

SYNTHESIS & CATALYSIS

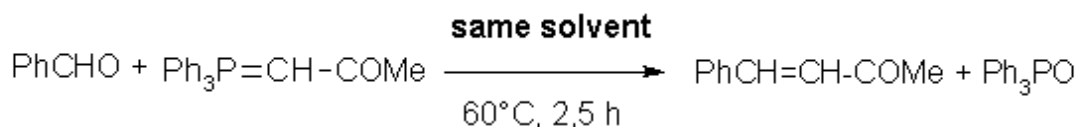
Wittig reaction

Wittig reaction is one of the most popular methods for the creation of C=C links, leading to good yields and good selectivities. However, the generation of the secondary product Ph_3PO is a problem in traditional solvents (separation by crystallization)

The use of the 1-Butyl-3-methylimidazolium tetrafluoroborate $[\text{Bmim}][\text{BF}_4]$ ionic liquid solves this problem¹. At the end of the reaction, the alkene is extracted with diethyl ether and Ph_3PO is extracted with toluene. This method makes it possible firstly to isolate the pure alkene with good yields (>80%), and secondly to recycle the ionic liquid used in a Wittig's reaction with another aldehyde (cf. diagram)

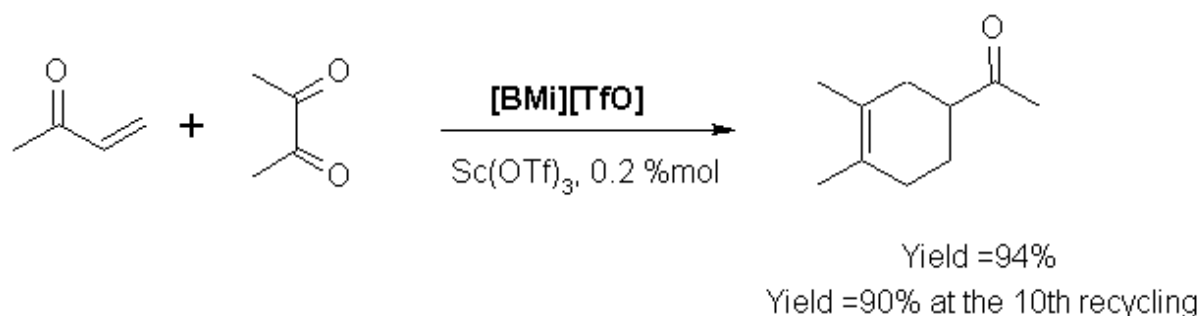


then



Diels-Alder reaction

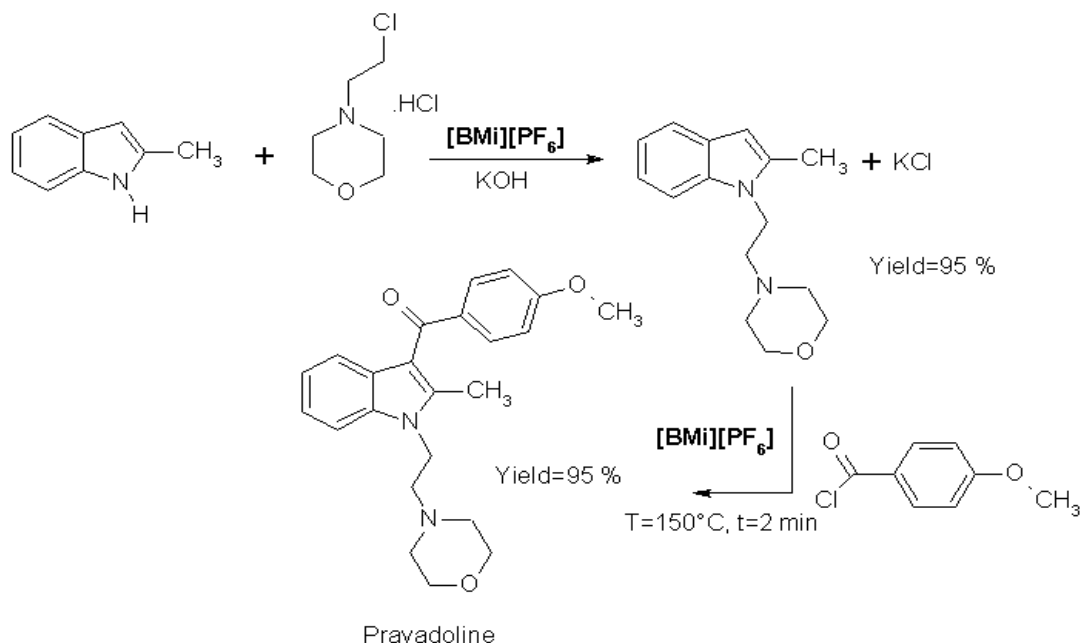
Diels-Alder is commonly used in organic chemistry to create a C-C link. This reaction may be catalyzed by scandium triflate which, when immobilized in the 1-Butyl-3-methylimidazolium trifluoromethanesulfonate $[\text{Bmim}][\text{CF}_3\text{SO}_3]$, ionic liquid, speeds up the reaction².



The reaction is completed in 2 hours at 20°C / 68°F with only 0.2% mol of catalyst (instead of 10% mol in traditional organic solvents). The product from the reaction may be extracted with ether, and the catalyst may be recycled without any activity loss (90% yield after the 10th recycling process).

Synthesis of the pravadoline³

Only one solvent for both steps : the ionic liquid 1-butyl-3-methylimidazolium hexafluorophosphate [Bmi][PF₆].



First step :

- Extraction with toluene at the end of the first step, the ionic liquid is then washed with water and dried under vacuum (few residual wastes (water + KCL) before being engaged in the next step.
- The reaction does not require heat supply.

At this step, the product is extracted from the solvent to know the yield and the selectivity of the reaction. These treatments are unnecessary in so far as, after the water washings, the ionic liquid can simply be dried under vacuum taking care of not distilling the product by checking the vacuum or the temperature.

Second step : (Friedel-Craft acylation)

- No need for catalyst, which is usually required in organic reactions
- Easy extraction of the product with toluene (the extraction of the product could also be carried out by scCO₂)
- Ionic liquid recycling.

REFERENCES	IONIC LIQUIDS
IM0403C	1-Butyl-3-methylimidazolium tetrafluoroborate , 98%
IM0205B	1-Butyl-3-methylimidazolium trifluoromethanesulfonate , 99.5%
IM0404C	1-Butyl-3-methylimidazolium hexafluorophosphate, 98%

¹ V. Le Boulaire, R. Grée Chem. Commun., 2000, 2195.

² C. E. Song, W. H. Shim, E. J. Roh, S. Lee, J. H. Choi Chem. Commun., 2001, 1122.

³ M. J. Earle, P. B. McCormac, K. R. Seddon Green Chem., 2000, 261