



Nouryon is exploring reactive distillation

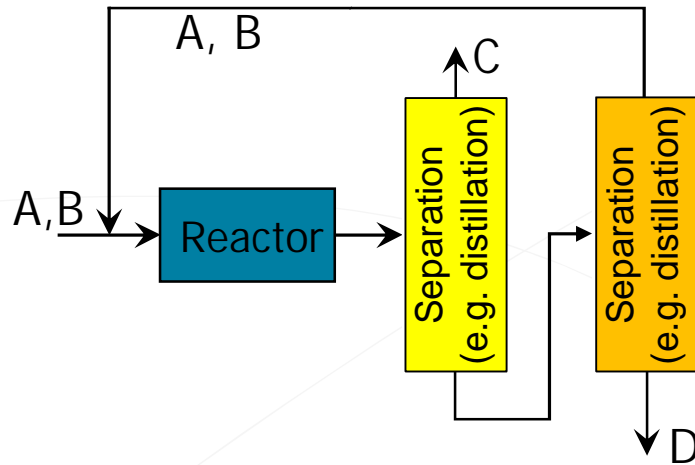
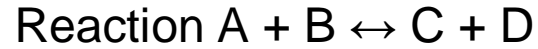
Ulf Schröder, Stijn Oudenhoven, Gerrald Bargeman, Antoon ten Kate
Nouryon – ECCD

EFCE WP Fluid Separations
Göteborg SE, 19 June 2019

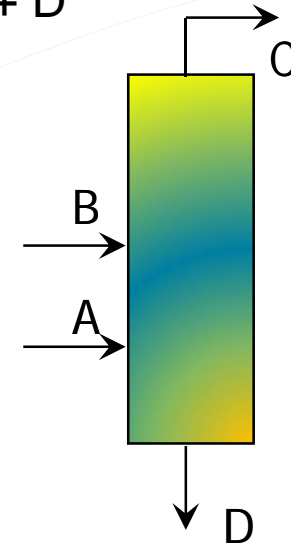
Nouryon

Reactive Distillation technology in short

Simultaneous removal of vapor products from a boiling, reacting mixture



Traditional process

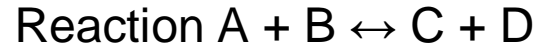


Reactive Distillation

Reactive Distillation

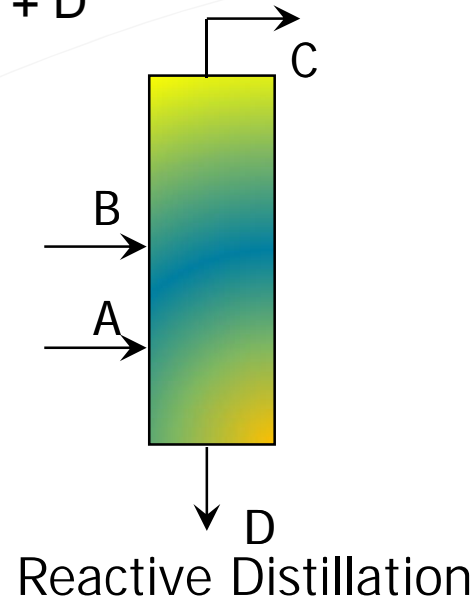
technology in short + benefits

Simultaneous removal of vapor products from a boiling, reacting mixture

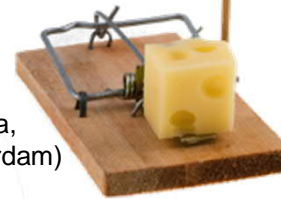
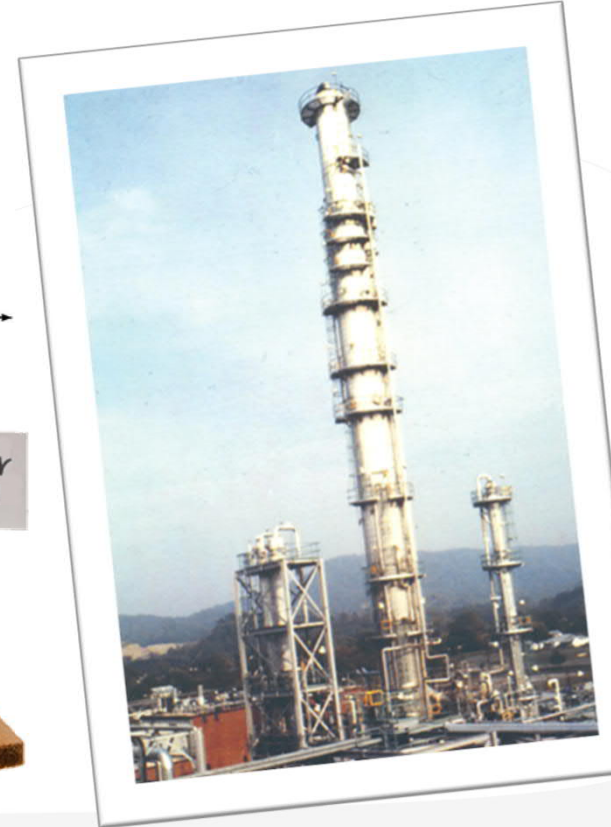
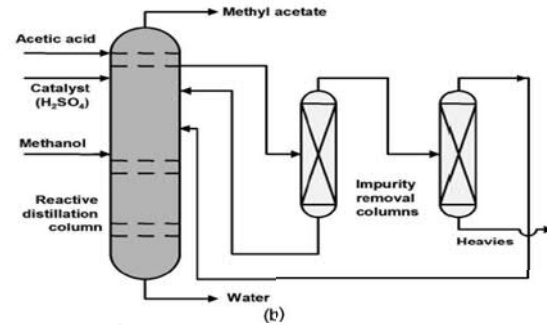
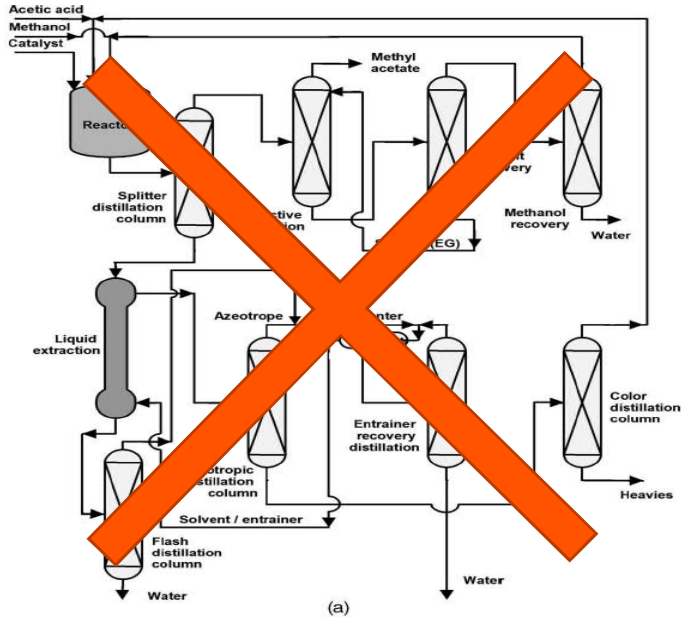


