CORNING

Advanced-Flow™ Reactors

Photochemistry

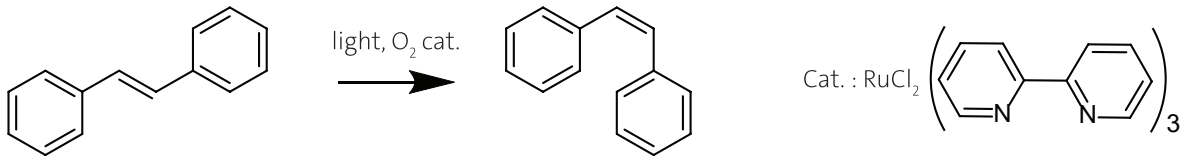
Application Note #3

Issued: February 2018

Setup: Corning® Lab Reactor with photo module

Model Reaction: Isomerization of stilbene

Adapted from Chem. Eur. J. 2015, 21, 5350 – 5354



Analytics: GC or ^{1}H-NMR

Safety:

Make sure you have read the MSDS of the chemicals and the safety notes in the Lab Reactor Manual. Collect the liquid at the reactor outlet in inert gas purged container. Risk of oxygen accumulation.

Feed Preparation:

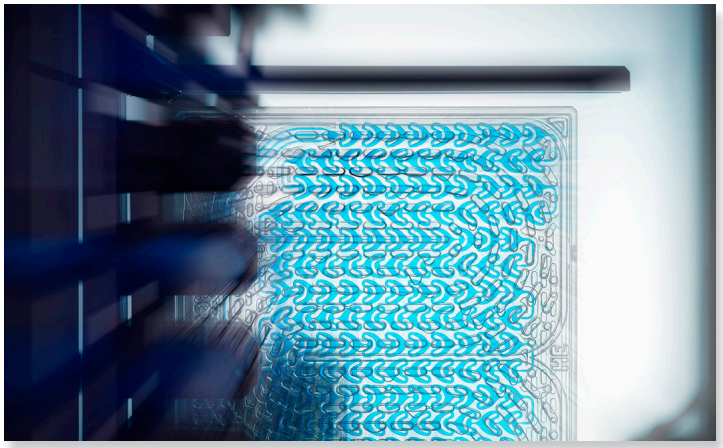
- Feed 1: 1.8 g (10 mmol) trans stilbene (CAS 103-30-0) and 224 mg (3 mol%) Ru-bipy (CAS 50525-27-4) are dissolved in 100 ml acetonitrile.
- Feed 2: Oxygen gas is connected to the gas mass flow controller.

Flow experiment:

The back pressure is regulated to 4 bar, Oxygen flow to 1 ml/min. The liquid is pumped with various flow rates through the photo module. The wavelength and power of the light can be varied.

Hint: Gas is compressible, so it will take a while for the system to stabilize. Start the gas flow first.

Cleaning: Stop the gas flow, switch off light. Replace feed solution with pure acetonitrile and pump @ 1 ml/min for at least 20 min.



Results:
Depending on flow rates, wavelength and light power different yields for cis stilbene will be achieved.

Light (nm)	365	385	405	485	485	485	485	485	white
Power (%)	100	100	100	10	50	100	100	100	100
Flow Rate (ml/min)	2	2	2	2	2	2	1.5	1	2
Yield (%)	23	29	50	6	25	52	60	83	7

Conclusion:
The AFR photo reactor provides more control over the photochemistry than a classic batch mercury lamp setup. The wavelength and power can be adjusted to the chemistry.

Tips & Tricks

Using LED in photochemistry requires knowledge of the absorption spectra of the starting material. A better overlap provides the best yields.

Example: The overlap of Ru-Bipy with different LEDs. 485 nm will be clearly a better choice than 365 nm.