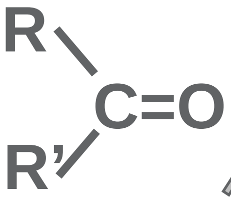
CORNING

Engineered reactors for Chemicals Industrial Production

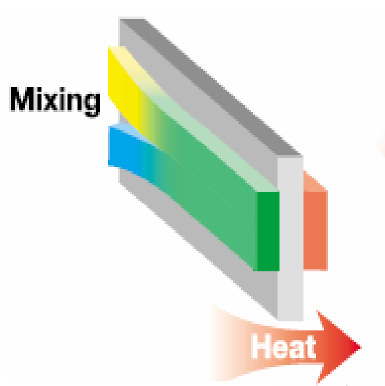
PIN meeting April 26th, 2007

Philippe CAZE

Corning focus:
The reactor and its integration into production system

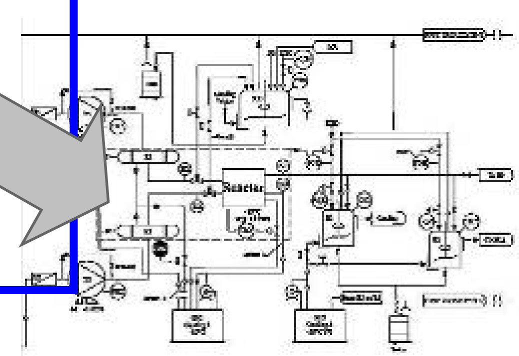


Chemistry



Mass and heat transfer

System engineering



Reactor engineering

Corning designs and manufactures From glass microstructures to engineered reactors

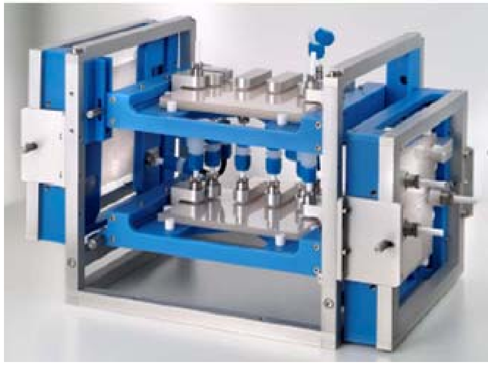
- **Glass compositions**
  - Chemical compatibility with chemicals and solvents
  - High chemical corrosion resistance with almost all chemicals

- **Design of micro structures**
  - Flexibility in design

- **Unique, proprietary microstructure manufacturing technology**

- **Reactor engineering**
  - Methodology and experience to leverage existing customer data/know-how to define the continuous reactor
  - Basic and detailed engineering from pilot runs to full scale production

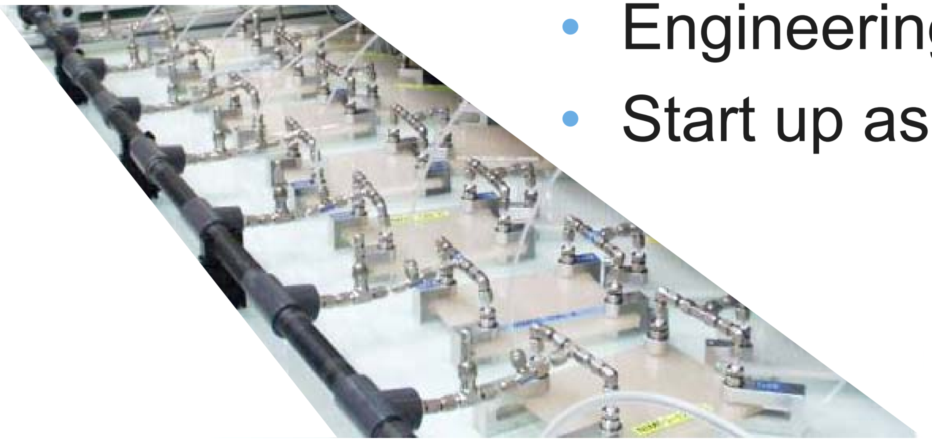
Corning product
Engineered reactor and service



Reactor

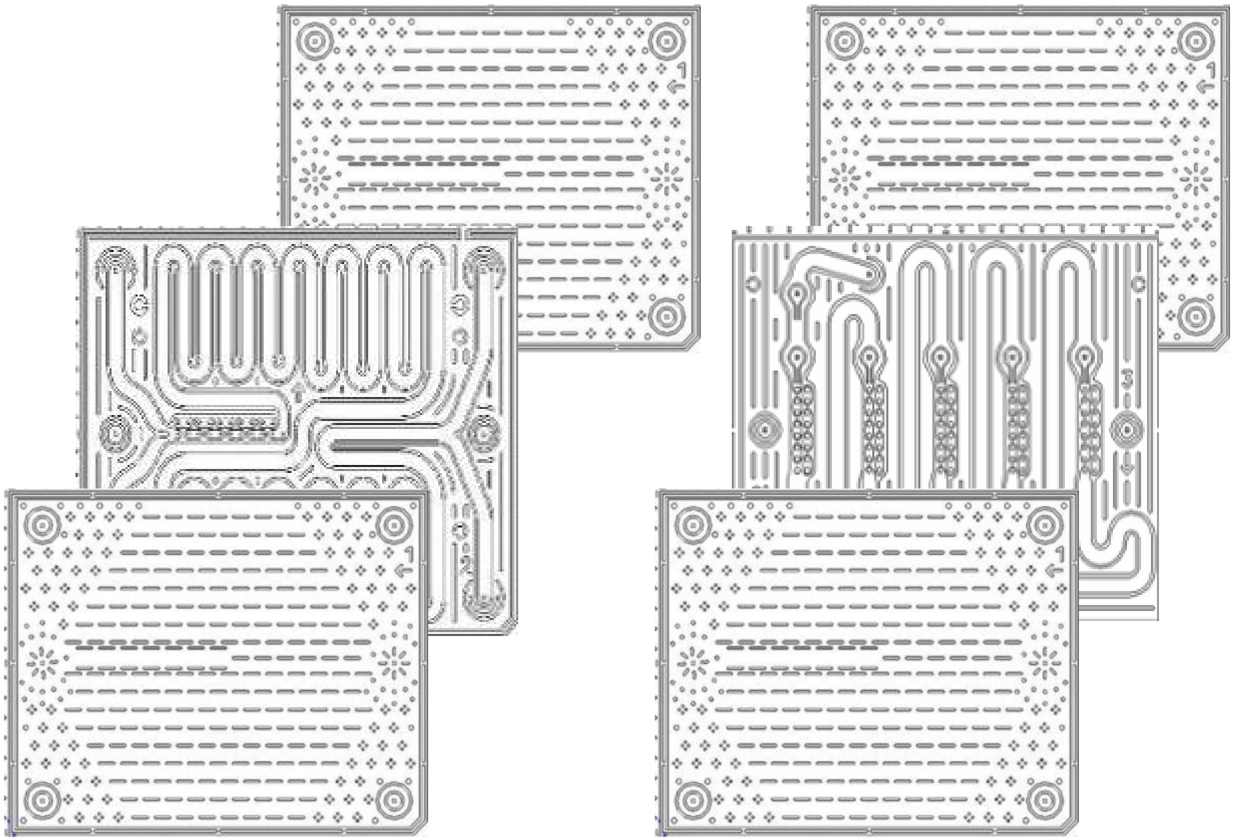
- Basic and detailed engineering
  - Reactor
  - Product synthesis module
  - Numbering up
- Feasibility studies
- Customization
- Engineering services
- Start up assistance

