O(8)	0.7277 (3)	0.2193 (1)	−0.1951 (1)	0.0436 (5)
O(211)	0.2768 (4)	0.0828 (2)	0.1458 (1)	0.0501 (6)
O(212)	0.5668 (4)	0.0541 (1)	0.2392 (1)	0.0564 (6)
N(1)	0.4093 (3)	−0.0817 (1)	0.0454 (1)	0.0282 (5)
N(2)	0.5754 (3)	0.0567 (1)	−0.1536 (1)	0.0302 (5)
C(1)	0.3175 (6)	−0.1675 (2)	0.1798 (2)	0.0503 (9)
C(2)	0.4817 (4)	−0.0846 (2)	0.1356 (1)	0.0299 (6)
C(3)	0.1880 (5)	−0.1398 (2)	0.0260 (2)	0.0389 (7)
C(4)	0.5214 (4)	−0.0117 (2)	−0.0075 (1)	0.0270 (6)
C(5)	0.4026 (4)	0.0116 (2)	−0.0933 (1)	0.0282 (6)
C(6)	0.2270 (4)	0.1084 (2)	−0.0818 (2)	0.0362 (6)
C(7)	0.3859 (5)	0.2081 (2)	−0.0970 (2)	0.0375 (7)
C(8)	0.5825 (4)	0.1651 (2)	−0.1535 (1)	0.0310 (6)
C(21)	0.4505 (4)	0.0260 (2)	0.1786 (1)	0.0329 (6)

† Occupation factor: 0.8.
‡ Occupation factor: 0.2.

Table 2. *Selected geometric parameters* (Å, °)

S(1)—C(1)	1.785 (3)	S(1)—C(3)	1.810 (3)
O(4)—C(4)	1.230 (3)	O(8)—C(8)	1.235 (3)
O(211)—C(21)	1.301 (3)	O(212)—C(21)	1.203 (3)
N(1)—C(2)	1.474 (3)	N(1)—C(3)	1.460 (3)
N(1)—C(4)	1.347 (3)	N(2)—C(5)	1.462 (3)
N(2)—C(8)	1.326 (3)	C(1)—C(2)	1.533 (4)
C(2)—C(21)	1.521 (3)	C(4)—C(5)	1.529 (3)
C(5)—C(6)	1.548 (3)	C(6)—C(7)	1.528 (3)
C(7)—C(8)	1.508 (3)		
C(1)—S(1)—C(3)	90.1 (1)	C(2)—N(1)—C(4)	118.7 (2)
C(3)—N(1)—C(4)	125.1 (2)	C(5)—N(2)—C(8)	113.3 (2)
C(2)—N(1)—C(3)	115.0 (2)	N(1)—C(2)—C(1)	106.6 (2)
S(1)—C(1)—C(2)	107.9 (2)	N(1)—C(2)—C(21)	111.9 (2)
C(1)—C(2)—C(21)	108.6 (2)	S(1)—C(3)—N(1)	103.4 (2)
N(1)—C(4)—C(5)	117.4 (2)	O(4)—C(4)—N(1)	121.3 (2)
N(2)—C(5)—C(4)	110.7 (2)	O(4)—C(4)—C(5)	121.1 (2)
N(2)—C(5)—C(6)	101.9 (2)	C(4)—C(5)—C(6)	108.4 (2)
C(6)—C(7)—C(8)	103.7 (2)	C(5)—C(6)—C(7)	102.9 (2)
O(8)—C(8)—C(7)	127.1 (2)	N(2)—C(8)—C(7)	109.1 (2)
O(212)—C(21)—C(2)	122.9 (2)	O(8)—C(8)—N(2)	123.7 (2)
O(211)—C(21)—O(212)	124.4 (2)	O(211)—C(21)—C(2)	112.6 (2)

Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). The structure was refined by full-matrix least squares with anisotropic displacement parameters for all non-H atoms. H atoms were located on a difference Fourier map, except for H(11) [bonded to C(1)] which was introduced at a calculated position (C—H = 1.080 Å, U_{iso} = 0.07 Å^2) and not refined; all other H atoms were refined isotropically. Program used to refine structure: *SHELX*76 (Sheldrick, 1976).

