Process Intensification and Green Chemistry

Batch-to-continuous

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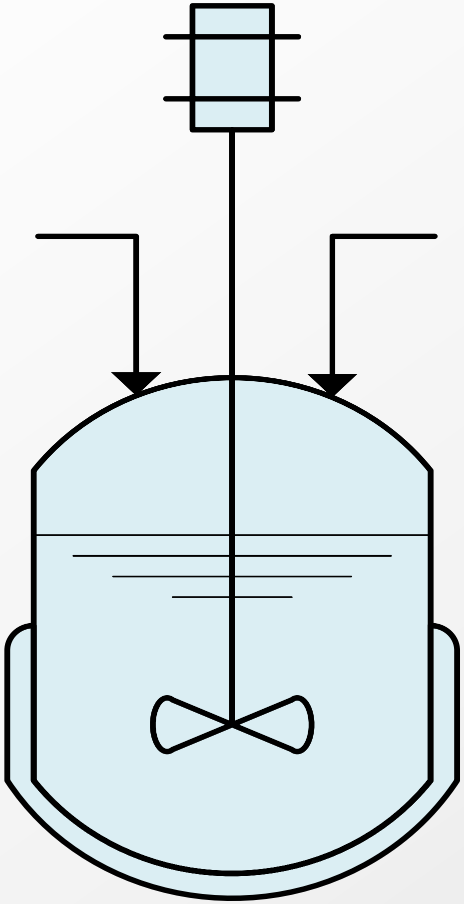
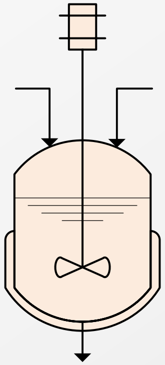
Content

1. Advantages and limitations of batch reactors
2. Advantages and limitations of continuous reactors
3. High pressure processing
4. Criteria for an implementation of continuous processing
5. Examples

Batch vs continuous process

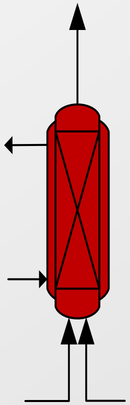


Batch reactor



Similar technology

CSTR or CSTR cascade



Intensified reactor

- Static mixer/heat exchanger
- Micro-reactor
- Spinning disk reactor
- Loop reactor
- Etc.

Large range of commercially available technologies

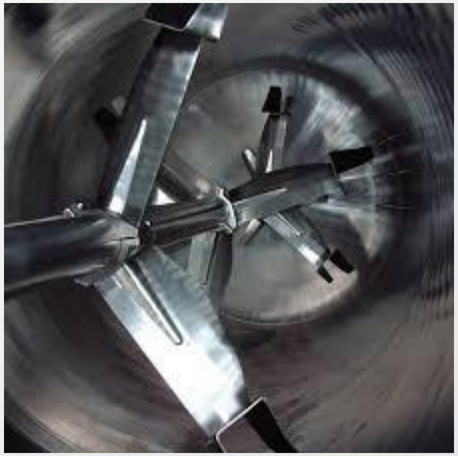
⇨ Opportunities for process intensification

1. Batch reactors

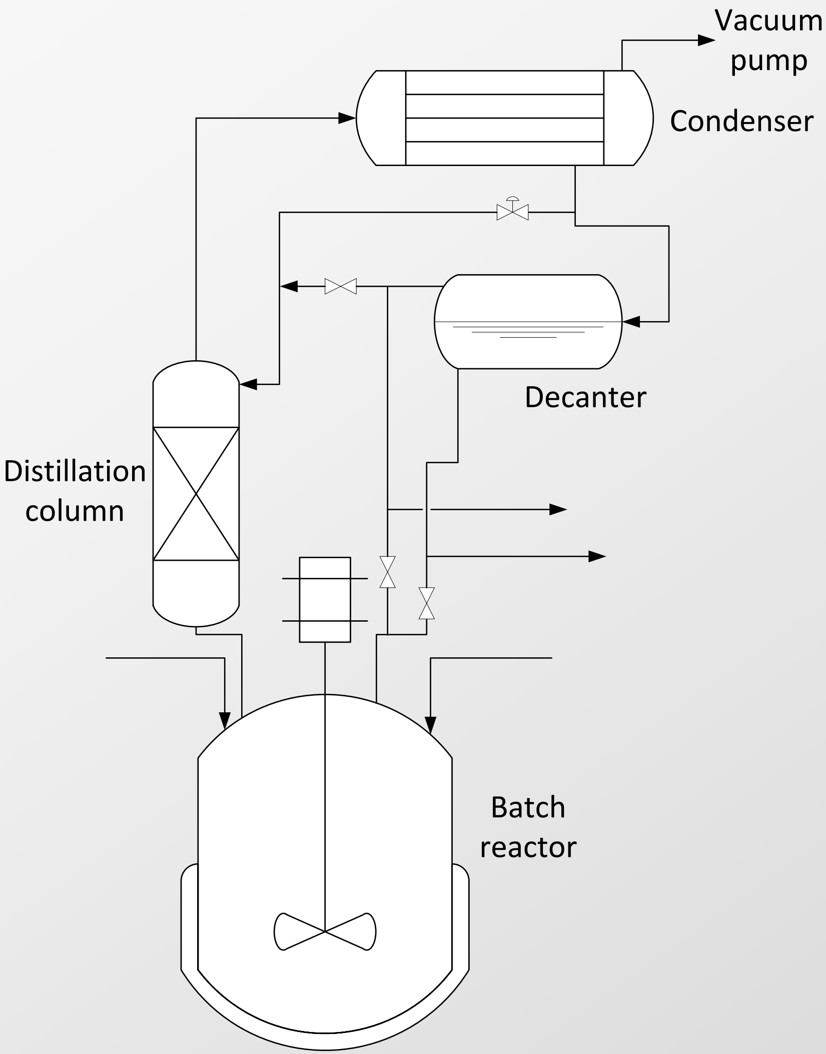
Advantages and limitations

Batch reactors

The workhorse of fine chemistry and pharma



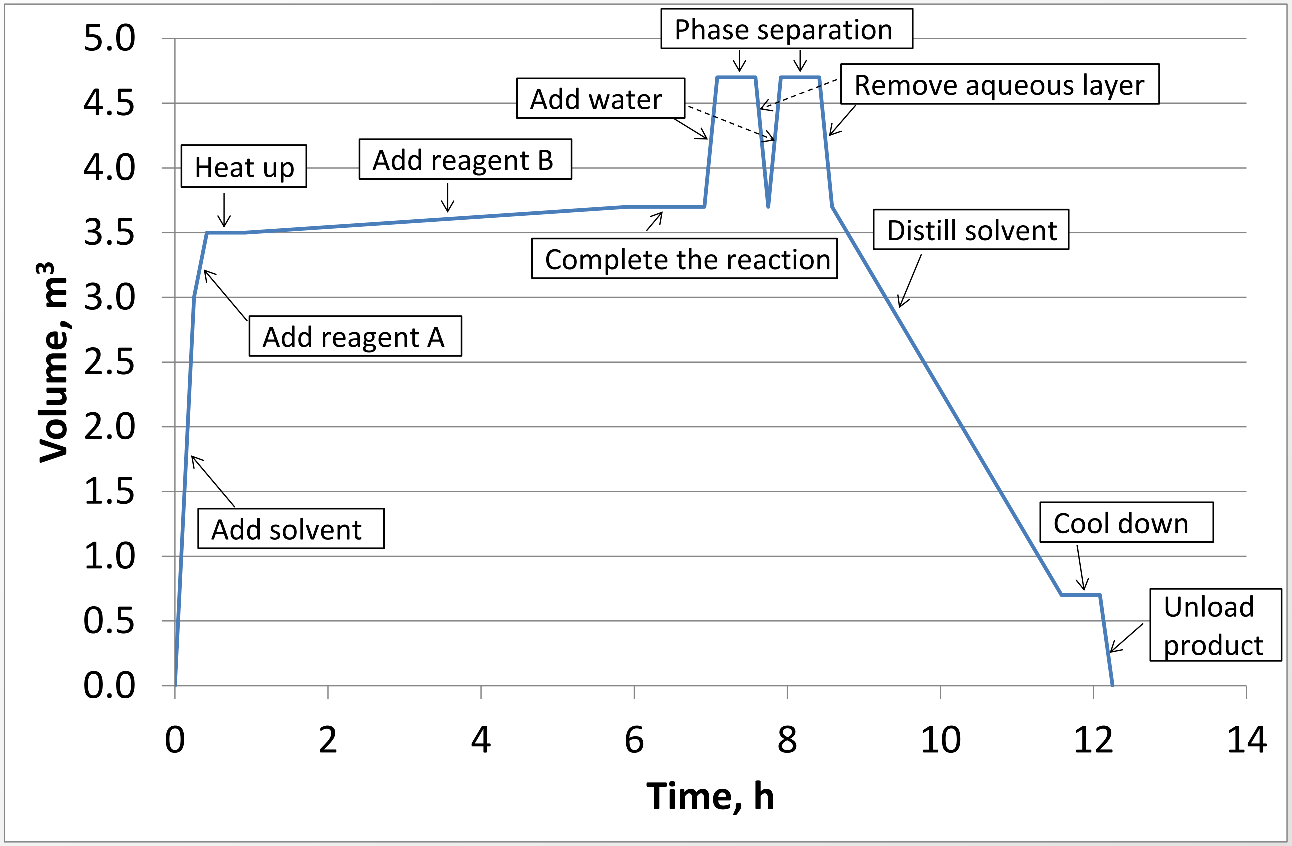
Schematic of typical batch reactor setup



Flexible but limited performance (mixing, mass and heat transfer)

Volume vs time in typical batch procedure

$A + B \rightarrow P$



Reasons for low volumetric productivity $\left(kg\ P/h \cdot m^3\right)$:

- The reactor is also used to perform other unit operations
- A lot of solvent must be added for thermal safety reasons
- Reagent B must be added slowly to allow for isothermal operation and minimize heat accumulation

Batch reactors

The workhorse of fine chemistry and pharma

- ✓ **Flexible**
  - ○ Large range of reaction times (hours to days)
  - ○ Down-stream steps can be carried-out (e.g., neutralization, 'washes', L/L extraction, solvent removal, crystallization,…)
- ✓ **Available**
  - ○ No investment required if plant operating below its full capacity
- ✘ **Limited heat-transfer rates**
  - ○ Need to operate semi-batch wise to control temperature
  - ○ Need to add enough solvent (safety, selectivity)
  - ⇨ Impact on volumetric productivity
  - ⇨ Impact on range of feasible reactions (safety, selectivity, cost)
- ✘ **Limited mass-transfer rates**
  - ○ Low volumetric productivity (G/L, L/L, L/S, G/L/S and L/L/S reactions)

Batch reactors

The workhorse of fine chemistry and pharma

- ✘ **Large volume**
  - ○ Decreased safety
- ✘ **High (micro)mixing time**
  - ○ Decrease in selectivity for fast competitive reactions
- ✘ **Large range of energy dissipation (dead-zones)**
  - ○ Potential negative impact on selectivity, quality (reactions, crystallizations, precipitations, emulsifications, …)
- ✘ **Scale-up complex due to inhomogeneous conditions**
  - ○ Temperature, concentration, energy dissipation rate, shear rate

Consequences of running processes in batch reactors

- Most **chemical reactions are deliberately slowed down** to match the batch equipment capabilities in terms of heat transfer, mass transfer and mixing

⇨Batch processes are mostly operated sub-optimally

- Some **chemical routes are not considered** during the route-scouting phase due to their incompatibility with batch plant equipment (hazardous reagents, highly exothermic reactions)

⇨ Missed opportunities for cost-effective, expeditive syntheses

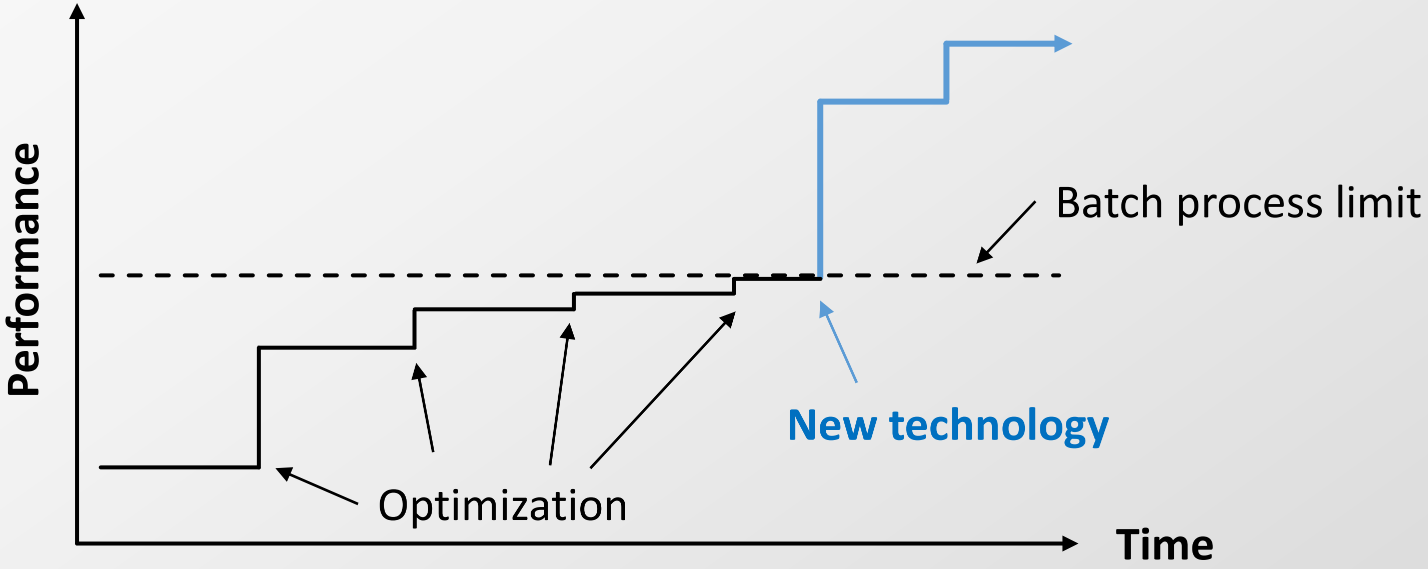
Common measures to slow down a chemical reaction & their implications

Measure to slow down reaction	Consequences
Dilute the reaction mixture (solvent)	Larger plant (reactor and peripherals) Larger volume of solvent to recycle
Slow reagent addition	Additional process control Lower plant capacity
Low temperature or pressure	Costly infrastructure Costly safety measures
Reflux cooling	Limited solvent choice
Use mild equivalents of hazardous reagents	Longer synthetic routes Higher reagent cost

