## Abstract

This work is divided into two parts that are focused on the synthesis and assessment of photophysical properties of two structurally related compounds: (i) 9,9'-spirobifluorenes (SBFs) and (ii) dispiroindeno[2,1-*c*]fluorenes (DS-IFs).

(i) In the first part, a five-step synthetic approach to SBFs was developed and as the crucial step was used an intermolecular [2+2+2] cyclotrimerization of symmetrically or unsymmetrically substituted diynols with alkynes catalyzed by Rh-complexes. Catalyst screening showed that Wilkinson's catalyst (RhCl(PPh<sub>3</sub>)<sub>3</sub>) had the highest efficiency in yielding 1,2,3,4-substituted fluorenols – the key intermediates. The fluorenols were then converted into SBFs bearing various electron-donating and -withdrawing groups, aromatic substituents, and  $\pi$ -extended aromatic hydrocarbon moieties (PAHs). Altogether 19 different SBFs were prepared and their photophysical properties screened. The fluorescent emission maxima  $\lambda_{em}$  were in the range of 315-389 nm with excellent quantum yields  $\Phi_s$  (up to 1.00). As far as the substituent effect is concerned the presence of electron-withdrawing substituents on the SBF scaffold results in the red-shift of the emission maxima.

(ii) In the second part, a similar synthetic strategy was applied for synthesis DS-IFs. In this instance was utilized again the Rh-complex catalyzed intramolecular [2+2+2] cyclotrimerization of triyndiols, which provided indeno[2,1-*c*]fluorenr-5,8-diols in high yields. These were then converted into the corresponding DS-IFs. In total, 9 symmetrically substituted DS-IFs at positions 6 and 7 and 2 unsymmetrically substituted DS-IFs at positions 3 and 10 were synthesized. Furthermore, to demonstrate the potential of this synthetic approach, three [7]-helical DS-IFs were prepared. Photophysical properties of all compounds were evaluated as well. Their fluorescent emission maxima  $\lambda_{em}$  were in the range of 351-428 nm with excellent quantum yields  $\Phi_s$  (up to 0.80). [7]-helical DS-IFs have the highest measured quantum yields among the class of helicene like compounds recorded thus far.