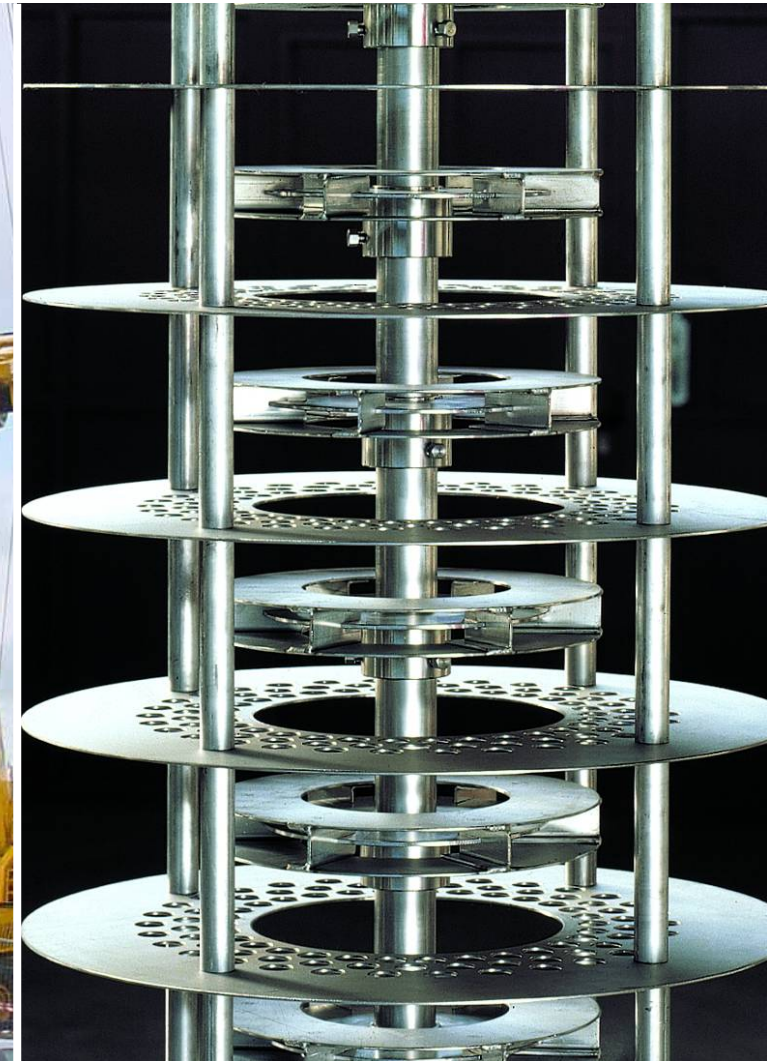


**PIN NL Meeting | November 10<sup>th</sup> 2010 | Utrecht**

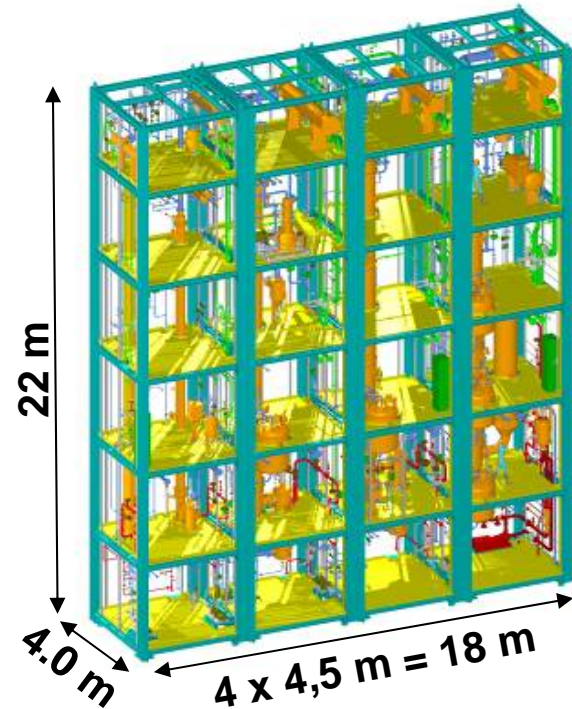
## **Title: Process Intensification In Real Life**