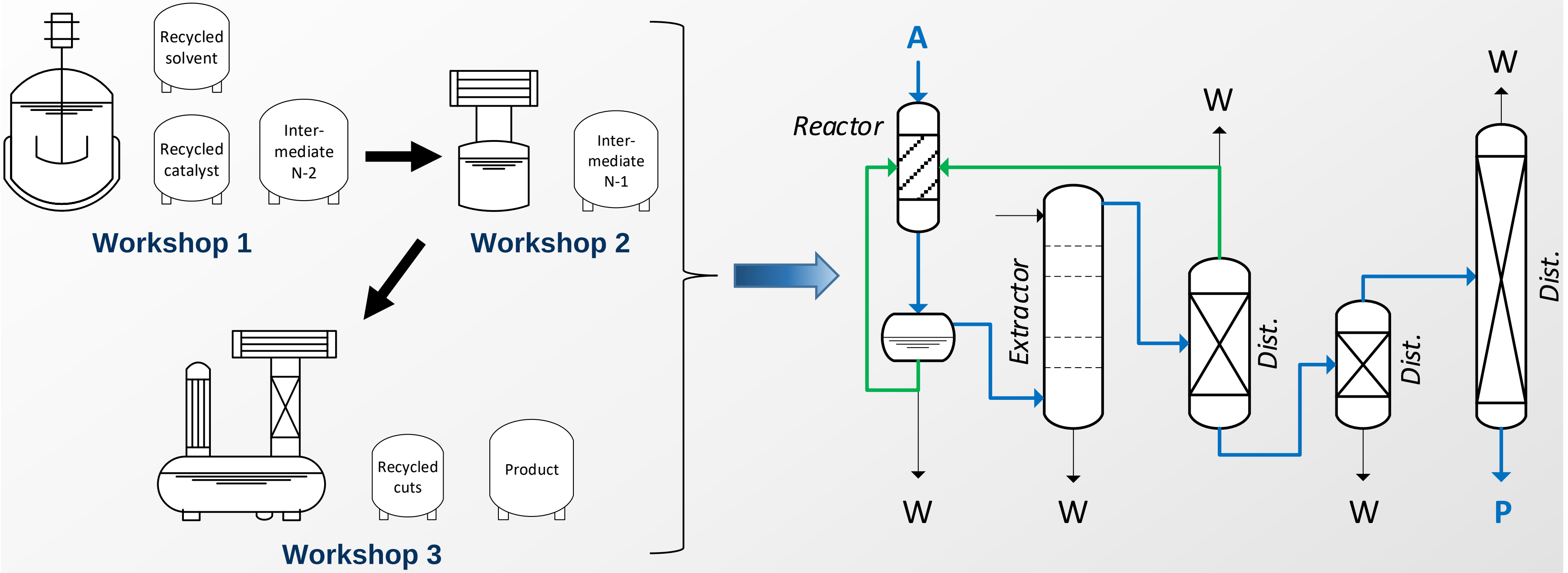
- ➢ Increased cost and complexity
- ➢ Increased plant footprint
- ➢ Reduced process intensity
- ➢ Reduced economic sustainability

Limits of batch reactor process optimization

- Optimization of a specific chemical system with a given batch reactor setup has an intrinsic limit
- At some point only a change in technology can bring significant process improvement



Batch to continuous



Main potential benefits:

- Raw materials and energy savings
- Enhanced product quality & consistency
- Lower inventory and labor
- Safety
- Shorter lead time

Cost
Quality
Safety
Environment

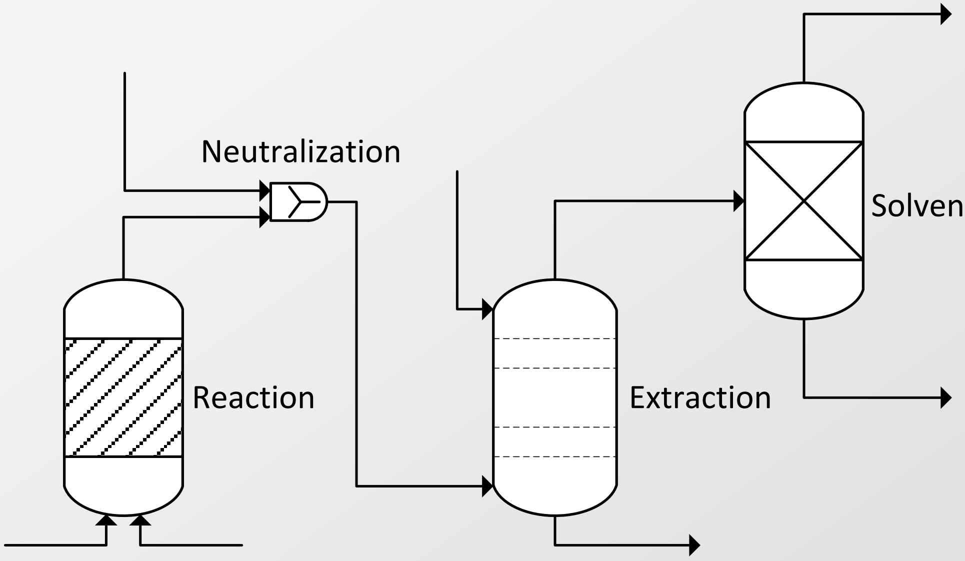
2. Continuous reactors

Continuous reactors

Potential advantages of continuous processing

Reasons for reduction in reactor size:

1. Reactor is only used for the reaction step
    - No process time utilized for charging and discharging raw materials/products, heating/cooling, phase separation, solvent distillation, etc.

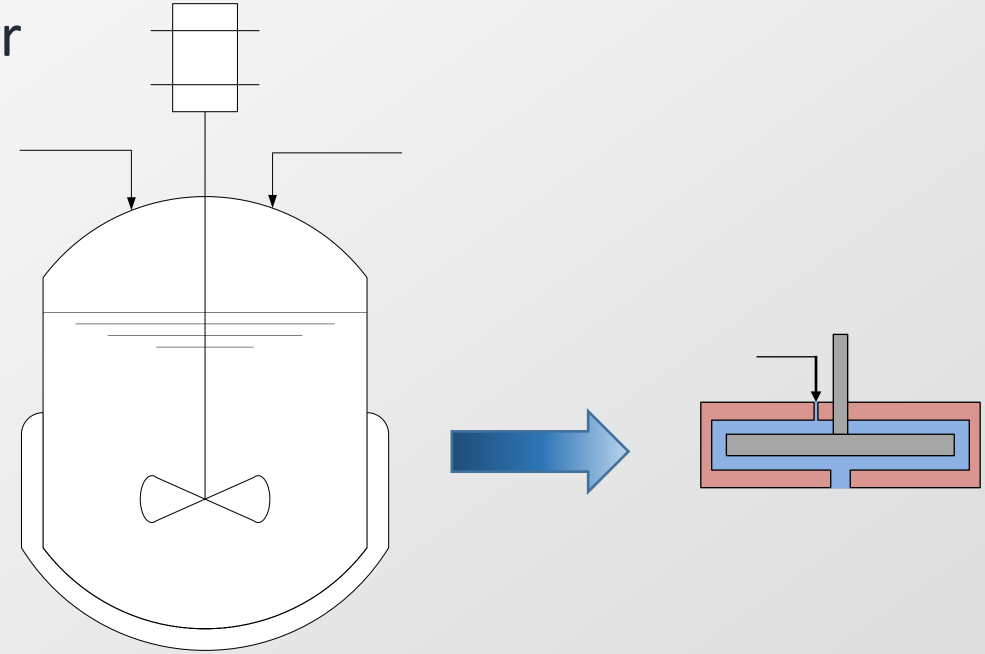


Continuous reactors

Potential advantages of continuous processing

Reasons for reduction in reactor size:

2. Potentially, adoption of an intensified technology when switching from batch to continuous
   - E.g., batch stirred tank reactor → spinning disk reactor or microreactor



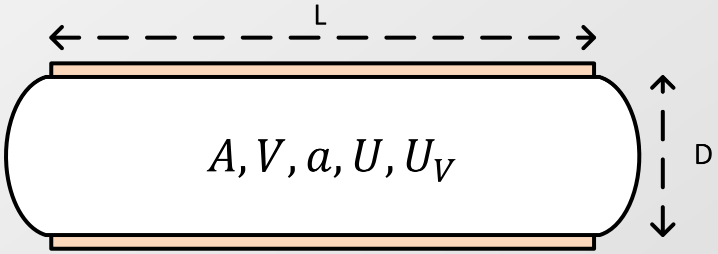
Continuous reactors

Potential advantages of continuous processing

Implications of reduced reactor size:

1. Increased surface/volume ratio ($a = \frac{A}{V}$)

- ✓ Increased specific heat transfer rate $U_V = U \cdot \boldsymbol{a}\ (W \cdot m^{-3} \cdot K^{-1})$
- ✓ Increased safety (improved temperature-control, faster heat removal)
- ✓ Increased volumetric productivity $(mol \cdot m^{-3} \cdot s^{-1})$



Continuous reactors

Potential advantages of continuous processing

Implications of reduced reactor size:

2. Increased mixing intensity
    - ✓ Higher energy dissipation rate $\boldsymbol{\varepsilon}$, higher shear rate $\dot{\boldsymbol{\gamma}}$
        - ✓ Decreased mixing time t_{mx}

          → increased selectivity and product quality
        - ✓ Increased specific mass transfer rate ($\boldsymbol{k_L \cdot a_{LG}, k_S \cdot a_{LS}, etc}$)

          → increased productivity
        - ✓ Increased $\boldsymbol{U} \cdot a$ (increase in U)

          → increased safety and productivity

Continuous reactors

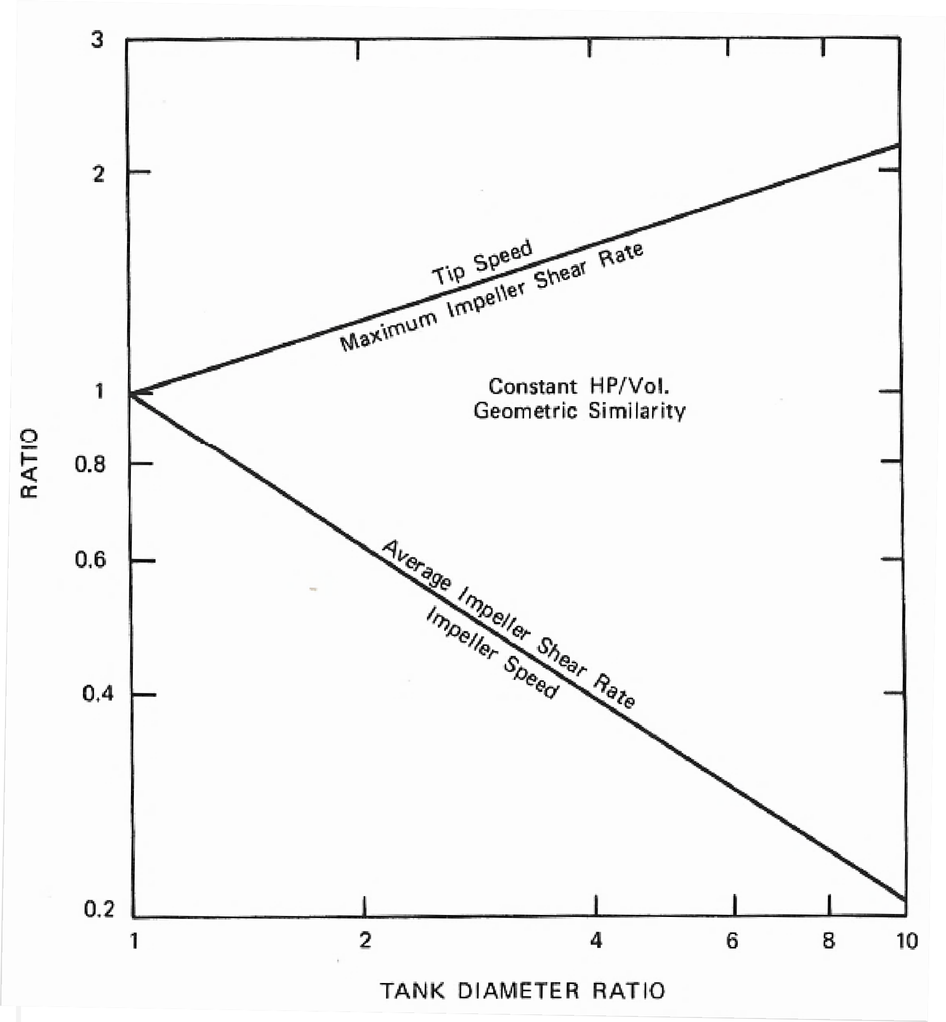
Potential advantages of continuous processing

Implications of reduced reactor size:

3. More homogeneous mixing
    - ✓Lower variance of energy dissipation rate and shear rate $\sigma^2(\varepsilon), \sigma^2(\dot{\gamma})$ (next slide)
        - ✓Increased selectivity and product quality
4. Lower holdup
    - ✓Increased safety (less residual heat, lower amount of toxic compounds)

Effect of vessel size on shear rate

Batch reactor, constant P/V, geometric similarity



- Maximum shear rate increases upon scaleup
- Average shear rate decreases upon scaleup

→ Greater variation in shear rate $\sigma^2(\dot{\gamma})$ in large tanks than in small tanks

A. Bisio and R. L. Kabel, Scaleup of Chemical Processes: Conversion from Laboratory Scale Tests to Successful Commercial Size Design, 1985, Wiley

Continuous reactors

Potential advantages of continuous processing

Opportunities

1. Reduced mixing time, homogeneous mixing conditions
    - Increased selectivity for fast competitive reactions
    - Increased quality e.g., for crystallization, precipitation and emulsification processes

Continuous reactors

Potential advantages of continuous processing

Opportunities

2. Enhanced heat and mass transfer, lower holdup, lower mixing time:

   ⇨Enables use of “forbidden” or “forgotten” reactions (i.e., considered too dangerous from a thermal safety point of view to be carried out in batch reactors)

   ⇨ Enables use of alternative conditions or a different (but cheaper/cleaner) synthesis with hazardous reagents