Technological benefits:

- **Beat low equilibrium** constants
- Enhance overall rates
- **Improve selectivity** & reduce byproducts
- Avoid/eliminate difficult separations
- React away impurities
- Reduce/eliminate solvents
- **Energy reduction** potential



Reactive Distillation methylacetate production



(Quote: prof. Krishna,
University of Amsterdam)

Sources:
Bob Huss, *Reactive Distillation: What, why, and how?*
East Tennessee AIChE Seminar Program (17 November 2015)
James Bielenberg, Michelle Bryner, *Realize the Potential of Process Intensification*,
CEP (March, 2018)

Structured approach for reactive distillation



route scouting

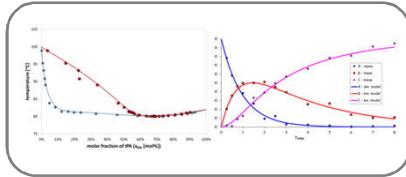
process development

engineering

CCS

construction, commissioning, start-up

data



model & parameters

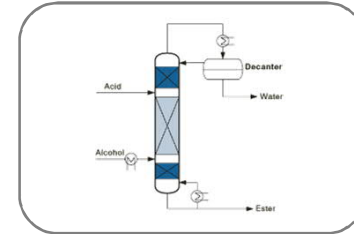
$$y_i \cdot p = \gamma_i \cdot x_i \cdot P_i^{sat}$$

