05.1-22
X-RAY DIFFUSE SCATTERING STUDY OF SPATIAL CORRELATIONS IN PARA-TERPHENYL. by T.R.Welberry. Research School of Chemistry, Australian National University, CANBERRA, and S.L.Mair, CSIRO Division of Materials Science and Technology, CLAYTON, Victoria, Australia.

In the monoclinic ($P2_1/a$) room-temperature phase of para-terphenyl the triple-ringed molecules are approximately planar, but the rings are rotationally disordered about the long axis of the molecule (Rietveld, Maslen and Clews, 1970; see also Baudour, Cailleau and Yelon, 1977). At temperature T_c(≈193K) the molecules become rotationally ordered and non-coplanar. The structure then is triclinic ($P\bar{1}$) with a pseudo-monoclinic cell (Baudour, Delugeard and Cailleau, 1976; Baudour and Charbonneau, 1974), related to the high-temperature phase by an approximate doubling of the a and b cell dimensions.
X-ray diffuse scattering intensities have been measured around superlattice reflections in the monoclinic phase of para-terphenyl for the temperature range T_c+1 to T_c+121 K. Near T_c the correlation lengths along the a, b and c directions, estimated from the peak shapes within the Ornstein-Zernike formalism, are found to be in the approximate ratios 2:6.5:1, respectively. The two larger correlations are along the directions in which the unit cell undergoes an approximate doubling of the lattice parameter at T_c. Over most of the temperature range the correlation lengths are proportional to $(T-T_c)^{-1/2}$, corresponding to the predictions of mean-field theory, but within 4 K of T_c a critical exponent, ν, greater than 0.5 is indicated.

Baudour J.L., Cailleau H. and Yelon W.B. (1977) Acta Cryst. B33, 1773-80.
Baudour J.L. & Charbonneau G.P. (1974) Acta Cryst. B30, 1379.
Baudour J.L., Delugeard Y. & Cailleau H. (1976) Acta Cryst. B32, 150-4.
Rietveld H.M., Maslen E.N. & Clews C.J.B. (1970) Acta Cryst. B26, 693-706.

05.1-23 ORDER-DISORDER PHASE TRANSITIONS: THE CRYSTAL STRUCTURE OF THE 1:1 DONOR:ACCEPTOR COMPLEX BETWEEN ANTHRACENE AND TETRACHLOROPHTHALIC ANHYDRIDE AS A FUNCTION OF TEMPERATURE. By John J. Stezowski* and Rolf-Dietrich Stigler*, W. Mühle† and J. U. von Schütz† Institut für Organische Chemie, Biochemie und *Isotopenforschung und †Physikalishes Institut, Teilinstitut 3 der Universität Stuttgart, D-7000 Stuttgart 80, FRG.

Order-disorder phase transitions are fairly common in molecular crystals, especially for those belonging to the class of π-donor:acceptor complexes. The complex between anthracene, A, and tetrachlorophthalic anhydride, TCPA, is a very interesting example of such a system. The specific heat, $C_p(T)$, measurements by differential scanning calorimetry techniques, reveal two phase transitions, one at 257 K and another at 194 K; the C_p vs. T curves are consistent with second order phase transitions. We have determined the crystal structure for this complex at three temperatures: 296, 230, and 120 K; that is for an example of the structure in each of the three phases accessible with our low temperature apparatus (Syntex LT-1). All three phases display $P\bar{1}$ symmetry: lattice parameters are at 296 K: a = 16.441(3), b = 8.913(1), c = 6.834(1) Å, α = 101.77(1), β = 91.06(1) and γ = 102.29(1)°; at 230 K: a = 16.425(5), b = 8.863(3), c = 13.539(5) Å, α = 101.62(3), β = 90.73(3) and γ = 102.54(3)° and at 120 K: a = 16.401(3), b = 8.810(2), c = 13.417(3) Å, α = 101.60(2), β = 90.62(2) and γ = 102.76(2)°. The doubling of the c-axis length on passing from the room temperature phase to the phase at 230 K, gives rise to two symmetry independent D:A complexes per asymmetric unit. The TCPA molecules display two fold orientational disorder in the room temperature phase, which persists in the 257-194 K phase. In this phase, the ·D:A:D:A· stacks contain symmetry nonequivalent adjacent D:A units. Upon crossing the second phase boundary (T = 194 K), the TCPA molecules reorient to give an ordered structure.