Jeffrey FELIX, | Sulzer Chemtech Ltd. | Process Technology



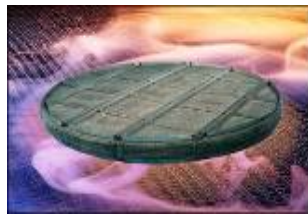
# Presentation overview

- Introduction of Sulzer Chemtech Process Technology
- Process Intensification by combining unit operations
- Concrete PI examples with pervaporation, distillation & crystallization technology





Structured packing  
Tray Technology



Separators  
Demisters

Maintenance, revamps  
& installation

### Tower Field Services



Shell Alliance



Random packing



Sulzer Chemtech

### Mass Transfer Technology

### Process Technology



Membrane  
Technology



Crystallization  
Technology



Multi-component dosing, mixing  
and application systems

### Mixpac Systems



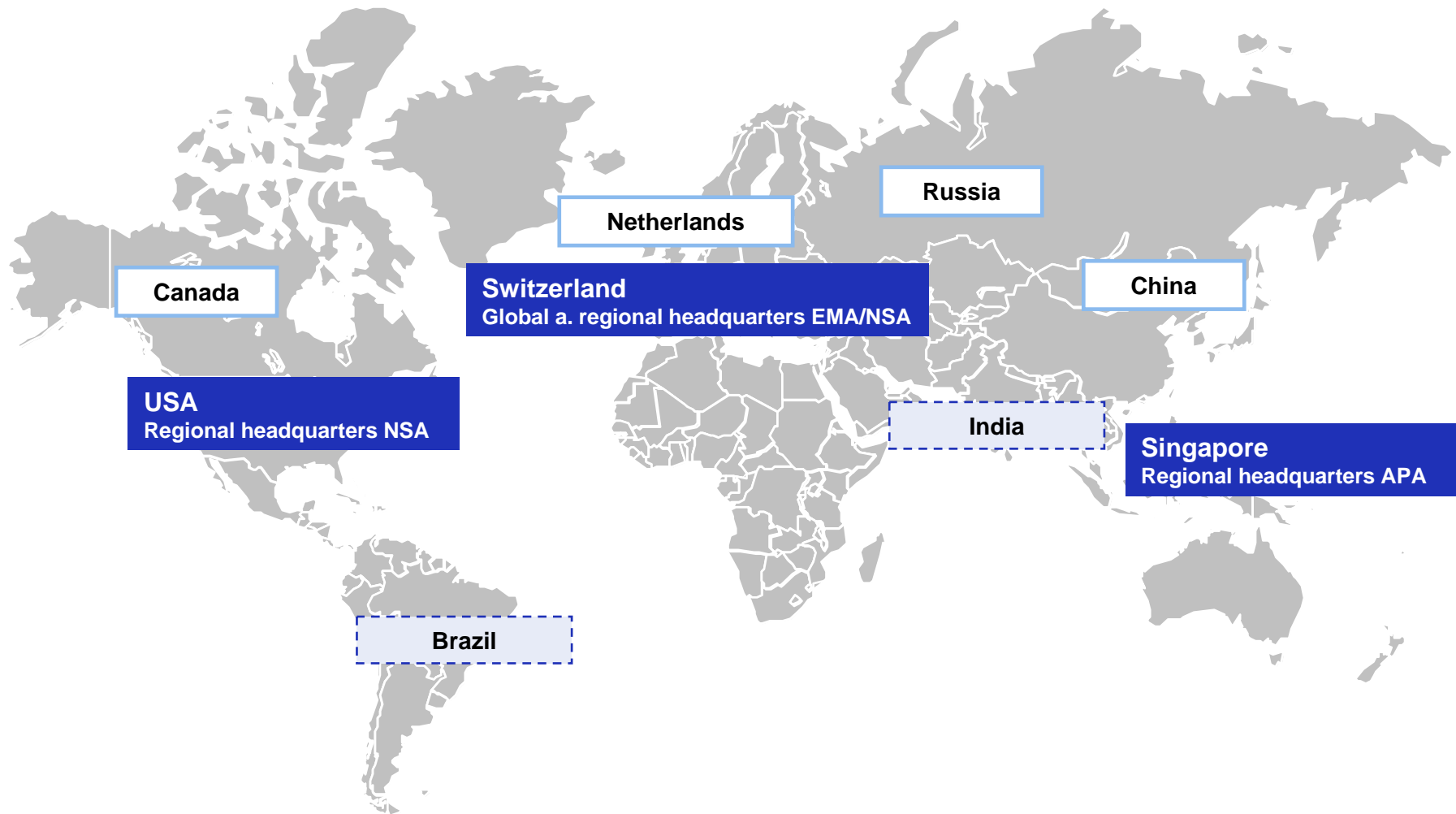
Hybrid Process  
Plants



Film Evaporation  
L/L-Extraction  
Reactive Distillation

# Process Technology

## Global organization



 PT locations

 PT presence

 PT potential expansions

### Technologies

Extraction, distillation, crystallization, membrane, evaporation, absorption, polymer & reaction technology

### Segments

Fine & specialty chemical, pharmaceutical process industry, food industry, polymer production

### Products and services

- Engineering services (conceptual, basic & detail)
- Customer testing, process validation, production of samples, toll production
- Process equipment, modular plants, skids

