Robinson Brothers
Excellence in Chemistry

STOLI chem

Doubling the yield of a gas-liquid acylation process with intensified continuous reactor - an industrial case study

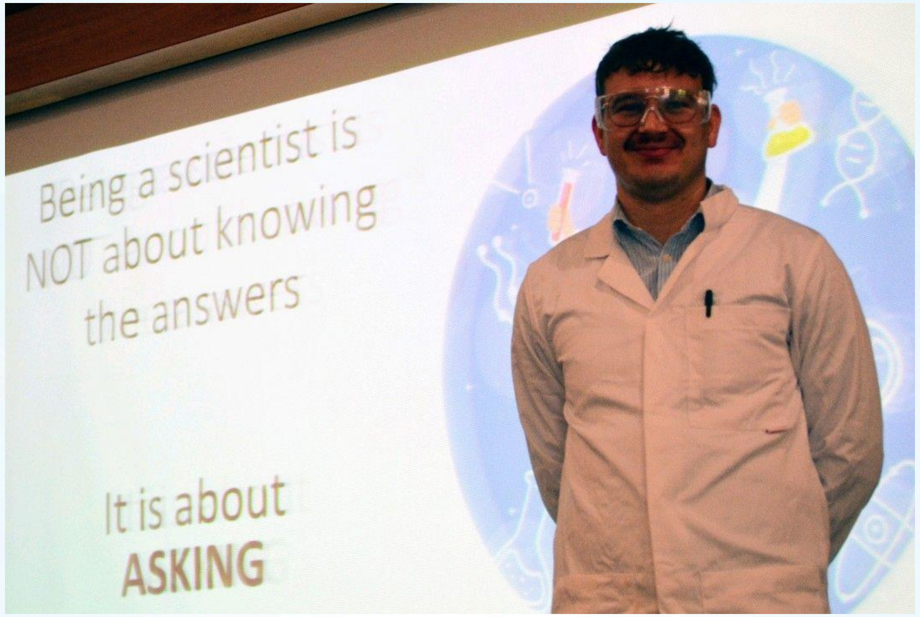
Producing a Valuable Product takes 5 days at only 40% yields

- Fragrance and flavouring product is highly valuable

- Product is sensitive to temperature but reaction is highly exothermic
- High possibility to overreact forming heavier components
- Energy intensive multi-step process