Continuous reactors

Potential advantages of continuous processing

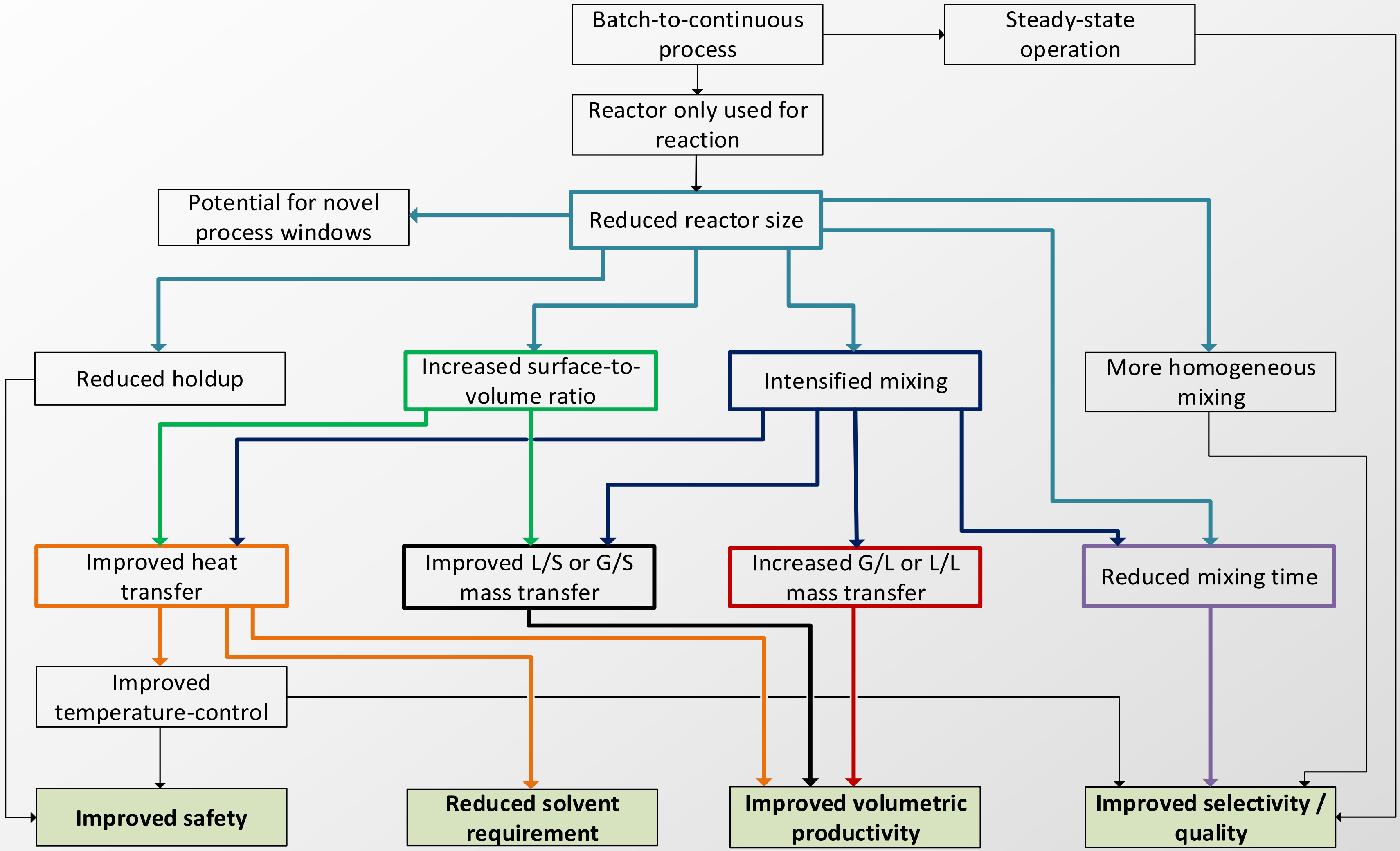
Opportunities

3. Enhanced heat and mass transfer, lower holdup, lower mixing time:

⇨ Enables the use of intensified process conditions (high T, p, c = "novel process windows")

- **High concentration** (e.g., solvent reduction or suppression)
- **High temperature** / short residence time under safe conditions due to high heat removal / low holdup of potentially unstable intermediates
- **High pressure** (high pressure apparatus easier to design for low-volume equipment) → see section 3

Some potential advantages of continuous reactors



Batch-to-continuous process

Steady-state operation

Reactor only used for reaction

Potential for novel process windows

Reduced reactor size

Reduced holdup

Increased surface-to-volume ratio

Intensified mixing

More homogeneous mixing

Improved heat transfer

Improved L/S or G/S mass transfer

Increased G/L or L/L mass transfer

Reduced mixing time

Improved temperature-control

Improved safety

Reduced solvent requirement

Improved volumetric productivity

Improved selectivity / quality

High level business drivers

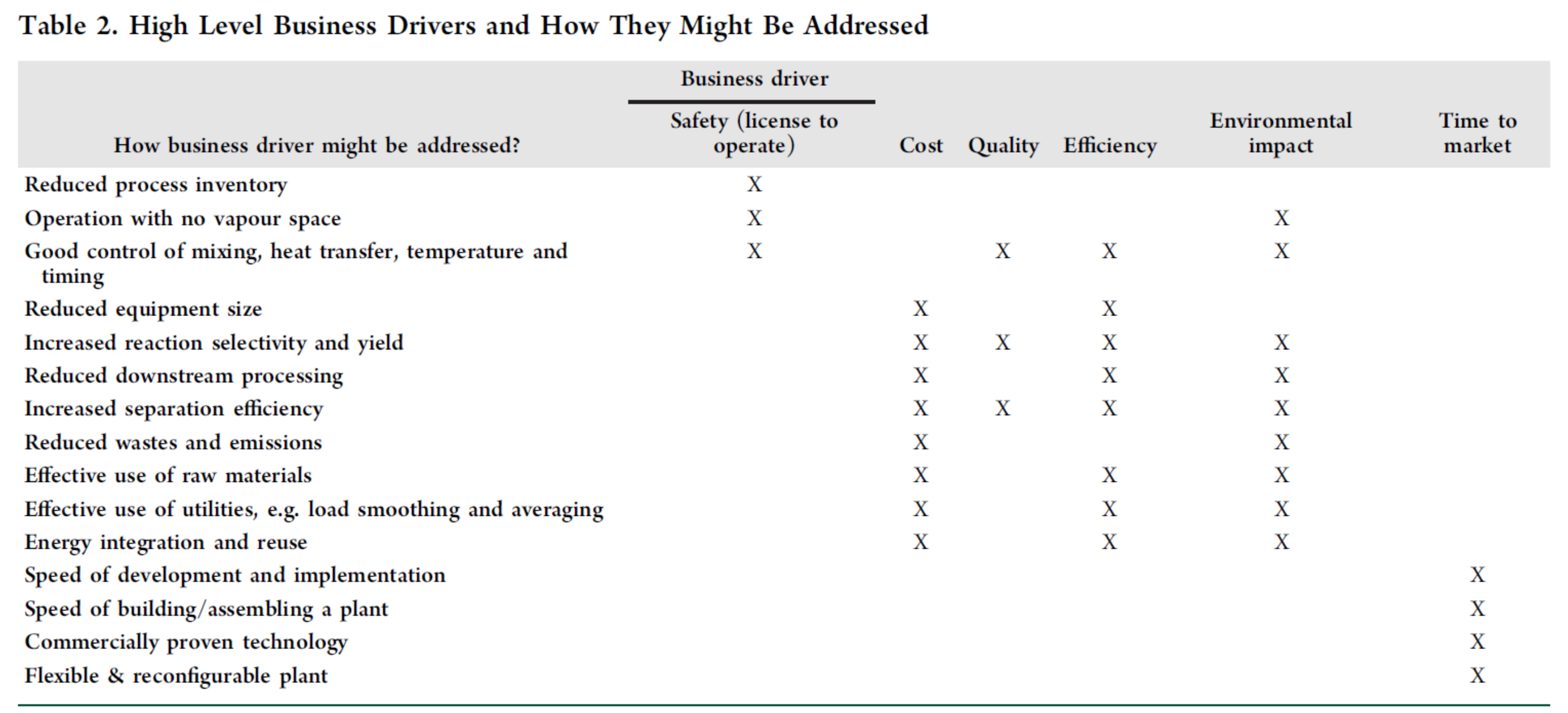


Table 2. High Level Business Drivers and How They Might Be Addressed

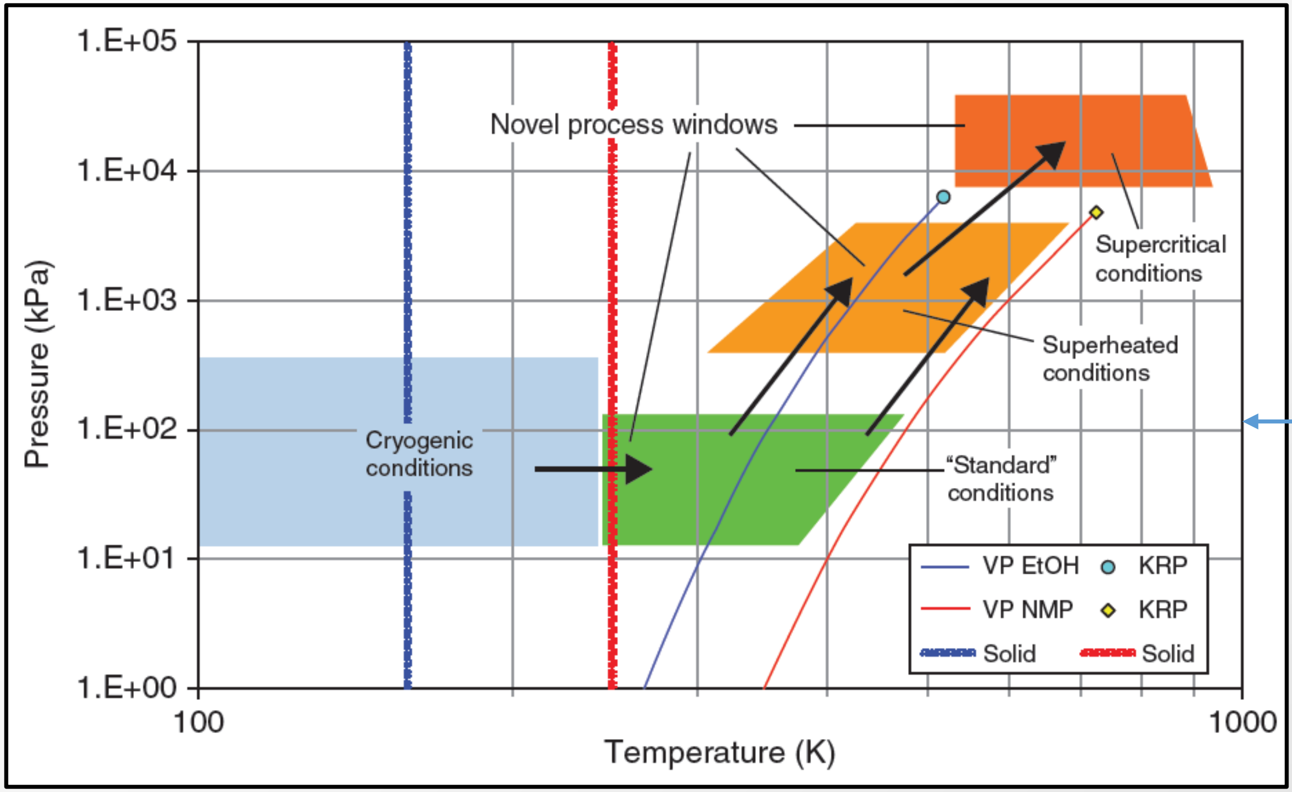
	Business driver					
How business driver might be addressed?	Safety (license to operate)	Cost	Quality	Efficiency	Environmental impact	Time to market
Reduced process inventory	X					
Operation with no vapour space	X				X	
Good control of mixing, heat transfer, temperature and timing	X		X	X	X	
Reduced equipment size		X		X		
Increased reaction selectivity and yield		X	X	X	X	
Reduced downstream processing		X		X	X	
Increased separation efficiency		X	X	X	X	
Reduced wastes and emissions		X			X	
Effective use of raw materials		X		X	X	
Effective use of utilities, e.g. load smoothing and averaging		X		X	X	
Energy integration and reuse		X		X	X	
Speed of development and implementation						X
Speed of building/assembling a plant						X
Commercially proven technology						X
Flexible & reconfigurable plant						X

Teoh et al., Org. Process Res. Dev. 2016, 20, 414−431

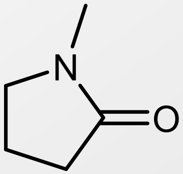
3. High pressure processing

High T-p conditions

“Novel process windows”



p_{atm}



NMP
(N-Methyl-2-pyrrolidone)

- Use of high temperature/short residence time
- Use of superheated, near-critical or supercritical conditions

Hessel, V., Kralisch, D. and N. Kockmann. Novel Process Windows, 2015. Wiley.

High pressure conditions

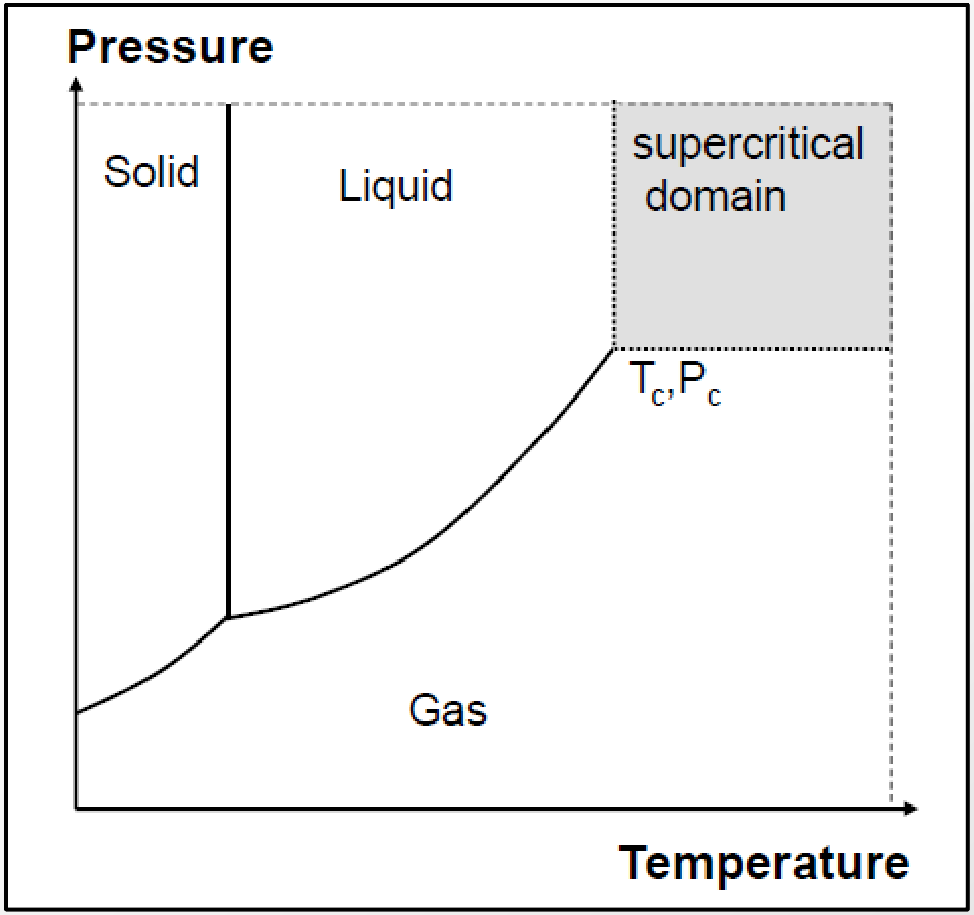
Overview of pressure effects

- Allows reaching superheated, near-critical or supercritical conditions (reactions above normal boiling point of the solvent)
- Influence on equilibrium constant
- Influence on rate constant
- Influence on selectivity (sterically hindered compounds, modification of chemo-, regio- or stereo-selectivity)
- Increased solubility of gases in liquids
- Increased miscibility of liquids
- Effect on physical properties
  - Density, viscosity, dielectric constant
- Effect on transport properties
  - Diffusion coefficient, thermal conductivity

Borukhova and Hessel: Micro Process Technology and Novel Process Windows – Three Intensification Fields, in: Boodhoo, K. and A. Harvey. Process Intensification for Green Chemistry: Engineering Solutions for Sustainable Chemical Processing. 2013. John Wiley & Sons Inc