### Process applications

- Solvent recovery
- Product purification
- Hybrid processes and solutions
- Temperature sensitive separations
- Polystyrene, PLA, EPS

### Key success factors

- Solving complex and difficult separation problems
- Supplying advanced, highly efficient process equipment and complete plants
- Extensive experience in combining unit operations

### Unique experience in unit operations

- Evaporation
  - Falling film (FFE)
  - Wiped thin film (TFE)
  - Short path (SPE)
- Distillation / rectification
  - Structured packing
  - Random packing
  - Trays
- Liquid / liquid extraction
  - Static columns
  - Stirred columns
- Fractional crystallization
  - Falling film
  - Static system
  - Suspension crystallization
- Membrane systems
  - Pervaporation / vapor permeation
  - Pressure driven membrane solutions



### ■ Process Engineering Studies

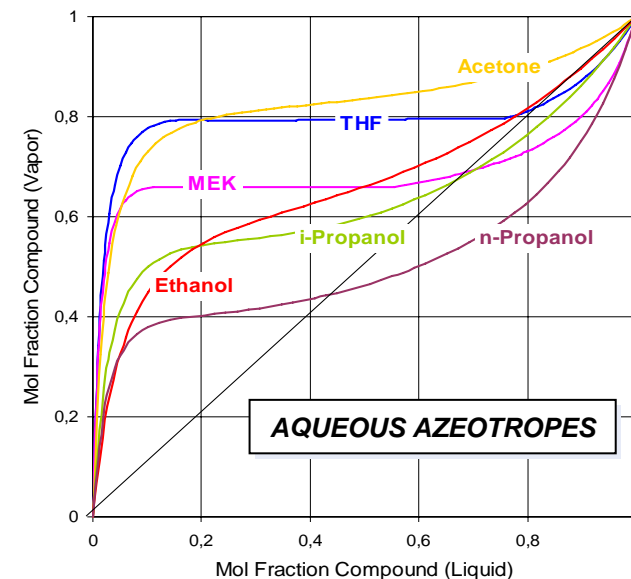
- Conceptual design
- Process optimizations / debottlenecking
- Process synthesis, development and simulation

### ■ Test Centre

- Standard laboratory and pilot equipment for development, pilot and toll processing
- Testing and validation of distillation, absorption, crystallization, liquid-liquid extraction, film evaporation and membrane processes as well as reaction, devolatilization and mixing tests for polymeric systems