Jonty Thornton

Chemical Engineer, Stoli Chem, UK



Ask me about

- Blues Guitar
- Arsenal
- Chemical Engineering

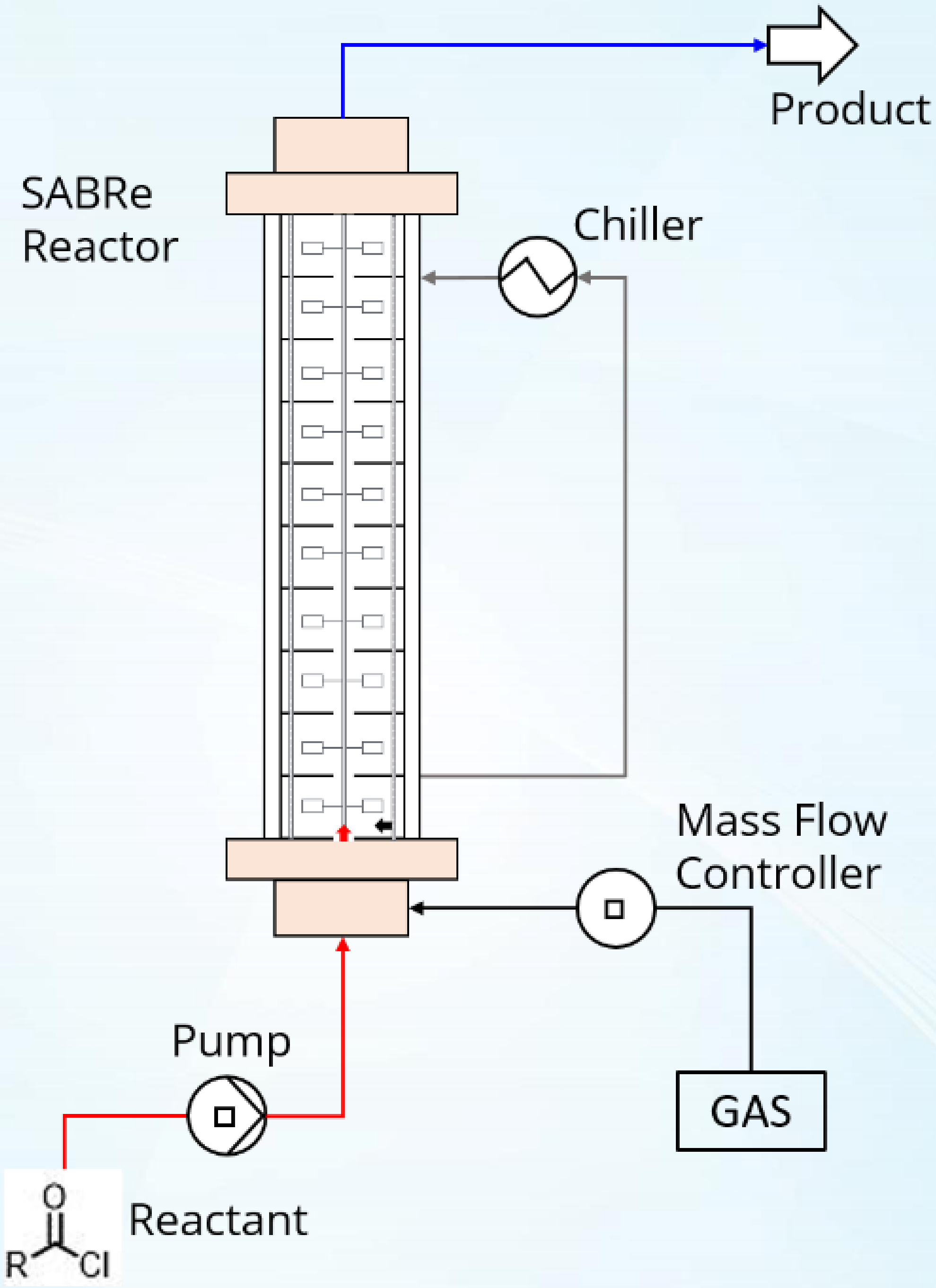
Education and career

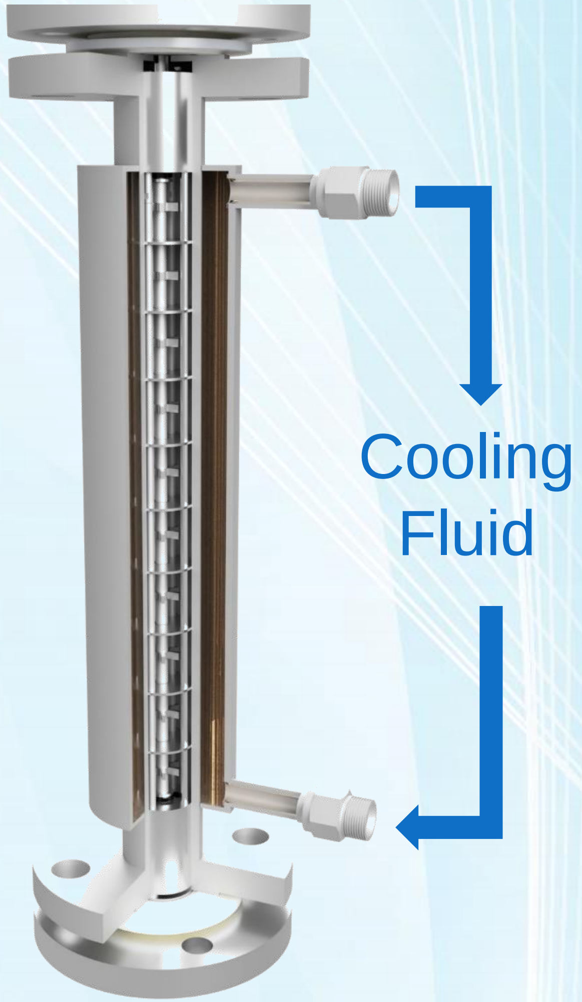
- Galaxy Kayaks – logistics operator
- University of Sheffield (2016-2021)
- Faurecia – Improvements Engineer (2019-2020)
- Quality Teach – Tutor (2021-2022)
- Stoli Chem – Chemical Engineer (2022-2024)