Product synthesis unit



Corning Microreaction technology

Mass and heat transfer are combined & integrated into each microstructure



Heat transfer

Mass transfer

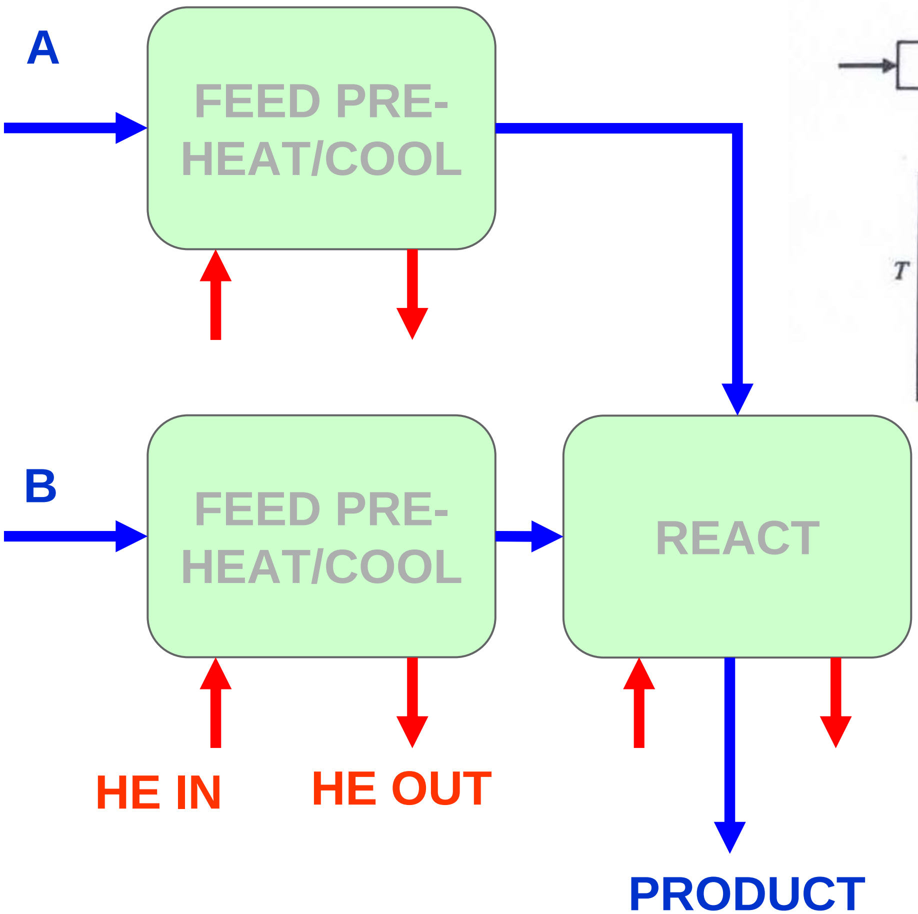
Heat transfer

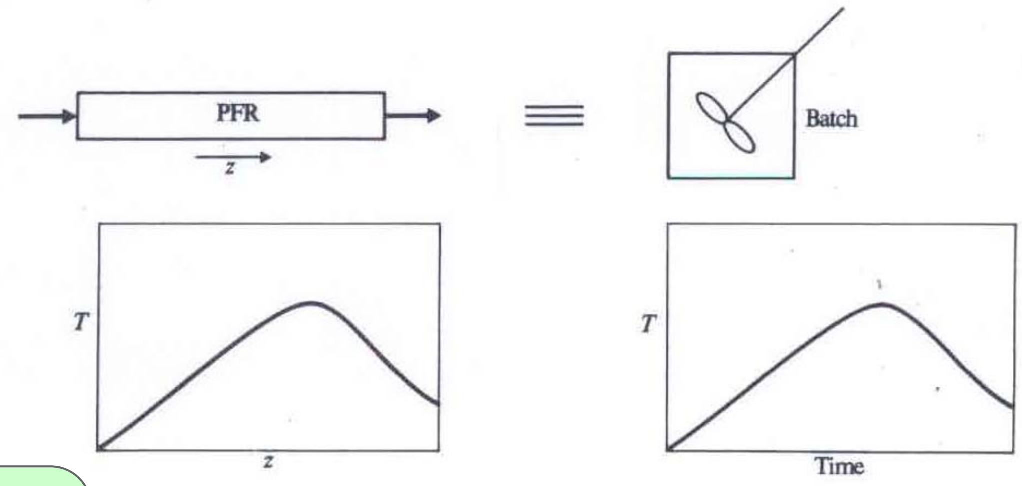
Micro reactor for Industrial Production

Targeted product Metric tons/Year per reactor	Flow rate (Kg/hour)					
Reactants concentration (wt%)	1	2	3	5	10	12
10	1	2	2	4	8	10
20	2	3	5	8	17	20
30	2	5	7	12	25	30
50	4	8	12	21	42	50
70	6	12	17	29	58	70
100	8	17	25	42	83	100

Assumptions	Conversion	100	%
	Selectivity	95	%

Single-injection reactor





$$\frac{dF_i}{dV} = \sum_{j=1}^{M_R} \nu_{ij} r_j$$

for constant flow rate :

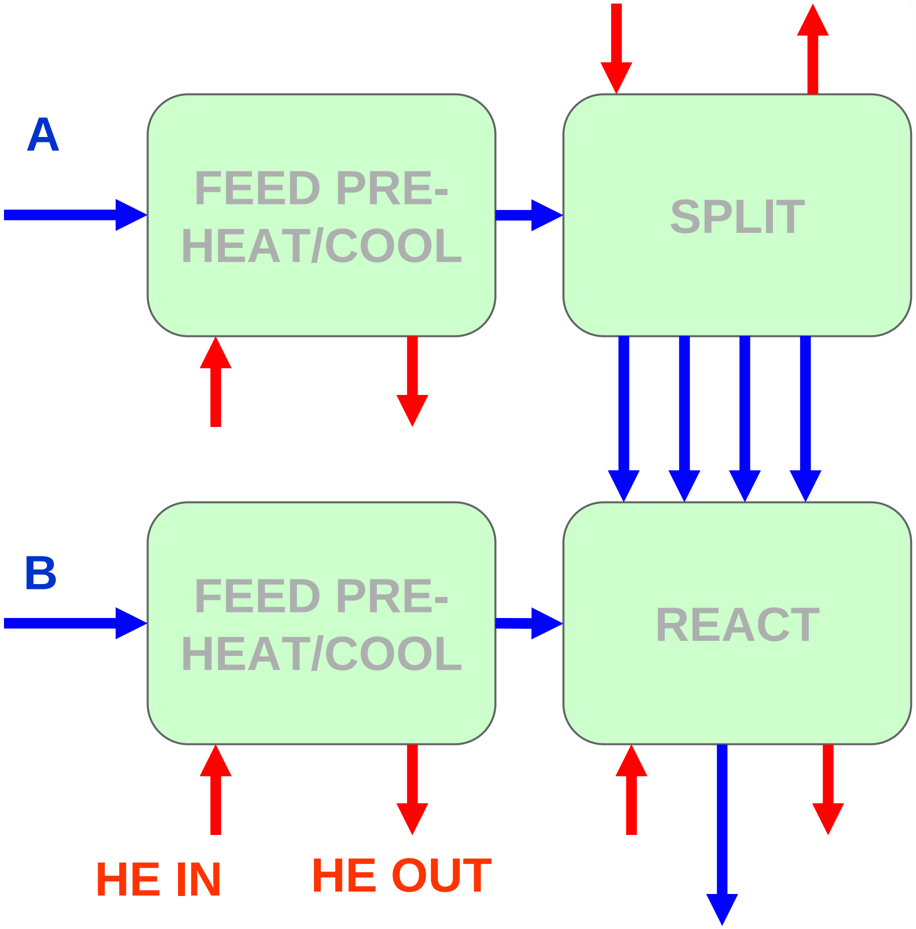
$$\frac{dC_i}{d\tau} = \sum_{j=1}^{M_R} \nu_{ij} r_j$$

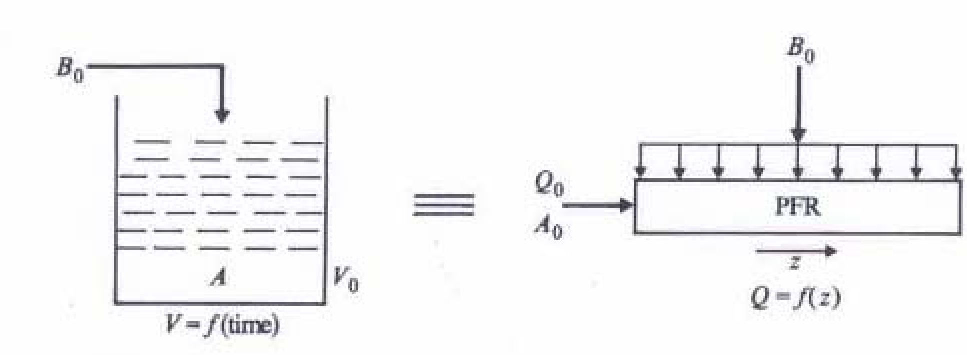
where $\tau = V/Q$

$$\frac{dC_i}{dt} = \sum_{j=1}^{M_R} \nu_{ij} r_j$$

where $C_i = N_i/V$

Multi-injection reactor





$$(1)\quad \frac{dx_B}{d(t/t_f)} = 1 - kC_A t_f (1 - x_B)$$

for the semi-batch reactor

and

$$(2)\quad \frac{dx_B}{d(z/L)} = 1 - kC_A \frac{V_P}{Q} (1 - x_B)$$

for the multi-injection reactor.

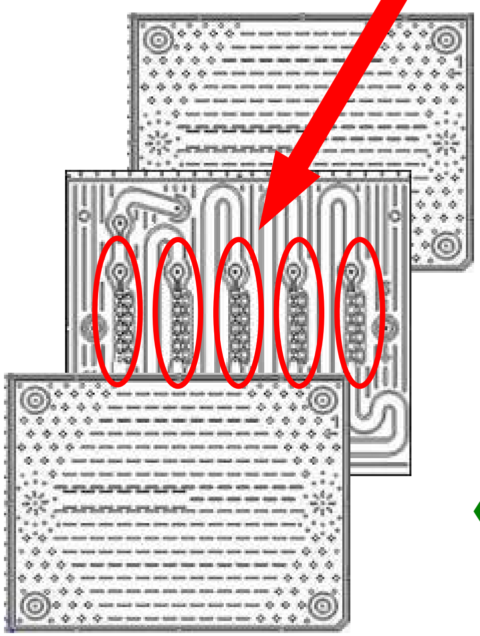
Acknowledgement:
Michael T. Klein, Dean School of Engineering
Rutgers, The State University of New Jersey

