1. Miller, W. L. *Z. physik. Chem. (Leipzig)* **69** (1909) 436.
2. Consden, R., Gordon, A. H. and Martin, A. J. P. *Biochem. J.* **40** (1946) 33.
3. Vestermark, A. *Sci. Tools* **17** (1970) 24.
4. Crestfield, A. M. and Allen, F. W. *Anal. Chem.* **216** (1955) 67.
5. Vestermark, A. and Sjödin, B. *J. Chromatog.* **73** (1972) 211.

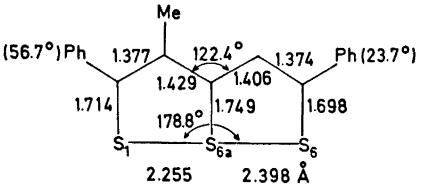
Received November 27, 1972.

The Structure of 2,5-Diphenyl-3,4-dimethyl-6a-thiathiophthene

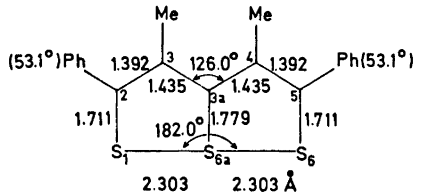
ASBJØRN HORDVIK, ODDVAR SJØLSET and LEIF J. SÆTHRE

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

The structure study of 2,5-diphenyl-3-methyl-6a-thiathiophthene (I)[1] showed that the sulphur-sulphur bonds there are unequal, *i.e.* S(1) – S(6a) = 2.255(1) Å and S(6a) – S(6) = 2.398(1) Å.



(I)



(II)

This agrees with the results from CNDO/2 calculations of mono-methyl and mono-phenyl substituted 6a-thiathiophthenes.[2]

A structure investigation of 2,5-diphenyl-3,4-dimethyl-6a-thiathiophthene (II) has been carried out in order to obtain further information about the degree to which methyl and phenyl substituents affect the S – S bonding in 6a-thiathiophthenes; the preliminary results are given.

The S(6a) – C(3a) bond in II lies along a crystallographic two-fold axis, and the two halves of the molecule are therefore exactly equal. The bond lengths in the 6a-thiathiophthene system of II are, S(1) – S(6a) = 2.303(1) Å, S(1) – C(2) = 1.711(4) Å, S(6a) – C(3a) = 1.779(4) Å, C(2) – C(3) = 1.392(4) Å, and C(3) – C(3a) = 1.435(4) Å. The phenyl groups are twisted 53.1° about the connecting bonds.

The S(6a) – C(3a) bond in II is 0.03 Å longer than that in I, and it is in fact the longest S(6a) – C(3a) bond which so far has been found in 6a-thiathiophthenes.

A sample of 2,5-diphenyl-3,4-dimethyl-6a-thiathiophthene was generously supplied by M. Stavaux.[3] The crystals are dark red and belong to the orthorhombic space group *Aba*2. The cell dimensions are a = 7.7023(17) Å, b = 29.6436(32) Å, and c = 7.2417(8) Å. There are four molecules per unit cell; density, calculated 1.367 g/cm³, found 1.362 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. 800 reflections were observed within $\theta = 28°$.

The structure was solved by the heavy atom (S) method and refined by full matrix least squares. The final R factor is 0.033.

The authors are indebted to Dr. M. Stavaux, Faculté des Science de Cáen, France, for providing a sample of 2,5-diphenyl-3,4-dimethyl-6a-thiathiophthene.

1. Hordvik, A., Sjølset, O. and Sæthre, L. J. *Acta Chem. Scand.* **26** (1972) 1297; and *to be published.*
2. Hansen, L. K., Hordvik, A. and Sæthre, L. J. *Chem. Commun.* **1972** 222.
3. Stavaux, M. and Lozac'h, N. *Bull. Soc. Chim. France* **1967** 2082.

Received December 13, 1972.