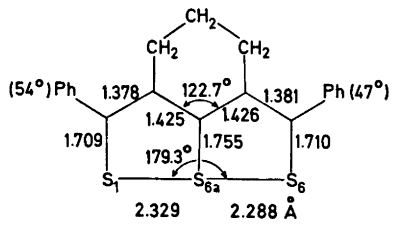
The Structure of 2,5-Diphenyl-3,4-dimethylene-6a-thia-thiophthene

BJØRN BIRKNES, ASBJØRN HORDVIK and LEIF J. SÆTHRE

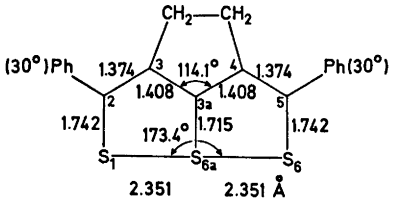
Chemical Institute, University of Bergen, N-5000 Bergen, Norway

2,5-Diphenyl-3,4-dimethylene-6a-thia-thiophthene (II) possesses intramolecular strain caused by the presence of the dimethylene bridge. A structure analysis of II has been carried out in order to find the degree to which the intramolecular strain affects the bonding in the 6a-thiathiophthene system of II relative to the bonding in the 6a-thiathiophthene system of 2,5-diphenyl-3,4-trimethylene-6a-thiathiophthene (I).[1] The preliminary results from this study are given.

The 2,5-diphenyl-3,4-dimethylene molecules lie across crystallographic mirror planes, and the two halves of the molecule



(I)



(II)

are therefore exactly equal. The bond lengths in the 6a-thiathiophthene system of II are, S(1)–S(6a) = 2.351(1) Å, S(1)–C(2) = 1.742(4) Å, S(6a)–C(3a) = 1.715(5) Å, C(2)–C(3) = 1.374(5) Å, and C(3)–C(3a) = 1.408(4) Å. The twist angles of the phenyl groups about the respective connecting bonds are 30°.

The central C–S bond in II is shorter than the terminal C–S bonds. This is opposite to what so far has been found for the C–S bonds in 6a-thiathiophthenes.[2] Furthermore, the dimethylene bridge has caused a lengthening of about 0.1 Å of the three-sulphur sequence in II relative to that in I.

A sample of 2,5-diphenyl-3,4-dimethylene-6a-thiathiophthene was generously supplied by M. Stavaux.[3] The crystals are very dark red and belong to the orthorhombic space group *Pnma*. The cell dimensions are, $a = 7.165(1)$ Å, $b = 29.704(4)$ Å, and $c = 7.372(3)$ Å. There are four molecules per unit cell; $D_c = 1.433$ g/cm³, $D_m = 1.427$ g/cm³.

The structure analysis is based on X-ray data collected on a paper-tape controlled Siemens AED diffractometer using Mo$K\alpha$ radiation. 812 reflections were observed within $\theta = 27°$.

The structure was solved by the heavy atom method and refined by full matrix least squares. The final R factor is 0.037.

We thank Dr. M. Stavaux, Departement de Chimie, Université de Caen, France, for a sample of 2,5-diphenyl-3,4-dimethylene-6a-thiathiophthene.

1. Birknes, B., Hordvik, A. and Sæthre, L. J. *Acta Chem. Scand.* **26** (1972) 2141.
2. Hansen, L. K. and Hordvik, A. *Acta Chem. Scand.* **27** (1973). *In press.*
3. Stavaux, M. and Lozac'h, N. *Bull. Soc. Chim France* **1967** 2082.

Received December 13, 1972.