Interest

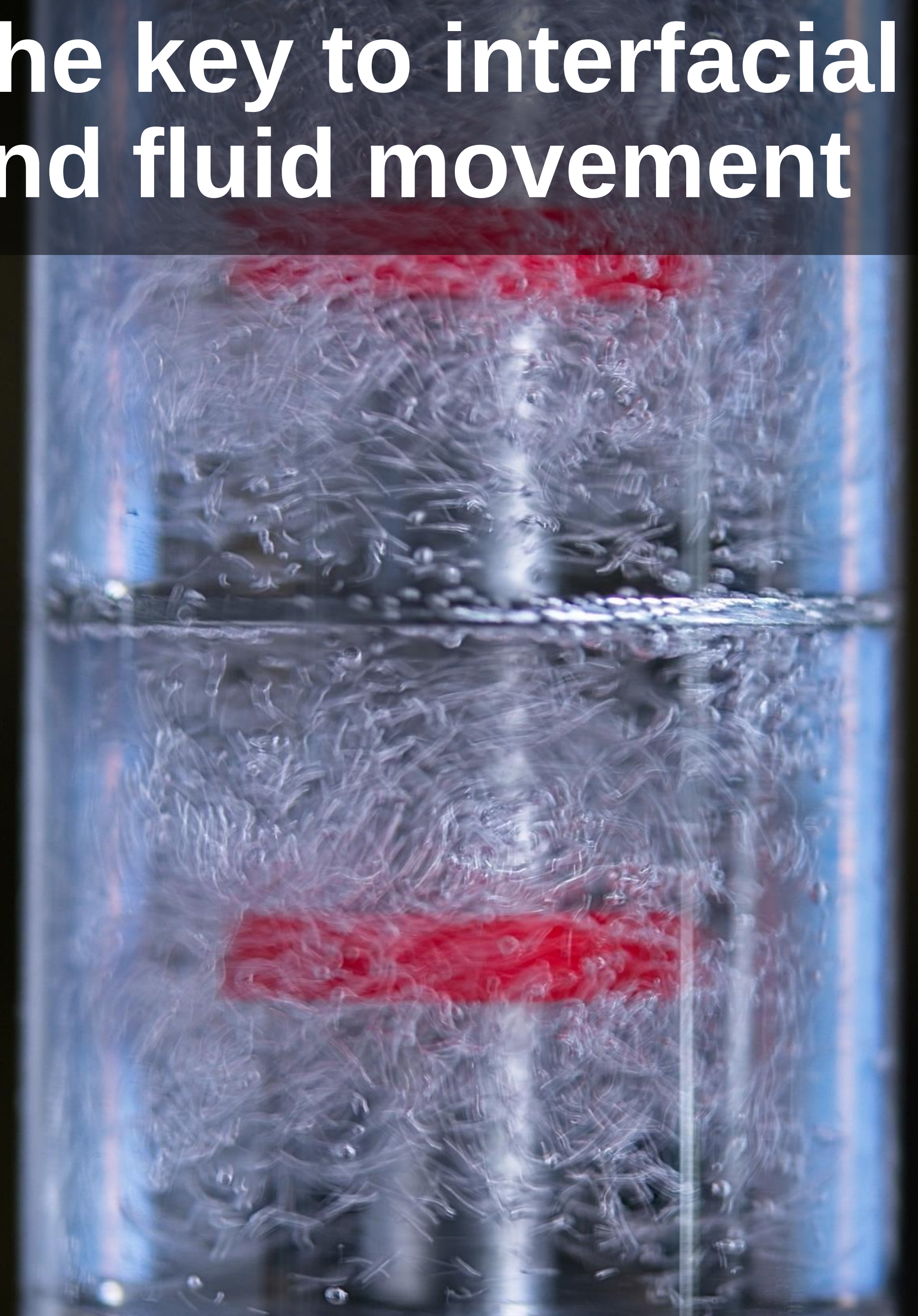
- Sports – football and cricket
- Cooking
- History
- Learning new things (science, music)