### ■ Process- & Basic Engineering

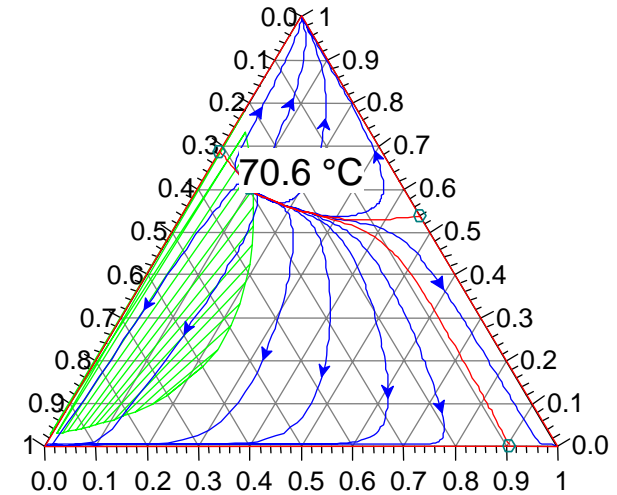
- Process design, simulation, PFD
- P&ID, Basic Engineering



# Process Technology

## Hybrid Approach

- Screening of possible unit operations
  - Experience / reference units / plants
  - In house know how
  - Literature / databases
  - Process simulation
  - Bench scale feasibility tests for promising unit operation
- Combination of unit operations to most cost effective hybrid process
- Validation
  - Process simulation
  - Pilot scale tests / sample generation
- Basic Engineering
- Detail Engineering
- Supply of (key)equipment or skid mounted unit
- Commissioning of turn key hybrid system





### Distillation

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- Robust process with high mass transfer rates in liquid and vapor phase
- Phase separation is rapid and complete

### Crystallisation

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- Separation at low (melting) temperatures
- Separation of mixtures with close boiling components
- Very high specific purification is possible

### Liquid-Liquid-Extraction

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- Low temperature selective separation @ at mostly ambient pressure
- High throughput @ low energy consumption

### Vapour permeation / Pervaporation

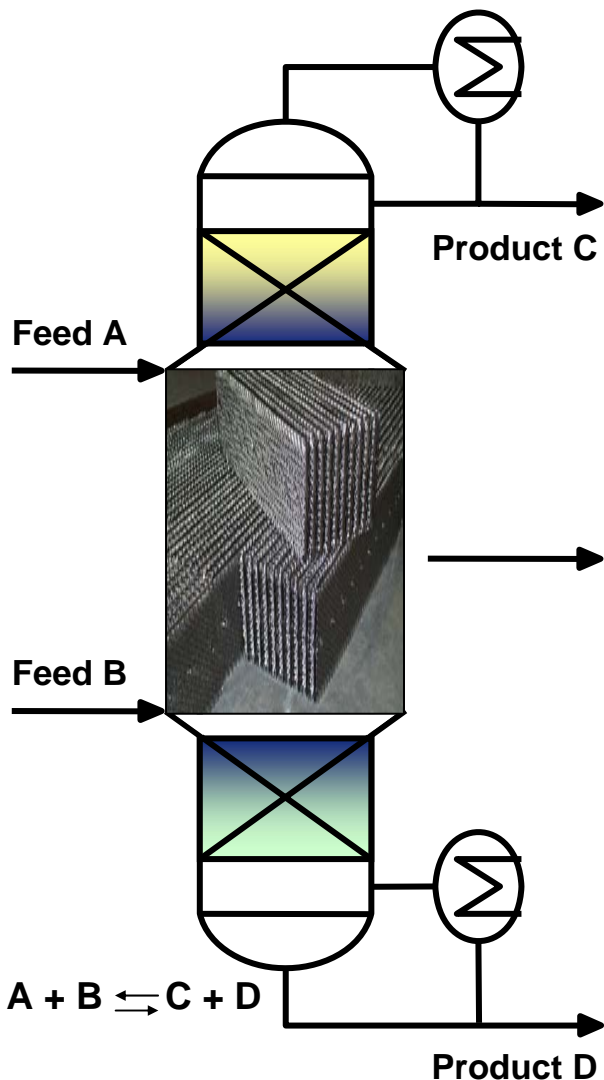
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- Independent of vapor–liquid equilibrium
- Direct solvent dehydration of azeotropics and close-boilers without entrainer

