distance of C(14) with respect to the plane formed by C(13), C(15), C(16) and C(17) is 0.604 (4) Å. The atoms C(13), C(16), C(17) and C(20) lie in the same plane. O(2) is displaced 0.230 (2) Å from this plane and the pyridine ring plane is almost perpendicular to it [at 81.0 (1)°]. The torsion angles (Table 2) around C(20) define a (20*S*) isomer of the steroid molecule (Fig. 1*b*). The twist along the length of the steroid molecule is negligible [C(19)—C(10)···C(13)—C(18) = 1.8 (4)°].

The packing arrangement (Fig. 2) shows screw-axis-related steroid molecules with head-to-tail hydrogen bonds through O(2)—H(2)···O(1). The columns thus formed, parallel to **b**, are hydrophobic. At the same time O(1) and O*W* are linked through hydrogen bonds forming a hydrophilic column up the *b* axis.

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Acta Cryst. (1992). C**48**, 501–503

Structure of the Head-to-Tail *cis-anti-cis* Photodimer of 2-Methylbenzo[*b*]thiophene 1,1-Dioxide

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(*Received* 18 *April* 1991; *accepted* 26 *July* 1991)

Abstract. *r*-1,*c*-2,*t*-3,*t*-4-Di(2′,3′-benzothiophene 1,1-dioxide)-1,3-dimethylcyclobutane (alternative name: 5a,10a-dimethyldibenzo[*d*:*d*′]cyclobuta[1,2-*b*:4,3-*b*′]-dithiophene 5,5,10,10-tetraoxide), $C_{18}H_{16}O_4S_2$, M_r = 360.4, monoclinic, $P2_1/a$, $a = 6.918$ (1), $b = 12.270$ (1), $c = 9.283$ (1) Å, $\beta = 104.13$ (2)°, $V = 764.1$ (2) Å^3, $Z = 2$, $D_x = 1.56$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 3.29$ mm^{-1}, $F(000) = 376$, $T = 293$ K, $R = 0.051$ for 1357 observed reflections. The molecular structure shows no significant differences in bond lengths and bond angles between the two benzo[*b*]thiophene groups. A crystallographic centre of symmetry coincides with the cyclobutane ring and this dimer possesses a *cis-anti-cis* structure.

Introduction. Photodimerization of 2-methylbenzo[*b*]thiophene 1,1-dioxide in benzene solution was first reported by Hopkinson, Schloman, Plummer, Wenkert & Raju (1979) and El Amoudi El Faghi, Geneste & Olivé (1981). Two photodimers were identified as being of the cyclobutane type and a specific stereochemistry, head-to-head and head-to-tail *cis-anti-cis*, was assigned to each dimer on the basis of ^{1}H and ^{13}C NMR. The first X-ray structure of a head-to-tail dimer, the *cis-syn-cis* isomer, was reported by El Amoudi El Faghi, Geneste, Olivé, Rambaud & Declercq (1987). This photodimer was obtained from irradiation of 2-methylbenzo[*b*]thiophene 1,1-dioxide in the solid state. In this article we report the X-ray structure of the head-to-tail dimer, *cis-anti-cis*, obtained by photodimerization of the same reactant in solution (Hopkinson *et al.*, 1979; El Amoudi El Faghi *et al.*, 1981).

Experimental. Colourless prismatic shaped crystals from chloroform at room temperature. Lattice

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parameters from 25 reflections in the range $10 < 2\theta < 38°$. Diffraction data measured on a Huber 424 + 511 diffractometer, using ω–2θ scans, graphite-monochromatized Cu $K\alpha$ radiation collected to $2\theta_{\max} = 135°$; ranges of *hkl*: −8 to 7, 0 to 14, 0 to 11 respectively. Standard reflection (040) checked every 50 reflections showed no significant deviation. No absorption correction was applied. 1385 independent reflections with $(\sin\theta)/\lambda \le 0.599$ Å^{-1}. 1357 were observed with $I \ge 2.5\sigma(I)$. The structure was solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement was carried out by full-matrix least squares with *SHELX*76 (Sheldrick, 1976) using F magnitudes. All H atoms located by a difference Fourier map and refined with isotropic thermal parameters. Non-H atoms of the half molecule were refined anisotropically. Weights $w = 1/[\sigma^2(F) + 0.01026F^2]$. Convergence reached at $R = 0.051$ and $wR = 0.065$, $S = 0.96$ for 1357 observed reflections. Maximum $\Delta/\sigma = 0.184$. Largest minimum and maximum $\Delta\rho$ on a difference Fourier map were −0.58 and 0.38 e Å^{-3}. 135 parameters were refined. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final atomic parameters are given in Table 1, bond lengths and angles in Table 2.*