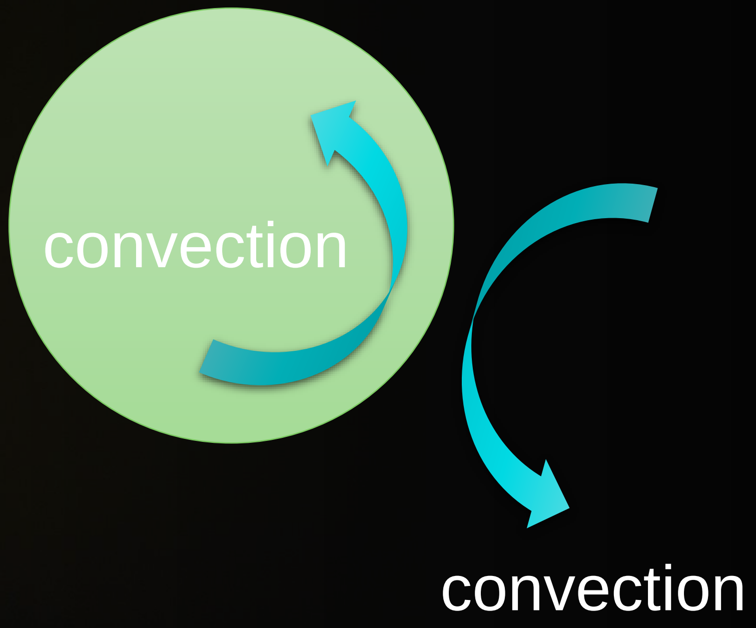
Using lab SABRe system as a potential solution





The key to interfacial mass transfer is large area and fluid movement





$$\text{rate} = k_L a\, \Delta C \qquad \sim \text{mixing energy}$$

Multi-dimensional Process Optimisation

- Mixing Energy
  - How does mixing intensity affect performance?
- Catalyst Amount
  - How much/little catalyst we can utilise to still be profitable, in solution and safe?
- Concentration of reactant in solvent
  - How are reaction rates and downstream purification affected by this?
- Residence time
  - How fast can the reaction get? What size reactor do we need at scale to meet customer demands?

Factors affecting gas concentration

- Gas Ratio
  - Does having a molar excess provide
- Temperature
  - How does varying temperature affect performance? Does gas solubility come into play?
- Pressure
  - What trade off can we afford between high pressure and expensive equipment?