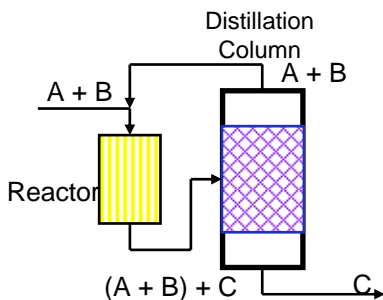
### Pressure driven membrane (UF/NF/RO)

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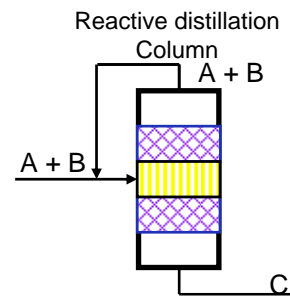
- No phase transition leads to low energy demand
- Separation based on molecule size



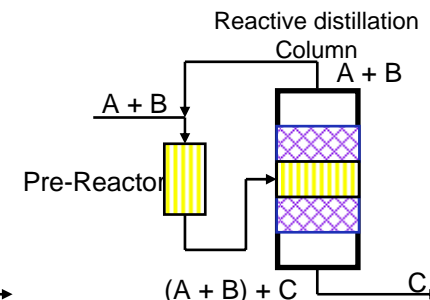
### Combine Reaction and Separation in ONE Column



Conventional Flowsheet for Reactor and Distillation



Reactive Distillation Flowsheet

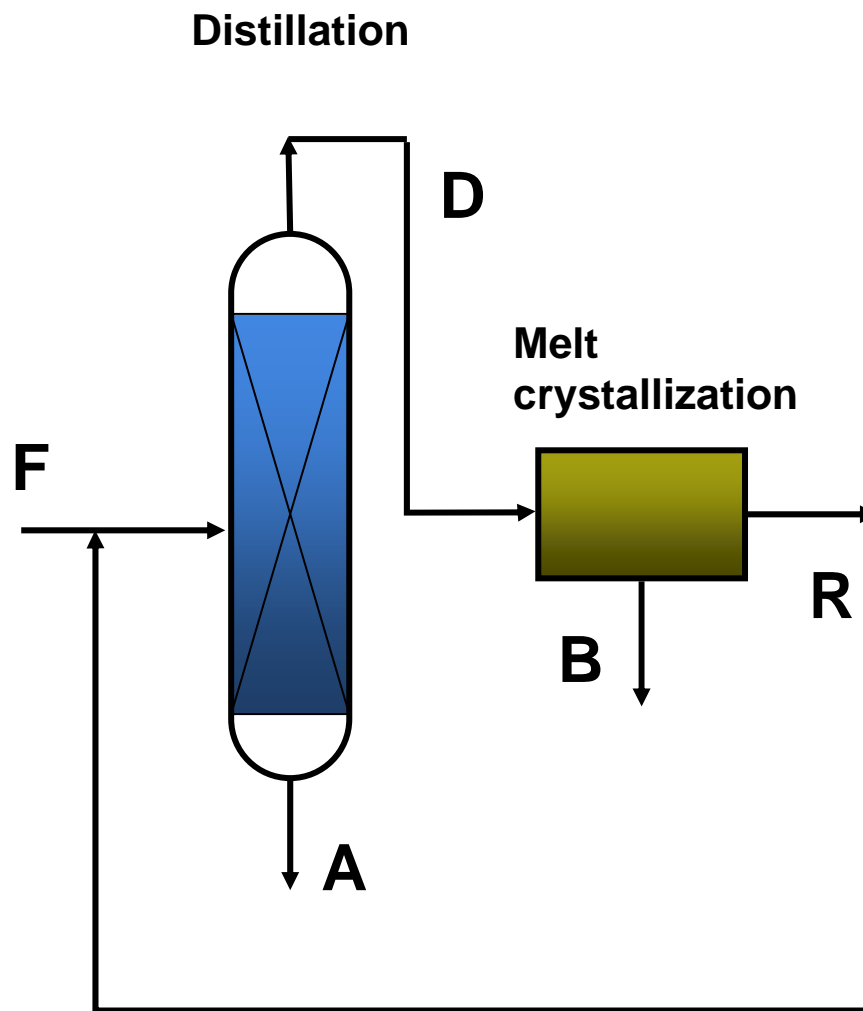
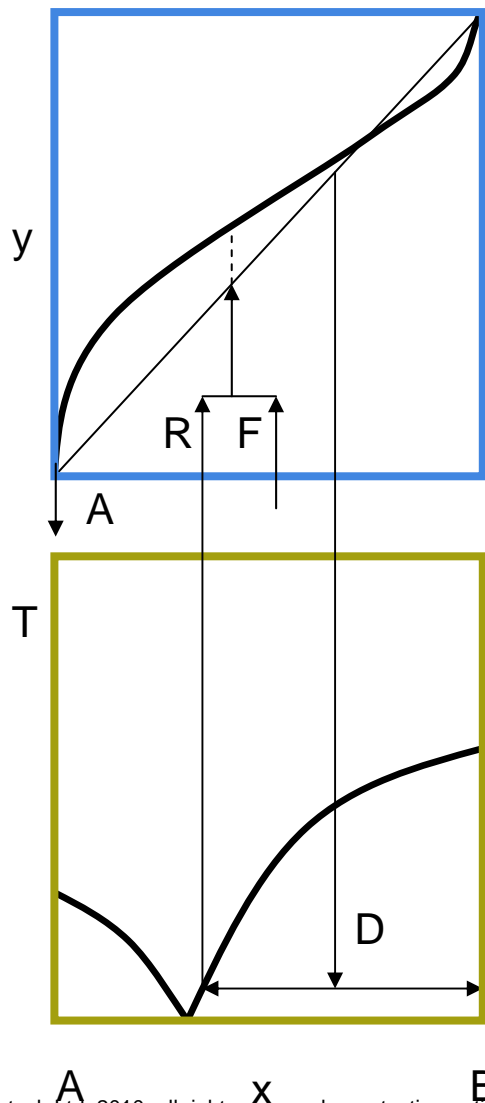