One of us (JDA) is much indebted to Conselho Nacional de Desenvolvimento Cientifico e Tecnologico (CNPq) for fellowship at the University of Milan.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Averbuch-Pouchot, M. T., Durif, A., Pera, M. H. & Hamri, A. (1990). *Acta Cryst.* C**46**, 299–301.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Keszler, D. A., Johnson, A. T. & White, J. D. (1991). *Acta Cryst.* C**47**, 1481–1482.
Parthasarathy, R., Paul, B. & Korytnyk, W. (1976). *J. Am. Chem. Soc.* **98**, 6634–6643.
Pugliese, A., Biglino, A., Uslenghi, C., Marinelli, L., Forno, B. & Girardello, R. (1992). *Int. J. Immunother.* **8**, 212–219.
Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Villani, V., Pucciariello, R., Crimella, T. & Stradi, R. (1993). *J. Mol. Struct.* **298**, 177–190.

©1995 International Union of Crystallography
Printed in Great Britain – all rights reserved

Acta Cryst. (1995). C**51**, 475–477

2-(*p*-Ethoxyphenyl)-5-hexyl-1,3-dithiane, $C_{18}H_{28}OS_2$

MICHIHIRO MIYAKE*

Department of Chemistry, Faculty of Engineering, Gunma University, Tenjin, Kiryu 376, Japan

YUICHIRO HARAMOTO

Department of Applied Chemistry and Biotechnology, Faculty of Engineering, Yamanshi University, Takeda, Kofu 400, Japan

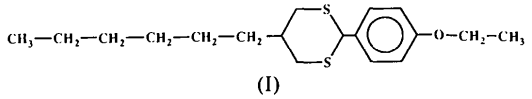
(*Received* 3 *August* 1993; *accepted* 28 *June* 1994)

Abstract

The molecules, which are *trans* isomers, are almost linearly extended and are stacked with their axes alternately oriented. The dithiane rings of the molecules are in chair conformations and are situated close together; the S···S interatomic distance is 3.006 (1) Å.

Comment

2,5-Disubstituted 1,3-dithianes, (R-$C_4H_6S_2$–C_6H_4-R'), have been reported as a new type of liquid-crystal compound (Haramoto & Kamogawa, 1983, 1985*a*,*b*; Haramoto, Nobe & Kamogawa, 1984; Haramoto, Akazawa & Kamogawa, 1984). The title compound, (I), with R = n-C_6H_{13} and R' = OC_2H_5, exhibits monotropic behaviour and the nematic liquid-crystal phase appears in the temperature range 316–305 K.



(I)

The molecule of compound (I) is shown in Fig. 1. The molecule, which is the *trans* isomer, is almost linearly extended. The S···S interatomic distance, the mean S—C bond length and the mean C—C bond length in the dithiane ring are 3.006 (1), 1.808 (4) and 1.516 (5) Å, re-

spectively. The dithiane ring is in a chair conformation and is twisted with respect to the benzene ring in order to avoid steric repulsion. The mean C—C bond length (excluding those of the benzene ring) and the mean C—O bond length are 1.513 (5) and 1.402 (5) Å, respectively; these C—C and C—O bonds are in the usual *anti* conformations. However, the *gauche* conformation, which is less stable than the *anti* conformation, has been observed in the compound with R = n-C_5H_{11} and R' = n-OC_4H_9 (Miyake, Haramoto & Kamogawa, 1994). This compound also shows monotropic liquid-crystal behaviour (Haramoto, Nobe & Kamogawa, 1994).