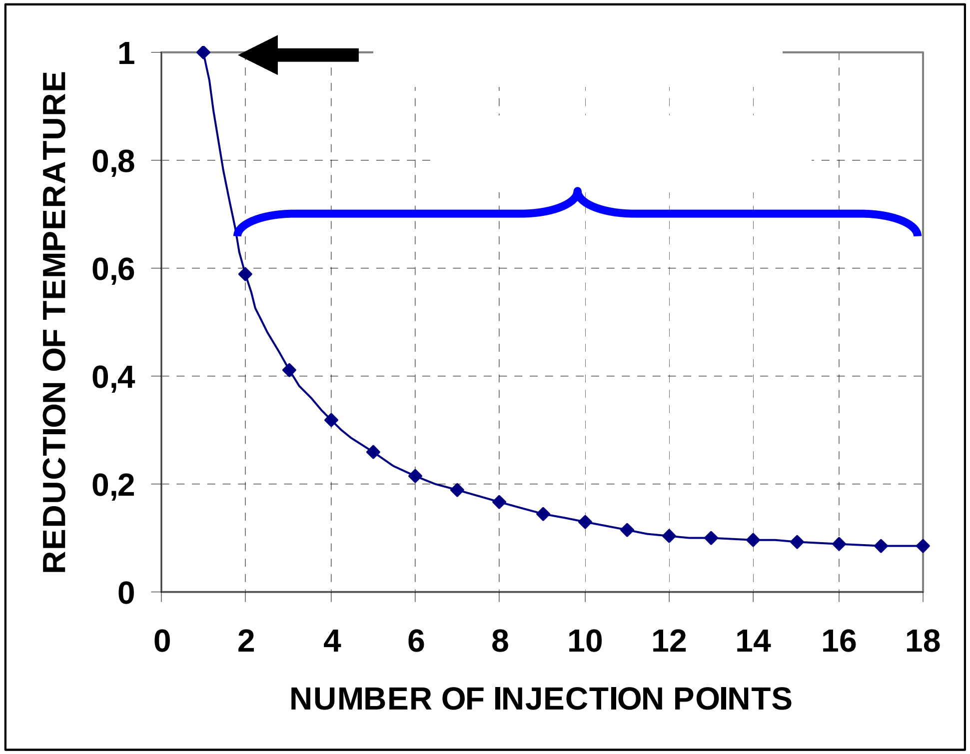
Multi-injection: Better temperature management along the flow path

$$\rho C_p \frac{dT}{d\tau} = kA(-\Delta H) - U\cdot\left(S/V\right)\cdot(T-T_c)$$

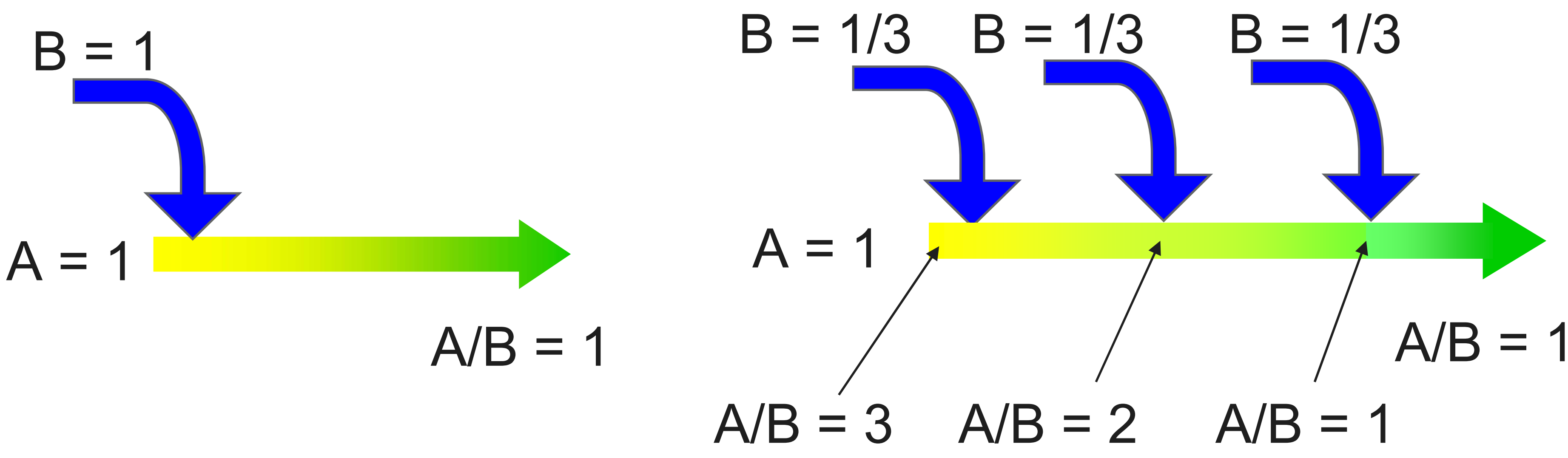
Heat removal

Heat generation



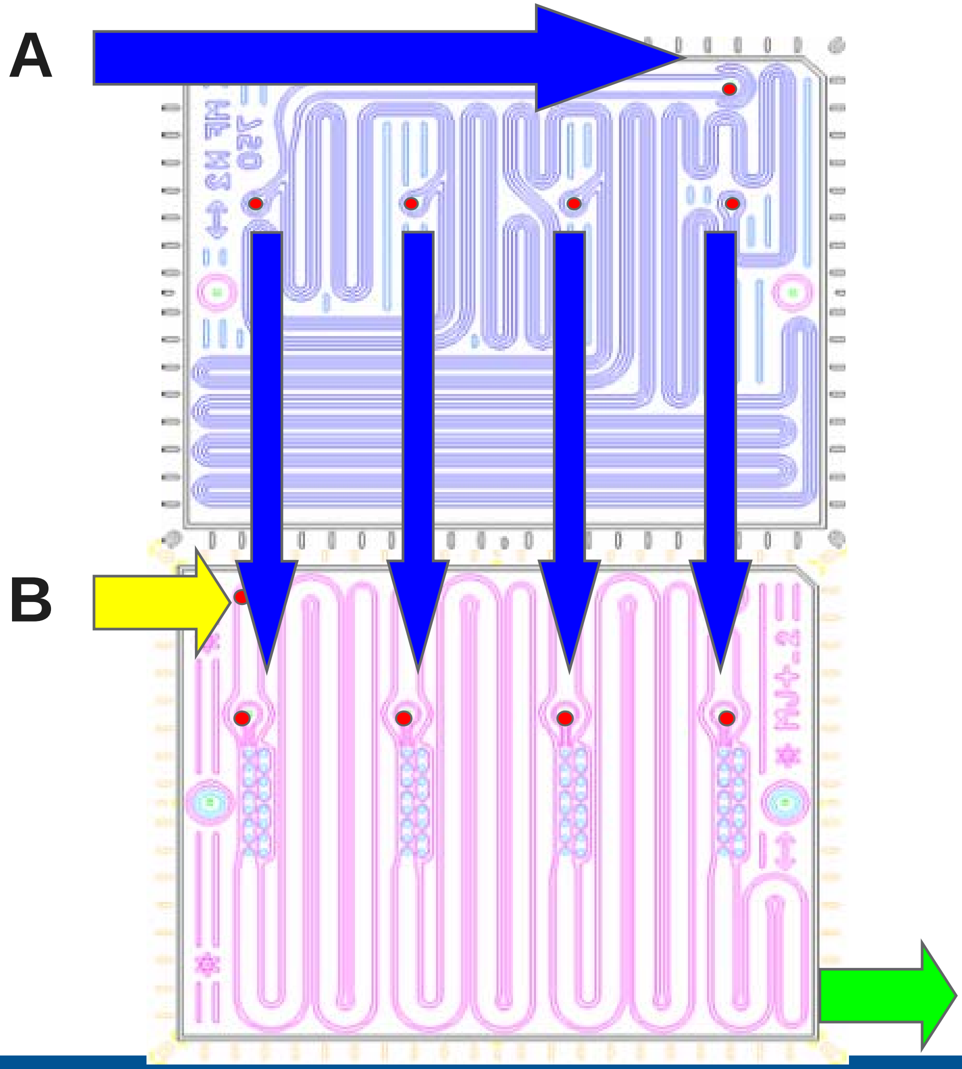


Local molar ratio management



In both cases, the incoming feeds molar ratio is the same

Multi-injection: Only two pumps are required

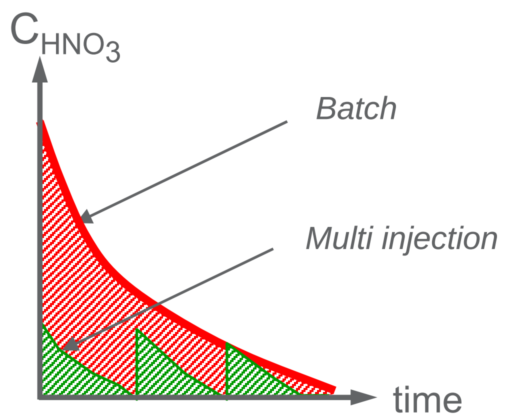


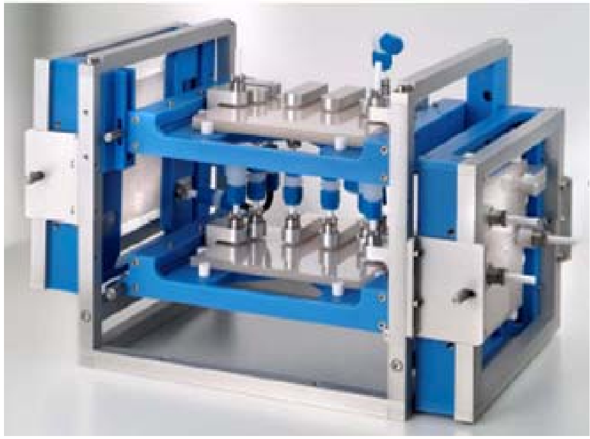
Pressure drop management and fluid split within the micro-structures