Table 1. *Atomic coordinates* (× 10^5) *and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = 8/3\pi^2\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
S(1)	75316 (6)	8137 (3)	87436 (4)	2.06
O(1)	87516 (21)	17757 (13)	90068 (18)	3.15
O(2)	84720 (21)	−1916 (13)	84644 (15)	2.87
C(2)	61618 (28)	6329 (14)	101790 (18)	1.93
C(3)	39211 (25)	5876 (15)	93490 (19)	1.87
C(4)	18777 (32)	8848 (15)	66488 (25)	2.58
C(5)	18923 (34)	11455 (20)	52166 (21)	2.95
C(6)	36451 (32)	13613 (17)	47968 (22)	2.95
C(7)	54387 (31)	12962 (14)	58484 (23)	2.61
C(3a)	36674 (36)	8296 (12)	77186 (25)	2.23
C(7a)	53953 (28)	10163 (16)	72926 (21)	2.13
C(21)	68117 (35)	14894 (16)	113919 (23)	2.62

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O(1)—S(1)	1.437 (1)	C(3a)—C(3)	1.510 (3)
O(2)—S(1)	1.447 (2)	C(5)—C(4)	1.370 (3)
C(2)—S(1)	1.827 (2)	C(3a)—C(4)	1.387 (3)
C(7a)—S(1)	1.757 (2)	C(6)—C(5)	1.387 (3)
C(3)—C(2)	1.554 (2)	C(7)—C(6)	1.381 (3)
C(2)—C(3′)	1.566 (2)	C(7a)—C(7)	1.391 (3)
C(21)—C(2)	1.526 (3)	C(7a)—C(3a)	1.367 (3)

O(2)—S(1)—O(1)	117.4 (1)	C(3a)—C(3)—C(2)	110.3 (2)
C(2)—S(1)—O(1)	111.3 (1)	C(3a)—C(4)—C(5)	119.3 (2)
C(2)—S(1)—O(2)	111.3 (1)	C(6)—C(5)—C(4)	122.1 (2)
C(7a)—S(1)—O(1)	111.3 (1)	C(7)—C(6)—C(5)	119.1 (2)
C(7a)—S(1)—O(2)	108.3 (1)	C(7a)—C(7)—C(6)	117.9 (2)
C(7a)—S(1)—C(2)	95.0 (1)	C(4)—C(3a)—C(3)	126.3 (2)
C(3)—C(2)—S(1)	105.9 (1)	C(7a)—C(3a)—C(3)	115.3 (2)
C(21)—C(2)—S(1)	110.0 (1)	C(7a)—C(3a)—C(4)	118.4 (2)
C(21)—C(2)—C(3)	118.0 (2)	C(7)—C(7a)—S(1)	124.2 (1)
C(3)—C(2)—C(3′)	90.3 (2)	C(3a)—C(7a)—S(1)	112.7 (2)
C(2)—C(3)—C(2′)	89.7 (2)	C(3a)—C(7a)—C(7)	123.1 (2)

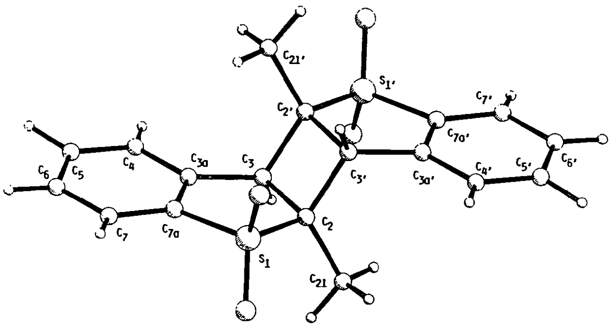

Fig. 1. View of the htt *cis-anti-cis* dimer of 2-methylbenzo[*b*]thiophene 1,1-dioxide showing the numbering of atoms (*PLUTO*; Motherwell & Clegg, 1978).

