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1,5-Bis(3-thienyloxy)-3-oxapentane: 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) = 0.003 Å
R factor = 0.033
wR factor = 0.072
Data-to-parameter ratio = 16.3

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

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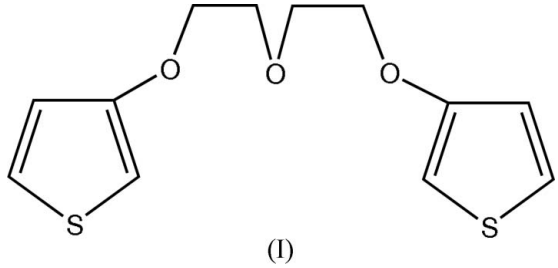
The title compound, $C_{12}H_{14}O_3S_2$, is composed of two thiophene rings bridged by an $-O(CH_2)_2O(CH_2)_2O-$ chain. The molecule is U-shaped, with the two thiophene rings inclined to one another by 83.21 (10)°. In the crystal structure, the molecules are bridged by C—H···S and C—H···O hydrogen bonds, forming a double-stranded polymer chain.

Comment

The preparation of a range of open-chain cryptand-like structures, incorporating thiophene rings, as precursors for azacryptand Mannich bases, was undertaken by Barker *et al.* (1993) and Chaffin *et al.* (2001, 2002). The title compound, (I), was synthesized by the reaction of methyl 3-hydroxythiophene-2-carboxylate with 1,5-bis(*p*-tolylsulfonyloxy)-3-oxapentane 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. The molecule is U-shaped and has pseudo-C_2 symmetry, with the central $-O(CH_2)_2O(CH_2)_2O-$ bridge having a *cis–cis* conformation. The two thiophene rings are inclined to one another by 83.21 (10)°. The thiophene bond lengths and bond angles are similar to those in an unsubstituted thiophene reported by Bonham & Momany (1963). The thienyloxy and other bond lengths and angles in (I) are in agreement with standard values (*International Tables for Crystallography*, Vol. C, 1995). In the crystal structure, symmetry-related molecules are bridged by C—H···S and C—H···O hydrogen bonds (Table 2), forming a double-stranded polymer chain (Fig. 2).

Experimental

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

Crystal data

$C_{12}H_{14}O_3S_2$
M_r = 270.35
Monoclinic, $P2_1/n$
a = 5.2998 (4) Å
b = 19.4005 (18) Å
c = 12.7277 (9) Å
β = 100.960 (8)°
V = 1284.78 (18) Å^3
Z = 4
D_x = 1.398 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
θ = 1.7–26.1°
μ = 0.41 mm^{-1}
T = 153 (2) K
Plate, colourless
0.50 × 0.25 × 0.10 mm

Data collection

Stoe IPDS diffractometer
ω scans
Absorption correction: none
10146 measured reflections
2509 independent reflections
1609 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 26.0°
$h = -6 \rightarrow 6$
$k = -23 \rightarrow 23$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.072
S = 0.85
2509 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0331P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C4	1.705 (2)	O3—C8	1.428 (2)
S1—C1	1.717 (2)	C1—C2	1.356 (3)
S2—C12	1.701 (2)	C3—C4	1.353 (3)
S2—C9	1.716 (3)	C5—C6	1.503 (3)
O1—C2	1.368 (2)	C7—C8	1.490 (3)
O1—C5	1.425 (2)	C9—C10	1.362 (3)
O2—C6	1.414 (3)	C10—C11	1.414 (3)
O2—C7	1.421 (2)	C11—C12	1.346 (3)
O3—C10	1.362 (3)		
C4—S1—C1	91.89 (10)	C3—C4—S1	112.15 (16)
C12—S2—C9	91.92 (11)	O1—C5—C6	107.58 (16)
C2—O1—C5	116.42 (15)	O2—C6—C5	108.33 (17)
C6—O2—C7	111.88 (16)	O2—C7—C8	109.35 (18)
C10—O3—C8	115.46 (16)	C10—C9—S2	110.66 (17)
C2—C1—S1	110.59 (15)	C9—C10—O3	128.5 (2)
C1—C2—O1	128.80 (18)	C9—C10—C11	112.9 (2)
C1—C2—C3	113.61 (18)	O3—C10—C11	118.64 (18)
O1—C2—C3	117.59 (17)	C12—C11—C10	112.3 (2)
C4—C3—C2	111.76 (19)	C11—C12—S2	112.15 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···O1^{i}	0.95	2.52	3.358 (2)	148
C12—H12A···S1^{ii}	0.95	2.86	3.787 (2)	164

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z+1$.

H atoms were included in calculated positions and treated as riding atoms, with C—H = 0.95–0.99 Å and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *EXPOSE* (Stoe & Cie, 2002); cell refinement: *CELL* (Stoe & Cie, 2002); data reduction: *INTEGRATE* (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*.

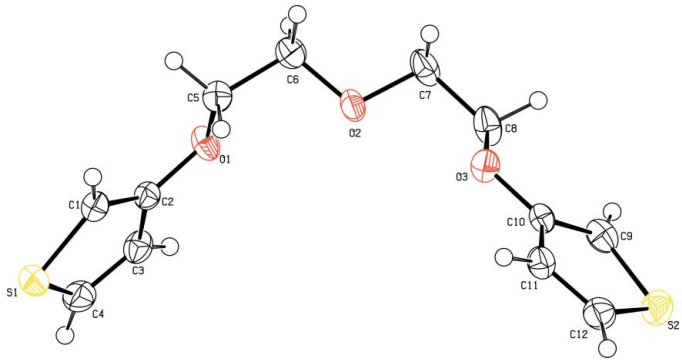


Figure 1
View of the molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

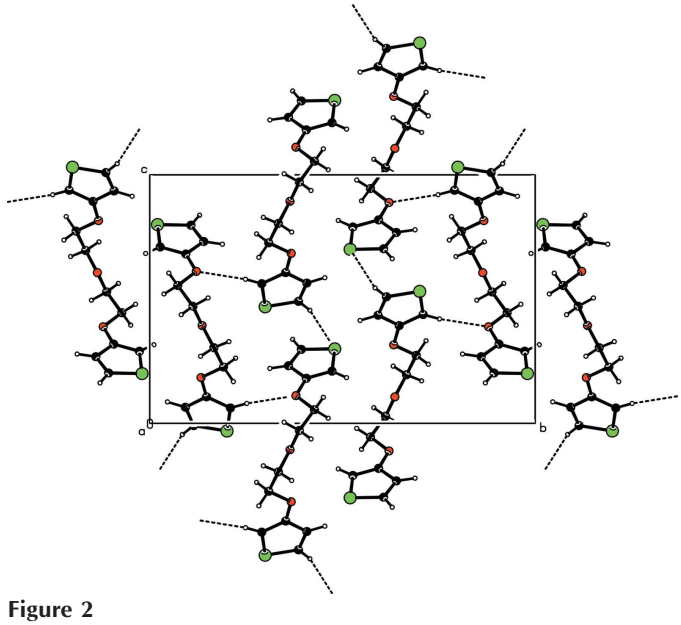


Figure 2
The crystal packing of compound (I), viewed down the *a* axis. C—H···S and C—H···O hydrogen bonds are shown as dashed lines.

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