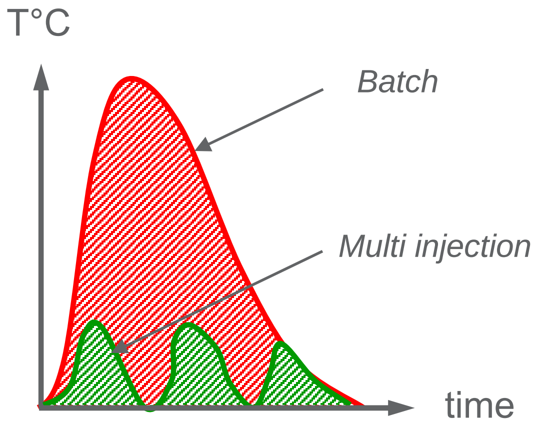
Multi injection reactor module

- Avoid local over concentration of active species
- Eliminate hot spots

→ Less by product formation



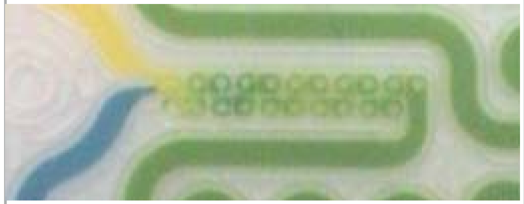
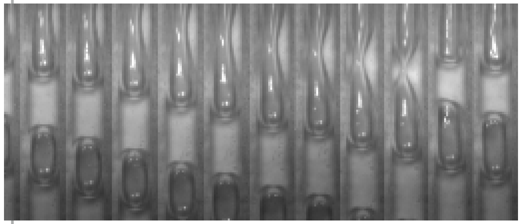




Example of use:

$$C_6H_6 \xrightarrow{HNO_3} C_6H_6NO_2 \xrightarrow{HNO_3} C_6H_6(NO_2)_2$$

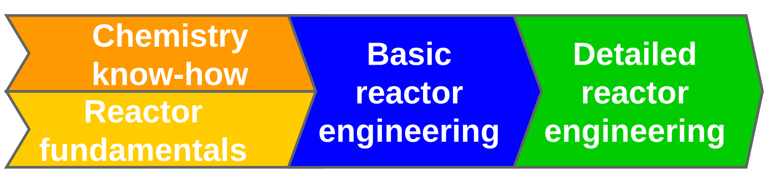
Multi-phase mass transfer

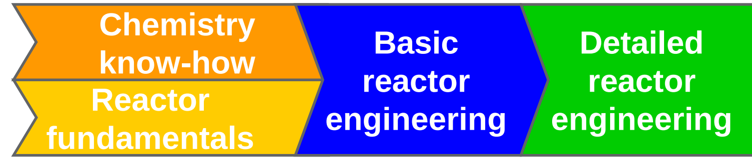
Feeds	Mixing test	Results	Hydrodynamic visualization
L	Villermaux method *Villermaux & all, AIChE Symp. Ser. 88 (1991) 6, 286.*	Mixing quality > 90% for flow rates > 1.8 L/h	
L/L	Polystyrene precipitation *Chem. Eng. Technol.* 2005, *28*, 324-330 *Proc. of the 10th APCChE Congress*, 2004, 4B-02	50-100 nm particle size	
L/G	Measure of slug size *Pressure drop in monolith reactors, P. Woehl, R.L. Cerro, Catalysis Today 69 (2001) 171-174* *Flow patterns in liquid slugs during bubble-train flow inside capillaries, Chem Eng Sci 52 (1997) 2947-2962*	0.5 - 10 mm Hydrodynamic regime adapted to needs	

End user application

Reactor engineering process

1. **Chemistry know-how and reactor fundamentals**
   - Chemistry know-how
     - Reaction network and feed distribution
     - Thermodynamic & Kinetics
     - Feed characteristics
   - Reactor fundamentals
2. **Basic engineering : Translate chemistry into a mass and heat transfer question**
   - Single injection / multi-injection
   - Mass and heat transfer principle
3. **Detailed reactor engineering**
   - Identify critical dimensions of the reactor
   - Design and sizing



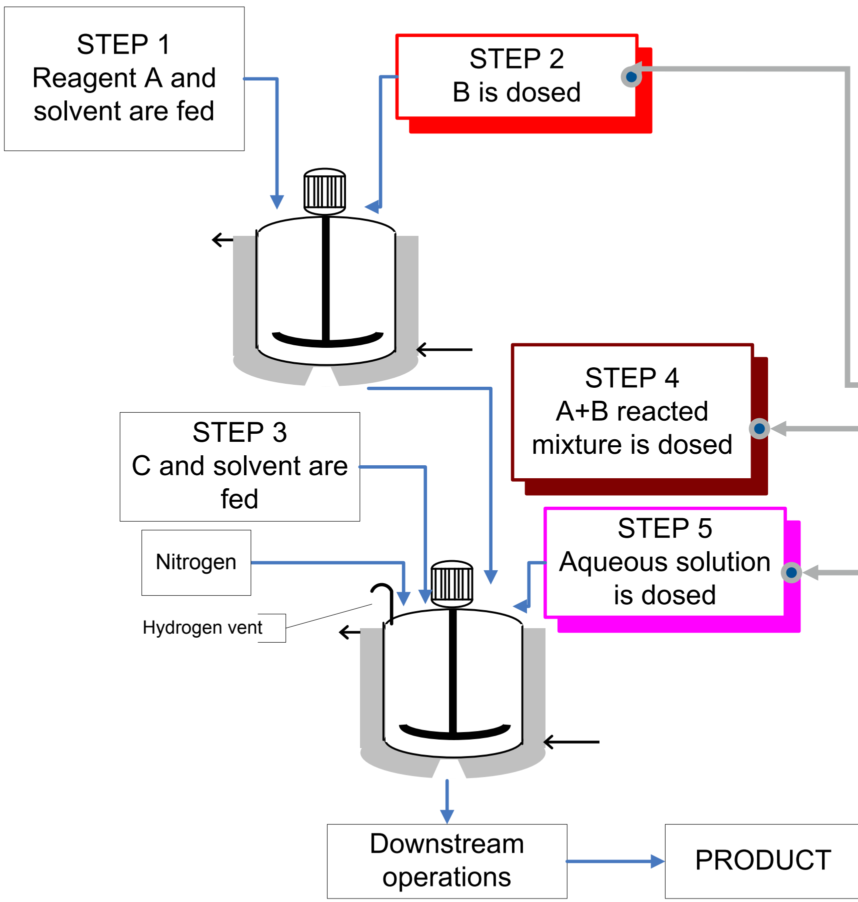


Customer needs

- Safe and smooth production of 40 kg / week / reactor
- Raw material cost > 500 €/kg
- More than 95% conversion
- Impurities below 2%

Chemistry know-how

The chemistry know-how



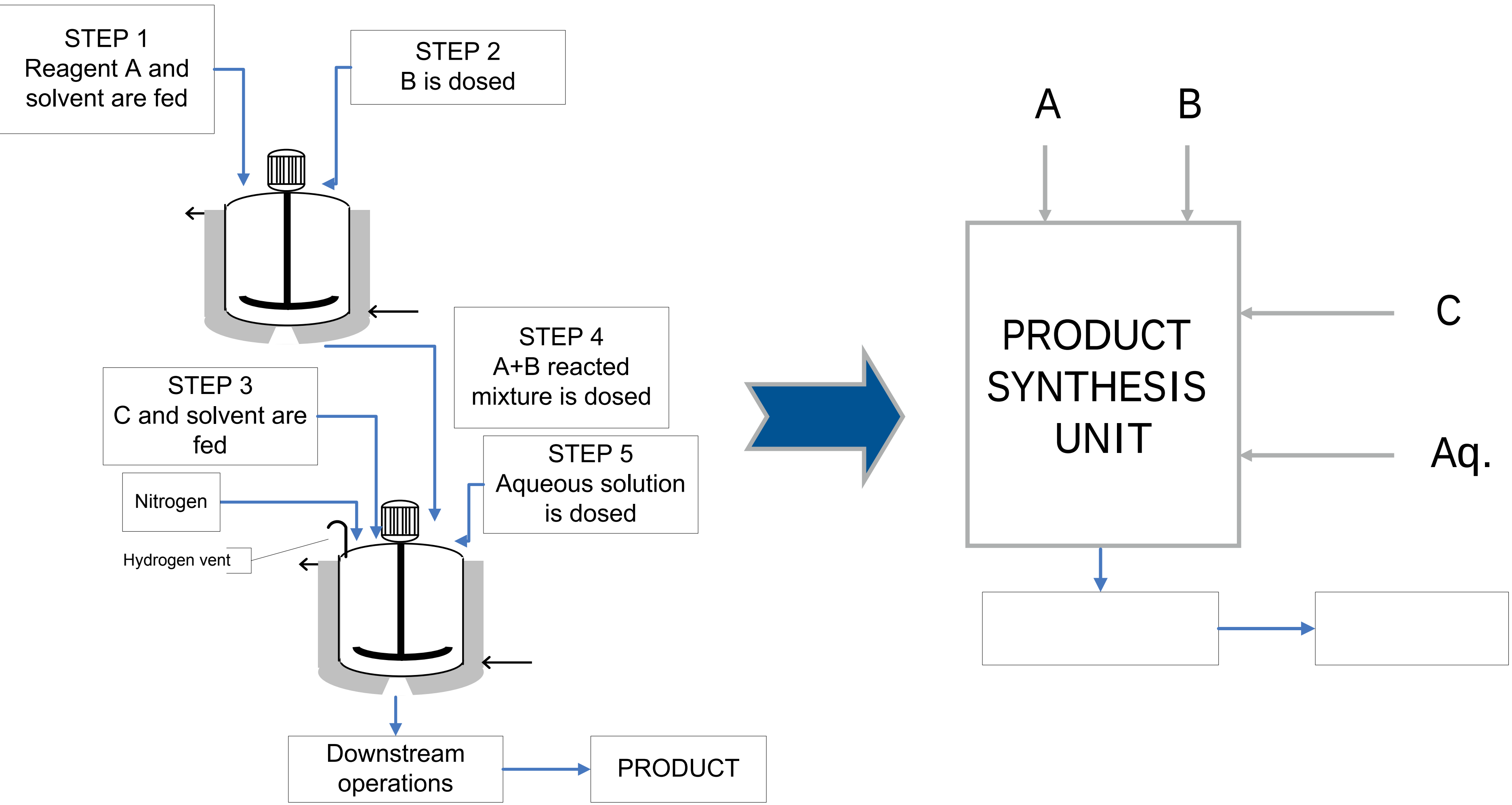
- $A + B \rightarrow C$
- $C + D \rightarrow E$
- $E + H_2 0 \rightarrow$ Product + H2

