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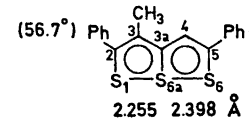
The Structure of 2,5-Diphenyl-3-methyl-6a-thiathiophthene

ASBJØRN HORDVIK, ODDVAR SJØLSET and LEIF J. SÆTHRE

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

CNDO/2 calculations on mono-methyl and mono-phenyl substituted 6a-thiathiophthenes show that a 3-methyl group causes a shortening of the S(1) – S(6a) bond relative to that in 6a-thiathiophthene, while a twisted 2-phenyl group causes a lengthening of this bond.[1,2] The lengthening effect of the 2-phenyl group on S(1) – S(6a) varies with the twist angle, being negligible at twist angle 0° and most pronounced at 90°.[1]

A structure investigation of 2,5-diphenyl-3-methyl-6a-thiathiophthene (I) has been carried out in order to test the CNDO/2 predictions; the preliminary results are given.



(I)

The sulphur – sulphur bonds in I are S(1) – S(6a) = 2.255(1) Å and S(6a) – S(6) = 2.398(1) Å, and the 2- and 5-phenyl groups are twisted 56.7 and 23.7° about the respective connecting bonds. Thus, the effect of phenyl group 2 on the S-S bonding is opposed by the effect of phenyl group 5, and it seems likely that it is the 3-methyl group which has caused the shortening of S(1) – S(6a) in agreement with the results from the CNDO/2 calculations.

Other bond lengths in the 6a-thiathiophthene system of I are: S(1) – C(2) = 1.714(2) Å, S(6a) – C(3a) = 1.749(2) Å, S(6) – C(5) = 1.698(2) Å, C(2) – C(3) = 1.377(2) Å, C(3) – C(3a) = 1.429(3) Å C(3a) – C(4) = 1.406(2) Å, and C(4) – C(5) = 1.374(3) Å.

A sample of 2,5-diphenyl-3-methyl-6a-thiathiophthene was generously supplied by M. Stavaux.[3] The crystals are dark red and belong to the monoclinic space group $P2_1/c$. The cell dimensions are $a = 15.463(2)$ Å, $b = 8.015(1)$ Å, $c = 13.076(2)$ Å, and $\beta = 106.38(1)°$. There are four molecules per unit cell; density, calculated 1.393 g/cm³, found 1.388 g/cm³.

The structure analysis is based on X-ray data collected on a paper-tape controlled Siemens AED diffractometer using Cu$K\alpha$ radiation. 2865 reflections were observed within $\theta = 71°$.

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

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