

molécules de *m*-chlorophényl-urée par l'intermédiaire des quatre ponts hydrogène et cela au niveau des atomes d'oxygène [O(1) O(4)] et [O(1') O(4')]. Les deux unités



des molécules de type *guest* forment un raccord entre des atomes d'oxygène adjacents, N(17)···O(4) = 2,94 (1) et N(20)···O(1) = 3,27 (2) Å. Les distances H(17)···O(4) = 2,11 (1) et H(20A)···O(1) = 2,49 (1) Å de même les angles O(4)···H(17) = 167,2 (1) et O(1)···H(20A)—N(20) = 144,8 (1)° confirment les liaisons hydrogène avec l'éther avec l'éther-couronne (Fig. 2). Une liaison hydrogène H(20B)···O(7') est exclue vu les paramètres défavorables O(7')···H(20B) = 2,80 (2) Å et O(7')···H(20B)—N(20) = 105,0 (2)°, et un lien 'bifide' H(20A) vers O(7') est improbable pour les mêmes raisons et de plus l'oxygène O(7') se trouve dans un autre plan que O(1) et O(4). Par ailleurs, les atomes d'oxygène adjacents sont trop proches pour former deux liaisons hydrogène avec un groupe NH₂.

Les deux molécules de *m*-chlorophényl-urée sont accrochées au 18-crown-6 de part et d'autre d'un plan moyen formé par les atomes d'oxygène O(7) et O(7'). Une coordination qui engage deux atomes d'oxygène adjacents dans le macrocycle semble favoriser la conformation biangulaire. L'élément de structure NH—C—NH



favorise certainement cette conformation.

Une particularité de cette structure par rapport aux autres complexes de l'urée et de la thiourée est une véritable association 1:2 de l'éther-couronne et du dérivé de l'urée. La conformation biangulaire est exploitée entièrement vu les quatre liaisons hydrogène

vers des atomes d'oxygène adjacents qui par paire se trouvent de part et d'autre d'un même plan.

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2-Ethylidene-1,3-dithiane 1,3-Dioxide*

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Abstract. C₆H₁₀O₂S₂, *M_r* = 178.3, monoclinic, *P*2₁/*a*, *a* = 9.9607 (4), *b* = 6.8842 (3), *c* = 11.6445 (5) Å, β = 100.952 (3)°, *V* = 783.94 Å³, *Z* = 4, *D_x* = 1.51 Mg m⁻³, λ(Mo Kα₁) = 0.70930 Å, μ =

0.59 mm⁻¹, *F*(000) = 375.97, *T* = 298 K, *R* = 0.025 for 919 observed terms. The molecule has a chair conformation with one S=O bond axial and the other equatorial. The S=O bond lengths are similar to those in dimethyl sulfoxide and dimethyl sulfone, and all other bond lengths are normal. There are no short intermolecular contacts.

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Table 1. Final atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses

	x	y	z	B_{eq}^* (\AA^2)
S1	0.48464 (6)	0.05987 (9)	0.72748 (5)	2.58 (3)
S2	0.68913 (7)	-0.25182 (10)	0.68804 (5)	2.71 (3)
O1	0.43715 (17)	0.1499 (3)	0.82932 (14)	3.65 (8)
O2	0.58886 (17)	-0.29435 (24)	0.57806 (13)	3.62 (8)
C1	0.59497 (23)	-0.1370 (3)	0.78494 (19)	2.06 (10)
C2	0.7850 (3)	-0.0413 (4)	0.65967 (25)	2.80 (13)
C3	0.6947 (3)	0.1249 (4)	0.60727 (23)	2.88 (13)
C4	0.6138 (3)	0.2173 (4)	0.69007 (24)	2.77 (13)
C5	0.5940 (3)	-0.2033 (4)	0.89161 (22)	2.91 (13)
C6	0.6730 (5)	-0.3724 (6)	0.9502 (3)	4.63 (19)
H21	0.8419 (23)	-0.007 (3)	0.7326 (20)	3.3 (6)
H22	0.8462 (23)	-0.087 (3)	0.6099 (20)	3.4 (6)
H31	0.6337 (23)	0.080 (3)	0.5384 (20)	3.1 (6)
H32	0.7497 (24)	0.217 (3)	0.5861 (19)	3.2 (6)
H41	0.5617 (21)	0.330 (3)	0.6559 (17)	2.5 (5)
H42	0.6691 (20)	0.260 (3)	0.7599 (17)	2.2 (5)
H51	0.5377 (23)	-0.138 (3)	0.9383 (19)	3.3 (6)
H61	0.733 (3)	-0.439 (4)	0.9068 (24)	5.7 (8)
H62	0.715 (3)	-0.343 (5)	1.018 (3)	8.4 (12)
H63	0.616 (4)	-0.470 (5)	0.967 (3)	9.9 (14)

