

CORNING

Advanced-Flow™ Reactors



Controlling Reaction Time and Temperature

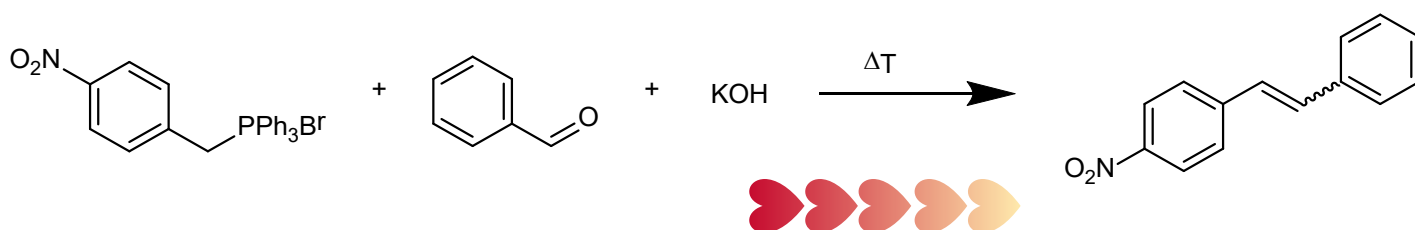
Application Note #1

Issued: February 2018

Setup: Corning® Lab Reactor with one module

Model Reaction: Wittig reaction

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



Analytics:

Human eye (optional online UVvis Spectroscopy)

Safety:

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

Feed Preparation:

- Feed 1: 478 mg (1 mmol) 4-Nitrobenzyltriphenylphosphonium bromide (CAS 2767-70-6) are dissolved in 100 ml Ethanol. 127 mg (1.2 mmol) of benzaldehyde (CAS 100-52-7) are added to the solution.
- Feed 2: 112 mg (2 mmol) KOH are dissolved in 100 ml Ethanol (Dissolution takes some time ~1 h).

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: When changing the temperature, wait until the measured temperature is stable before taking a sample.

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

Tips & Tricks: How to calculate residence time and flow rates.

$$\text{Residence time} = \frac{\text{Reactor volume} \times 60\text{s/min}}{\text{Flow rate}_{\text{total}}}$$

$$\text{Flow rate}_{\text{total}} = \frac{\text{Reactor volume} \times 60\text{s/min}}{\text{Residence time}}$$

Flow rate_{total} = Flow rate Pump 1 (ml/min) + Flow rate Pump 2 (ml/min)

