CORNING

Advanced-Flow™ Reactors

Liquid/Liquid Reaction

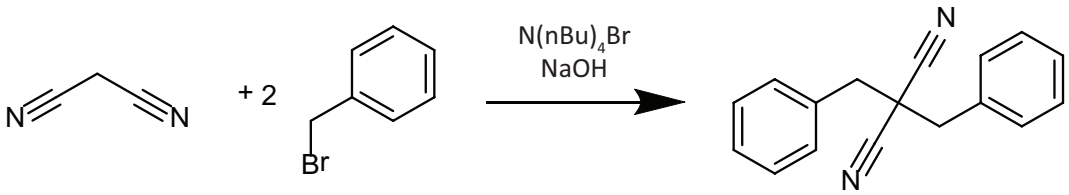
Application Note #5

Issued: February 2018

Setup: Corning® Lab Reactor with one module

Model Reaction: Alkylation of Malonitrile

Adapted to flow from Chemometr. Intell. Lab ,1990, 9, 287-292 and Chem. Commun. 2003, 936–937



Analytics: Conversion by ^{1}H-NMR or GC

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

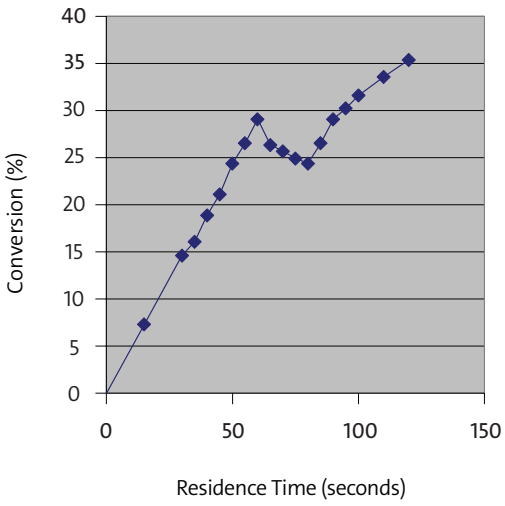
Feed Preparation:

- Feed 1: 0.991 g (0.15 mol) of malonitrile and 5.13 g (0.3 mol) of benzyl bromide (CAS 100-39-0) are dissolved in 100 ml dichloromethane.
- Feed 2: 1.8 g (0.45 mol) of NaOH and 0.121 g (2.5 mol%) of tetrabutyl ammonium bromide are dissolved in water

Flow experiment:
The chiller is set to 25°C. The solutions are pumped with similar flow rates (e.g. 1 ml/min per pump) through the module. The analytic samples are quenched with HCl (1N). The organic phase is separated and dried over Calcium Chloride. Alternatively a phase separator can be used.

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

Results:
This biphasic reaction is sensitive to mixing. The drop in conversion at 60 s is related to less effective mixing, which is at lower flow rates compensated by longer reaction times.



Conclusion:
Biphasic reactions benefit from mixing by the HEART design, make sure you work with the right flow rates.

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Tips & Tricks

You can easily qualitatively monitor the mixing by looking at the reactor outlet. Large slugs are an indicator for less good mixing while short slugs show good mixing. Very good mixing will lead to slugs which are still slightly turbid and not transparent at the reactor exit.

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