Antoine - pars
BIP

$$\frac{dc_i}{dt} = k_j \prod a_i$$

$k_{j,0}$
 $E_{A,j}$

conceptual design



Validation

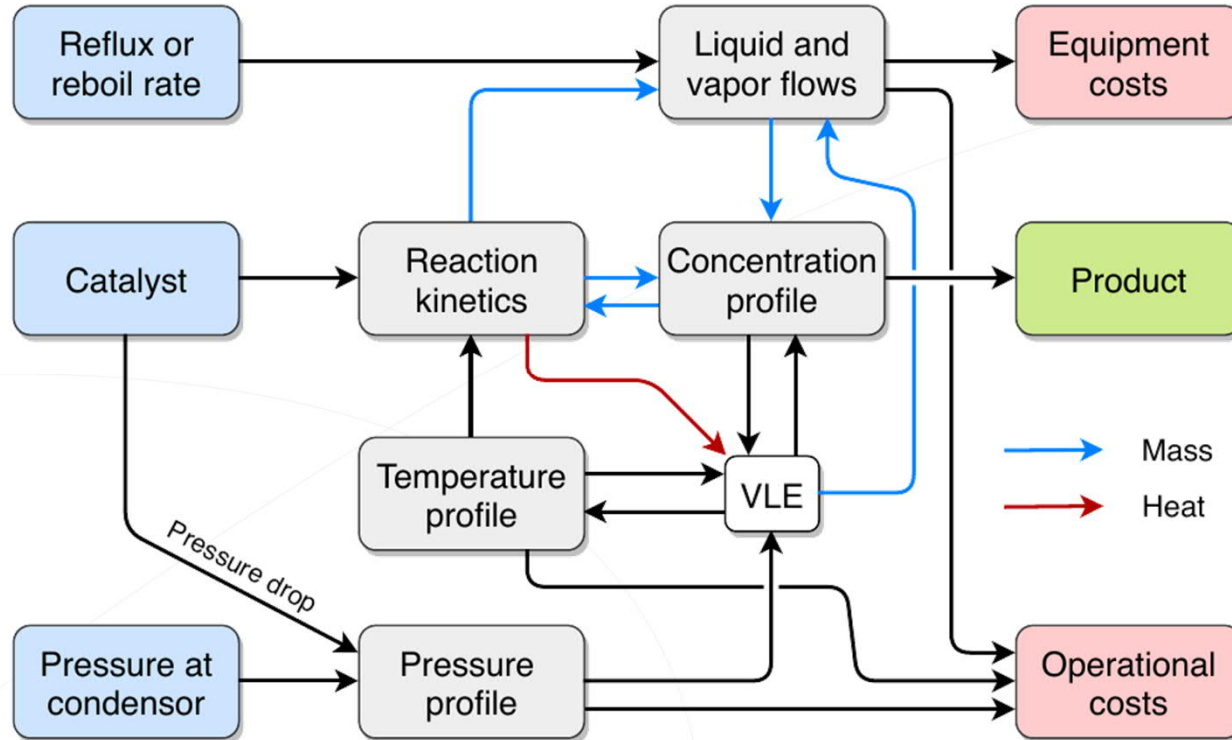


experiment

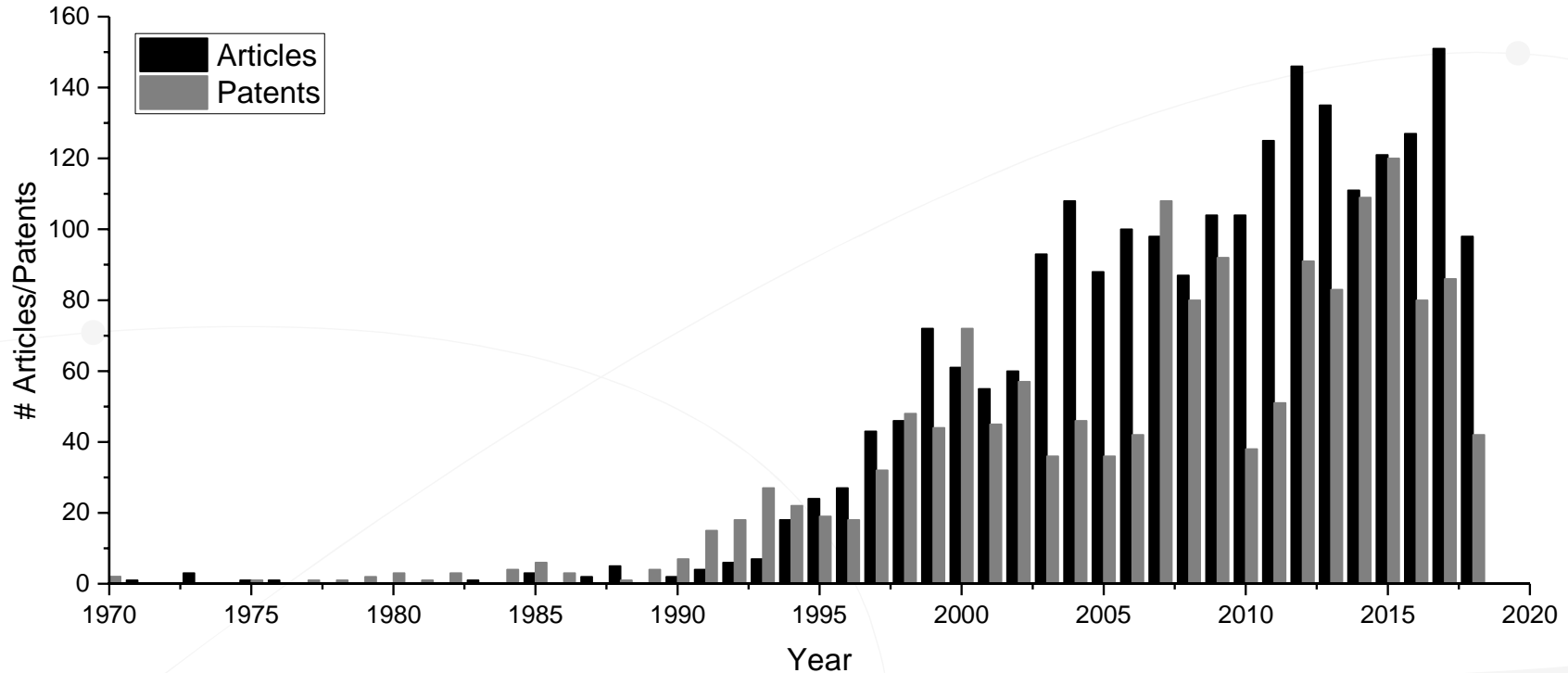


Complexity in reactive distillation

An artist impression



Literature & patent review Bibliography (1970 - Mid 2018)