- Exothermic
- Highly reactive intermediate
- No major side products

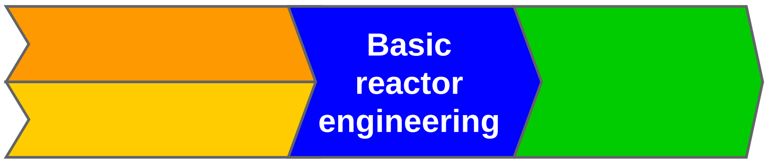
- Exothermic
- Maximum temperature 10°C
- Safety limit : 50 L batch vessel
- Excess of C = Selectivity issue

- Exothermic
- Hydrogen release

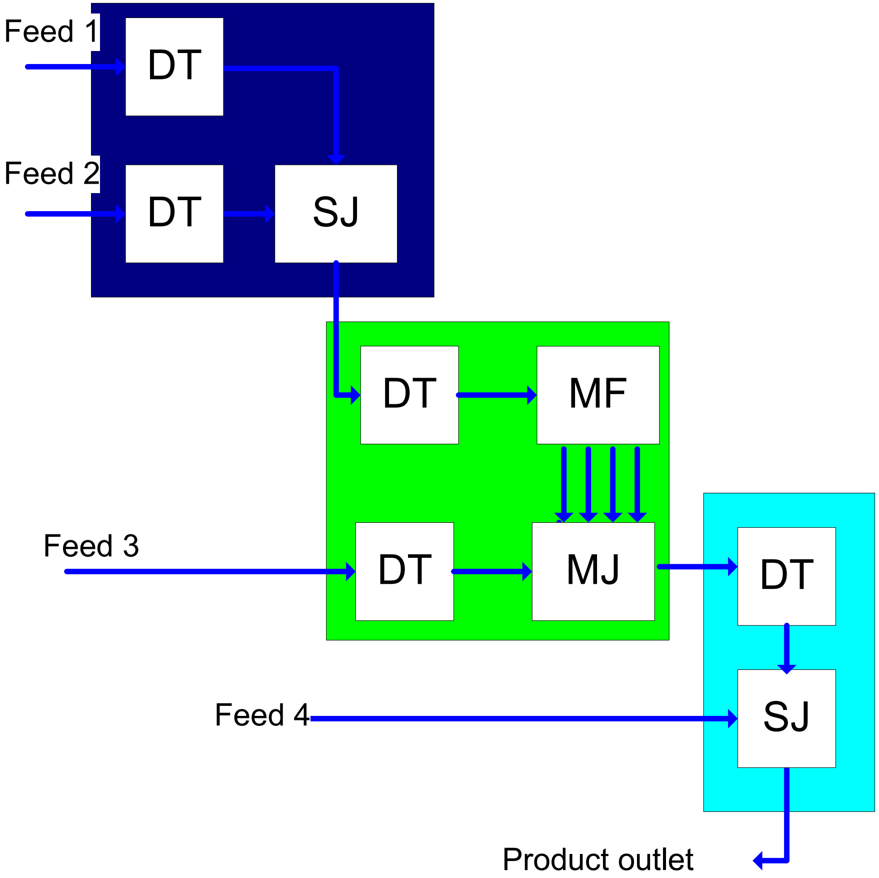
Product synthesis unit



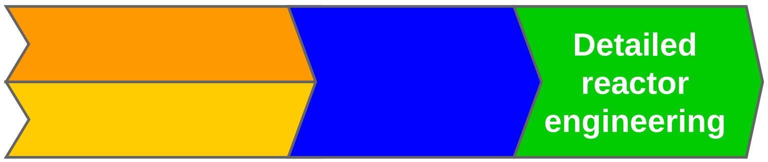
Translation into a mass and heat transfer question

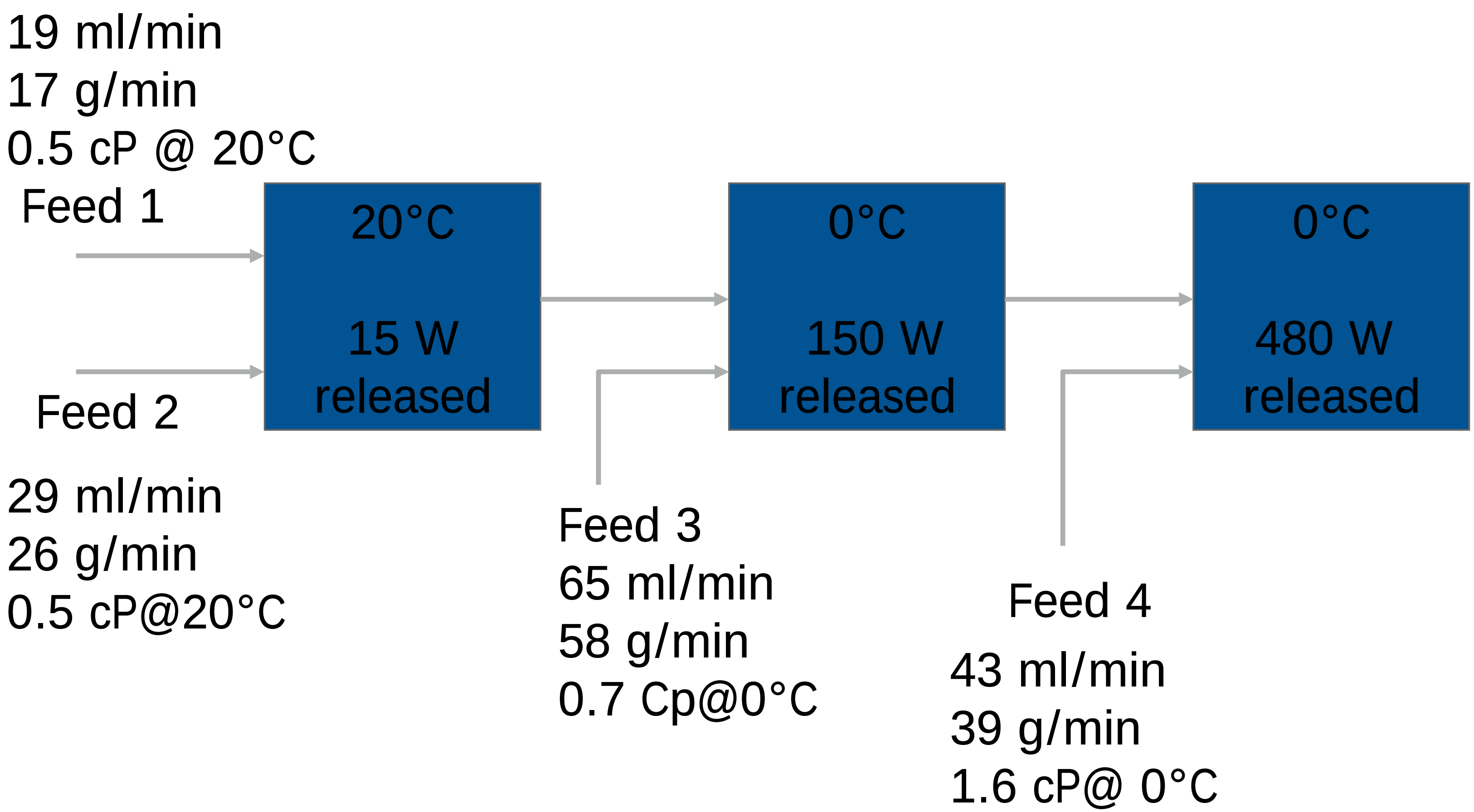


- Step 1 :
  - Mixing and heat exchange integrated
  - Single injection
- Step 2 :
  Mixing and heat exchange integrated
  No excess of C
- Step 3:
  - Mixing and heat exchange integrated
  - Single injection

