

CORNING

Advanced-Flow™ Reactors



Controlling a Reaction: Mixing, Residence Time and Temperature

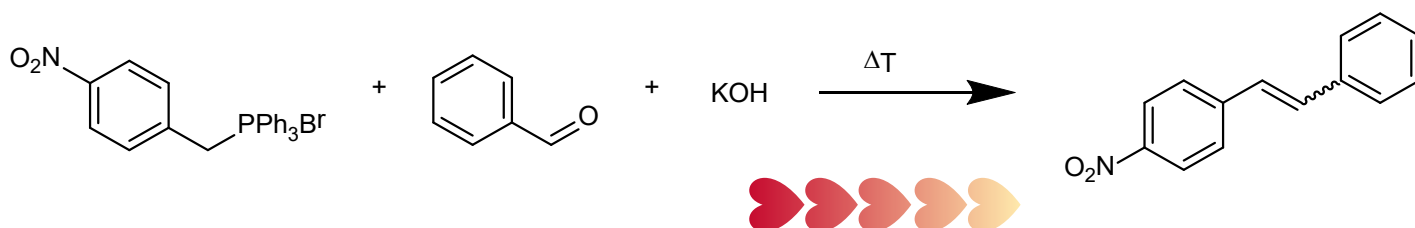
Application Note #6

Issued: February 2018

Setup: Corning® Lab Reactor with one module

Model Reaction: Wittig reaction

Adapted from: Analyst, 2001, 126, 7–10



Analytcs: Human eye

Safety:

Make sure you have read the MSDS of the chemicals and the safety notes in the Lab Reactor Manual.

Feed Preparation:

- Feed 1: 956 mg (2 mmol) 4-Nitrobenzyltriphenylphosphonium bromide (CAS 2767-70-6) are dissolved in 200 ml dichloromethane, 254 mg (2.4 mmol) of benzaldehyde (CAS 100-52-7) are added to the solution.
- Feed 2: 224 mg (4 mmol) KOH are dissolved in 200 ml water

Flow experiment:

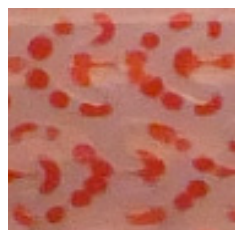
The solutions are pumped with similar flow rates (e.g. 1 ml/min per pump) through the module. The flow rates and temperature can be varied in order to optimize conversion.

Hint: Pressurize the backpressure regulator and start with 50°C ,

Cleaning: Replace both feed solutions with Ethanol and pump @ 1 ml/min for at least 20 min

Results:

Red color disappears as reaction proceeds through mixing cells. Depending on your flow rates, residence time and temperature you will obtain more or less HEART cells with droplets of various sizes.



0.5/0.5 ml/min



1/1 ml/min



2/2 ml/min



70°C

Conclusion:

Working with biphasic solutions is complex. You have to know the impact of the mass transfer on your reaction kinetics. Reducing the flow rates in order to get complete conversion might not work, since it will give you less good mixing (less mass transfer) and therefore, the conversion might not go down despite having more reaction time. A solution is to increase temperature instead of lowering the flow rates in order to accelerate the reaction.

Tips & Tricks

This reaction is an extremely good demonstration reaction to showcase flow chemistry.

- @ 50° C 1/1 ml/min reaction not complete
- @ 50° C 0.5/0.5 ml/min reaction is not complete, mixing is not so good droplets can be seen
- @ 70° C 1/1 ml/min reaction is complete at the end of the module
- @ 80° C 1/1 ml/min reaction is over after 2-3 HEART cells