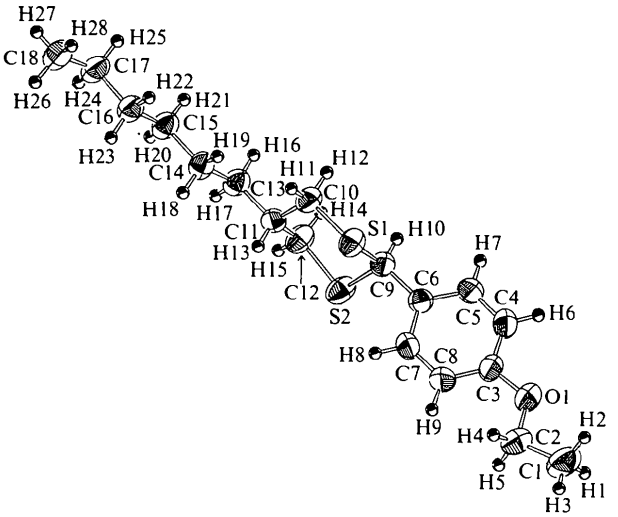


Fig. 1. Perspective drawing (*ORTEP*II; Johnson, 1976) of the title compound indicating the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

The packing is shown in Fig. 2. The molecules are stacked with their axes alternately oriented. The dithiane rings in neighbouring molecules are close together and the S(1)···S(1)($1-x$, $\pm\frac{1}{2}+y$, $\frac{3}{2}-z$), S(1)···S(2)(x, $1+y$, z) and S(2)···S(2)(x, $\pm 1+y$, z) interatomic distances are 4.085 (2), 4.971 (2) and 5.492 (3) Å, respectively. This feature has also been observed in the compound with R = n-C_5H_{11} and R' = n-OC_4H_9.

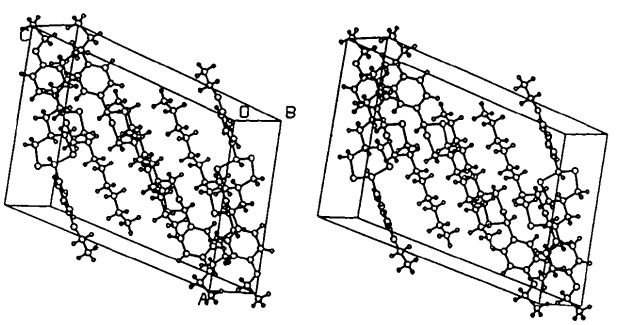


Fig. 2. Stereoscopic view (*PLUTO*; Motherwell & Clegg, 1978) of the unit cell.

Liquid-crystal compounds with a dithiane ring show high viscosity and slow response time compared with those containing a dioxane ring or a cyclohexane ring (Haramoto & Kamogawa, 1985*b*). Provided that the structure of the crystal is maintained approximately in the structure of the liquid crystal, these characteristics may be attributed to the short interatomic distances between the S atoms of neighbouring molecules, which have a larger dispersion force than the O and C atoms.

Experimental

The synthesis of the title compound has been reported previously (Haramoto, Nobe & Kamogawa, 1984). The compound was recrystallized from chloroform.

Crystal data

$C_{18}H_{28}OS_2$
M_r = 324.54
Monoclinic
$P2_1/c$
a = 14.539 (3) Å
b = 5.492 (2) Å
c = 23.452 (2) Å
β = 98.12 (1)°
V = 1853.8 (9) Å^3
Z = 4
D_x = 1.163 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 15.0–16.4°
μ = 0.273 mm^{-1}
T = 296 K
Rectangular
0.3 × 0.2 × 0.15 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: none
4876 measured reflections
4698 independent reflections
1998 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.032
θ_{max} = 27.5°
h = 0 → 18
k = 0 → 7
l = −30 → 30
3 standard reflections monitored every 150 reflections
intensity decay: 3%

