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(5*Z*,14*Z*)-1,10-Dioxa-4,7,13,15-tetrathiaoctadeca-5,14-diene

BY JOHN C. BARNES* AND JOHN D. PATON

Chemistry Department, The University, Dundee DD1 4HN, Scotland

C. KEITH PROUT AND JAMES C. G. TURNER

Chemical Crystallography Laboratory, Parks Road, Oxford, England

AND W. SCHROTH

Chemistry Department, Martin Luther Universität, Halle, 4020 *German Democratic Republic*

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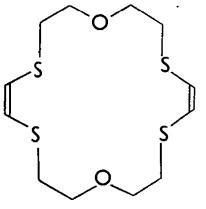
Abstract. $C_{12}H_{20}O_2S_4$, $M_r = 324{\cdot}52$, monoclinic, $P2_1/a$, $a = 9{\cdot}106$ (2), $b = 10{\cdot}033$ (1), $c = 17{\cdot}307$ (3) Å, $\beta = 97{\cdot}51$ (2)°, $V = 1567{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}38$ Mg m^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}5418$ Å, $\mu = 53{\cdot}2$ cm^{-1}, $F(000) = 688$, $T = 293$ K, $R = 0{\cdot}034$ for 2460 reflexions ($F_o > 2\sigma_F$). The conformation at each C—S—C=C group is *anti* whereas each C—S—C—C group is *gauche*. Each of the O—C—C—S groups is *gauche*. There is not even approximate symmetry within the molecule in any direction. The angle between the normals to the S—C=C—S mean planes is 81·4 (2)°.

Experimental. The preparation and characterization of the title compound (I) will be reported separately. After preliminary photographs, unit-cell dimensions were refined from 25 accurately centred reflections with $\theta \simeq 30°$ using an Enraf–Nonius CAD-4F diffractometer.

Data were collected on one quadrant of a colourless crystal $0{\cdot}3 \times 0{\cdot}5 \times 0{\cdot}1$ mm for $\theta \leq 75°$. 4492 measured reflexions gave 3212 unique reflexions (R_{int} 0·051) of which 2460 with $F > 2\sigma(F)$ were used in the refinement. Ranges of indices $-11 \leq h \leq 11$, $0 \leq k \leq 12$, $0 \leq l \leq 21$. The intensities of two standard reflexions were checked every hour and the orientation of the crystal was verified every 200 reflexions. Data were corrected for the Lorentz and polarization terms and for absorption using a ψ-scan routine. Correction factors ranged from 1·00 to 1·48.

The structure was solved using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement was carried out using *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *SHELX*76. In the final refinement in two blocks all non-hydrogen atoms had



(I)

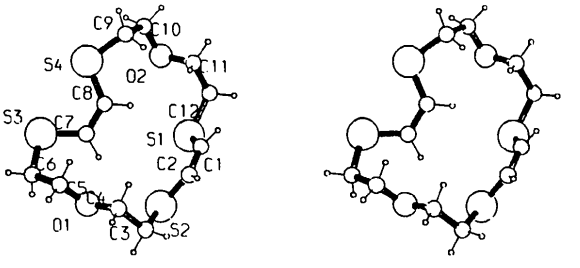


Fig. 1. Stereoview of the title compound normal to the plane S1, S3, S4, showing atomic numbering.

* To whom correspondence should be addressed.

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Table 1. *Coordinates* ($\times 10^5$) *for non-hydrogen atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}(Å^2 × 10^3)
S1	15917 (6)	39157 (5)	59200 (3)	46 (1)
S2	16401 (6)	15594 (5)	72629 (3)	49 (1)
S3	29144 (11)	55482 (7)	92690 (4)	78 (1)
S4	44491 (7)	70552 (6)	79310 (3)	58 (1)
O1	29046 (18)	21700 (17)	90151 (9)	55 (1)
O2	31311 (16)	68217 (14)	60266 (9)	48 (1)
C1	29947 (24)	27430 (22)	61307 (13)	49 (1)
C2	30277 (25)	18124 (21)	66782 (13)	49 (1)
C3	25837 (30)	5341 (21)	80252 (14)	56 (1)
C4	36887 (28)	12583 (24)	86036 (15)	58 (1)
C5	38539 (28)	29225 (26)	95805 (13)	59 (1)
C6	29682 (32)	40521 (31)	98489 (14)	69 (1)
C7	28125 (28)	49542 (24)	83134 (14)	57 (1)
C8	34239 (27)	55830 (21)	77694 (12)	52 (1)
C9	52689 (25)	71971 (25)	70404 (16)	58 (1)
C10	42668 (27)	77239 (22)	63421 (15)	55 (1)
C11	36588 (24)	58178 (22)	55622 (13)	48 (1)
C12	23872 (25)	49435 (23)	52241 (12)	49 (1)