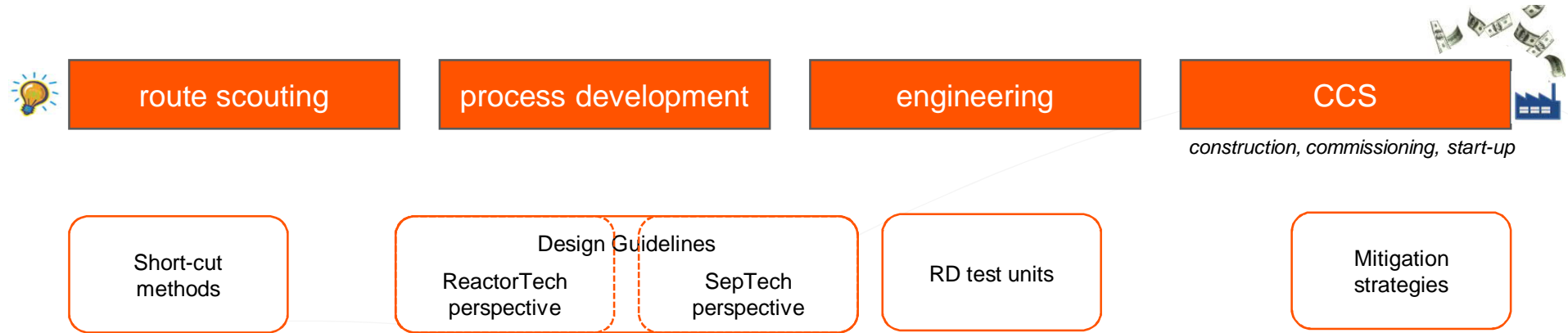


Literature & patent review

Observations

- RD methods typically developed from SepTech perspective:
 - Strong need for **ReactorTech** perspective.
- Process design software appears (far) less developed for RD than it is for distillation, which quite easily can lead to annoying & significant errors (a number of examples in lit.)
 - Need for more **advanced tools** for RD in **simulation** software
- Most (engineering) literature is based on simulations only (>95%)
 - Need for **experimental validation**
- Focus (mainly) on RD column as such (or aspects thereof); less on integral process
 - Need focus on **(integral) process** or at least **RD section** (RD unit \pm 1 unit operation)
- No clear design procedure
 - Need for clear **design procedure & guideline**, especially for relatively slow reactions.

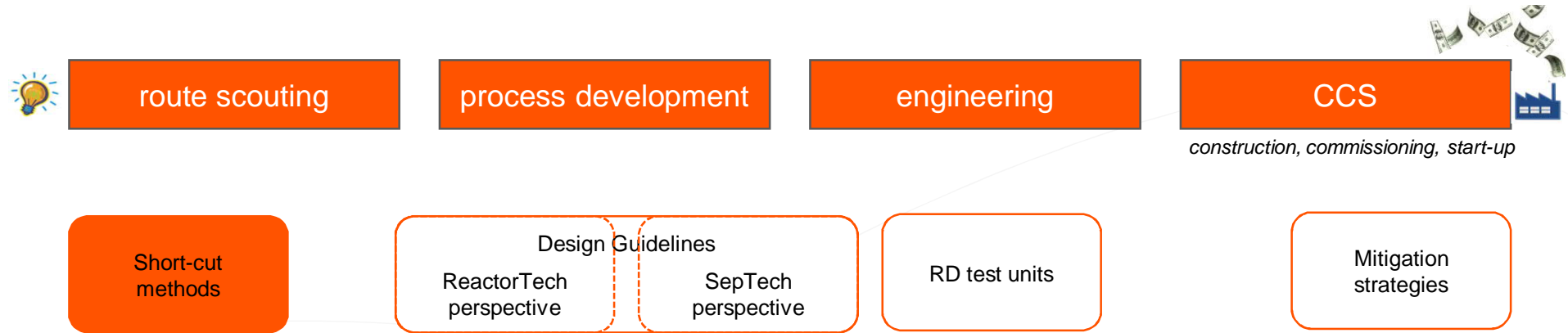
Collaboration on Reactive Distillation purpose



Cooperation with

- Universities (UCL, prof E. Sorensen; Univ Twente, prof S. Kersten)
- Software vendors (AspenTech)
- Hardware suppliers (Montz)

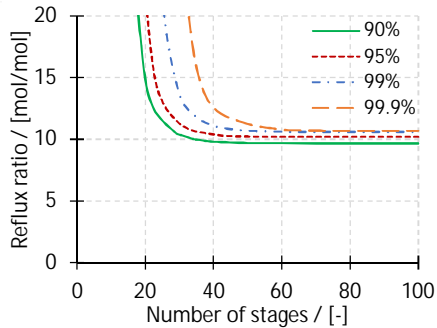
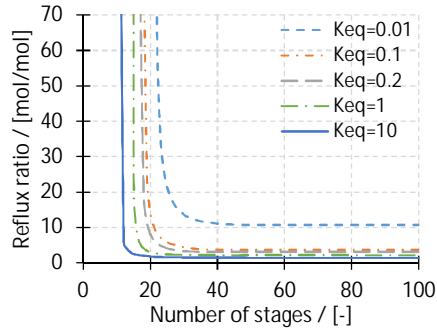
Collaboration on Reactive Distillation purpose



Cooperation with

- Universities (UCL, prof E. Sorensen; Univ Twente, prof S. Kersten)
- Software vendors (AspenTech)
- Hardware suppliers (Montz)