Supercritical fluids as solvents

Critical coordinates for common fluids

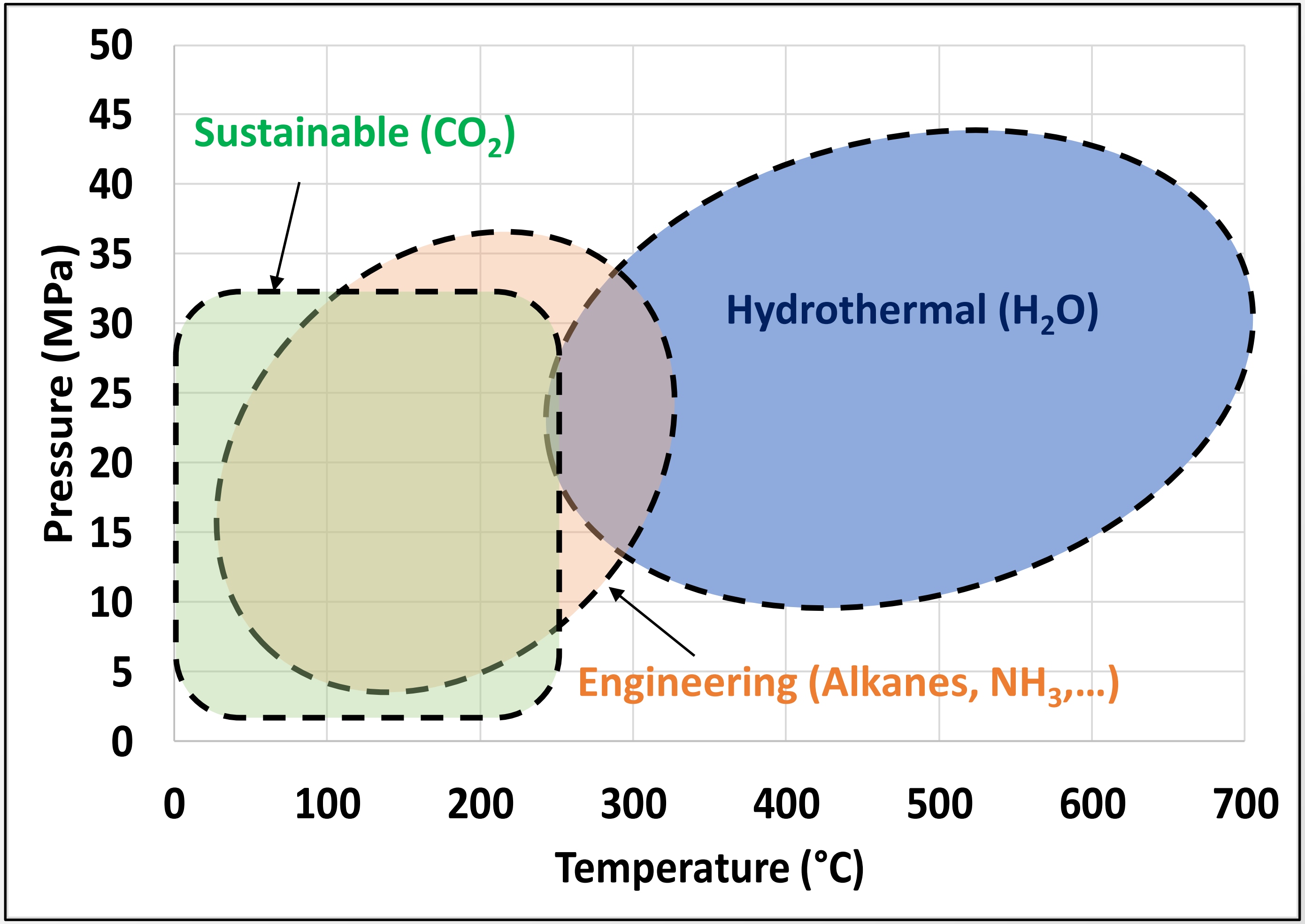


Solvent	T_c (°C)	p_c (bar)
Water	**374**	**218**
CO_2	**31**	**74**
Propane	97	42
Ethane	32	48
Butane	152	38
Acetone	235	47
Ethanol	243	63

Danielle Barth, Séverine Camy and Jean-Stéphane Condoret: Supercritical CO2, the Key Solvent for Sustainable Processes in Poux, M., P. Cognet and C. Gourdon. Green Process Engineering. 2015. CRC Press.

Supercritical fluids

Operational regions of the three classes of SCF



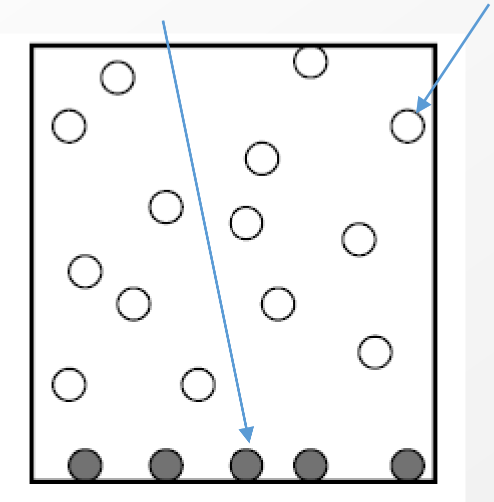
Borukhova and Hessel: Micro Process Technology and Novel Process Windows – Three Intensification Fields, in: Boodhoo, K. and A. Harvey. Process Intensification for Green Chemistry: Engineering Solutions for Sustainable Chemical Processing. 2013. John Wiley & Sons Inc

Supercritical fluids

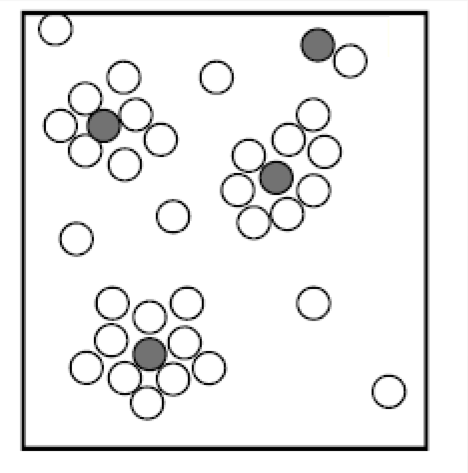
Schematic of molecular organization of solvent in the presence of a solute

Solute molecule

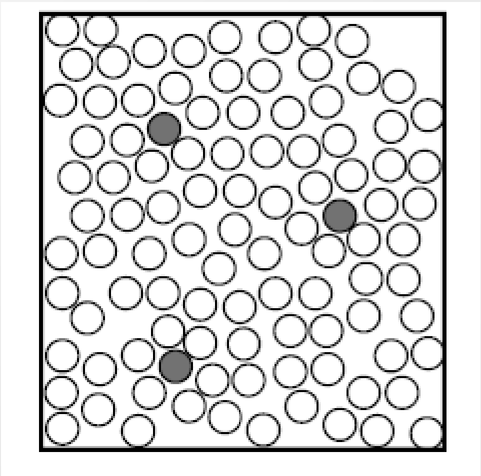
Solvent molecule



Gas → no solvation



SCF → aggregates or clusters



Liquid → solvation

Danielle Barth, Séverine Camy and Jean-Stéphane Condoret: Supercritical CO2, the Key Solvent for Sustainable Processes in Poux, M., P. Cognet and C. Gourdon. Green Process Engineering. 2015. CRC Press.

Supercritical fluids

Key properties and advantages

Good solvation power (increases with density). Density is tunable (p↑, T↓)

	Gas	SCF	Liquid
$\rho\ (kg \cdot m^{-3})$	1	$\mathbf{700}$	1000
$\mu\ (Pa \cdot s)$	10^{-5}	$\mathbf{10^{-4}}$	10^{-3}
$D_m\ (m^2 \cdot s^{-1})$	10^{-4}	$\mathbf{10^{-7}}$	10^{-9}

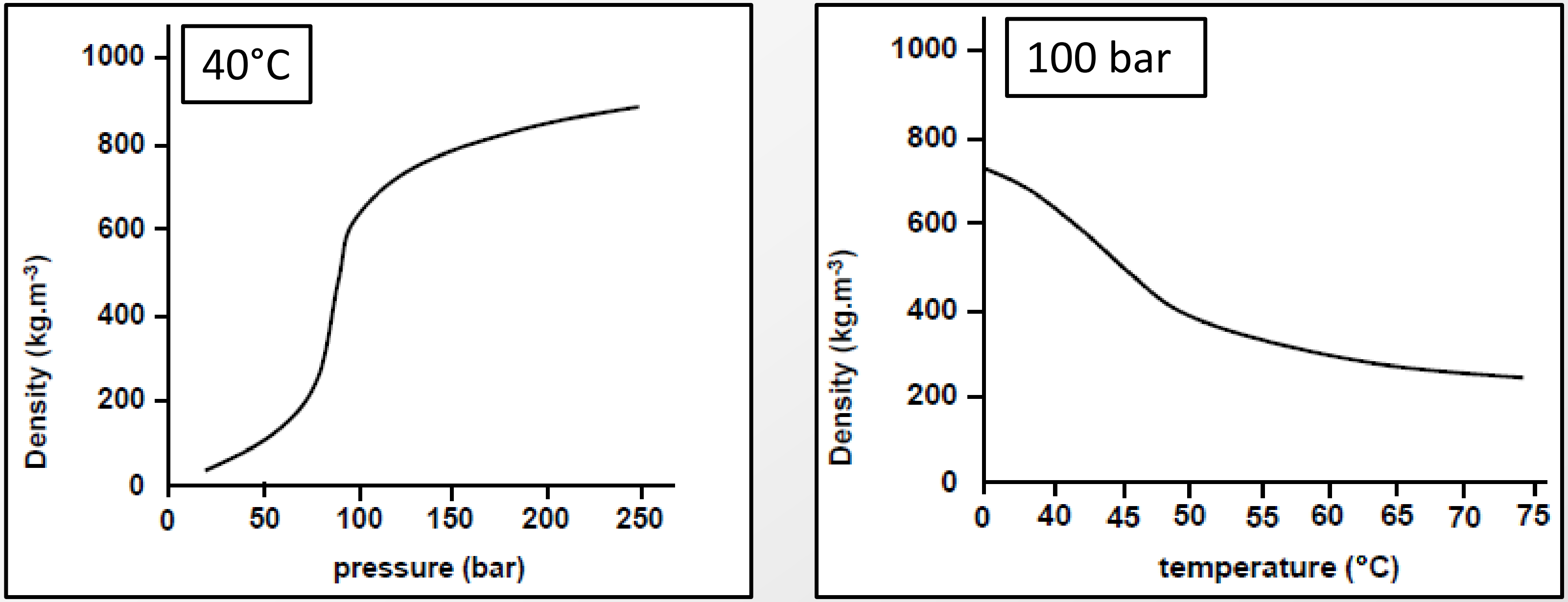
Good mass transfer properties
(lower μ and higher D_m than liquid state)

SCF: no surface tension effect → good penetration into porous media

SCF: kinematic viscosity $\nu = \dfrac{\mu}{\rho}$ is low $\rightarrow D_m$ is high

Supercritical fluids

Density of CO_2 as a function of p and T



- Solvation power increases with density, i.e. with pressure
  - ➢ Tunable solvation power (e.g. selective extraction)
- Depressurization causes precipitation (easy separation after extraction or reaction)

Supercritical solvents

Advantages of CO_2

- Inert
- Low critical coordinates (31°C, 74 bar)
- Not flammable
- Does not contaminate aqueous phases
- Low T_c gases (H_2, CO, O_2) are totally soluble in SC-CO_2 → intensification of gas-liquid reactions by making them single-phase
- Low viscosity → easy penetration into complex structures
- But: not a very good solvent for polar compounds (low dielectric constant). Alcohols or water can be added to increase the solvent power.

Supercritical fluids (SCF) as solvents

Overview of some of their advantages

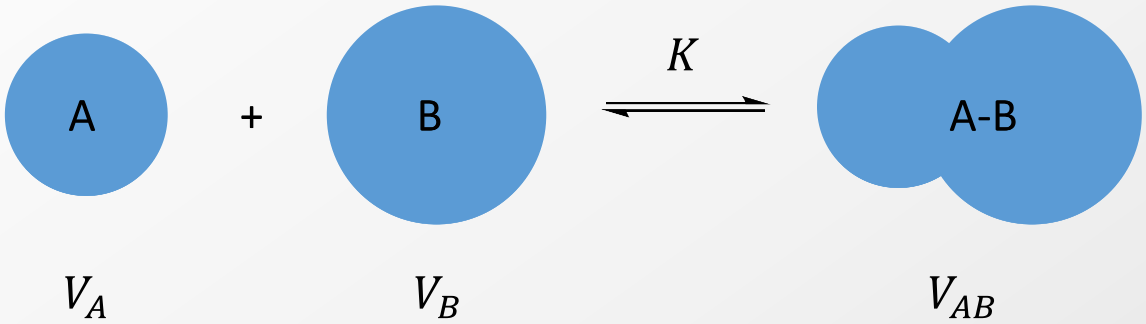
- Transition of laminar to turbulent conditions (increase Re by decreasing μ without significantly changing ρ)
- Increase in diffusion coefficient D_m → rapid mixing
- High thermal conductivity λ → fast heat transfer
- Low μ and high D_m → Low D_{ax} (narrow RTD)
- Mostly water and CO_2 considered for chemical processing, due to their abundance, low cost, non-flammability, chemical inertness, easy recovery, non-toxicity
- Disadvantages of SC-water include high energy requirements, high corrosive power, high T_c preventing its use as a solvent for thermally labile components
- Alcohols are also used (but they are flammable, implying costly safety measures)

High pressure conditions

Volume effects

Thermodynamic effect

$$Reactants \underset{}{\overset{K}{\rightleftharpoons}} Products$$



$$\Delta V_r = V_{AB} - (V_A + V_B) < 0$$

$$\left(\frac{\partial \Delta G}{\partial p}\right)_T = \Delta V_r = -RT\left(\frac{\partial lnK}{\partial p}\right)_T$$

$$\rightarrow \left(\frac{\partial lnK}{\partial p}\right)_T = -\frac{\Delta V_r}{RT}$$

Negative volume of reaction $\Delta V_r < 0$ → K increases with p

High pressure conditions

Volume effects

Thermodynamic effect

- Occurs for condensation reactions such as cycloadditions (e.g., Diels-Alder)

- Effect significant only at very high pressures (several 100s of bar)
- ⚠ ΔV_r is a function of pressure→ $\left(\frac{\partial lnK}{\partial p}\right)_T = -\frac{\Delta V_r}{RT}$ cannot be integrated directly
- Linear approximation over small p range: $lnK = lnK_0 - \frac{\Delta V_r}{RT}p$