* B_{eq} is the mean of the principal axes of the thermal ellipsoid.

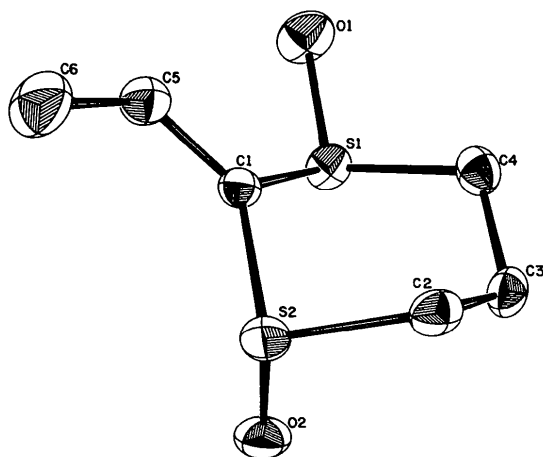


Fig. 1. Perspective view of the molecule with ellipsoids scaled to 50% probability.

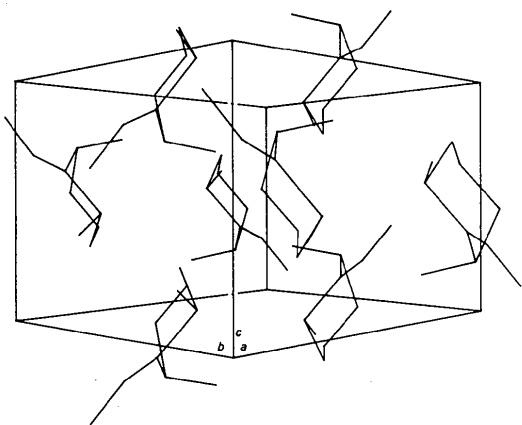


Fig. 2. The crystal packing.

Table 2. Selected bond lengths (\AA) and bond angles ($^\circ$) with e.s.d.'s in parentheses

S1—O1	1.4925 (17)	S2—C2	1.800 (3)
S1—C1	1.7924 (24)	C1—C5	1.325 (3)
S1—C4	1.798 (3)	C2—C3	1.511 (4)
S2—O2	1.4957 (16)	C3—C4	1.509 (4)
S2—C1	1.7832 (24)	C5—C6	1.496 (4)
O1—S1—C1	106.37 (10)	S1—C1—C5	119.81 (20)
O1—S1—C4	106.94 (12)	S2—C1—C5	122.62 (20)
C1—S1—C4	97.47 (12)	S2—C2—C3	112.82 (19)
O2—S2—C1	106.42 (10)	C2—C3—C4	114.23 (23)
O2—S2—C2	106.96 (12)	S1—C4—C3	113.19 (19)
C1—S2—C2	96.91 (12)	C1—C5—C6	126.8 (3)
S1—C1—S2	117.25 (13)		

Introduction. 1,3-Dithianes (ketene dithioacetals) are well known synthetic intermediates in organic chemistry (Groebel & Seebach, 1977), whereas the corresponding dioxides have received relatively little attention. The title compound was synthesized as a potential synthon and its structure determined as a result of its unexpected properties.