Initial yields of only 20%

- Initial experimentation indicated low valuable product

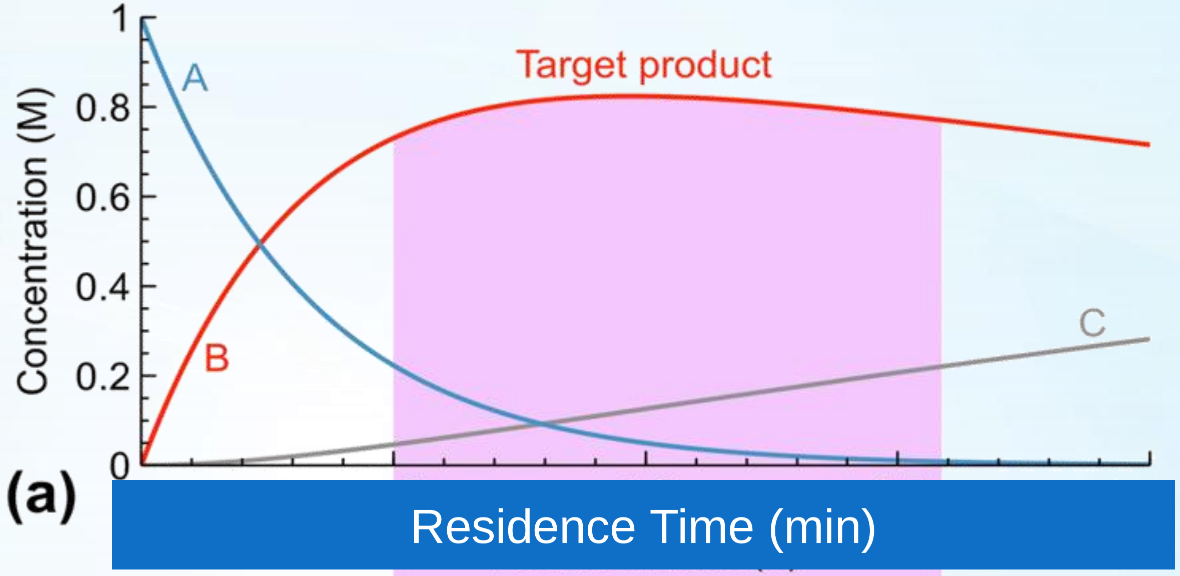
Concentration (wt%)	Residence time (min)	Temperature (oC)	Mixing RPMs	Catalyst Molar Ratio	Molar Ratio of Propylene	Pressure (bar)
8	30	0	300	0.75	1	Ambient

Conversion (%)	Selectivity (%)	Yield (%)
80	37	20

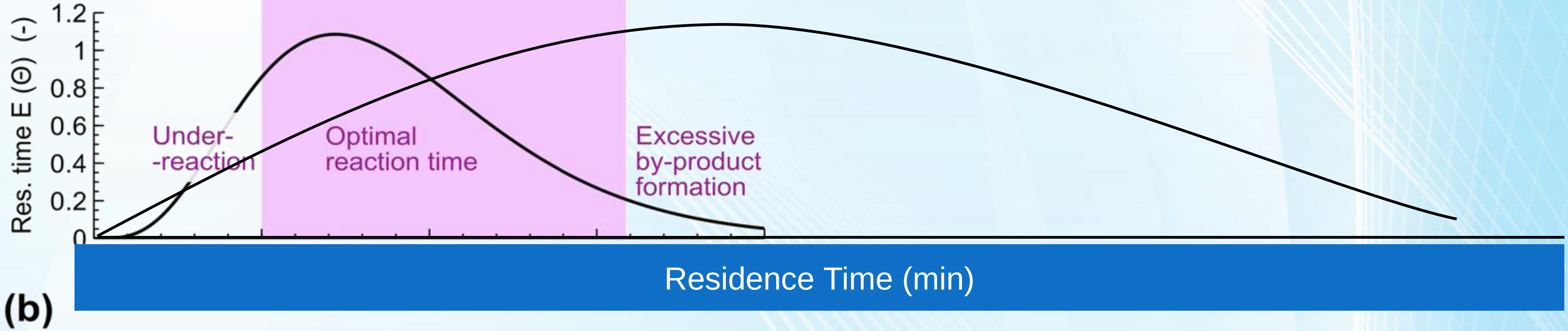
E)-Selective Friedel-Crafts acylation of alkynes to β-chlorovinyl ketones: Defying isomerizations in batch reactions by flow chemistry approaches