Mapping of applicability window

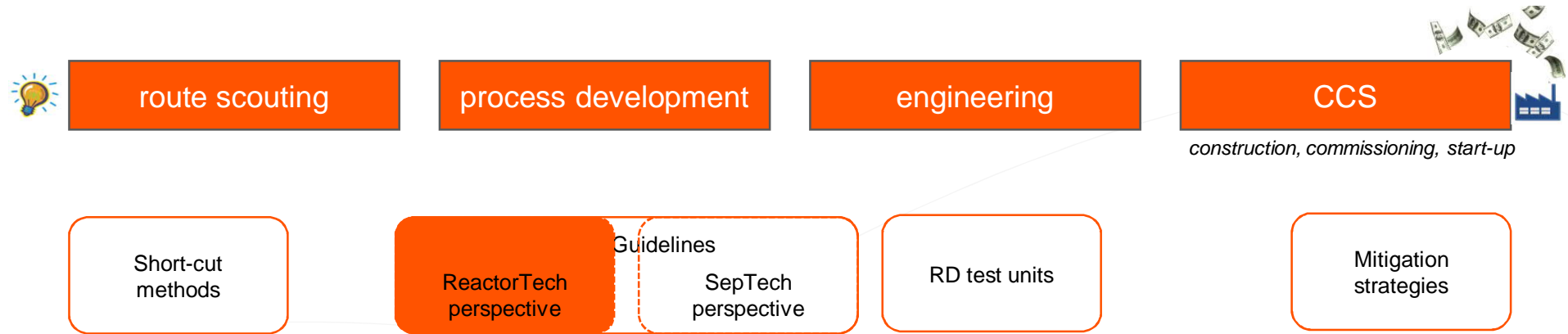


Study*) initiated on applicability window of reactive distillation. Results showed:

- there is a **window of feasible RD configurations** that provide in-spec product streams
- the feasibility window is characterized by minimum reflux ratio (RR_{min}) as well as minimum number of stages (N_{min}), (similar to that of distillation)

*) **Novel method for mapping the applicability of reactive distillation**
 Rahma Muthia et al
 Chemical Engineering and Processing - Process Intensification
 Volume 128, June 2018, Pages 263-275

Collaboration on Reactive Distillation purpose

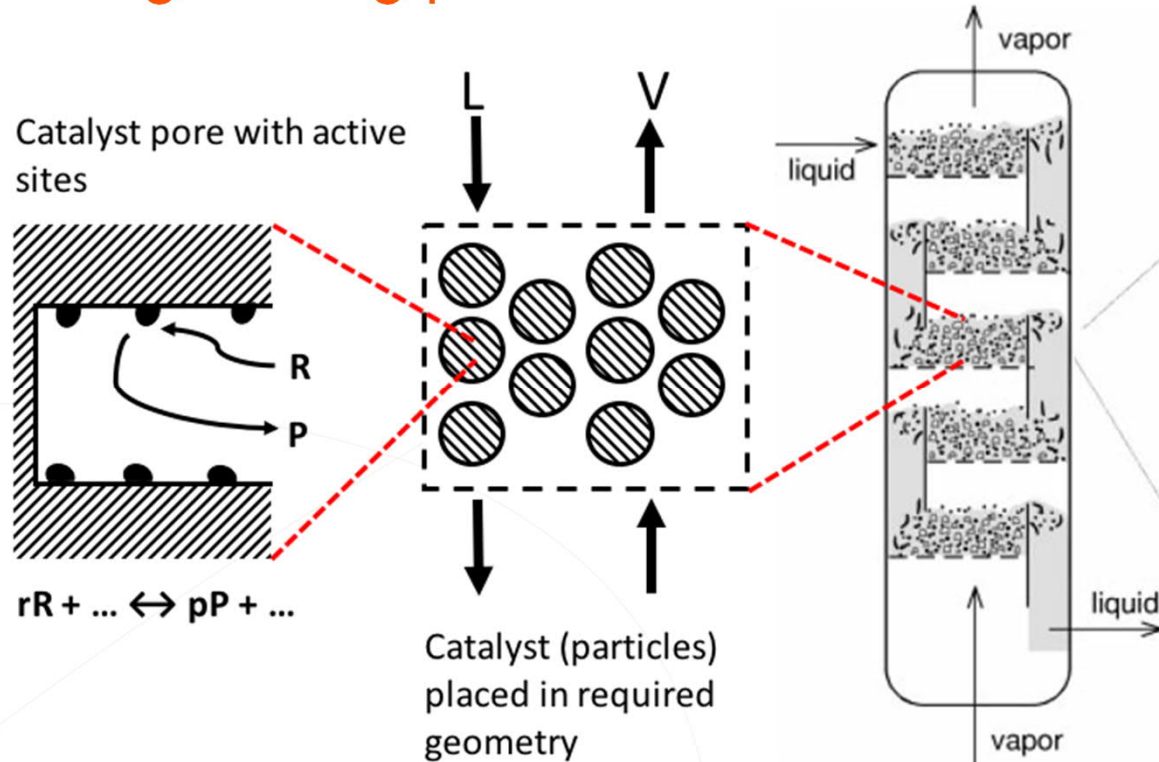


Cooperation with

- Universities (UCL, prof E. Sorensen; Univ Twente, prof S. Kersten)
- Software vendors (AspenTech)
- Hardware suppliers (Montz)

