

Synthesis and Characterization of New Thermotropic Monomers - Containing Phosphorus

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Abstract : New phosphorus-containing monomers having methoxy end functional groups were prepared from methyl 4-hydroxybenzoate and two different dichlorides with phosphorus, namely phenyl phosphonic dichloride and phenyl dichlorophosphate. The structures of the monomers were confirmed by FTIR and NMR spectroscopy. The assignments for the ^1H , ^{13}C and ^{31}P chemical shifts are based on 1D and 2D NMR homo- and heteronuclear correlations (^1H , ^1H -COSY (Correlation Spectroscopy), ^1H , ^{13}C -HMQC (Heteronuclear Multiple Quantum Correlation and ^1H , ^{13}C -HMBC (Heteronuclear Multiple Bond Correlation)) and ^{31}P - ^{13}C couplings. The monomers exhibited good solubility in common organic solvents. Dimethyl sulfoxide was to be a good solvent to grow crystals of considerable size which were investigated by X-ray analysis. One of these two new monomers presented thermotropic liquid crystalline behaviour, as revealed by differential scanning calorimetry (DSC), polarized light microscopy (PLM) and X-ray diffraction (XRD). The transition temperature from crystal to liquid crystalline state (K \rightarrow LC) was 143°C and from the LC to isotropic state (LC \rightarrow I) was 167°C. Upon heating, bis(4-(methoxycarbonyl)phenyl) formed fine textures, difficult to be ascribed to smectic or nematic phases. Upon cooling from the isotropic state, bis(4-(methoxycarbonyl)phenyl) exhibited a mosaic-type texture. X-ray diffraction measurements at small angles (SAXS) of bis(4-(methoxycarbonyl)phenyl) showed two peaks at 1.8 Å and 3.5 Å, respectively suggesting organization at supramolecular level.

Keywords : phosphorus-containing monomers, polarized light microscopy, structure investigation, thermotropic liquid crystalline properties

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