CORNING

Advanced-Flow™ Reactors

Gas/Liquid Reaction

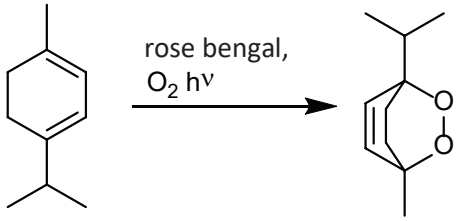
Application Note #2

Issued: February 2018

Setup: Corning® Lab Reactor with photo module

Model Reaction: Oxidation of *a*-terpinene

Org. Process Res. Dev., 2017, 21 (9), pp 1435–1438



Analytics: ^{1}H-NMR

Safety:

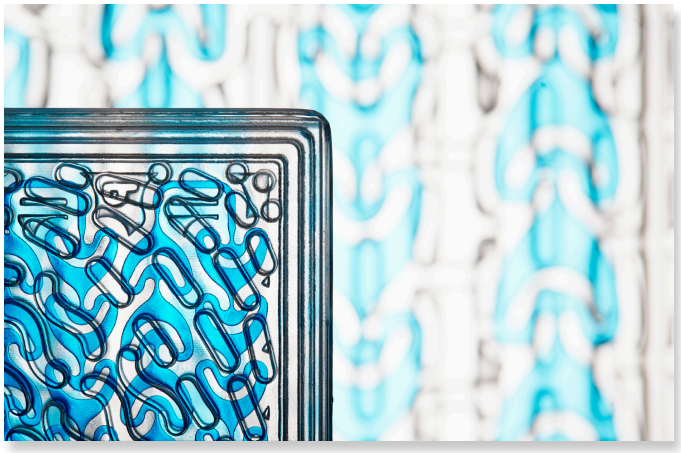
Make sure you have read the MSDS of the chemicals and the safety notes in the Lab Reactor Manual. The solution collected at the reactor outlet must be purged with an inert gas. Risk of oxygen accumulation.

Feed Preparation:

- Feed 1: 3.41 g (0.25 mol) alpha-terpinene (CAS 99-86-5) and 250 mg (1 mol%) rose bengal (CAS 632-69-9) are dissolved in 100 ml methanol.
- Feed 2: Oxygen gas connected to the gas inlet of the dosing module.

Flow experiment:

The 4000K LED is switched on. The back pressure regulator (BPR) is regulated to 5 bar. Switch chiller to 20 °C.
The oxygen flow is regulated to 10 ml/min (0.44 mmol/min) Feed 1 is pumped with various flow rates.
The oxygen feed is varied to find the best condition.
Hint: When working with a gas, if possible start the gas first, as it will take while before the system is stable as gas is compressible.
Cleaning: Switch off gas and light and rinse with pure methanol for 20 min, keep the back pressure regulator at 5 bar.



Results:
To obtain the best results for a Gas/Liquid reaction, pressure and flow rates should be screened.

Flow Rate L (ml/min)	Flow Rate G (ml/min)	Pressure (Barg)	Time (s)	Yield (%)	Gas Bubbles (BPR) before	Gas Bubbles (BPR) after
2	5	5	57	56	No	No
2	10	5	44	Quantitative	No	Some
2	20	5	30	Quantitative	Yes	Yes
4	20	5	22	60	Some	Yes
4	20	2	15	47	Yes	Yes
4	20	0	7	41	Slugs	Yes

Conclusion:
Gas handling in flow differs a lot from batch. It is a mass flow controlled feed so a precise dosage is possible . Pressure is a very important factor as it impacts several parameters like the solubility and residence time.

Tips & Tricks

An exact calculation of the residence time is very difficult when gases are involved. In general the residence time given in the literature is the minimum residence time based on the assumption that no gas is consumed. The min. residence time is calculated as in application note 1. The flow rate for the gas feed can be calculated with the following formula:

$$V_{real\,flow} = \frac{V_{mass\,flow} * 1\,bar * TReactor}{P_{BPR} * 273.15\,K}$$

Example: A reaction at 8 bar, 25 °C and gas flow (mass flow controller setting) 7.5 ml/min has a real gas flow rate of 1 ml/min.

$$V_{real\,flow} = \frac{7.5\,ml * 1\,bar * 298.15\,K}{8\,bar * 273.15\,K}$$