Discussion. The bond distances for the benzene ring in the benzo[*b*]thiophene groups show a shortening of the C(3a)—C(7a) bond [1.367 (3) Å] compared with the same bond in the 2- or 3-methylbenzo[*b*]thiophene 1,1-dioxide [1.388 (3) and 1.395 (3) Å respectively] (El Amoudi El Faghi, Geneste, Olivé, Rambaud & Declercq, 1988) and another significantly longer bond C(2)—C(3) [1.554 (2) Å compared with 1.319 (3) and 1.321 (4) Å respectively] owing to the dimerization. A comparable result was observed with the head-to-tail *cis-syn-cis* dimer (El Amoudi El Faghi *et al.*, 1987). The cyclobutane ring shows simple C—C bonds, but compared with the head-to-tail *cis-syn-cis* dimer, the values of its angles are significantly different, 89.7 (1) and 90.2 (1)° instead of 87.3 (2) and 88.7 (2)°.

As seen in Fig. 1, the sulfone groups are on the opposite side of the molecule and one benzothiophene moiety is bonded up while the other is bonded down with respect to the cyclobutane plane. The conformation is that of a chair and the molecule shows a crystallographic centre of symmetry which constrains the cyclobutane ring to be planar. Least-squares-planes calculations (*XANADU*; Roberts & Sheldrick, 1975) give a dihedral angle of 120 (0.5)° between the benzothiophene group and cyclobutane ring. As in the other benzo[*b*]thiophene 1,1-dioxides the crystal contains many van der Waals contacts but no hydrogen bonds. Thus, the *r-ctt* htt dimer according to the *Rules for the Nomenclature of*

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54476 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0473]

Organic Chemistry, Section E (IUPAC, 1976) (*i.e.* head-to-tail *cis-anti-cis* dimer) possesses a '*Z* structure' compared to the '*U* structure' of the htt *cis-syn-cis* dimer (El Amoudi El Faghi *et al.*, 1987).

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Acta Cryst. (1992). C**48**, 503–507

Structures of 1:1 Addition Compounds of Methyl Cholate with Methanol and with 2-Propanol

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(*Received* 7 *June* 1991; *accepted* 30 *July* 1991)

Abstract. (1): Methyl cholate–methanol (1/1), $C_{25}H_{42}O_5.CH_4O$, $M_r = 454.65$, monoclinic, $C2$, $a = 25.184\,(3)$, $b = 7.797\,(1)$, $c = 15.174\,(3)$ Å, $\beta = 121.05\,(1)^\circ$, $U = 2552.8\,(7)$ Å^3, $Z = 4$, $D_x = 1.183$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.5418$ Å, $\mu = 6.23$ cm^{-1}, $F(000) = 1000$, $T = 295$ K, $R = 0.033$ for 1844 observed reflections. (2): Methyl cholate–2-propanol (1/1), $C_{25}H_{42}O_5.C_3H_8O$, $M_r = 482.70$, monoclinic, $C2$, $a = 25.835\,(2)$, $b = 8.137\,(1)$, $c = 15.553\,(2)$ Å, $\beta = 121.08\,(1)^\circ$, $U = 2800.3\,(3)$ Å^3, $Z = 4$, $D_x = 1.145$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.5418$ Å, $\mu = 5.92$ cm^{-1}, $F(000) = 1064$, $T = 295$ K, $R = 0.067$ for 1944 observed reflections. The molar ratio between methyl cholate (MC) and the guest molecules (methanol and 2-propanol) is 1:1. The crystal structures are isomorphous. The MC molecules form layered arrangements, however, which provide no hydrophobic channel spaces. Alcohol molecules are accommodated by the host steroidal molecules with hydrogen bonding.

Introduction. It is known that the typical steroidal bile acid, deoxycholic acid (3α,12α-dihydroxy-5β-cholan-24-oic acid; DCA) accommodates a variety of

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0108-2701/92/030503-05$03.00