material formed was then extracted from the cold reaction mixture into chloroform. Evaporation of the washed and dried solution left an oily material which was converted to the solid HBr salt by addition of HBr to an ethyl acetate solution and the precipitated material recrystallized from ethyl acetate/acetone; yield 0.8 g (49 %), m.p. 170°C (decomp). (Found: C 38.85; H 4.19; N 5.65. Calc. for $C_8H_9NOS \cdot HBr$: C 38.72; H 4.06; N 5.46.)

NMR spectrum in TFA: 7.30 τ ($-CH_3$, singlet), 6.54 and 5.39 τ ($-S-CH_2-CH_2-O-$ triplets), 2.25–2.63 (H^7-H^8, AB-quartet with $J=9$ cps). UV maxima in 0.1 N HCl/EtOH at 338 nm (log $\varepsilon=4.10$) and at 256 nm (3.76); in 0.1 N NaOH/EtOH at 311 nm (log $\varepsilon=4.07$) and at 250 nm (3.90).

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The Structure of 2-Methyl-6a-thiathiophthene

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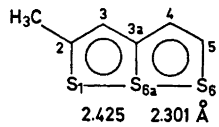
The results from CNDO/2 calculations on mono-methyl substituted 6a-thiathiophthenes show that a 2-methyl group causes a lengthening of the S(1)–S(6a) bond.[1] A structure study of 2-methyl-6a-thiathiophthene (I) has been carried out in order to test the CNDO/2 predictions, and the preliminary results from this study are given.



(I)

The sulphur–sulphur bonds in I are S(1)–S(6a) = 2.425(2) Å and S(6a)–S(6) = 2.301(2) Å in agreement with the results from the CNDO/2 calculations.[1]

Other bond lengths in the molecule are S(1)–C(2) = 1.688(4) Å, S(6a)–C(3a) = 1.747(5) Å, S(6)–C(5) = 1.672(5) Å, C(2)–C(3) = 1.353(7) Å, C(3)–C(3a) = 1.403(6) Å, C(3a)–C(4) = 1.425(7) Å, C(4)–C(5) = 1.365(7) Å and C(2)–C(6) = 1.520(8) Å.

A sample of I was generously supplied by Reid.[2] Crystallisation from cyclohexane yielded orange red plates with {001} predominant. The crystals belong to the monoclinic space group $P2_1/c$ with unit cell dimensions $a=5.9558(9)$ Å, $b=7.8563(8)$ Å, $c=16.014(4)$ Å, and $\beta=91.31(2)°$. There are four molecules per unit cell; $D_c=1.545$ g cm^{-3}, $D_m=1.54$ g cm^{-3}.

The structure was solved by direct methods[3] and refined by full matrix least squares. With anisotropic temperature factor coefficients for all atoms except hydrogen, the present R is 0.05.

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