Refinement

Refinement on F
R = 0.044
wR = 0.047
S = 1.30
1998 reflections
303 parameters
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ = 0.31
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 0.1165 × 10^{-5}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	B_{eq}
S(1)	0.60188 (6)	0.2083 (2)	0.77467 (4)	4.55 (4)
S(2)	0.72092 (6)	−0.0318 (2)	0.87412 (4)	5.22 (5)
O(1)	0.9516 (2)	−0.1215 (5)	0.6534 (1)	5.5 (1)

C(1)	1.0677 (4)	0.005 (2)	0.5999 (2)	7.5 (3)
C(2)	1.0170 (3)	0.070 (1)	0.6491 (2)	5.4 (2)
C(3)	0.8884 (2)	−0.0947 (7)	0.6911 (1)	4.1 (2)
C(4)	0.8218 (3)	−0.2745 (8)	0.6891 (2)	4.8 (2)
C(5)	0.7539 (2)	−0.2626 (7)	0.7245 (2)	4.3 (2)
C(6)	0.7506 (2)	−0.0718 (6)	0.7624 (1)	3.8 (1)
C(7)	0.8192 (3)	0.1040 (7)	0.7645 (2)	4.8 (2)
C(8)	0.8881 (2)	0.0943 (8)	0.7289 (2)	4.7 (2)
C(9)	0.6736 (2)	−0.0514 (7)	0.7988 (1)	4.1 (2)
C(10)	0.5162 (2)	0.1966 (9)	0.8233 (2)	4.4 (2)
C(11)	0.5537 (2)	0.2103 (7)	0.8869 (1)	3.8 (1)
C(12)	0.6149 (3)	−0.0064 (8)	0.9060 (2)	4.8 (2)
C(13)	0.4736 (3)	0.2245 (8)	0.9229 (2)	4.6 (2)
C(14)	0.4150 (3)	0.4529 (7)	0.9154 (2)	4.6 (2)
C(15)	0.3349 (3)	0.4554 (7)	0.9507 (2)	4.7 (2)
C(16)	0.2756 (3)	0.6803 (7)	0.9439 (2)	4.6 (2)
C(17)	0.1956 (3)	0.6782 (8)	0.9793 (2)	5.2 (2)
C(18)	0.1416 (4)	0.910 (1)	0.9767 (2)	6.2 (2)

Table 2. *Selected geometric parameters* (Å, °)

S(1)···S(2)	3.006 (1)	S(1)—C(9)	1.809 (4)
S(1)—C(10)	1.805 (4)	S(2)—C(9)	1.806 (4)
S(2)—C(12)	1.811 (4)	O(1)—C(2)	1.433 (5)
O(1)—C(3)	1.371 (4)	C(1)—C(2)	1.499 (6)
C(3)—C(4)	1.379 (5)	C(3)—C(8)	1.365 (5)
C(4)—C(5)	1.376 (5)	C(5)—C(6)	1.380 (4)
C(6)—C(7)	1.384 (5)	C(6)—C(9)	1.505 (4)
C(7)—C(8)	1.392 (5)	C(10)—C(11)	1.516 (5)
C(11)—C(12)	1.515 (5)	C(11)—C(13)	1.534 (4)
C(13)—C(14)	1.512 (5)	C(14)—C(15)	1.522 (5)
C(15)—C(16)	1.502 (5)	C(16)—C(17)	1.523 (5)
C(17)—C(18)	1.491 (6)		
C(9)—S(1)—C(10)	101.1 (2)	C(9)—S(2)—C(12)	100.3 (2)
C(2)—O(1)—C(3)	118.6 (3)	O(1)—C(2)—C(1)	106.4 (4)
O(1)—C(3)—C(4)	115.7 (3)	O(1)—C(3)—C(8)	124.3 (3)
C(4)—C(3)—C(8)	120.0 (3)	C(3)—C(4)—C(5)	120.3 (4)
C(4)—C(5)—C(6)	121.2 (4)	C(5)—C(6)—C(7)	117.5 (3)
C(5)—C(6)—C(9)	121.3 (3)	C(7)—C(6)—C(9)	121.2 (3)
C(6)—C(7)—C(8)	121.9 (4)	C(3)—C(8)—C(7)	119.1 (4)
S(1)—C(9)—S(2)	112.5 (2)	S(1)—C(9)—C(6)	108.8 (2)
S(2)—C(9)—C(6)	110.3 (2)	S(1)—C(10)—C(11)	115.8 (2)
C(10)—C(11)—C(12)	111.5 (3)	C(10)—C(11)—C(13)	110.3 (3)
C(12)—C(11)—C(13)	109.7 (3)	S(2)—C(12)—C(11)	115.9 (3)
C(11)—C(13)—C(14)	115.9 (3)	C(13)—C(14)—C(15)	114.0 (3)
C(14)—C(15)—C(16)	114.9 (3)	C(15)—C(16)—C(17)	114.0 (3)
C(16)—C(17)—C(18)	114.1 (4)		