High pressure conditions

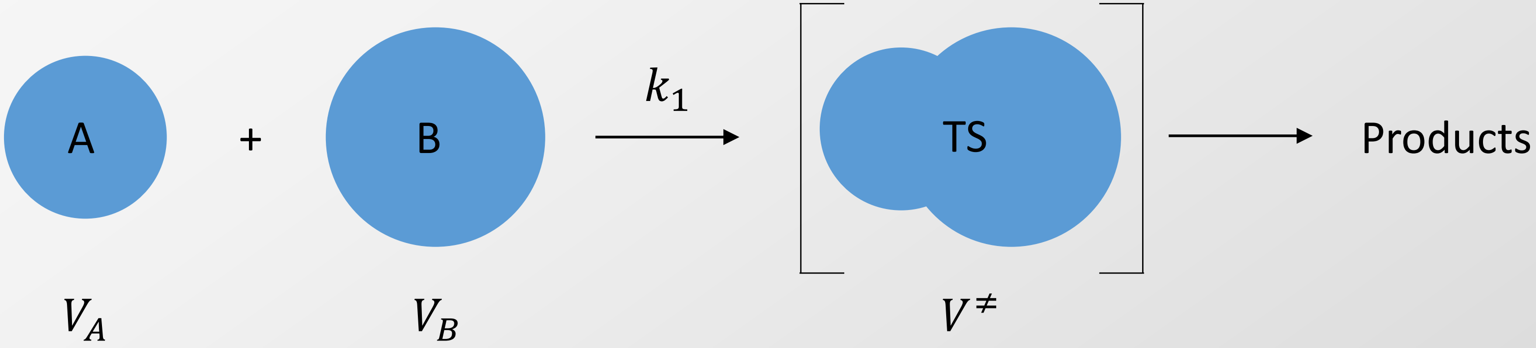
Volume effects

Kinetic effect

$$Reactants \overset{\Delta V_1^{\neq}}{\xrightarrow{k_1}} Transition\ state \rightarrow Products$$

$$\left(\frac{\partial \Delta G^{\neq}}{\partial p}\right)_T = \Delta V^{\neq} = -RT\left(\frac{\partial \ln k}{\partial p}\right)_T \qquad \rightarrow \left(\frac{\partial \ln k_1}{\partial p}\right)_T = -\frac{\Delta V_1^{\neq}}{RT}$$

Negative volume of activation: $\Delta V_r < 0 \rightarrow k_1$ increases with p



Activation volume $\Delta V_1^{\neq} = V^{\neq} - (V_A + V_B) < 0$

High pressure conditions

Volume effects

Kinetic effect

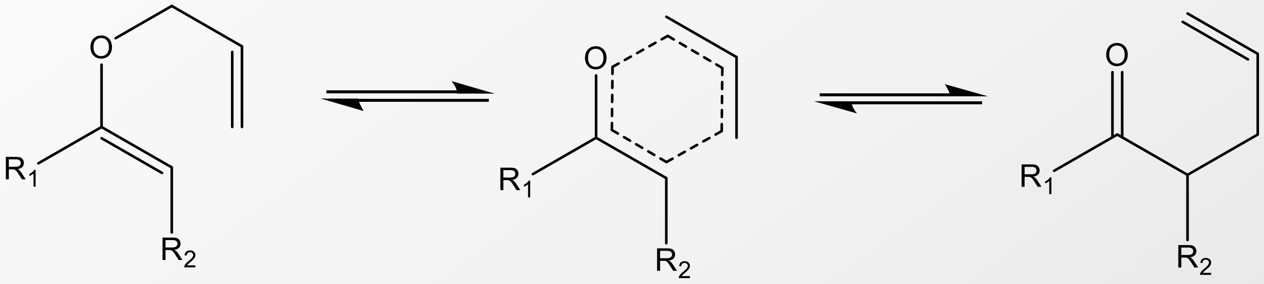
- Negative activation volume $\Delta V_1^{\neq} < 0 \rightarrow k_1$ increases with p
- Linear approximation $lnk = lnk_0 - \frac{\Delta V^{\neq}}{RT} p$ used to measure $\Delta V^{\neq}$

High pressure conditions

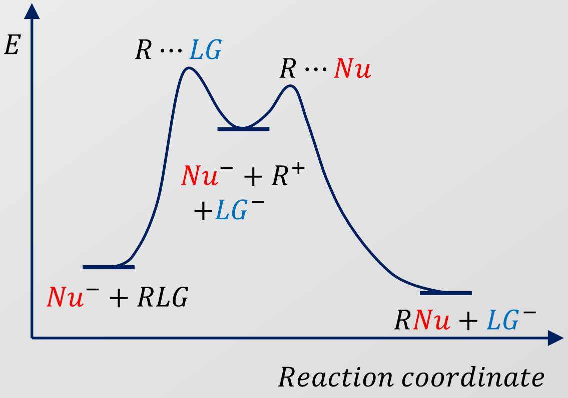
Volume effects

Kinetic effect: some examples

- Reactions with cyclic transition states (such as Claisen or Cope rearrangements)



- SN1 reactions in polar solvents, where activated complex involves charge separation → solvent molecules alignment in the vicinity of the activated complex → volume contraction (electrostriction)
- Reactions with high steric hindrance



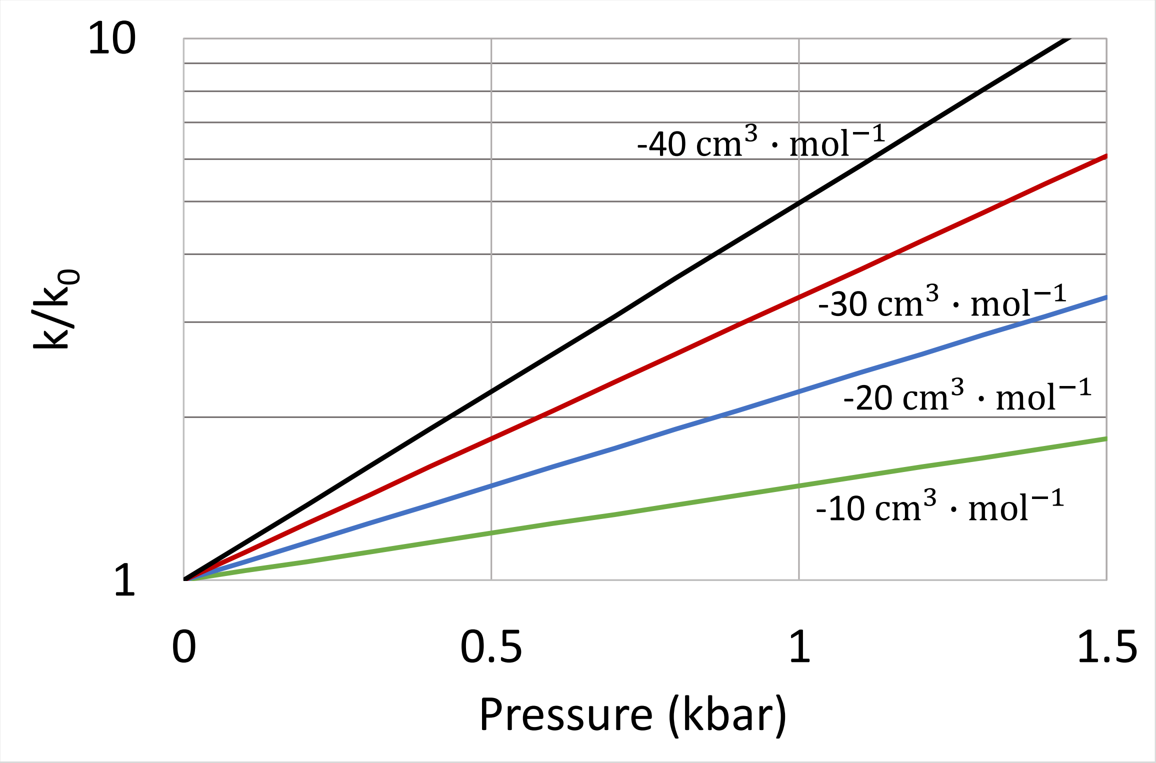
Nu: *nucleophile* *LG*: *leaving group*

High pressure conditions

Volume effects

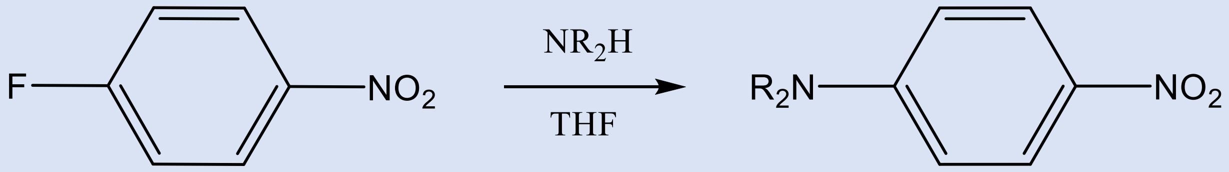
Kinetic effect: typical values of $\Delta V^{\neq}$

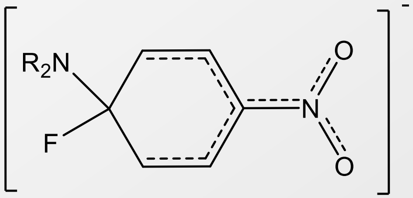
Reaction	$\Delta V^{\neq}$ $(cm^3 mol^{-1})$
Cycloaddition	-25 to -50
Ene reaction	-25 to -50
Aza-Michael addition	-40 to -50
Cope and Claisen	-10



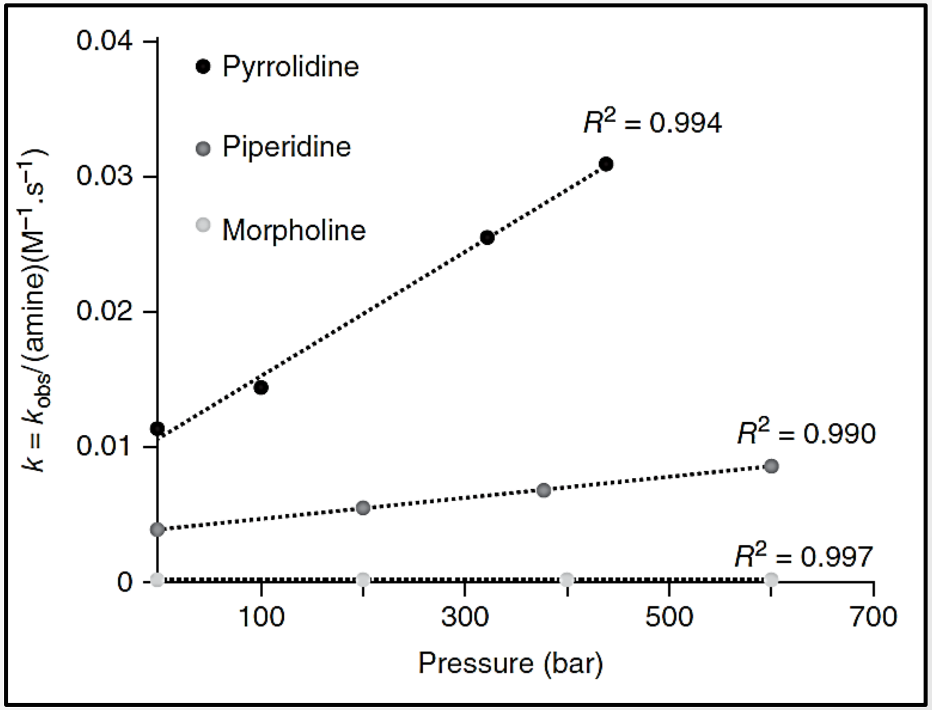
Examples of pressure-enhanced reactions

Aromatic nucleophilic substitution

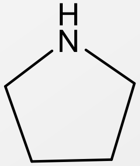




Meisenheimer intermediate



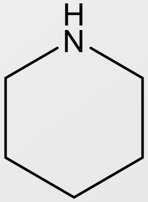
Benito-Lopez et al., Chem. Commun., 2005, 2857–2859



Pyrrolidine

$$\Delta V^{\neq} = -58\ cm^3 mol^{-1}$$

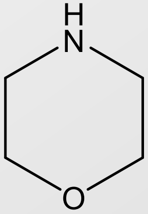
$$\varepsilon_r = 8.3$$



Piperidine

$$\Delta V^{\neq} = -32\ cm^3 mol^{-1}$$

$$\varepsilon_r = 5.8$$



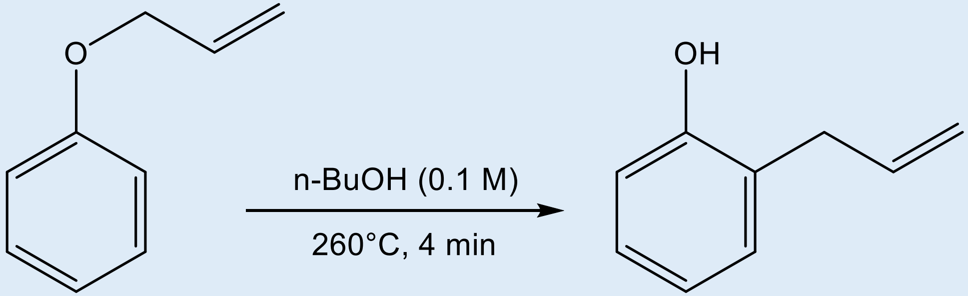
Morpholine

$$\Delta V^{\neq} = -22\ cm^3 mol^{-1}$$

$$\varepsilon_r = 7.3$$

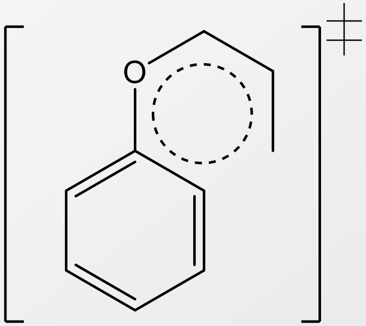
Examples of pressure-enhanced reactions

Claisen rearrangement

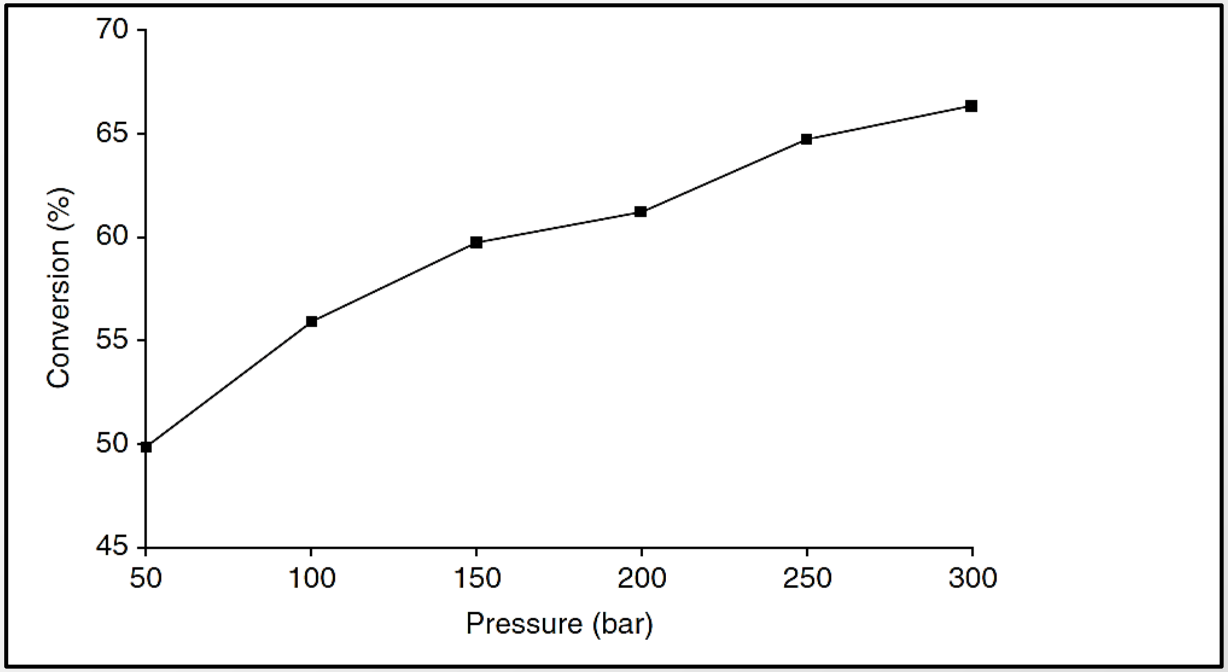


Allyl phenyl ether

2-allyl phenol



Aromatic transition state

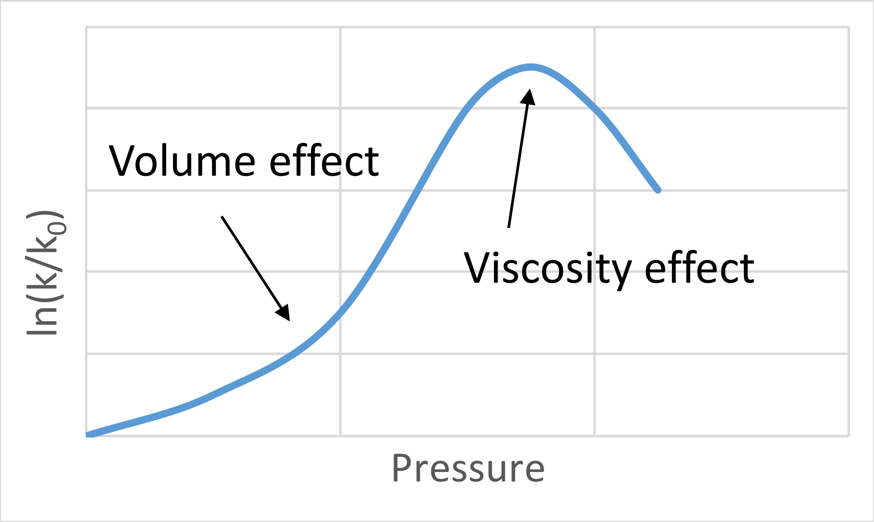


Hessel, V., Kralisch, D. and N. Kockmann. Novel Process Windows, chapter 7, 2015. Wiley.

