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Entitled

PREPARATION AND CHARACTERIZATION OF NOVEL AZO-CINNAMATES AND STEROIDAL AZO ETHERS

by

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Abstract

This thesis is concerned with the synthesis of novel azo-cinnamates and of steroidal azo ethers and with the analysis of the spectroscopic and thermal properties of these materials. The compounds were identified as possible photoswitchable liquid crystals. The molecules were prepared by multi-step sequences, involving etherification, azo coupling and Appel-type reactions as the key steps in the case of the azo-cinnamates, and Finkelstejn reactions and Williamson-type etherification reactions as the major transformations in the case of the steroidal azo ethers. The preparation of the azo-cinnamates was a further example of an esterification reaction run under Appel-type conditions using $BrCCl_3/PPh_3$. The azo-cinnamates were shown to be photoswitchable under photoirradiation at λ = 350 nm, where the thermally stable trans-azo compounds isomerized photochemically to the cis-isomers. Subsequently, the energetically less stable cis-isomers isomerized thermally back to the trans-isomers. The azo-cinnamates were found to exhibit thermotropic liquid crystalline behavior as evidenced by differential scanning calorimetric analysis and by optical texture analysis under the polarization microscope of selected samples. Azocinnamates have been found to exhibit liquid crystalline behavior before, however, it is commonly accepted that in systems containing three (or more) aromatic rings, in the compounds that exhibit liquid crystalline (eg., nematic) behavior, the ring systems normally are positioned in close proximity to each other, preferably with less flexible linkers connecting them. In the presently studied compounds, however, the cinnamate unit is separated from the azobenzene substructure by flexible C9- and C11-alkyl chains, and so it is exciting to see that also these compounds exhibit (albeit in a narrow temperature range) thermotropic behavior.

Keywords: Azo cinnamates, steroidal azo compounds, photoswitching, liquid crystals, single X-ray crystal structure.