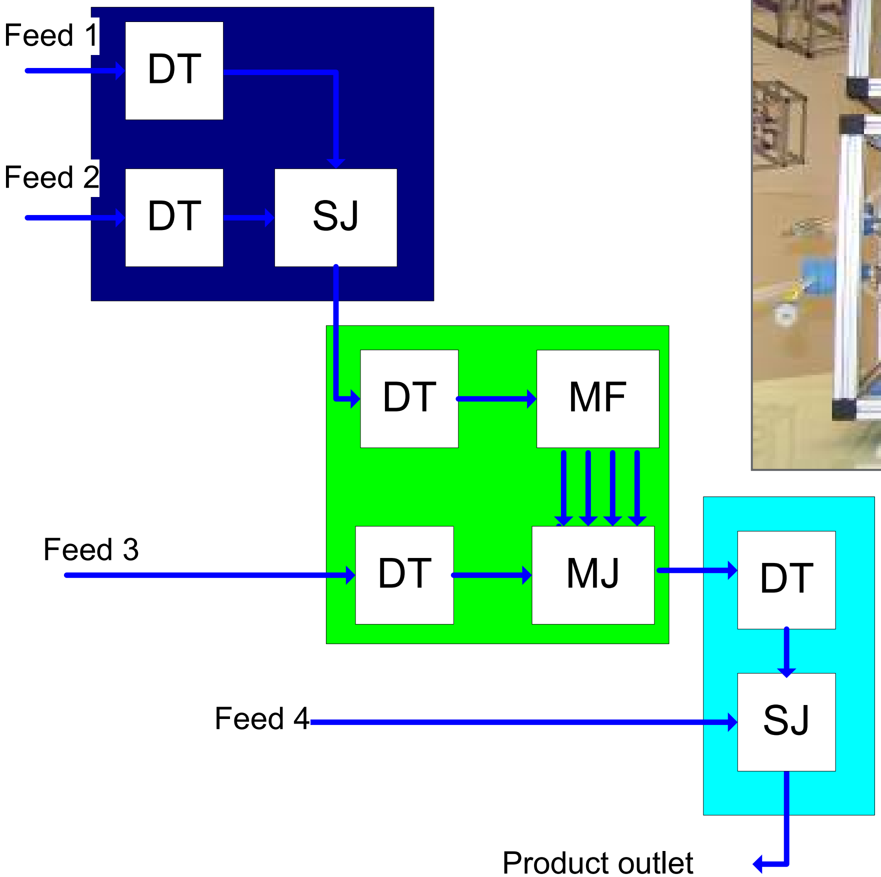


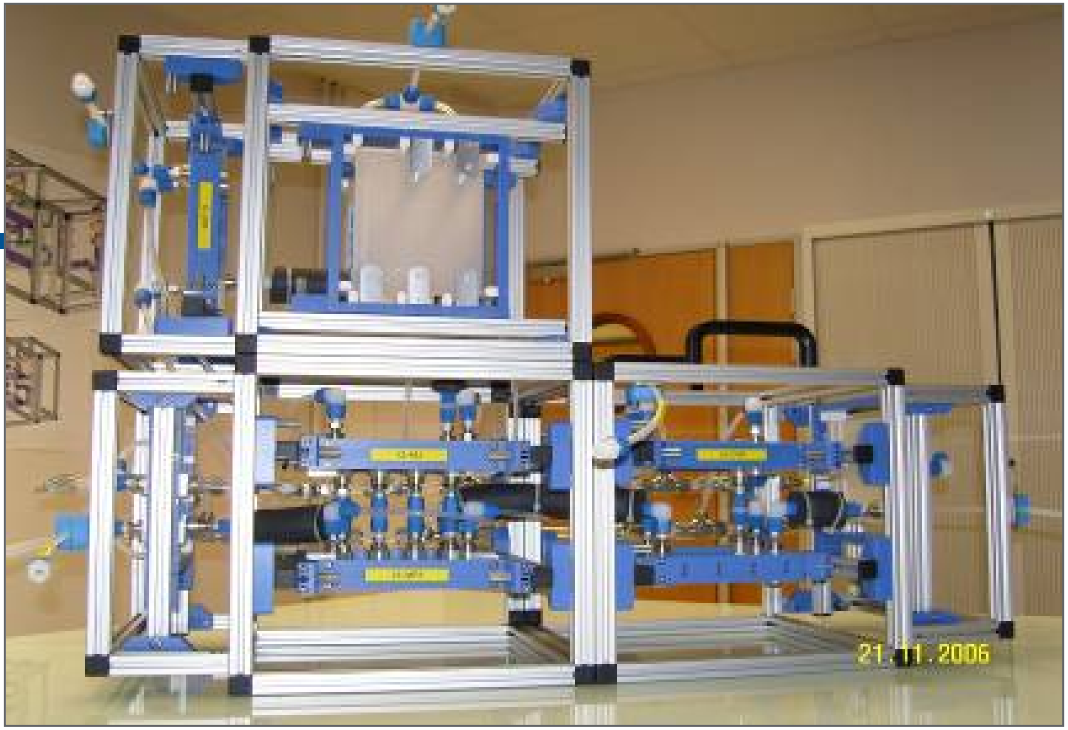
Mass and heat balance: data for reactor sizing





Product synthesis unit



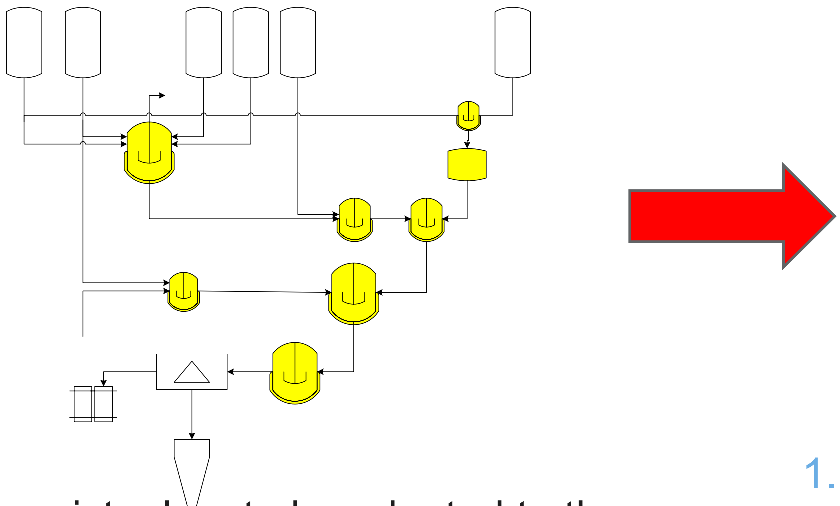


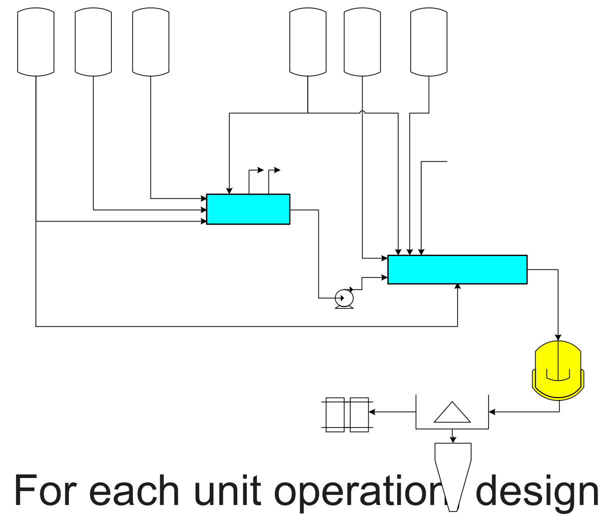
Chemistry know-how / Reactor fundamentals → Basic reactor engineering → Detailed reactor engineering

Throughput: 40 kg / week
99 % conversion
Impurities < 1 %

Pressure: Up to 18 bars
Temperature : -50°C to 40°C
Internal volume : 70 ml

Product synthesis unit





1. Chemistry has to be adapted to the equipment

2. Cascade of discontinuous unit operations in agitated vessels in order to make the product

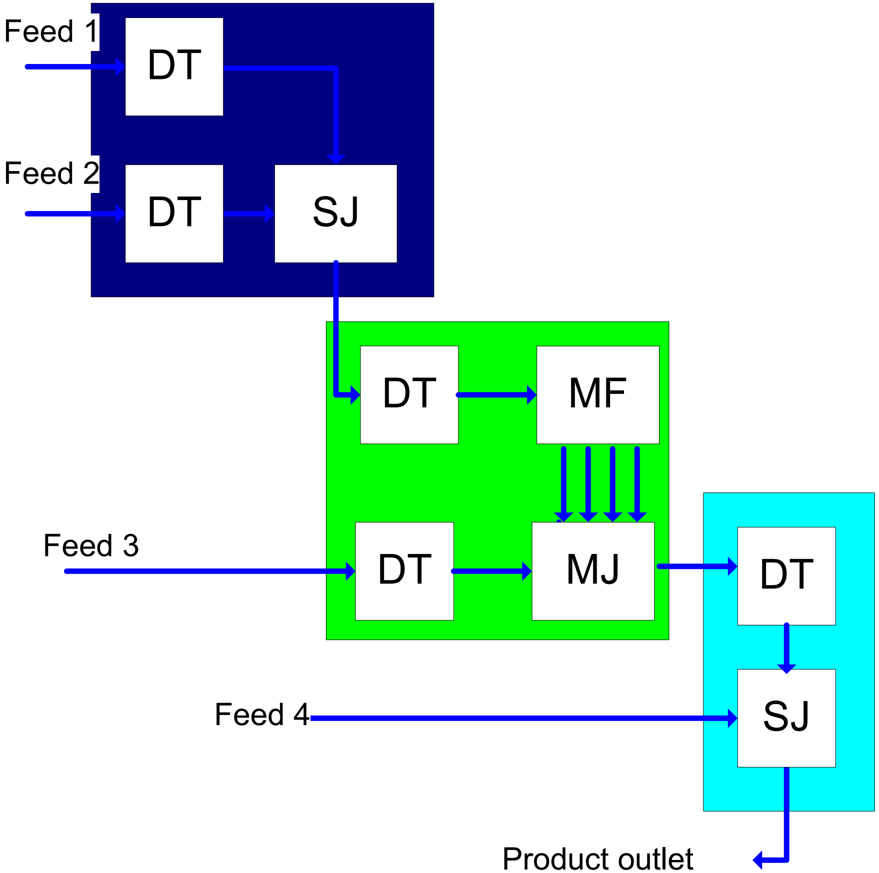
3. Manufacturing capacity is obtained through complex plant and manufacturing train management in campaign

