

09. STRUCTURES OF ORGANIC, ORGANOMETALLIC AND COORDINATION COMPOUNDS

The results obtained show a deformation of phenyl rings: the angles at C atoms bonded with a sulphur atom are smaller than those at neighbouring carbon atoms. Comparison of the molecular structures of 1, 2 and 3 indicates that the investigated compounds have various angles between phenyl planes. Difference in conformation of 1 and 2 and in particularly asymmetric structure of 3 was explained on the basis of energetic calculations. It was determined that in 3, for example, the energetic barrier during rotation around C-S bond is relatively low /approx. 2-3 kcal/mol/. It indicates a possibility of a conformational change in the compounds in diluted solution and at rising temperature. The determines valence angles, bond lengths as well as angles determining molecular structures are different from those found for poly-p-phenylenesulphide. The arrangement of molecules in unit cells confirms that only these model compounds which, similarly to related macromolecules, have not molecular symmetry C_1 can be used for the study of intermolecular interactions in polymers.

half forming the asymmetric unit.

The structure was solved using the direct method and then refined by least squares method using full matrix approximation with anisotropic thermal parameters for individual atoms to $R_w = 0.05$.

The dimer was found to have a centre of symmetry at the middle point of the bond connecting the two dimer halves. In each dimer half the thienyl ring was found to be coplanar with the pyridazinone ring. The deviations from this planarity were calculated for each atom.

09.2-53 Crystal Structure of 4,5-dihydro-6-(2-thienyl)-3(2H)pyridazinoneazine, $C_{16}H_{16}N_6S_2$.

By

G. Will, I. S. Ahmed Farag and M. El-Kordy.

(National Research Centre, Dokki, Cairo.)

The compound was prepared from a direct unusual reaction of 6-(2-thienyl)-2,3,4,5-tetrahydropyridazin-3-one with hydrazine hydrate. The crystals of this compound are monoclinic with unit cell constants: $a=5.567(1)_0$; $b=7.857(2)$; $c=19.194(10)_0$; $\beta=99.97(3)$ and $z=2$. The systematic absences and the statistical distribution of the normalized structure amplitudes showed that the unit cell should acquire a centre of symmetry and hence the space group was considered to be $P2_1/c$. The molecule behaves in the structure as a dimer with

09.2-54 COMPARATIVE STUDY OF THE HIGH PRESSURE AND LOW TEMPERATURE $(IMTSE)_2PF_6$ STRUCTURES.

J. Gaultier, C. Hauw, B. Gallois
Laboratoire de Cristallographie et de Physique Cristalline
LA 144 CNRS, 351, Cours de la Libération - 33405 TALENCE
CEDEX (France)

The crystal structure of $2 C_{10}H_{12}Se_4^{1/2+} . PF_6^-$, a unidimensional organic conductor which exhibits superconductivity at 12 Kbars below 0.9 K, has been determined respectively at 4 K and under an 7 Kbars hydrostatic pressure.

The space group and the crystal packing are the same than at ambient pressure and room temperature (Thorup et al 1981, Acta Cryst, B37). However the intra and interstack distances are perturbed. In particular, Se-Se distances of great interest for the understanding of conducting properties, decrease strongly. Thus, the dimensional character is increased.

	1 Kbar 300 K	7 Kbars 300 K	1 bar 4 K
Intrastack Se-Se distances			
d_1	3,66 Å	3,56 Å	3,55 Å
d_2	3,63 Å	3,56 Å	3,53 Å
Interstack Se-Se distances			
d'_1	3,88 Å	3,74 Å	3,71 Å
d'_2	3,93 Å	3,78 Å	3,74 Å
d'_3	3,96 Å	3,83 Å	3,86 Å
Shortest Se-F distance			
d'_4	3,23 Å	3,09 Å	3,06 Å

At room temperature, the PF_6^- anion is disordered. Under pressure, this disorder strongly weakens and at 4 K, the anion is perfectly ordered.