Residence time control allows for improved reaction

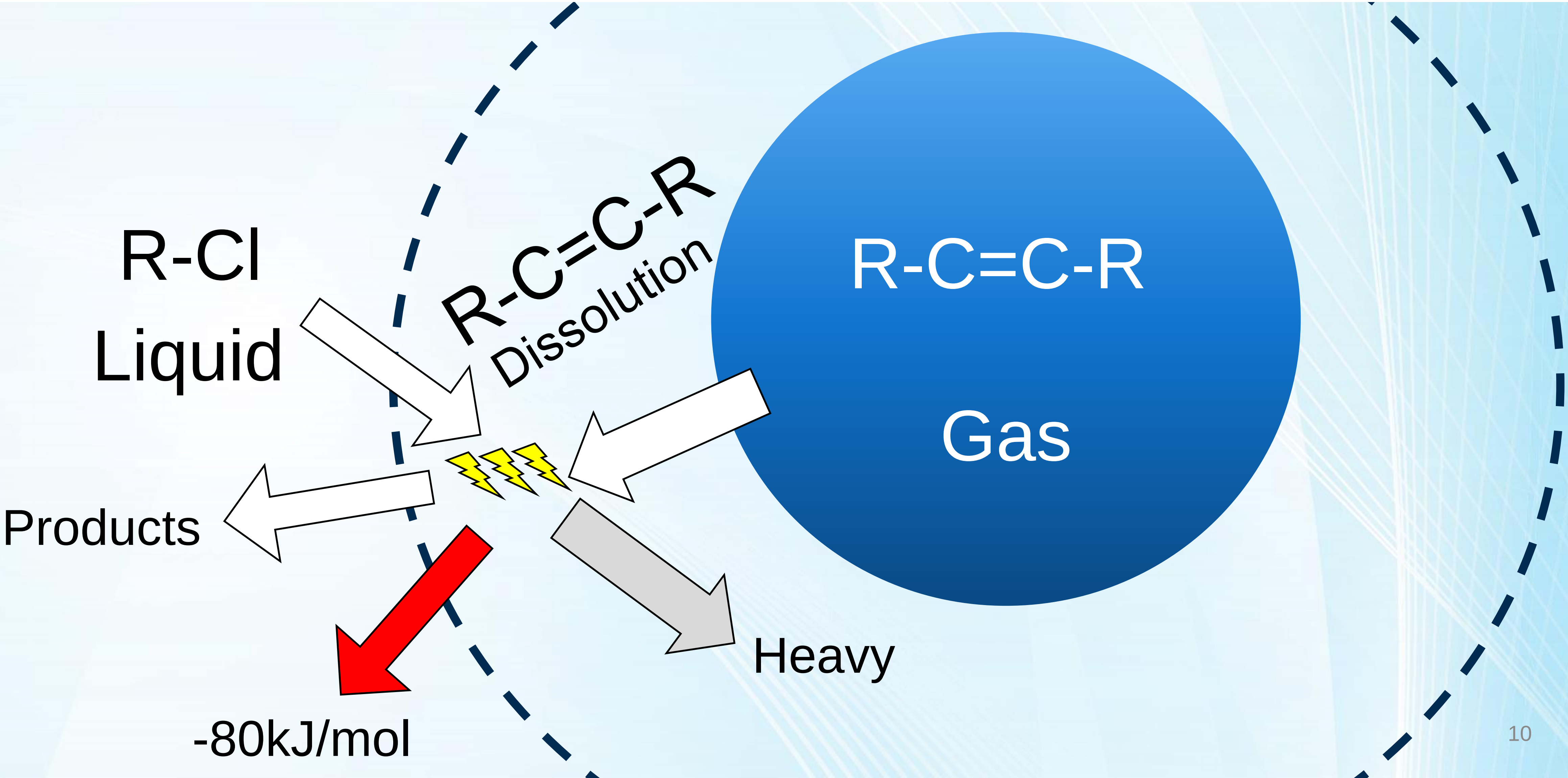
A → Target → C



Conversion (%)	Selectivity (%)	Yield (%)
89	66	59



Mass transfer enables reaction
Heat transfer enables fast residence times



Catalyst amount – an engineering compromise

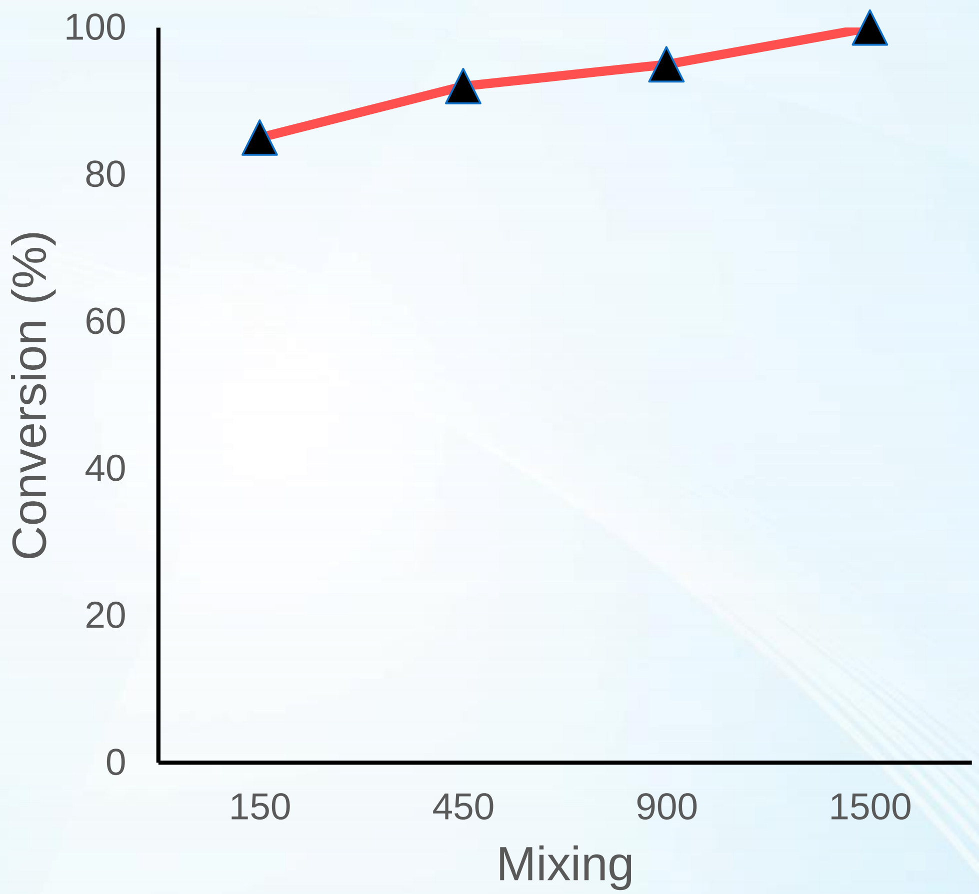
- Gambacorta and co suggests that high stoichiometry amounts typical for reaction.
- 1.2 molar ratio is our cap for catalyst – since solution is saturated
- On testing this out – just like with penicillin, Teflon and Vaseline

Conversion (%)	Selectivity (%)	Yield (%)
99.9	82	82

- More catalyst = 20% increase in yield. No brainer

Gambacorta G, Sharley JS, Baxendale IR. A comprehensive review of flow chemistry techniques tailored to the flavours and fragrances industries. Beilstein J Org Chem. 2021;17:1181–312.

Kinetically limited regime



- Reducing mixing rpms 10 fold does not drop conversion equivalently
- gas flowrate does a lot of the heavy lifting with regards to mixing compared to the stirrer
- velocity that disturbs the liquid enough to act as a mixer itself

