

09.2-19 A COMPARATIVE ANALYSIS OF OXINDOLE COMPOUNDS. By D.K.Chakraborty & S.K.Talapatra, X-ray Lab., Dept. of Physics, Jadavpur University Calcutta-700032, INDIA.

Indole and its various substituted products have long been known for their interesting chemical and physiological activities. Recently the indole chemistry has received special attention and a number of substituted indole compounds have been synthesized for the investigation of their physico-chemical and biological activities. In the literature there are only a few X-ray structures of oxindole compounds comprising light atoms. But there are no X-ray data available for halogen substituted oxindole compounds. In our course of studies, we took two halogen substituted oxindole compounds namely (i) 5-chloro-3-hydroxy-2(3H)indolone (ii) 3,5,7-Trichloro-3-methyl-2(3H)indolone (Chakraborty et al, 1985). Together with these two and 6-hydroxy-2-(2-methyl propyl)-3-spirotetro hydroxyrrolidino-oxindole (James, et al, 1972) were taken for comparative analysis. The first two were obtained when skatole was treated with sulphuryl chloride in presence of acetic acid and the third one was isolated from the dried root bark of *Eleagnus commutata*. Considering different parameters (i.e. space group, measured density, Av.C-C distances etc. planarity, dihedral angles, hydrogen bonding and packing) of the three compounds we found that the molecular structures have similar conformations. The shortening of the N-C bond was noticed in all the cases and verified from MO calculations. All the three compounds are characterized by strong intermolecular hydrogen bonding. It is further apparent that the substitution of halogen in the oxindole system (either in the benzene ring or five-membered ring) does not affect the symmetry of the Crystalline lattice

09.2-20 ANALYSIS OF RIGID BODY MOTION OF MOLECULES IN CRYSTALS. By D.K.Chakraborty & S.K.Talapatra: X-ray Lab., Dept. of Physics, Jadavpur University, Calcutta-700032, INDIA.

The rigid body motion analysis of crystals was carried out by the program THMV 9 (Trueblood, 85) for the two oxindole structures (i) 5-chloro-3-hydroxy-3-methyl-2(3H)-indolone (ii) 3,5,7-Trichloro-3-methyl-2(3H)indolone (Chakraborty et al, 1985). Since bond-stretching vibrations for atoms other than hydrogen and deuterium are normally of much smaller amplitude, the MSVA of a pair of bonded atoms of comparable mass should be equal along the bonding directions, even though they may be widely different in other directions (Rosenfield, Trueblood, Dunitz, 1978). As thermal ellipsoids derived from X-ray analysis are genuine, Hirshfeld (1976) has pointed out that this provides a necessary condition for the validity of the rigid body model, but it is by no means sufficient since $\Delta A, B$ (difference of MSVA of atom A along B) is unaffected by vibration components perpendicular to the AB direction. Gross violation of the "near equality" for bonded pairs are indications that the atomic vibration tensors (U_{ij} values) derived from least squares treatment are contaminated by experimental errors in the F-values or by charge deformation densities or by absorption or by disorder or by combination of these. Using the U_{ij} values the validity of the rigid body model has been checked for the two structures excluding H-atoms. The anisotropic thermal parameters also have been analysed in terms of assumed over all rigid body motion together with non-rigid librations of molecular fragments about selected bonds as axes. The average e.s.d. of individual U_{ij} is .0010 \AA^2 and .0015 \AA^2 for the 1st and 2nd molecu-

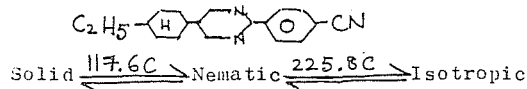
le respectively. The corresponding average difference of MSVA evaluated along the directions of the interatomic lines are .0023 \AA^2 & .0033 \AA^2 . The average e.s.d. of these differences are .0017 \AA^2 & .0025 \AA^2 . The av. difference in MSVA for the bonded atoms along the nine unique C-C bonds are .0015 \AA^2 & .0020 \AA^2 and the av. e.s.d. of these differences are .0018 \AA^2 & .0027 \AA^2 respectively. So the test of rigidity of the first molecule is meaningful but the second one does not stand this test. These observations are also confirmed by the rigid body motion parameters.

	Av. $ \Delta(\text{MSVA}) $	R_{WU}	R.M.S. ΔU_{ij}	Av. ϵ_{ij}
1st molecule	.0023	.067	.0014	.0017
2nd molecule	.0033	.096	.0041	.0025

In the first one excluding terminal methyl carbon C₉ and O₁ atoms the agreement is much better than the earlier ones ($R_{\text{WU}} = .046$, R.M.S. $\Delta U_{ij} = .0009$). Considering the internal motions of C₉ about C5-C8, O₁ about N₁-C7, C₁ about C2-C₁ bonds as axes, the corresponding mean squares amplitudes are 23.4(8.9), 90.7(16.2), 15.3(10.1) deg.² ($R_{\text{WU}} = .044$, R.M.S. $\Delta U_{ij} = .0009$). For the 2nd one, the internal motion of C₁₃ about C5-C8, C₁₁ about C2-C₁, C₉ about C7-C8, O₁ about N₁-C7 give the mean square amplitudes as 37.4, 30.0, 20.0 & 133.0 deg.² ($R_{\text{WU}} = .085$, R.M.S. $\Delta U_{ij} = .0036$). The analysis of Z^{A, B} values indicates that non-bonded links within the individual groups are not significantly less rigid than the bond themselves except for a few cases for the two molecules. For the entire molecule, the rigid body motion parameters, eigenvectors and eigenvalues of L & T and normal mode calculations in the I-frame have been done. The correction for interatomic distances for overall motion and that for atomic coordinates due to libration have been evaluated.

09.2-21 CRYSTAL STRUCTURE OF 5-(4-ETHYLCYCLOHEXYL)-2-(4-CYANOPHENYL-PYRIMIDINE). By P.Mandal, B. Majumdar and S. Paul, Dept. of Physics, North Bengal University, India.

The title compound is a nematogen and has got the following molecular structure and transition temperatures.



Average intermolecular distance, apparent molecular length and orientational order parameters of magnetically aligned sample in the liquid crystalline phase have been determined using different techniques by us. For a proper understanding and interpretation of the physical properties of the liquid crystal a knowledge of the molecular arrangement in the crystalline state is useful, therefore, we have started investigating its crystal structure. Plate shaped crystals, obtained by slow evaporation from a solution in acetone, belong to monoclinic system with space group $P2_1/n$ and cell parameters $a = 15.959$, $b = 33.469$, $c = 6.210$, $\beta = 90.34$ and $Z = 8$. A total of 5623 reflections were collected in a CAD-4 diffractometer on a $\theta - 2\theta$ scan mode. The structure has been solved by direct method program package MULTAN. The correct model containing the 44 non-hydrogen atoms has been built up from successive weighted Fourier synthesis. The refinement is in progress. The structural details and a correlation between the molecular organisations in the solid and mesomorphic phases will be presented.