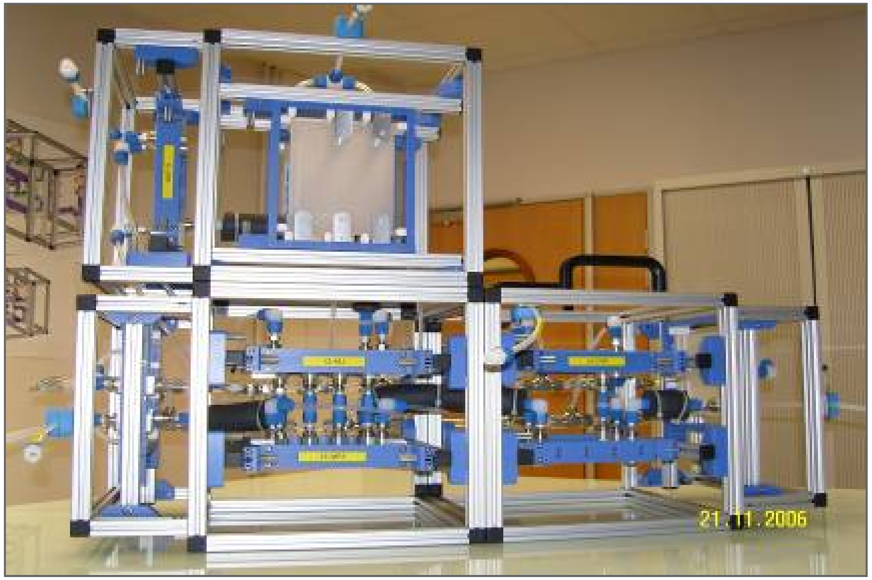
1. For each unit operation design the best engineered reactor module to fit your chemistry

2. Combine several engineered reactor modules into an optimized continuous operation

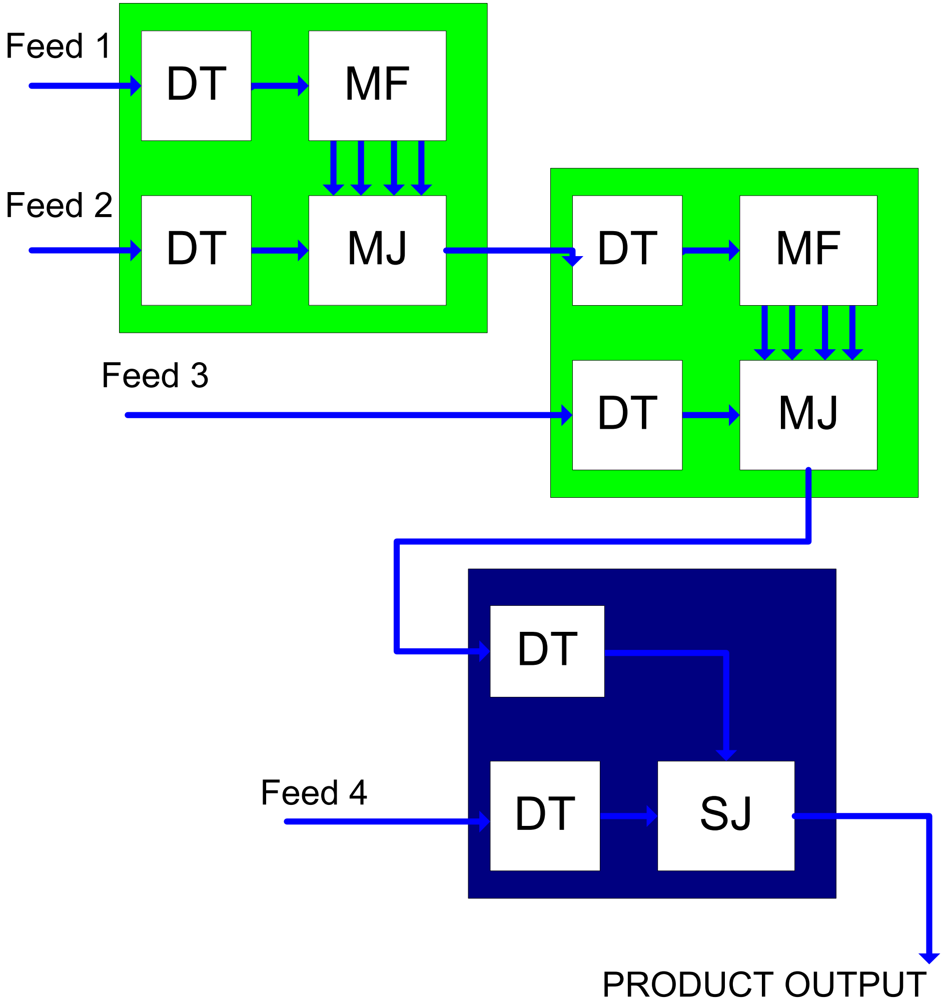
3. Combine several product synthesis module in order to achieve the capacity according to your needs