Some ReactorTech Perspectives

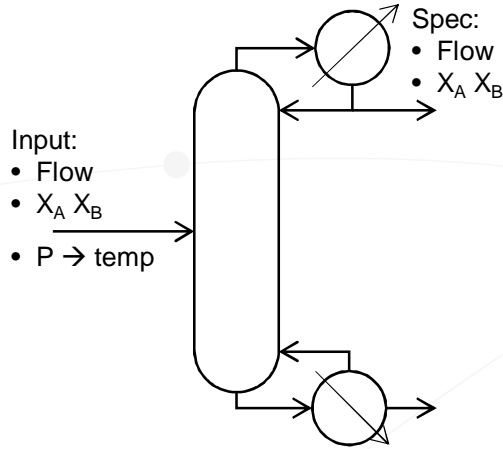
The reactor engineering point of view



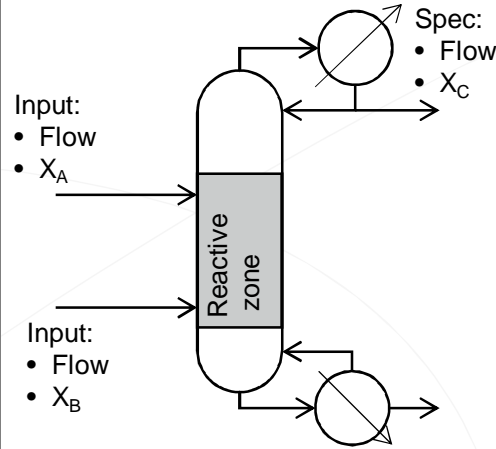
Some Integral Process Perspectives

Reactive distillation is not limited to stoichiometric feed and distillation

Conventional distillation

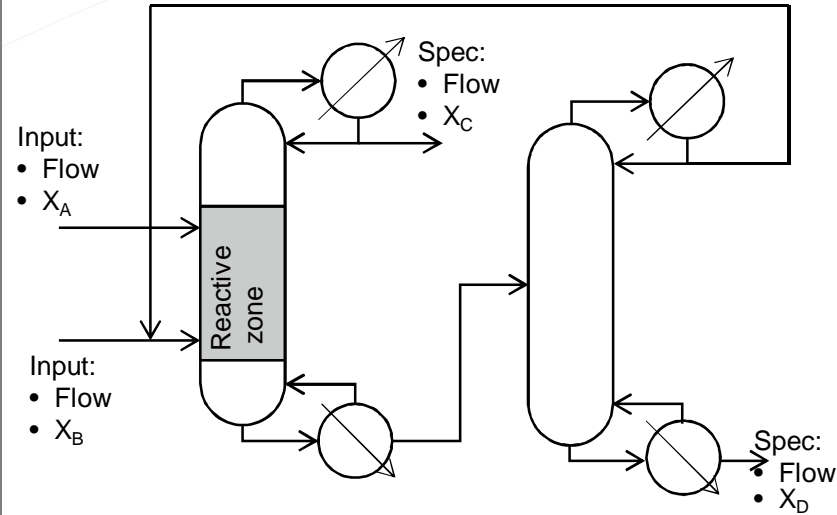


Reactive distillation

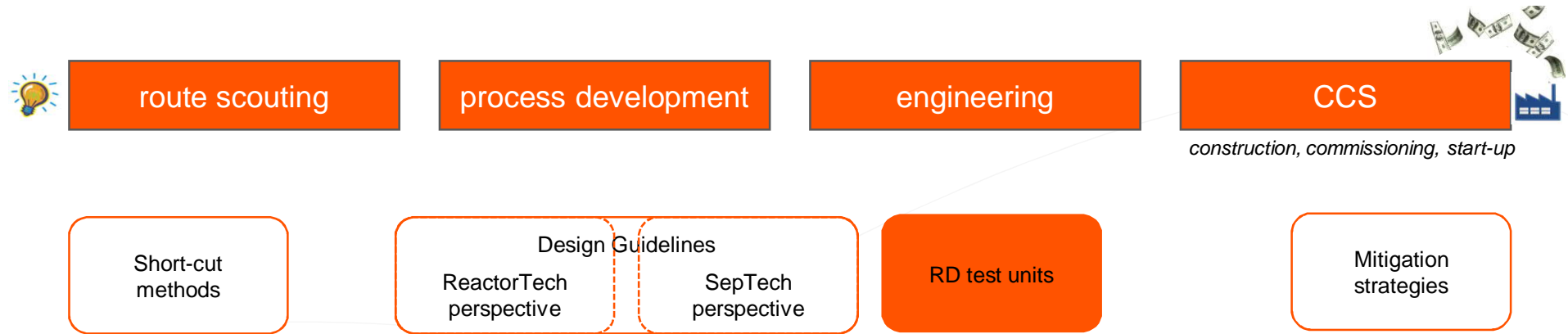


Excess reactant operation

- increased reaction rate
- Suppression of undesired side reactions
- enhanced operability & controllability



Collaboration on Reactive Distillation purpose



Cooperation with

- Universities (UCL, prof E. Sorensen; Univ Twente, prof S. Kersten)
- Software vendors (AspenTech)
- Hardware suppliers (Montz)

RD test units impression

Purpose:

- validate RD design

Applications:

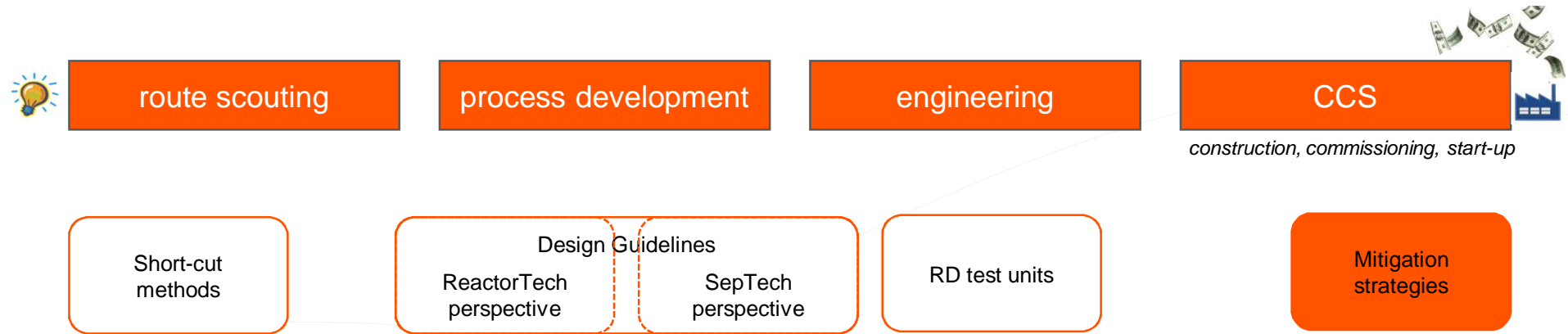
- Various chemistries (alkaline, acid)
- Slow, fast & instantaneous reactions