Experimental. The title compound was synthesized from the corresponding dithiane (Ziegler & Chan, 1978) as follows. Sodium periodate (5.39 g, 25.2 mmol, in 40 ml water) was added dropwise with stirring to 2-ethylidene-1,3-dithiane [1.22 g, 8.4 mmol, in 30 ml tetrahydrofuran (THF)]. A white precipitate was formed in a mildly exothermic reaction. The suspension was stirred for 12 h. The reaction mixture was extracted with dichloromethane (5 \times 25 ml), the combined extracts were dried (MgSO_4), filtered, and concentrated under vacuum to give a yellow oily solid. The latter was washed with THF and recrystallized from a mixture of THF and dichloromethane to give the dioxide, 0.98 g (65%), m.p. 437–438.6 K, ^1H NMR (90 MHz, CDCl_3 solution) δ 2.16 (*d*, 3H, $J = 3.5$ Hz, $\text{CH}_3\text{—CH}$), 2.2–2.9 (*m*, 4H), 3.06 (*m*, 1H, H—CHSO), 3.13 (*m*, 1H, H—CHSO), 6.76 (*q*, 1H, $J = 3.5$ Hz, $\text{CH}_3\text{—CH=}$). A crystal *ca* 0.30 \times 0.30 \times 0.30 mm was aligned on an Enraf–Nonius CAD-4 diffractometer; crystals shown to be monoclinic, space group $P2_1/a$ by systematic absences; cell parameters determined by least squares from 160 reflections ($40.00 < 2\theta < 45.00^\circ$) with Mo $K\alpha$ radiation (graphite-crystal monochromator). Integrated intensities by $\theta/2\theta$ scan mode of 1004 non-equivalent terms, measured to $2\theta_{\text{max}} = 45^\circ$ ($h-10-10$, $k 0-6$, $l 0-12$), 919 for which $I_{\text{net}} \geq 2.5\sigma I_{\text{net}}$ considered observed and used for structure refinement; reference reflections monitored during the data collection showed no decrease in intensity; intensities corrected for Lorentz and polarization effects, but not for absorption.

Structure solved by direct and Fourier methods. Least-squares refinements on F with anisotropic factors given to non-H atoms converged to $R = 0.025$, $wR = 0.022$, $S = 5.120$; weights based on counting

statistics, $w = 1/\sigma^2(F_o)$; Δ/σ (max.) = 0.101; largest peaks on final difference map +0.210 and -0.200 e Å⁻³. All calculations were made with the NRC VAX *Crystal Structure System* (Gabe, Lee & Le Page, 1985). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Final atomic coordinates are given in Table 1;* Fig. 1, which contains the atomic numbering, has been prepared from the output of *ORTEPII* (Johnson, 1976), and Fig. 2 from the output of *PACKER*, part of the NRC VAX *Crystal Structure System* (Gabe, Lee & Le Page, 1985). Table 2 contains selected bond lengths and bond angles. The molecule has the chair conformation with one S=O bond axial and the other equatorial. The S=O bond lengths are similar to those in dimethyl sulfoxide [1.47 (4) Å] and dimethyl sulfone

* Lists of structure amplitudes and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44436 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[1.44 (3) Å] (Allen & Sutton, 1950) and there are no unusual bond lengths involving carbon. It appears that this is the first determination of the crystal structure of a cyclic dithiane dioxide.

The crystal packing is shown in Fig. 2. All intermolecular contacts are normal.

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Low- and High-Temperature Structure of (*E*)-2,2'-Dimethylstilbene

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Abstract. C₁₆H₁₆, $M_r = 208.29$, monoclinic, $P2_1/n$, $Z = 2$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $F(000) = 224$. At 296 K: $a = 10.886$ (2), $b = 6.698$ (2), $c = 8.683$ (1) Å, $\beta = 98.53$ (1)°, $V = 626.1$ (2) Å³, $D_x = 1.105$ Mg m⁻³, $\mu = 0.0577$ mm⁻¹, $R = 0.047$ for 892 independent

reflections. At 118 K: $a = 10.784$ (2), $b = 6.622$ (1), $c = 8.565$ (1) Å, $\beta = 97.78$ (1)°, $V = 606.0$ (2) Å³, $D_x = 1.142$ Mg m⁻³, $\mu = 0.0596$ mm⁻¹, $R = 0.045$ for 1319 independent reflections. The molecule is centrosymmetric in the crystal. The length of the ethylenic