Azimuthal scans of several reflections revealed no need for an absorption correction; intensities were corrected for Lorentz–polarization effects.

The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). The non-H atoms were refined with anisotropic displacement parameters. H atoms, located by difference Fourier synthesis, were refined with isotropic displacement parameters. All calculations were performed using the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985). *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978) were used to prepare Figs. 1 and 2.

We thank Messrs T. Hori, S. Yoshimachi and R. Yokoyama of Rigaku Corporation for their kind help.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: OH1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Beurskens, P. T. (1984). *DIRDIF. Direct Methods for Difference Structures – an Automatic Procedure for Phase Extension and Refinement of Difference Structure Factors.* Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Gilmore, C. J. (1984). *J. Appl. Cryst.* **17**, 42–46.

Haramoto, Y., Akazawa, K. & Kamogawa, H. (1984). *Bull. Chem. Soc. Jpn*, **57**, 3173–3176.

Haramoto, Y. & Kamogawa, H. (1983). *J. Chem. Soc. Chem. Commun.* pp. 75–76.

Haramoto, Y. & Kamogawa, H. (1985*a*). *Bull. Chem. Soc. Jpn*, **58**, 477–480.

Haramoto, Y. & Kamogawa, H. (1985*b*). *Mol. Cryst. Liq. Cryst.* **131**, 101–107.

Haramoto, Y., Nobe, A. & Kamogawa, H. (1984). *Bull. Chem. Soc. Jpn*, **57**, 1966–1969.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Miyake, M., Haramoto, Y. & Kamogawa, H. (1994). *Acta Cryst.* **C50**, 805–807.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package.* MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* Univ. of Cambridge, England.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1995). C**51**, 477–479

(2*S*,3*R*,4*S*)-4-(1,1-Dimethylethoxycarbonylamino)-2,3-epoxy-5-phenylpentanoic Acid Ethyl Ester

H. P. Weber

Sandoz Pharma AG, Preclinical Research, CH-4002 Basel, Switzerland

D. Scholz, H. Schmidt and H. Gstach

Sandoz Forschungsinstitut, Brunnerstrasse 59, A-1235 Vienna, Austria

(*Received* 17 *December* 1993; *accepted* 16 *September* 1994)

Abstract

The X-ray analysis of the title compound [alternative name: ethyl 4-(*tert*-butoxycarboxamido)-2,3-epoxy-5-phenylpentanoate, $C_{18}H_{25}NO_5$, was performed in order to prove the structure of the synthetic product with respect to the existence of a *trans* epoxide and to determine the relative configurations of atoms C(2) (*S*) and C(3) (*R*) with respect to the known chirality of atom C(4) (*S*). There are no unusual structural features in the crystal although the torsion angles between the epoxide and the carboxamido groups are noteworthy.

© 1995 International Union of Crystallography
Printed in Great Britain – all rights reserved