Features:

- Minimum residence time in reboiler
- Dedicated column wall temperature control to compensate for heat losses
- Flexible feed points
- Multiple sampling and temperature points
- Vacuum, atmospheric to 6-10 bara
- MoC: glass, glass-lined and SS316
- Atex



Collaboration on Reactive Distillation purpose



Cooperation with

- Universities (UCL, prof E. Sorensen; Univ Twente, prof S. Kersten)
- Software vendors (AspenTech)
- Hardware suppliers (Montz)

Mitigation and process control

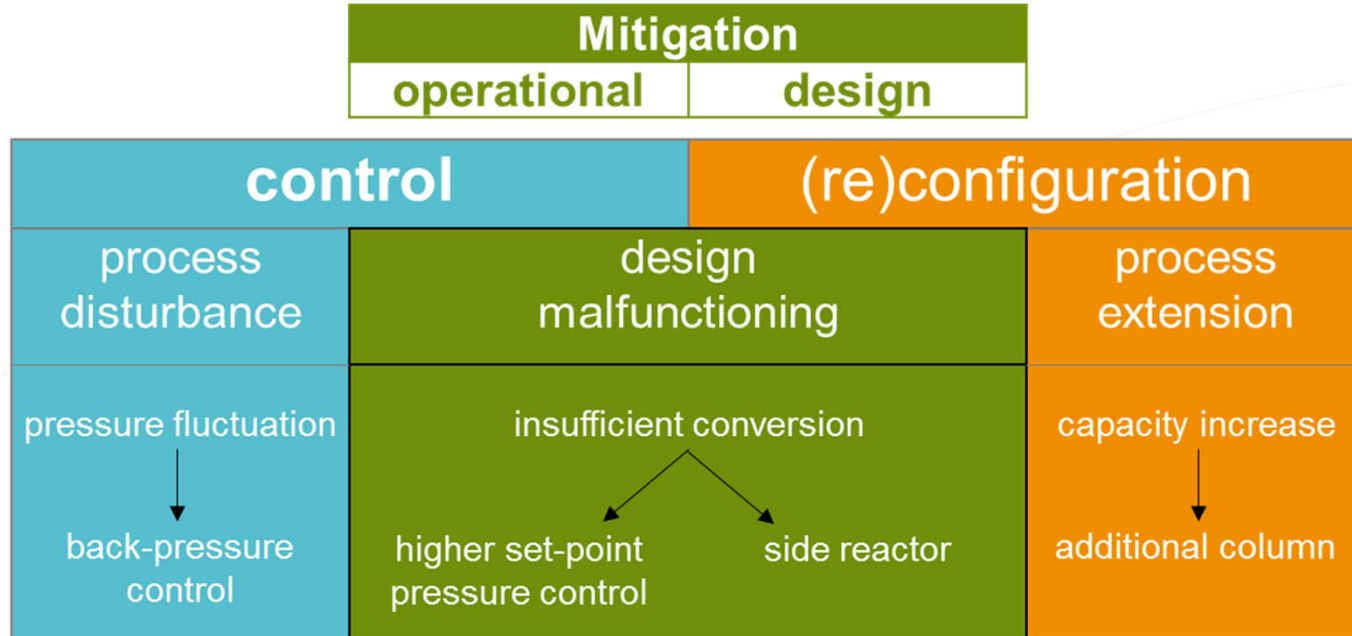
Considerations

- *Experiments have errors in process parameters and analysis.*
- *Models are simplified representation of reality.*
- *Strict design of RD column (Overdesign can lead to higher OPEX)*
- *Pilot testing expensive and time consuming*

Need for guidelines to mitigate uncertainties, thereby increasing plant acceptance and speeding up design and implementation process.

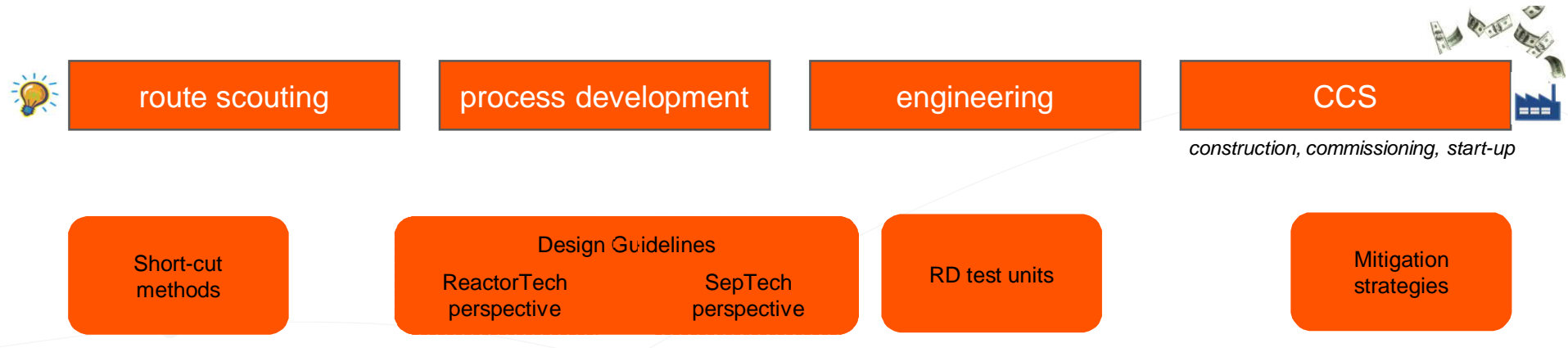
Mitigation and process control

Interconnection between both topics



Design mitigation required when solely control cannot mitigate process deviation.