High pressure conditions

Viscosity effects

- It is generally agreed that transition state theory may be invalid in highly viscous media, where diffusional processes play an important part
- Viscosity of liquids increases exponentially with pressure
- At high viscosity, diffusional phenomena may dominate and decrease the reaction rate, leading to an optimum operational pressure
- Beware of decreasing solvent freezing point and decreasing solubility of solids with increasing pressure!



High pressure conditions

Optional

Dielectric constant effects

- Dielectric constant ε weak function of temperature but strongly depends on pressure

$$\frac{\varepsilon_{p,0}}{\varepsilon_p} = 1 - A \cdot ln\left[\frac{B + p}{B + 1}\right]$$

$\varepsilon_{p,0}$: at 1 atm
ε_p: at pressure p

- A and B are liquid-dependent, pressure-independent.
- B decreases with increasing temperature.
- ε determines magnitude of electrostriction induced by solvent in the vicinity of charged species
- ε influences the activation volume

High pressure conditions

Solubility effects

- **Thermodynamic effect of pressure**

$$\left(\frac{\partial ln x_S}{\partial p}\right)_T = -\frac{\Delta V_{sol}}{RT}$$

  - Negative volume of solution $\Delta V_{sol} < 0$

    $\Delta V_{sol} < 0 \ \rightarrow$ solubility x_S increases with p

- **Solubility of gases and liquids increases with pressure**
  - Gas-liquid reactions can be performed at high concentration of dissolved gases or even under homogeneous conditions
  - Miscibility of liquids can be enhanced, due to changes in density and polarity with pressure

4. Criteria for the implementation of continuous processing

Significant criteria for the choice of an industrializable technology

Safety

Reliability / robustness

Cost

Quality

Productivity

Significant criteria for the choice of an industrializable technology

- **Safety**
  - ✓ Increased heat transfer
  - ✓ Decreased holdup of reaction mixture
- **Technology reliability/robustness**
  - ✗ Uncertainties (maintenance, availability, cost, scale-up…) related to new technologies
  - ✓ Relative ease of scale-up for certain technologies, speed in process R&D
- **Cost**
  - ✓ Reduction in manpower, energy, inventory
  - ✓ Increased safety → reduction in safety-related costs
  - ✓ Potential reduction in raw material costs (less solvent, less reactants/reagents if intensified reaction & selectivity improvements)
  - ✗ Investment cost might be high

Significant criteria for the choice of an industrializable technology

- **Quality**
  - ✓ Steady-state → constant quality
  - ✓ Potential increase in selectivity/quality due to intensified reaction conditions (mixing intensity/homogeneity, mass and heat transfers)
- **Productivity**
  - ✓ Increase due to enhanced heat/mass transfer
  - ✓ Increase due to potentially intensified conditions (less or no solvent, higher pressure, higher temperature,…)

Some criteria considering a transfer from batch to continuous

- Highly exothermic / endothermic reaction
- Selectivity highly dependent on temperature
- Selectivity sensitive to mixing
- Existence of an unstable intermediate
- Requirement for high dilution
- Presence of hazardous reagent, intermediate or product
- Large volume product

Reaction classification

- **Type A**
    - Very fast ($t_r < 1s$)
    - Mostly influenced or completely controlled by the mixing process
    - Reaction and heat production take place near the entrance in the mixing zone
    - Intensity of mixing controls the heat production
    - Examples: reactive species: Cl_2, Br_2, amines, RCOCl, organometallic reactions (Grignard and lithium-type chemistries). Often require cryogenic conditions
- **Type B**
    - Fast ($1s < t_r < 10\ min$)
    - Mainly kinetically controlled
    - Temperature control critical for systems with high reaction enthalpy
- **Type C**
    - Slow ($t_r > 10\ min$)
    - Normally carried out in batch
    - Millireactors may be advantageous if safety or product quality are important (thermal hazard and/or autocatalytic behavior)

Roberge (2004) Org. Process Res. Dev., 8 (6), 1049–1053.

Examples of type A, B and C reactions*

Optional

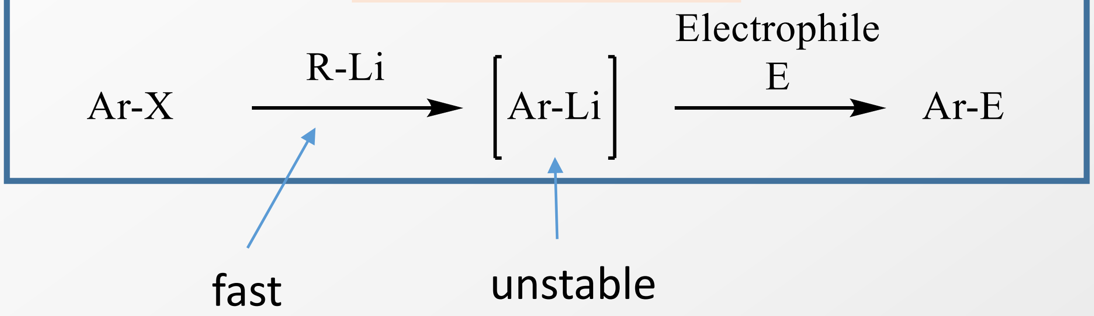
Type A	Type B	Type C
Grignard reaction	Simmons-Smith reaction	Nitrations
Organolithium reaction	Wittig reaction	Amination
Alkylation	Diketene reactions	Methylation
Bromination	Oxidation	Saponification
Chlorination	BOC-protection	Dehydrogenation
Nitrosation		Hydrogenation
DIBAL reduction		
Ozonolysis		

*Lonza classification

Optional

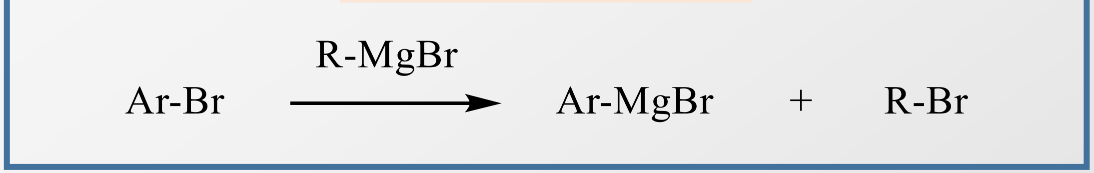
Type A reaction examples

Lithium exchange



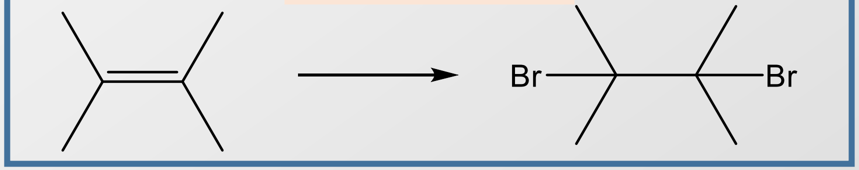
$$\text{Ar-X} \xrightarrow{\text{R-Li}} \left[\text{Ar-Li}\right] \xrightarrow{\substack{\text{Electrophile}\\ \text{E}}} \text{Ar-E}$$

Grignard exchange



$$\text{Ar-Br} \xrightarrow{\text{R-MgBr}} \text{Ar-MgBr} + \text{R-Br}$$

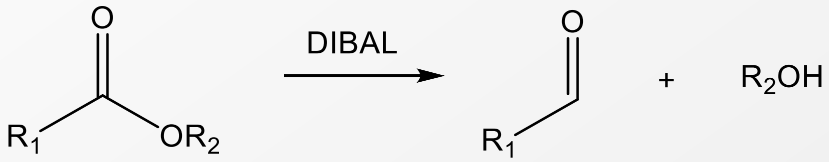
Bromination

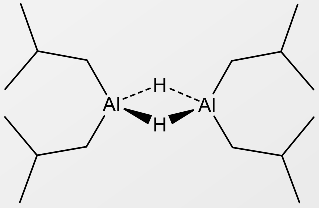


Optional

Type A reaction examples

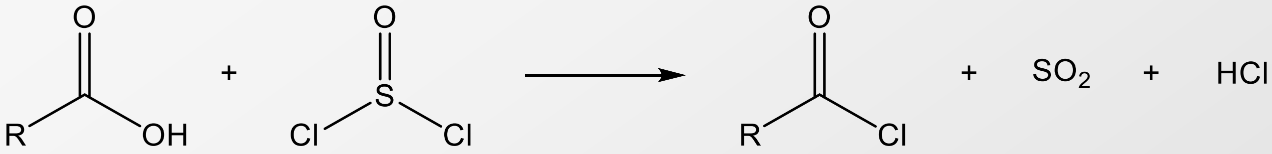
DIBAL reduction



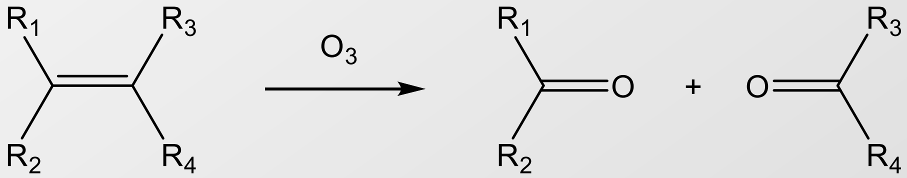


DIBAL

Chlorination

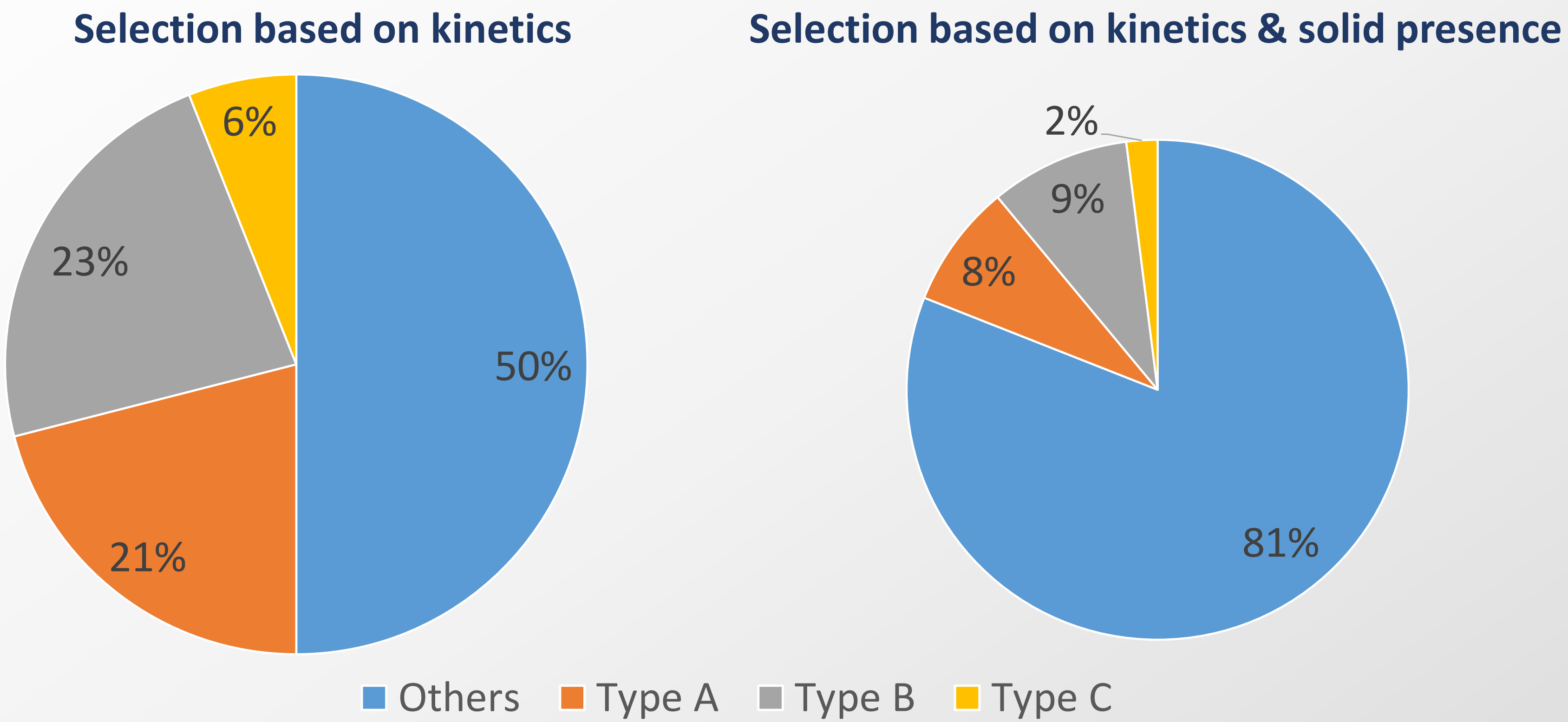


Ozonolysis



Lonza analysis*

Candidates for transfer from batch to continuous using plate millireactors



50% reactions would benefit from continuous reactor
62% of the 50% contain solids, hindering the use of microreactors
⇨ **19% would benefit from continuous reactor**

