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1,2-Bis(3-thienyloxy)ethane: a thiophene-based precursor for thiophene-based azacryptand Mannich bases

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Key indicators

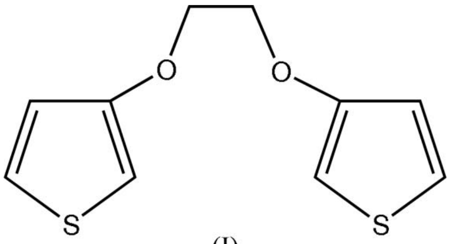
Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.090
Data-to-parameter ratio = 21.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{10}H_{12}O_2S_2$, is composed of two thiophene rings bridged by an –O(CH$_2$)$_2$O– chain in a *trans* arrangement. The molecule possesses C_2 symmetry with the twofold axis bisecting the central C—C bond. In the crystal structure, molecules related by a centre of symmetry are bridged by C—H$\cdots$O hydrogen bonds, forming a zigzag one-dimensional chain extending in the c-axis direction.

Comment

The preparation of a range of open-chain cryptand-like structures, incorporating thiophene rings, as precursors for azacryptand Mannich bases, has been described by Barker *et al.* (1993) and Chaffin *et al.* (2001, 2002). The title compound, (I), was prepared by the reaction of methyl 3-hydroxythiophene-2-carboxylate with 1,2-dichloroethane and anhydrous potassium carbonate in anhydrous *N*,*N*-dimethylformamide, followed by saponification and decarboxylation.



(I)

The molecular structure of (I) is illustrated in Fig. 1, and selected bond distances and angles are given in Table 1. In compound (I), two thiophene rings are bridged by an –O(CH$_2$)$_2$O– chain in a *trans* arrangement. A twofold axis bisects the central ethane bond [C5—C5$(1 - x, y, \frac{1}{2} - z)$] and each half of the molecule is almost planar, with C5—O1—C2—C1 and C5—O1—C2—C3 torsion angles of 0.00 (18) and −178.45 (11)°, respectively. The bond lengths and angles (Table 1) are similar to those in an unsubstituted thiophene described by Bonham & Momany (1963).

The crystal packing of compound (I) is illustrated in Fig. 2. The molecules related by centres of symmetry are linked by C—H$\cdots$O hydrogen bonds; details are given in Table 2. It can be seen that the molecules are arranged in a such a way as to form a zigzag one-dimensional polymer extending in the crystallographic c-axis direction.

Experimental

Compound (I) was synthesized according to the procedure described by Chaffin *et al.* (2001). Suitable crystals for X-ray crystallography analysis were obtained by slow evaporation of a 1:1 ethanol–dichloromethane solution.

Crystal data

$C_{10}H_{10}O_2S_2$
$M_r = 226.30$
Monoclinic, $C2/c$
$a = 22.175$ (3) Å
$b = 5.3918$ (4) Å
$c = 9.0831$ (11) Å
$\beta = 111.362$ (9)°
$V = 1011.39$ (19) Å^3
$Z = 4$
$D_x = 1.486$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9090 reflections
$\theta = 1.9$–29.6°
$\mu = 0.49$ mm^{-1}
$T = 153$ (2) K
Plate, colourless
0.5 × 0.5 × 0.2 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: none
9441 measured reflections
1398 independent reflections
1302 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.053$
$\theta_{\rm max} = 29.4°$
$h = -30 \rightarrow 30$
$k = -7 \rightarrow 7$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.090$
$S = 1.03$
1398 reflections
64 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 1.0038P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.35$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C4	1.7129 (14)	C3—C4	1.3674 (19)
S1—C1	1.7178 (13)	C3—C2	1.4227 (17)
O1—C2	1.3597 (15)	C2—C1	1.3672 (17)
O1—C5	1.4288 (15)	C5—C5$^{\rm i}$	1.500 (2)
C4—S1—C1	92.55 (6)	O1—C2—C3	118.83 (11)
C2—O1—C5	115.12 (10)	C3—C4—S1	111.56 (10)
C4—C3—C2	111.88 (11)	O1—C5—C5$^{\rm i}$	108.13 (9)
C1—C2—O1	127.57 (12)	C2—C1—S1	110.43 (10)
C1—C2—C3	113.59 (12)		

Symmetry code: (i) $-x + 1, y, -z + \frac{1}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1$^{\rm ii}$	1.00	2.41	3.3940 (16)	170

Symmetry code: (ii) $-x + 1, -y, -z$.

H atoms were located in difference Fourier maps and held fixed with $U_{\rm iso}$(H) = 0.05 Å^2 and C—H = 0.94–1.05 Å.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

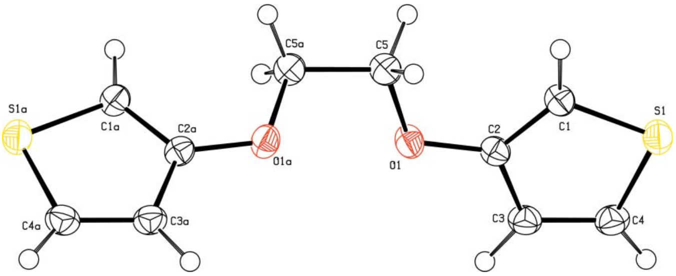


Figure 1
View of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (*a*) $1 - x, y, \frac{1}{2} - z$.]

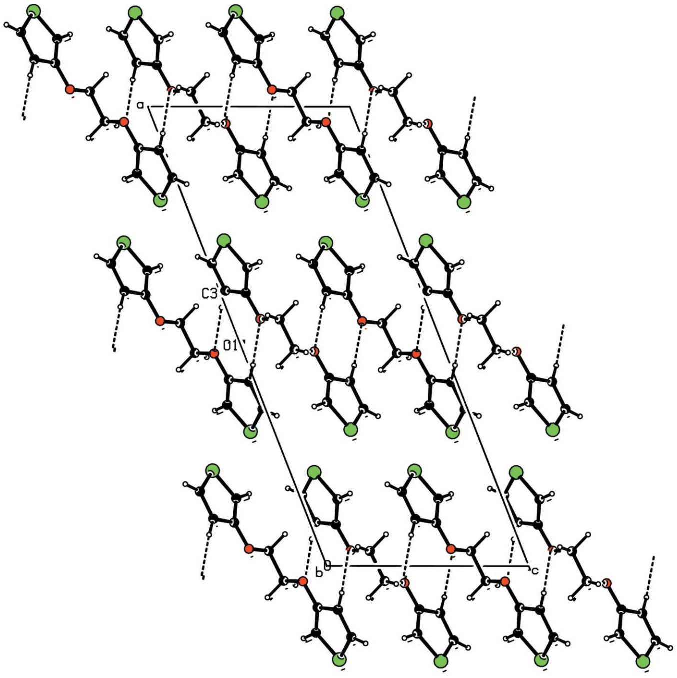


Figure 2
The crystal packing of compound (I), viewed down the *b* axis. C—H···O hydrogen bonds are shown as dashed lines (details are given in Table 2).

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