Reduction reaction

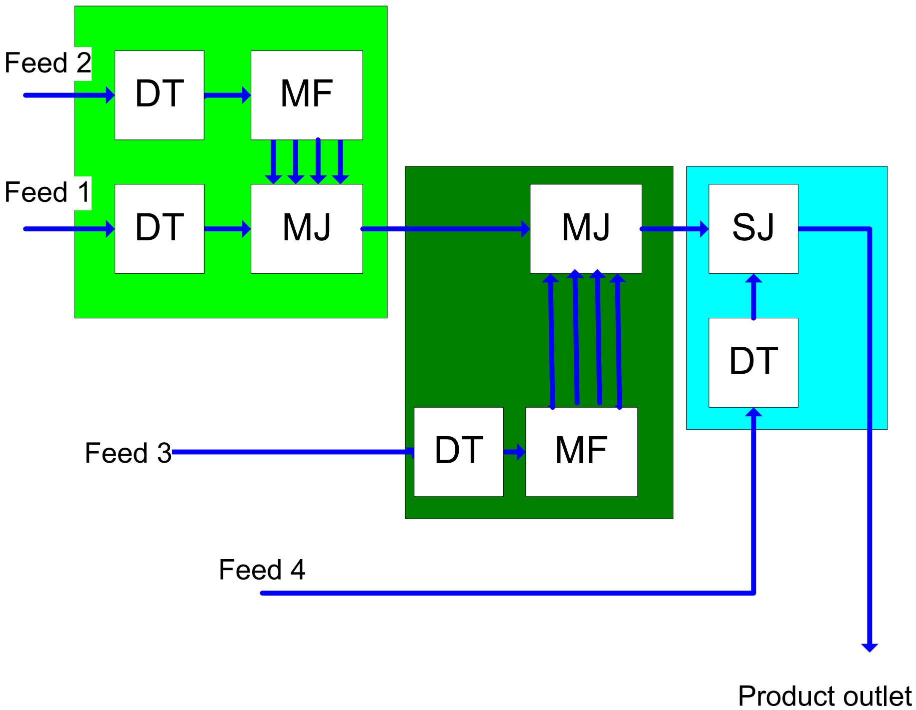


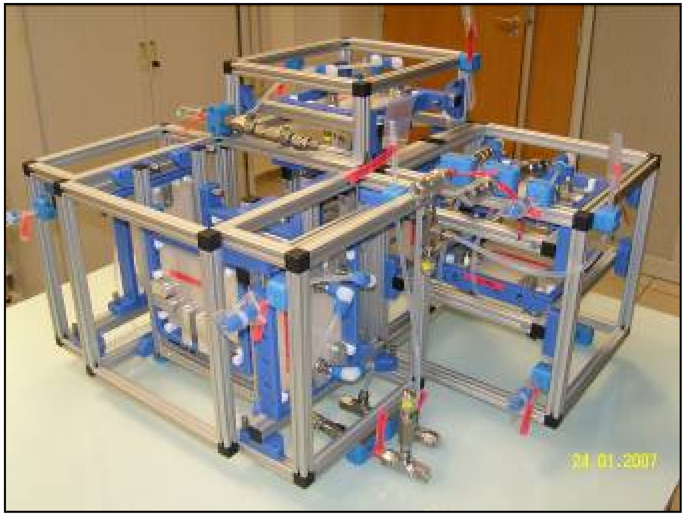


Nitration


Feed 1
DT
MF
Feed 2
DT
MJ
DT
MF
Feed 3
DT
MJ
DT
Feed 4
DT
SJ
PRODUCT OUTPUT

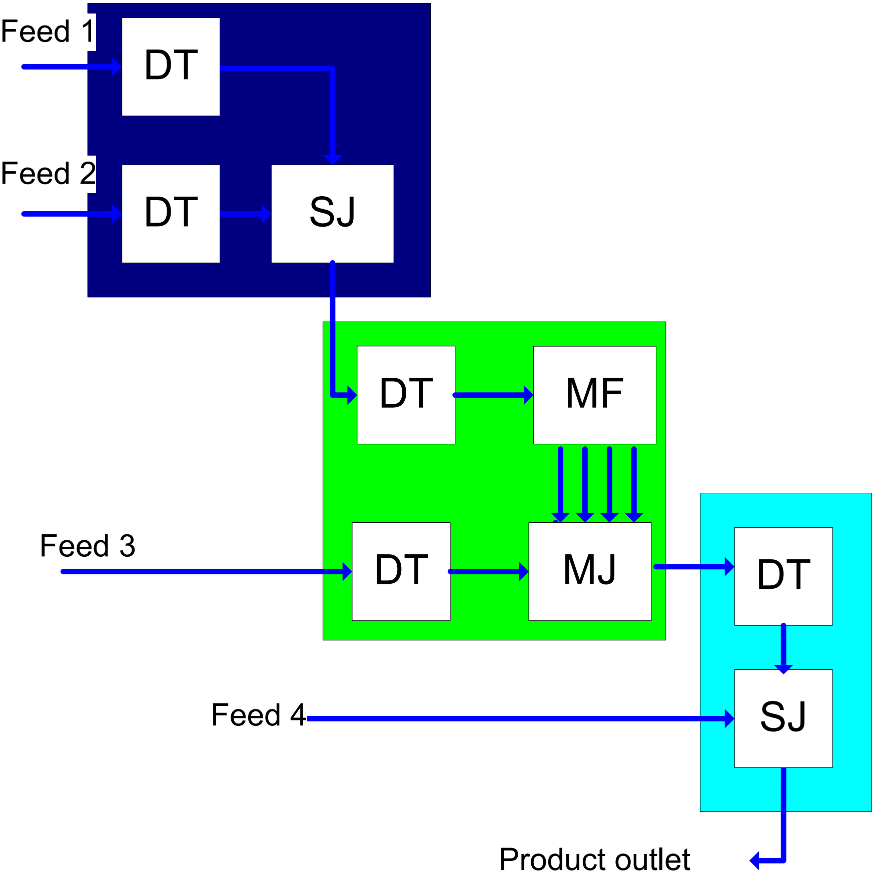
Organometalic reaction



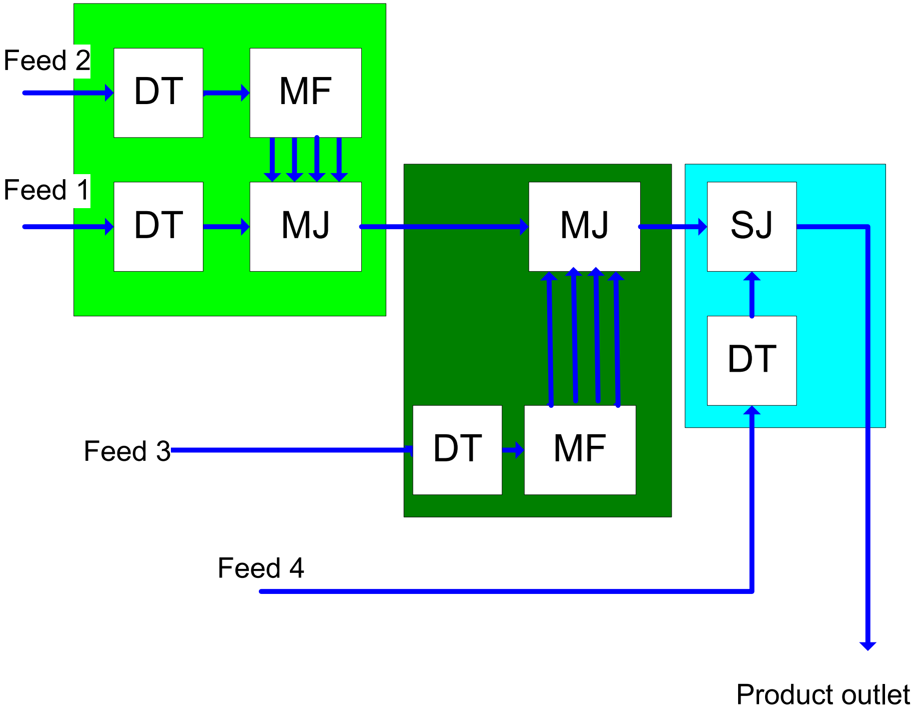


Multipurpose production

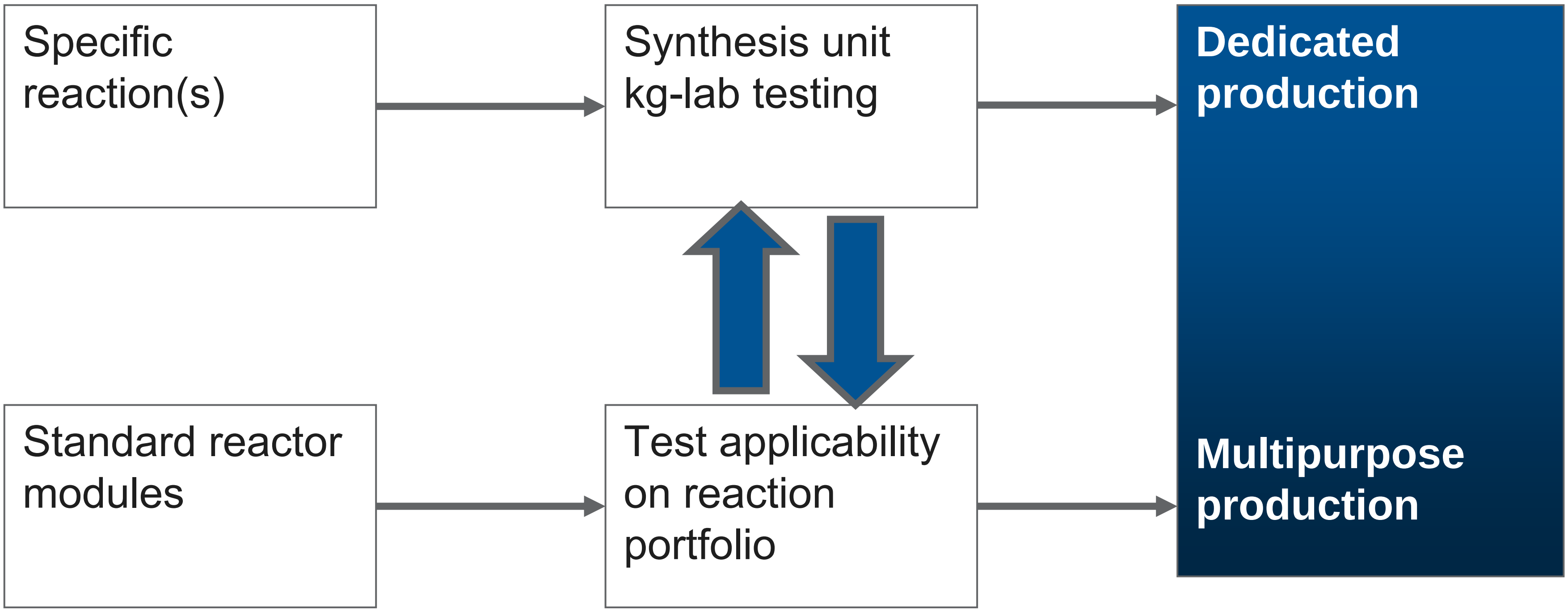
PRODUCTION 1
CAMPAIGN 1



PRODUCTION 1
CAMPAIGN 2

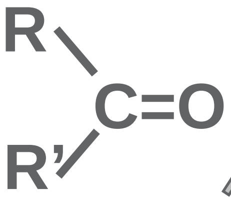


Multipurpose or dedicated production

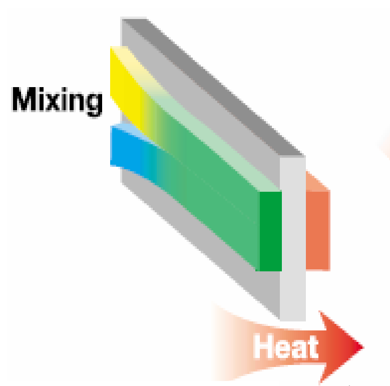


- Flexibility for process development
- Flexibility for Multipurpose production
- Flexibility for adapting production capacity to needs

Integration into production system

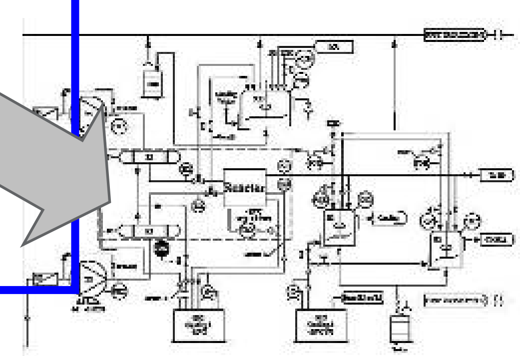


Chemistry



Mass and heat transfer

System engineering



Reactor engineering

Integrated approach in process development, reactor design & system engineering

- Changing to continuous requires a different approach from lab, through piloting to processing at the commercial scale
  - Corning & Zeton bring this approach in practice
  - Support of customer in their testing of chemistry in lab, pilot and production phase
  - Dedicated or multi purpose reactors by Corning
  - Overall engineering, automation, process integration and construction of facilities by Zeton

ZETON PILOT PLANT TECHNOLOGY

CORNING

- Zeton capabilities
  - Engineering & Construction of highly automated built plants
  - Support in development of projects through basic design studies
  - Custom design – fit for purpose
  - 20 years experience in continuous processing
  - Design low flow & severe conditions
  - Delivery conform ATEX & PED
  - Fast track project approach
  - Smart solution on small scale

- Corning capabilities
  - Microprocessing technology using a process intensified approach
  - Reactor design conform process & chemistry requirements
  - Assistance in the development from batch to continuous processing
  - Support in testing of chemistry at customer's facility
  - Strength in R&D and innovation
  - 150 years experience in materials and process

Industrial mobile multi purpose microreactor unit

- Independent operation
- Highly automated
- Adaptable to most chemistries

CORNING

ZETON PILOT PLANT TECHNOLOGY

- 3 liquid feeds
- 2 gas feeds
- ATEX EEx Zone II
- T= -80 / +250°C
- Output up to 50 t/y
- Data collection

Thank you for your attention