Table 2. *Interatomic distances* (Å) *and angles* (°)

C1–S1	1·740 (2)	C12–S1–C1	100·3 (1)
C12–S1	1·807 (2)	C3–S2–C2	101·3 (1)
C2–S2	1·738 (2)	C7–S3–C6	103·7 (1)
C3–S2	1·800 (2)	C9–S4–C8	101·6 (1)
C6–S3	1·803 (3)	C5–O1–C4	112·8 (2)
C7–S3	1·749 (2)	C11–O2–C10	112·7 (2)
C8–S4	1·750 (2)	C2–C1–S1	124·9 (2)
C9–S4	1·804 (3)	C1–C2–S2	124·6 (2)
C4–O1	1·410 (3)	C4–C3–S2	115·1 (2)
C5–O1	1·433 (3)	C3–C4–O1	108·0 (2)
C10–O2	1·428 (3)	C6–C5–O1	108·0 (2)
C11–O2	1·411 (3)	C5–C6–S3	115·7 (2)
C2–C1	1·328 (3)	C8–C7–S3	122·6 (2)
C4–C3	1·510 (3)	C7–C8–S4	123·9 (2)
C6–C5	1·499 (4)	C10–C9–S4	115·9 (2)
C8–C7	1·316 (3)	C9–C10–O2	114·5 (2)
C10–C9	1·511 (3)	C12–C11–O2	109·6 (2)
C12–C11	1·509 (3)	C11–C12–S1	115·3 (1)

Table 3. *Torsion angles* (°)

C12–S1–C1–C2	173·18	C4–O1–C5–C6	167·42
C1–S1–C12–C11	−72·36	C11–O2–C10–C9	−78·33
C3–S2–C2–C1	−164·41	C10–O2–C11–C12	−177·63
C2–S2–C3–C4	74·33	S1–C1–C2–S2	2·34
C7–S3–C6–C5	38·10	S2–C3–C4–O1	65·54
C6–S3–C7–C8	−147·01	O1–C5–C6–S3	−86·96
C9–S4–C8–C7	168·78	S3–C7–C8–S4	1·72
C8–S4–C9–C10	78·58	S4–C9–C10–O2	−71·13
C5–O1–C4–C3	180·00	O2–C11–C12–S1	−68·57

anisotropic temperature parameters, H atoms located on difference syntheses were refined with isotropic temperature parameters. A correction for secondary extinction (*SHELX*76) was applied. There is no evidence of disorder or high thermal motion. Final refinement (minimizing $\sum w|F_o - |F_c|^2$) 246 refined parameters, $R = 0{\cdot}034$, $wR = 0{\cdot}062$, $w = 0{\cdot}0025/[\sigma^2(F) + 0{\cdot}427544\,F^2]$, mean shift/e.s.d. = 0·185, max. shift/e.s.d. = 0·496, max. diff. peak = 0·284, max. negative peak = −0·361 e Å^{-3}.

Final atomic coordinates are given in Table 1, with bond lengths and angles in Table 2 and torsion angles in Table 3.* The molecule is shown in Fig. 1.

Related literature. The S atoms in crown thioethers may all point out of the crown ring as in tetrathia-12-crown-4 (Dalley, Larson, Smith, Matheson, Izatt & Christensen, 1981) or some inwards and others outwards as in hexathia-18-crown-6 (Hartman, Wolf, Foxman & Cooper, 1983) but in each of these saturated rings all C–S–C–C groups have *gauche* conformations. In the present molecule the additional constraints imposed by the C=C units result in *anti* conformations at C–S–C=C.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44695 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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