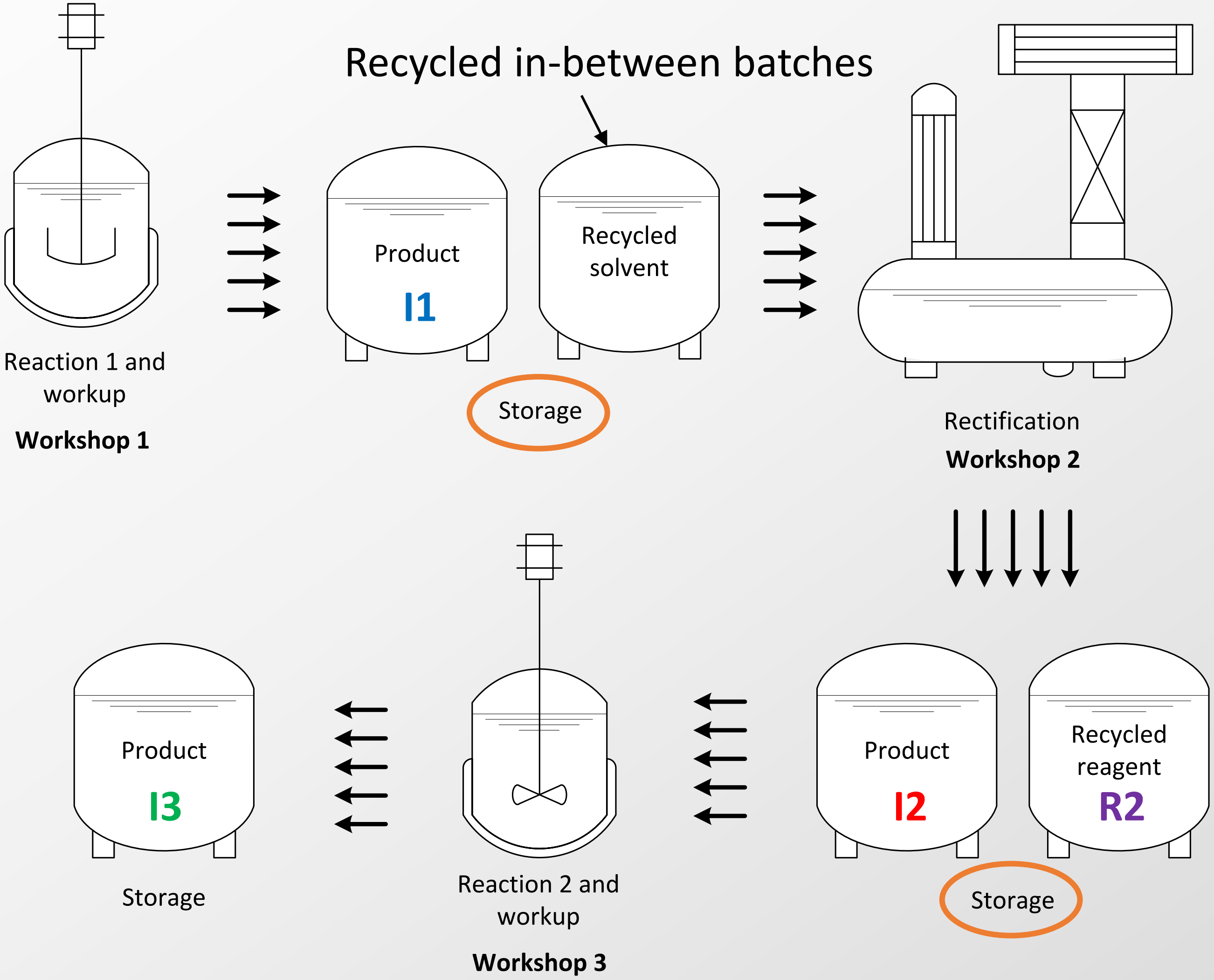
**Roberge et al., Chem. Eng. Technol. 2005, 28, 3, 318-323.* *Roberge, Organic Process Research & Development 2004, 8, 1049-1053*

Other advantages of continuous processing

- Suppression of intermediate storage → reduced **inventory** → cost savings (next slides)
- Reduced **lead-time** (no campaigns)
- Heat integration → **energy** savings (next slides)
- Reduced **manpower** requirements (more automation, steady state operation)
- Reduced plant **footprint** (more compact equipment)
- Increased selectivity → reduced impurity levels → reduced **separation** costs

Batch processing by campaigns

Inventory vs time



Recycled in-between batches

Reaction 1 and workup

Workshop 1

Product

I1

Recycled solvent

Storage

Rectification

Workshop 2

Product

I2

Recycled reagent

R2

Storage

Reaction 2 and workup

Workshop 3

Product

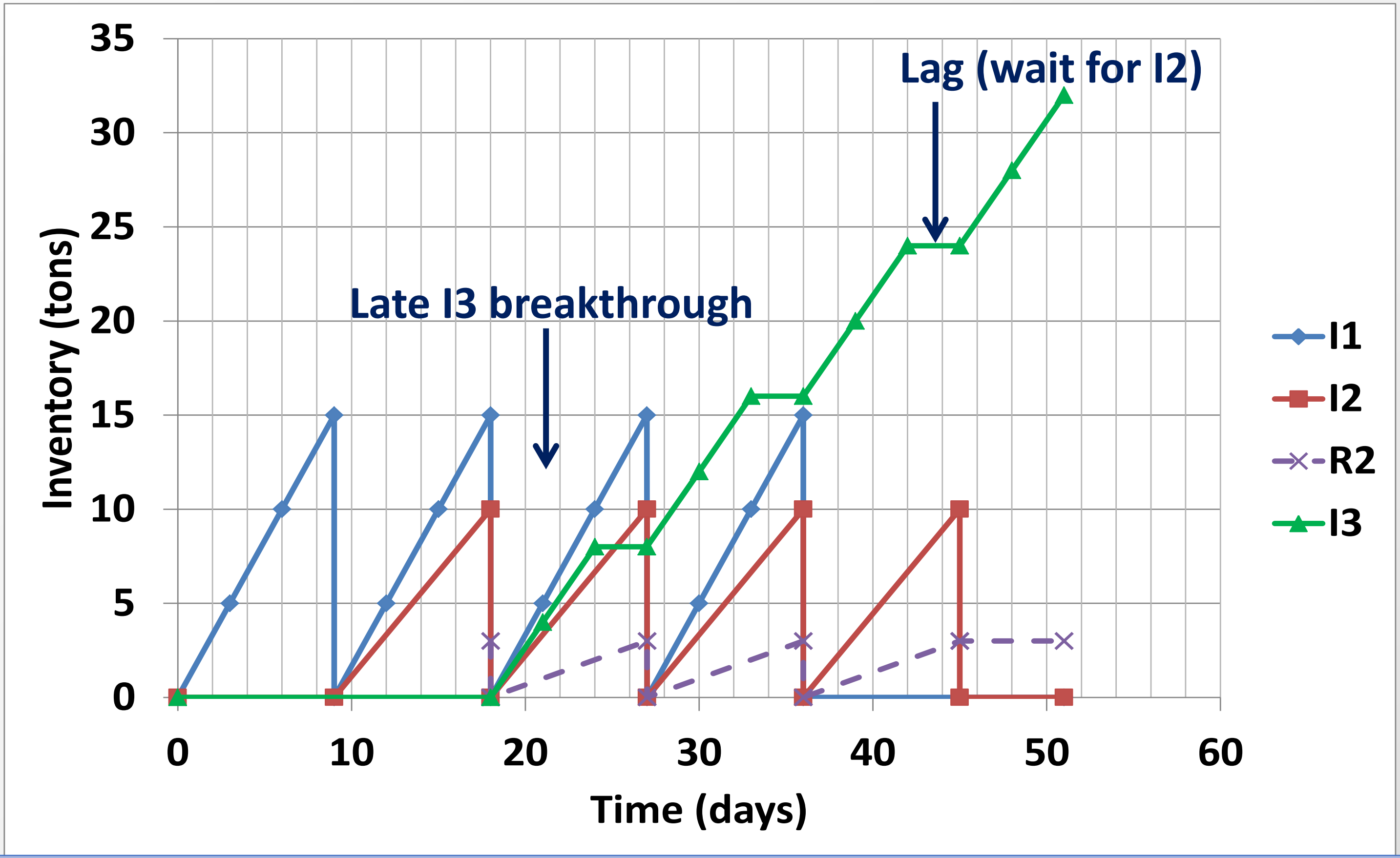
I3

Storage

Batch processing by campaigns

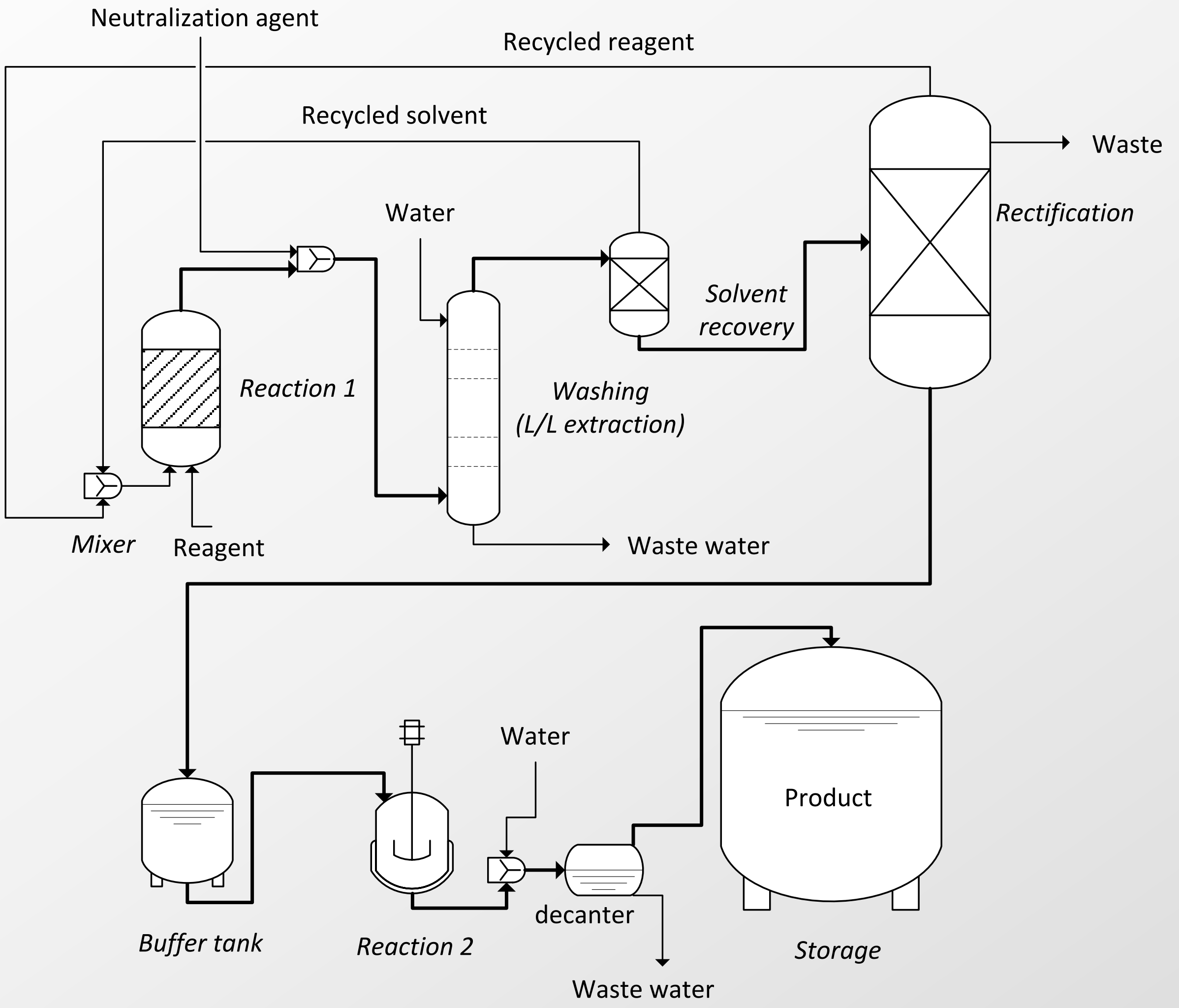
Inventory of key intermediates and recycled reagent vs time

3 × reaction 1 (3 days/batch) → 1 × rectification (9 days/batch) → 2 × reaction 2 (3 days/batch)



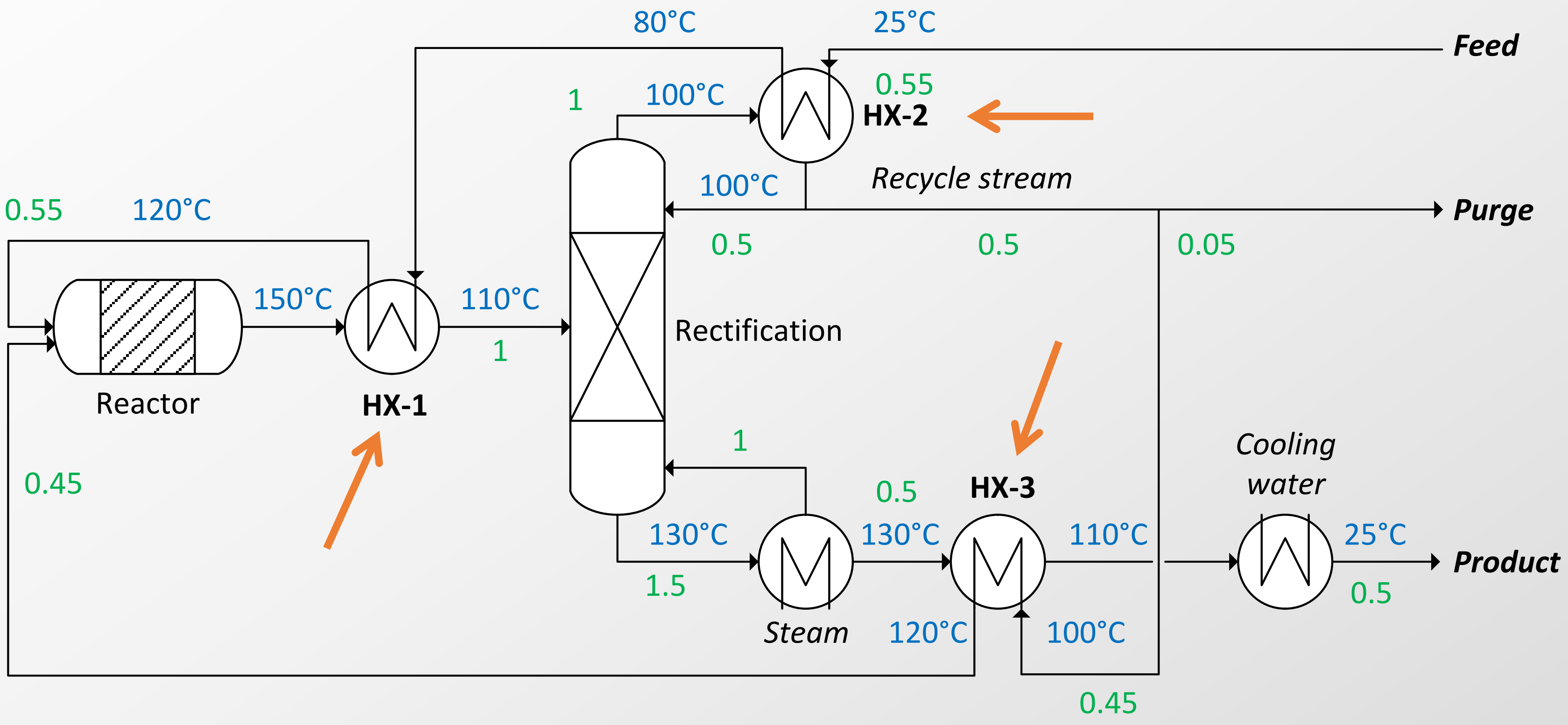
- Large quantities of I1, I2 and R2 are immobilized (cost)
- High lead-time (late product breakthrough, lag times if imperfect synchronization)

