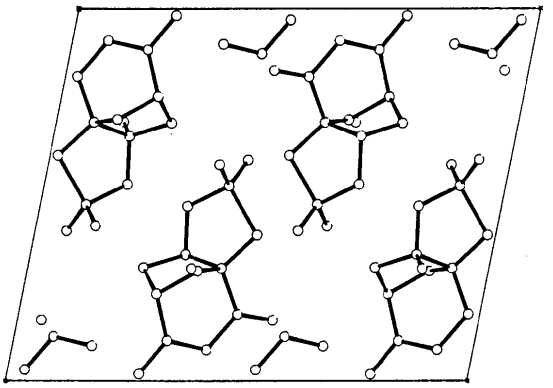

Fig. 2. *b*-axial projection of the unit-cell contents.

The structure determination confirms the molecular structure shown for (5) and provides the first example of a saturated cyclic sultone, although there are two examples where partly unsaturated sultone rings have been reported (Barnett, Newton & McCormack, 1972; Beetz, Kellogg, Kiers & Piepenbroek, 1975). The mean acyclic S—O bond is 1·417 (4) Å which compares with values of 1·43 and 1·41 Å quoted by Beetz *et al.* The cyclic S—O bond length of 1·559 (3) Å is slightly shorter than the values found by Barnett *et al.* and by Beetz *et al.*, 1·577 and 1·59 Å, respectively. The S—C bond length of 1·772 (7) Å compares with that of 1·727 Å reported by Barnett *et al.* There were no abnormal intermolecular contact distances.

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4-Hydroxy-4-phenylpentanamide, $C_{11}H_{15}NO_2$, a Moderately Active Anticonvulsant

BY J. ZUKERMAN-SCHPECTOR, E. E. CASTELLANO AND G. OLIVA

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

AND G. CARVAJAL

Instituto Politécnico Nacional, Escuela Nacional de Ciencias Biológicas, Departamento de Bioquímica, México 17, *DF, Mexico*

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Abstract. $M_r = 193{\cdot}25$, monoclinic, $P2_1/n$, $a = 24{\cdot}215$ (4), $b = 6{\cdot}981$ (2), $c = 6{\cdot}147$ (1) Å, $\beta = 91{\cdot}7$ (1)°, $V = 1038{\cdot}7$ (6) Å^3, $Z = 4$, $D_m = 1{\cdot}23$, $D_x = 1{\cdot}24$ Mg m^{-3}, graphite-monochromatized Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}61$ mm^{-1}, $F(000) = 416$, $T = 296$ K, $R = 0{\cdot}058$, $R_w = 0{\cdot}053$ for 1091 observed reflections ($I > 3\sigma$) and 187 refined parameters. The molecule adopts a pseudocyclic conformation through hydroxyl···amide oxygen intramolecular hydrogen bonding. The distance of 6·10 (1) Å between the centroid of the phenyl ring and the electron-donating oxygen atom is in the range found for other anticonvulsants.

Introduction. The title compound [also known as γ-hydroxy-γ-methyl-γ-phenylbutyramide (HMPB)] has long been known for its anticonvulsant activity. It produces a reduction in the duration of convulsions on

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