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Synthesis and X-Ray Crystal and Molecular Structure of 1,6-Bis(3-thienyl)hexane

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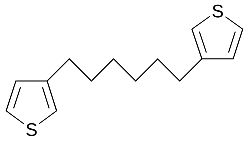
1,6-Bis(3-thienyl)hexane has been prepared and characterised in an X-ray diffraction study.

There is considerable interest in polythiophenes and oligothiophenes because they are conducting when doped and may be used for third harmonic generation.[1] We report here that 1,6-bis(3-thienyl)hexane (**1**) is prepared by the coupling of 1,6-bis(bromomagnesiohexane) with 3-bromothiophene in the presence of [$NiCl_2$(dppp)],[14]† the product being isolated by distillation followed by low temperature recrystallisation. It did not prove possible to cyclise **1** under oxidative conditions using palladium(II).[16] Bromination of **1** (NBS–DMF†,[17]) gave 1,6-bis(2-bromo-3-thienyl)hexane in reasonable yield, but successful cyclisation of the bromo compound also proved elusive.



1

The structure of **1** was determined (direct methods, SHELXS-86, SHELXL-93, $R_1 = 0.070$, $wR_2 = 0.175$) by X-ray crystallography (Fig. 1); the molecule lies across a crystallographic inversion centre. The thiophene ring is planar and unstrained, and the molecular dimensions are similar to those in bithiophene.[2]

Techniques used: ^{1}H and ^{13}C NMR spectroscopy, X-ray diffraction

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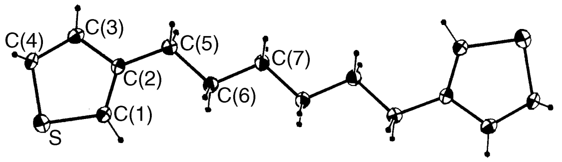

Fig. 1 Structure of 1,6-bis(3-thienyl)hexane) (**1**). $C_{14}H_{18}S_2$, $M = 250.4$, monoclinic, $a = 5.960(2)$, $b = 17.952(7)$, $c = 6.629(2)$ Å, $\beta = 107.36(3)$°, $V = 677.0(4)$ Å^3, space group $P2_1/n$ (non-standard No. 14), $Z = 2$, $D_x = 1.23$ g cm^{-3}. $R_1 = 0.070$, $wR_2 = 0.175$. Selected bond lengths (Å) and angles (°): S–C(1) 1.704(4); S–C(4) 1.700(4); C(1)–C(2) 1.360(5); C(2)–C(3) 1.411(5); C(3)–C(4) 1.390(5) Å. C(1)–S–C(4) 92.7(2); S–C(1)–C(2) 113.1(3); C(1)–C(2)–C(3) 110.0(4); C(2)–C(3)–C(4) 114.9(4); S–C(4)–C(3) 109.2(3)

Fig. 2: Packing diagram for **1**

Tables 1, 2: Atomic coordinates, bond lengths and angles for **1**

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†Abbreviations used: dppp = 1,3-(diphenylphosphino)propane; NBS = *N*-bromosuccinimide.