Continuous processing



No more storage of I1, I2 and R1. Only small buffer tanks for operational continuity

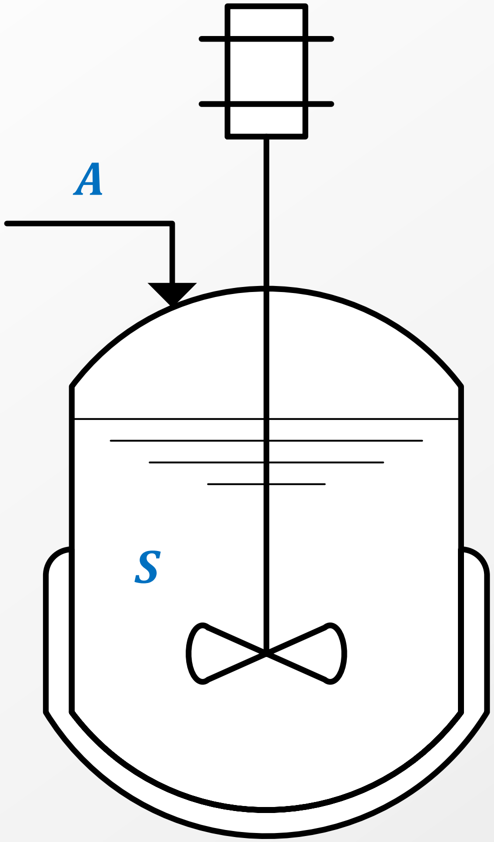
Example of heat integration in a continuous process



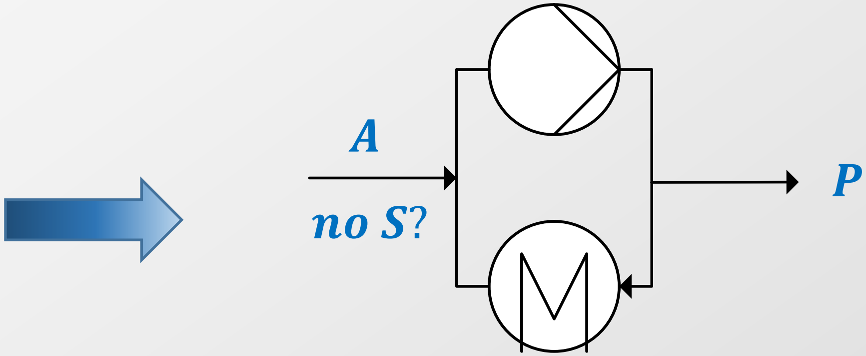
5. Examples

Example (exercise 9)

$\boxed{A \xrightarrow{S} P}$ Fast exothermic reaction of type B



Batch reactor



Loop reactor

Example (exercise 10)

Optional

$$\boxed{A + B \xrightarrow{r_1} C \xrightarrow{r_2} D}$$

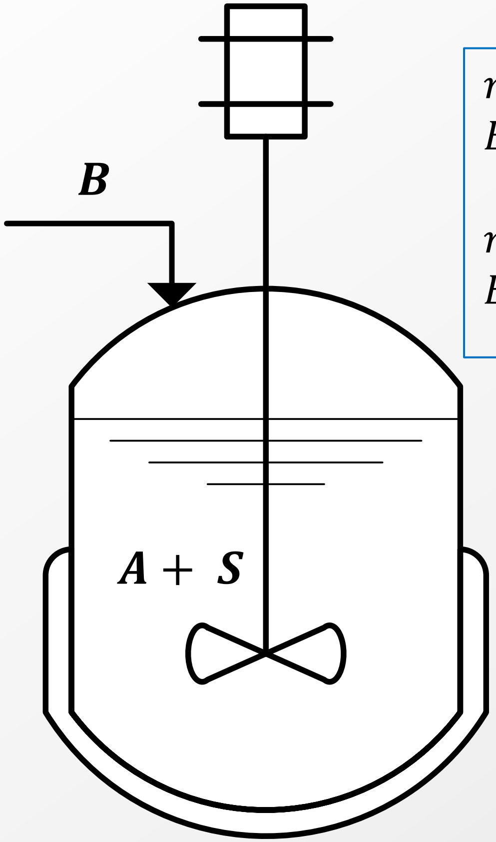
C is an unstable hydroperoxide

$r_1 = k_1 c_A c_B \; (mol \cdot m^{-3} \cdot s^{-1})$ $\quad k_{01} = 4 \cdot 10^4 \; m^3 \cdot mol^{-1} \cdot s^{-1}$

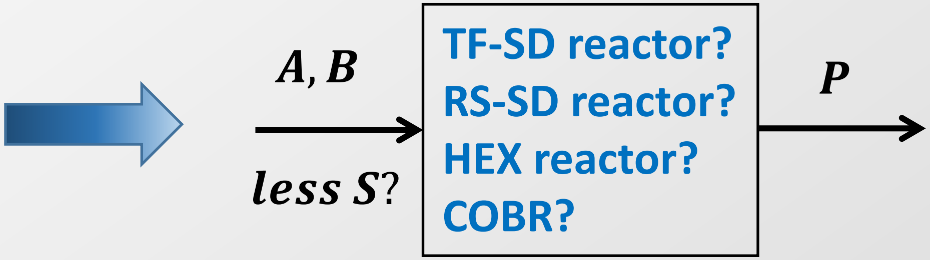
$E_{a1} = 50 \; kJ \cdot mol^{-1}$ $\quad \Delta H_{r1} = -50 \; kJ \cdot mol^{-1}$

$r_2 = k_1 c_C \; (mol \cdot m^{-3} \cdot s^{-1})$ $\quad k_{02} = 10^{15} \; s^{-1}$

$E_{a2} = 120 \; kJ \cdot mol^{-1}$ $\quad \Delta H_{r2} = -150 \; kJ \cdot mol^{-1}$



Batch reactor

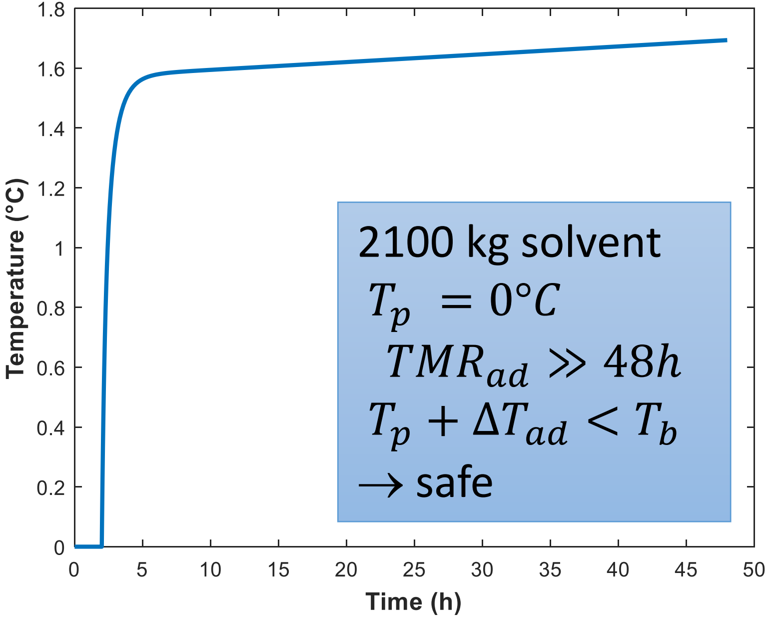


TF-SD reactor?
RS-SD reactor?
HEX reactor?
COBR?

Example (exercise 10)

Optional

Temperature profile with cooling failure at end of addition



2100 kg solvent

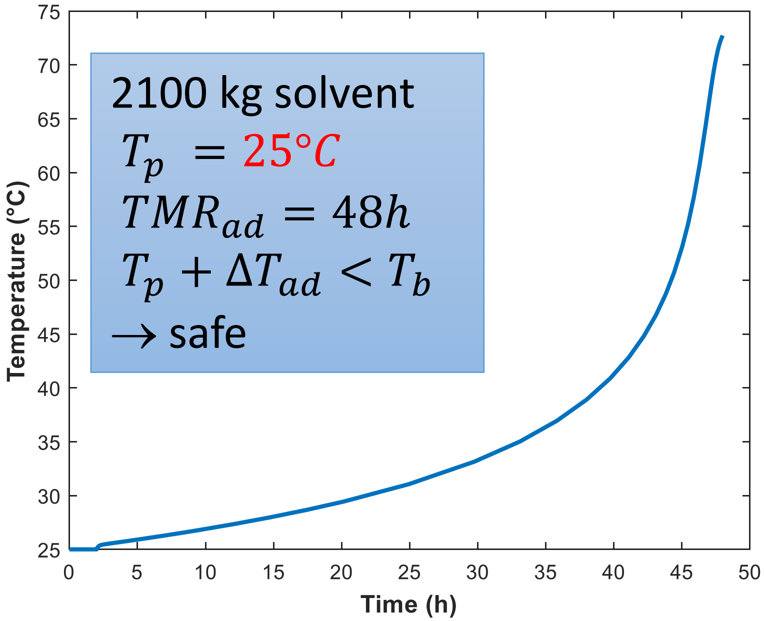
$T_p = 0^\circ C$

$TMR_{ad} \gg 48h$

$T_p + \Delta T_{ad} < T_b$

→ safe

Higher process temp.



2100 kg solvent

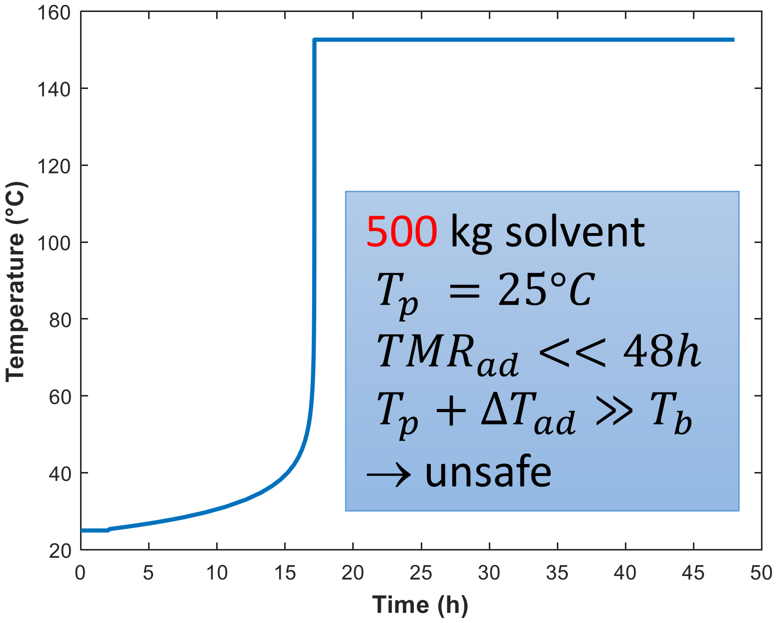
$T_p = 25^\circ C$

$TMR_{ad} = 48h$

$T_p + \Delta T_{ad} < T_b$

→ safe

Less solvent



500 kg solvent

$T_p = 25^\circ C$

$TMR_{ad} << 48h$

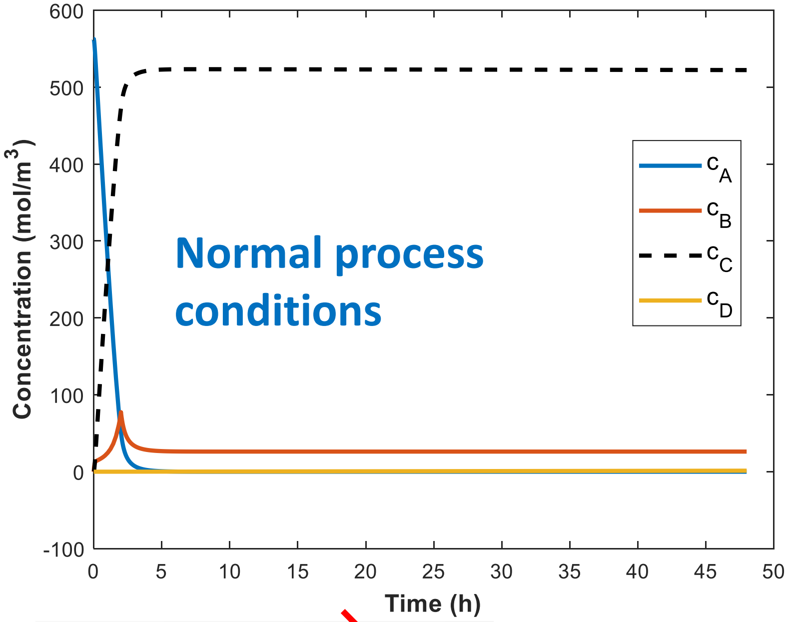
$T_p + \Delta T_{ad} \gg T_b$

→ unsafe

Example (exercise 10)

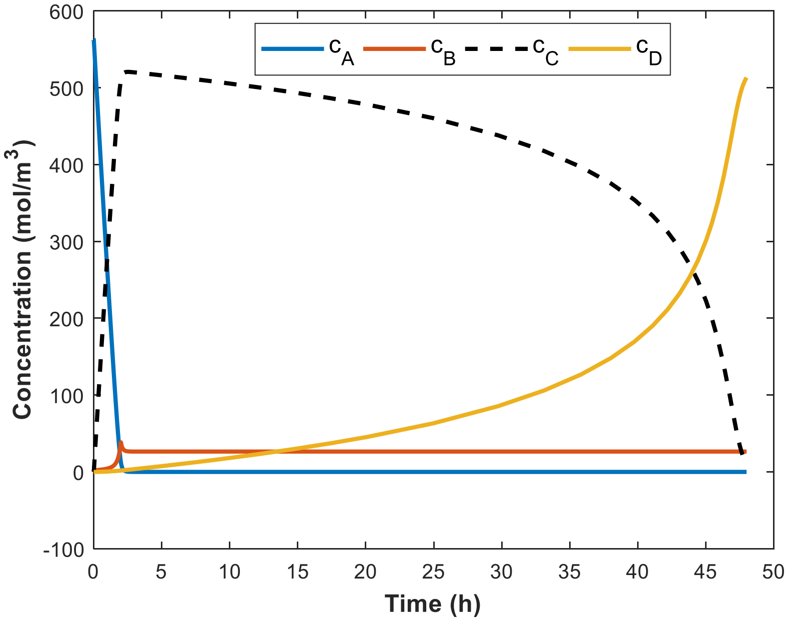
Optional

Concentration profiles with cooling failure at end of addition



Normal process conditions

Higher process temp.



Less solvent