Reactor volume = 1 module = 2.7 ml

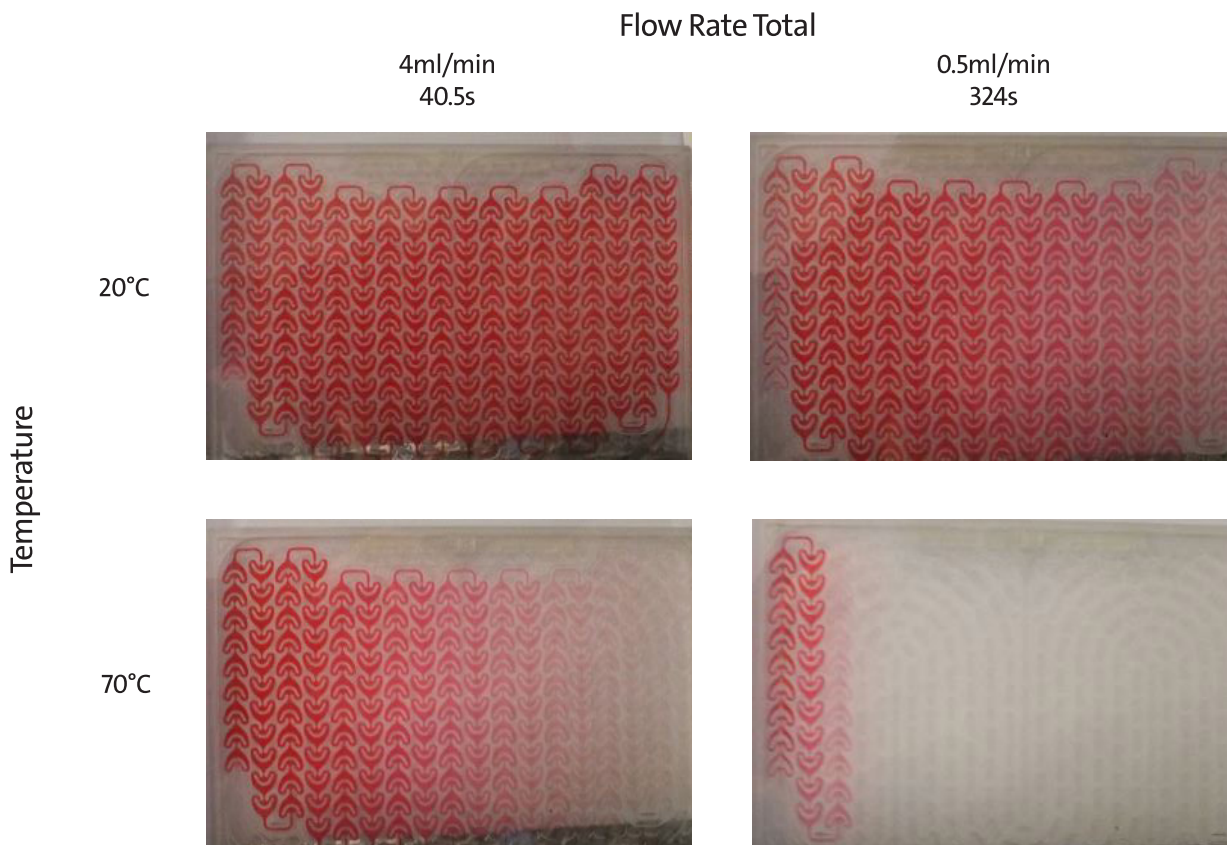
Residence time = time of the liquid from entrance to exit

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Results:

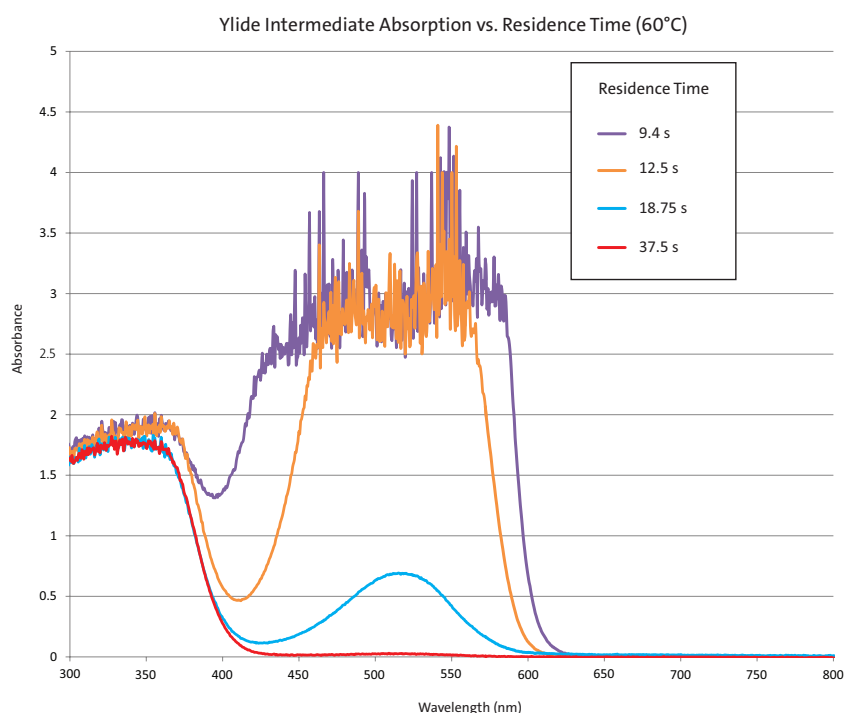
Red color disappears as reaction proceeds through mixing cells. Depending on your flow rates and temperature you will obtain more or less HEART cells with a red color.



Conclusion:

Yields/Conversion are controlled via two parameters: temperature and time.

Time is controlled via the flow rates of the pump. Normally the target should be the highest yield with the shortest reaction time.



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