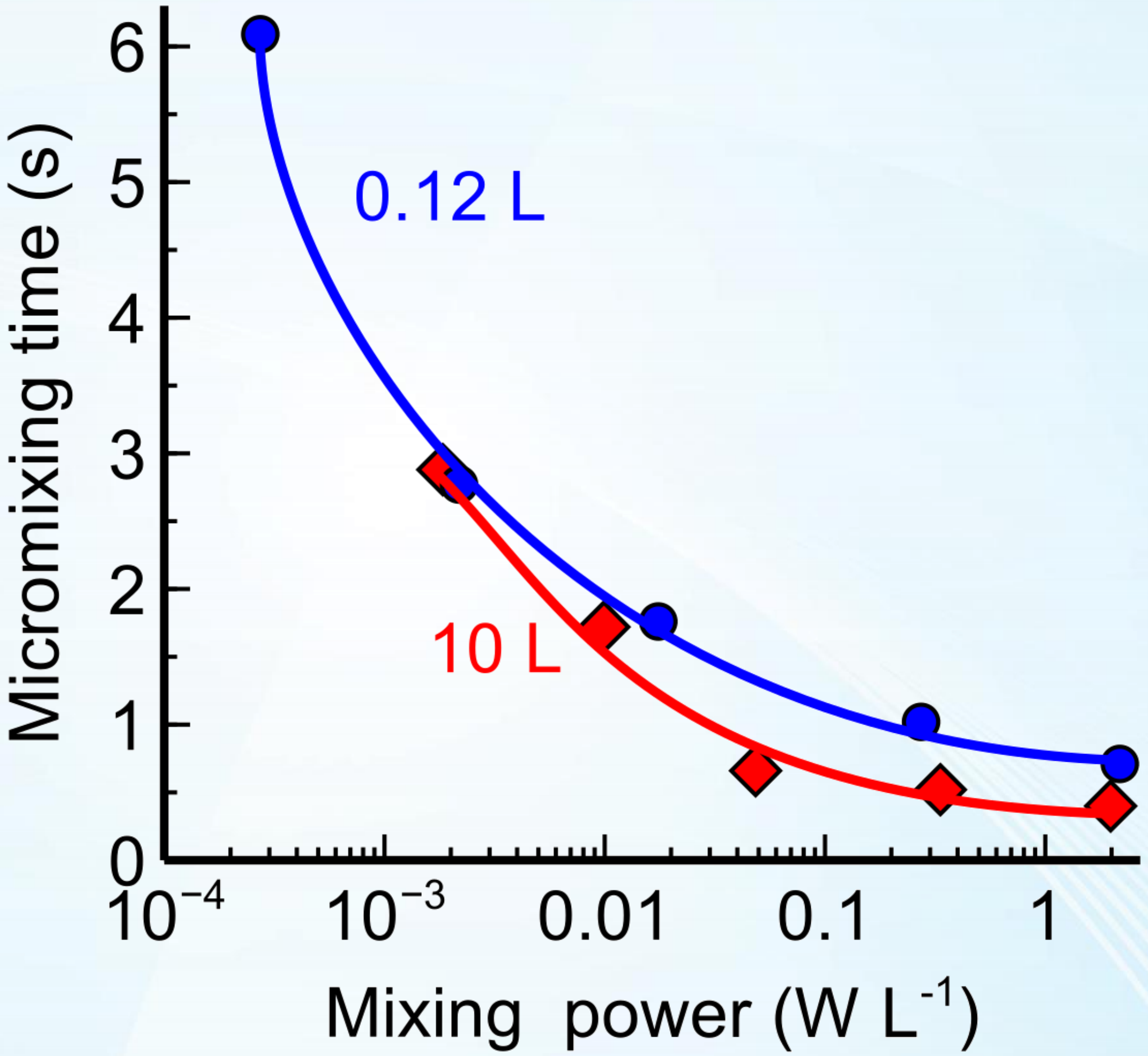
How much energy can we save?

- Scaled concentration until performance dropped – managed to increase substrate up to 20%.
- This means that we can reduce distillation energy costs by 60%

- Lab scale tests and model indicates that:
  - 140 kWh/kg of product using current procedure
  - 13 kWh/kg of product using developed flow

- That’s an 11 fold reduction in energy this step

Next Step: Scaling



Current scaled design utilises similar number of tanks and baffled interior so that micro mixing time is matched at scale

Fortunately managed to optimised conditions without significant pressure
- Allows for CAPex saved

Pilot testing begins in Feb!

Process only takes 48 hours

Yield Increased to 80%

Revenue doubled

11 fold Energy reduction

Scaling up in Feb!