Collaboration on Reactive Distillation purpose



Cooperation with

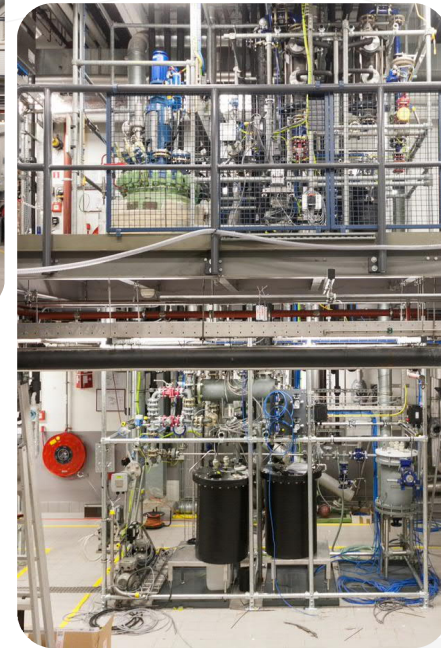
- Universities (UCL, prof E. Sorensen; Univ Twente, prof S. Kersten)
- Software vendors (AspenTech)
- Hardware suppliers (Montz)

ECCD Process Technology






reactive distillation

- Delivering world-class support in development of reactive distillation process:
 - Structured process development approach
 - for fast and slow reactions
 - from ideation until pilot scale
 - kinetics, VLE, conceptual design, validation
 - experimental + modeling, incl physprops
 - RD columns for validation
 - for acid, neutral and alkaline systems (glass-lined; SS316)
 - capacity: ~ 0.5 - 5 kg/h throughput
 - Close connection with key suppliers
 - RD column suppliers
 - Simulation software

Visit us in Deventer or our website



Expert Capability Center Deventer

- Who?  Over 80 skilled, highly educated and experienced scientists
- What?  Process & product technology, analytical science
- Where?  Deventer, NL
- With?  State of the art laboratories and equipment, including ATEX
- What else? 
- Broad knowledge of chemical products and processes
 - Fast response
 - Collaboration with universities, knowledge institutes and Nouryon RD&I groups
 - REACH notification, GLP accreditation

Expert Capability Center Deventer

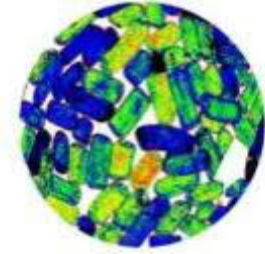
Solving your challenges using our expertise

Your challenges:

- Developing new and improved products
- Developing and implementing new and optimized technologies and processes

Our supporting role:

- Multi-disciplinary approach in industrial R&D perspective
- Using in/online analytical techniques to control processes
- Developing models for improved process control
- Performing dedicated trials at lab, pilot or plant scale
- Analyzing in complex matrices
- Failure analysis



Acknowledgement

University Twente, NL:

Prof S. Kersten;

V. Kroeze, PhD student; L. Noll, PhD student;

F. Kooijman, MSc student; K. Zijp, MSc student

University College London, UK:

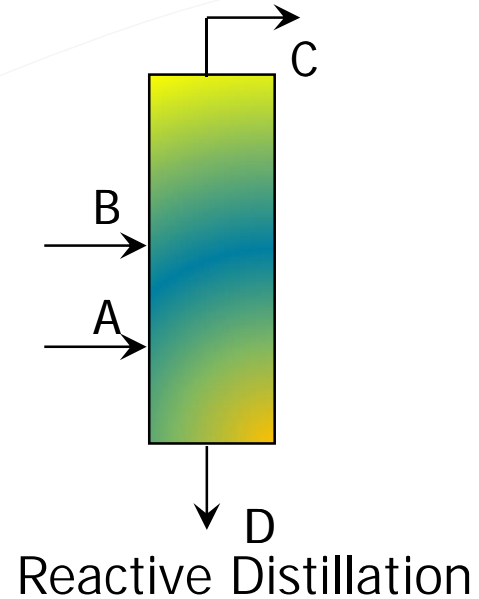
Prof E. Sorensen; A. Tsatse, PhD student

University Manchester, UK:

Prof T. Kiss, R. Muthia, PhD student

AspenTech, USA: Dr D. Tremblay

Montz, DE: Dr R. Schulz; Dr M. Krapoth



ECCD Process Technology

contact information

ECCD PT Lead scientist Antoon ten Kate
antoon.tenkate@nouryon.com
+31 (0)6 2933 6596

ECCD PT Leader Gerrald Bargeman
gerrald.bargeman@nouryon.com
+31 (0)6 2306 4091

Visiting address Zutphenseweg 10
7418 AJ Deventer
The Netherlands

Website <https://www.nouryon.com/company/innovation/eccd>

