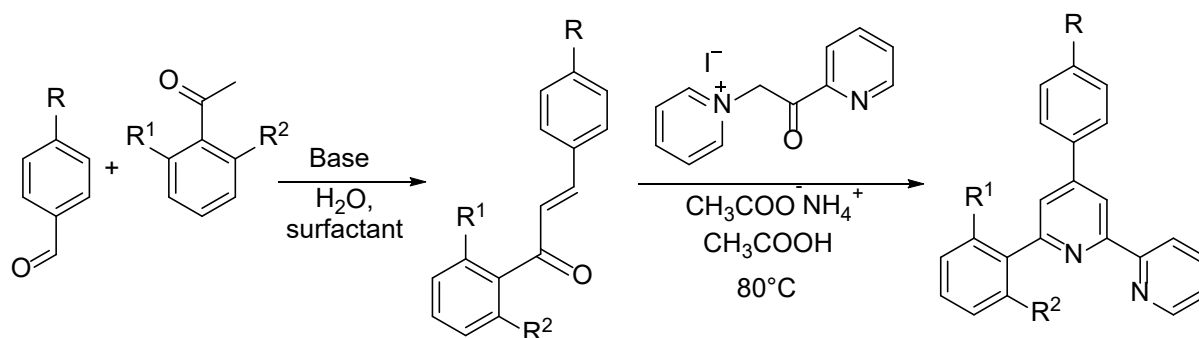


## Micellar Assisted Synthesis of Chalcones as Intermediates for Bipyridines

P. Quagliotto, M. M. H. Desoky, N. Barbero, C. Barolo, G. Viscardi

Dipartimento di Chimica e Centro NIS "Superfici ed Interfacce Nanostrutturate", via P. Giuria 7, Torino  
 pierluigi.quagliotto@unito.it

Chalcones are well known molecules, having several applications as intermediates and drugs.<sup>1</sup> They are often easily obtained by performing aldol reaction in solvents, with good yields. They are essential intermediates for the Kröhnke reaction, which give access to pyridines, bipyridines and terpyridines.<sup>2</sup> The bipyridine structural motif is often exploited as a ligand to form metal complexes which showed electrochemical CO<sub>2</sub> reduction activity.<sup>3</sup> In order to produce chalcones for the preparation of bipyridine ligands, we tried a green chemistry approach. While the solvents used for this reaction are in general mild (often alcohols are used), the use of water by surfactant assistance is an attractive alternative, which is not new but in the last few years it reached important results from the preparative point of view.<sup>4</sup> The aldol reaction was studied and applied to the synthesis of a few chalcones, useful for the preparation of bipyridine ligands. Several surfactants were used and among them, PTS, Nok and TPGS-750-M are truly green surfactants.<sup>4</sup> The use of surfactants shortened the reaction time and increased the yield. Whether the chalcone is sufficiently soluble in the micellar medium, the reaction can proceed with a Michael addition on the chalcone. The chalcone was easily isolated by filtration when it was slightly soluble in the micellar solution. Reactions performed in presence of a cosolvent (10% ethanol), showed an increased yield and gave pure solids by simple filtration (yields: 76-91%). The scope of the reaction was studied, and also the less deactivated substrates reacted easily. The chalcones were used to prepare some bipyridine derivatives, with Kröhnke reaction, under both standard conditions (acetic acid and ammonium acetate at 80°C) and an original and green way, performed in water.



**Figure 1:** Synthetic Pathway to chalcones in surfactant medium and to bipyridines by Krohnke reaction.

### References:

- [1] M. N. Gomes, E. N. Muratov, M. Pereira, J. C. Peixoto, L. P. Rosseto, P. V. L. Cravo, C. H. Andrade, B. J. Neves, *Molecules* **2017**, *22* (8), 1210.  
 [2] I. Sasaki, *Synthesis* **2016**, *48* (13), 1974-1992.  
 [3] a) C. Sun, R. Gobetto, C. Nervi, *New J. Chem.* **2016**, *40* (7), 5656-5661; b) C. Sun, S. Prosperini, P. Quagliotto, G. Viscardi, S. S. Yoon, R. Gobetto, C. Nervi, *Dalton Trans.* **2016**, *45* (37), 14678-14688.  
 [4] B. H. Lipshutz, *J. Org. Chem.* **2017**, *82* (6), 2806-2816.