05.1-24
STRUCTURAL PHASE TRANSITIONS IN CHLOROALKYLAMMONIUM-METALLATES. By H. Fuess, I. Pabst, M. Körfer, H. Ben Ghozlen and M. Czjzek, Institut für Kristallographie, University Frankfurt, Federal Republic of Germany.

Several chloroalkylammoniummetallates of di-, tri-, and tetravalent metals have been investigated by diffraction and spectroscopy.
Structural phase transitions were detected in $[(C_2H_5)_2NH_2]_2SnCl_6$ ($P2_1 \overset{330K}{\rightleftharpoons} P2_1/n$) and $[(C_2H_5)_4N]_2SnCl_6$ ($C2/c \overset{264K}{\rightleftharpoons} P2_1/c$). The octahedral coordination of the Sn-ions is not affected by the change in crystal structure.
A continuous transition previously described in the literature could not be confirmed for $(CH_3NH_3)_2CuCl_4$ which is in fact monoclinic throughout the temperature range 100 < T < 348K but presents a transition to an orthorhombic phase at T_t = 348K. The high temperature phase is characterized by orthorhombic distorted $CuCl_6$ octahedra due to the Jahn-Teller-effect of Cu-ions. The ferro/paraelectric phase transition in $CH_3NH_3HgCl_3$ at 61°C has been studies extensively. It has been shown that the $CH_3NH_3^{\oplus}$ group rotates almost freely in the high temperature monoclinic structure (space group C2) whereas these dipoles form an angle with the threefold axis in the trigonal ferroelectric phase (sp. gr. $P3_2$). D-NMR-measurements on single crystals between 80K and 340 showed that the angle between the long axis of the $CH_3NH_3^+$-group and the polar threefold axis is gradually increasing.
Inelastic and quasielastic neutron scattering gave some evidence of a jump rotation model of the CH_3-and NH_3-groups around the C-N-axis in the ferroelectric phase.
The compound $(CH_3NH_3)_4YbCl_7$ is the first in this group of compounds with a rate earth ion. The structure consists of isolated $[YbCl_6]^{3-}$octahedra in space group C2 and one Cl-ion is connected to the ammonium group via hydrogen bonds. The compound is paramagnetic down to 5K.

05.1-25 CHARACTERIZATION OF THE PHASE TYPES OF A SERIES OF PERALKANOYLATED GLUCOPYRANOSIDES: CHIRAL, NONAROMATIC, DISK SHAPED MOLECULES WHICH FORM COLUMNAR PHASES. By Nancy L. Morris, Richard G. Weiss, and Geoffrey B. Jameson, Department of Chemistry, Georgetown University, Washington, D.C. 20057.

Samples of peralkanoylated α- and β-glucopyranosides with alkanoyl chains from C_{10} to C_{18} were investigated by X-ray diffractometry to characterize their thermotropic liquid crystalline phase types. Various non-hexagonal columnar phases, D_{rd} and D_{ro} in Destrade's terminology, (C. Destrade, P. Foucher, H. Gasparoux, Nguyen Huu Tinh, A.-M. Levelut, and J. Malthete, Mol. Cryst. Liq. Cryst., 106 (1984) 121-146) and a nematic phase can be prepared depending on the phase preparation method. A preliminary photographic study of an α-C_{16} oriented phase on a glass slide showed a second order ring at high angle (d_{002} = 4.2Å) corresponding to stacking of disks. Intercolumnar spacings inferred from low angle Bragg peaks increase regularly in a homologous series except for α-C_{10} and α-C_{11}, which are anomalous. The α-series increases at 2.67(15)Å/CH_2 and the β-series at 2.17(7)Å/CH_2. The predicted core radius from the intercept of plots of chain length vs. intercolumnar distance is 10.0(9)Å for the β-series and 0.0(7) for the α-series, neglecting α-C_{10} and α-C_{11}. Thus, it has been shown for the first time that chiral molecules with no aromatic core can produce oriented, columnar phases of both ordered and disordered types and that α-anomers of the series investigated may also have a nematic phase. Additionally, the columnar background structure is retained throughout the D_{rd}, D_{ro}, and crystalline phases.