Reactive Distillation with Pre-Reactor

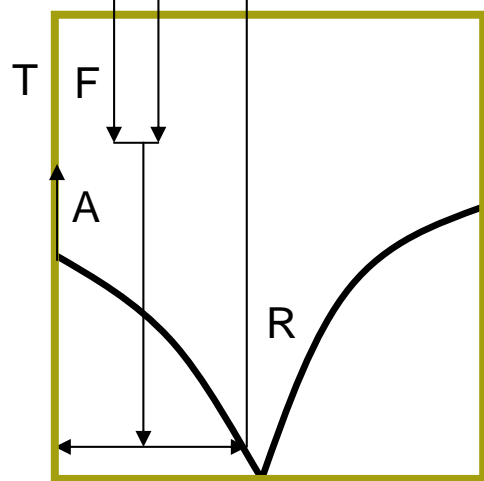
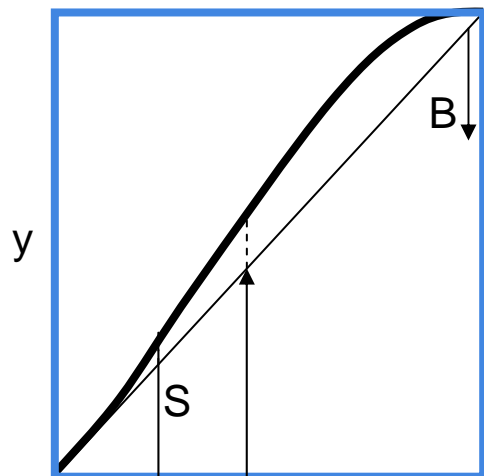
### Reactive Distillation Processes

- **Acetate Technology**
  - Synthesis of Methyl, Ethyl & Butyl Acetate
  - Hydrolysis of Methyl Acetate
- **Acetalisation**
  - Synthesis of Methylal
  - Removal methanol from Formaldehyde
- **Fatty Acid Esters**

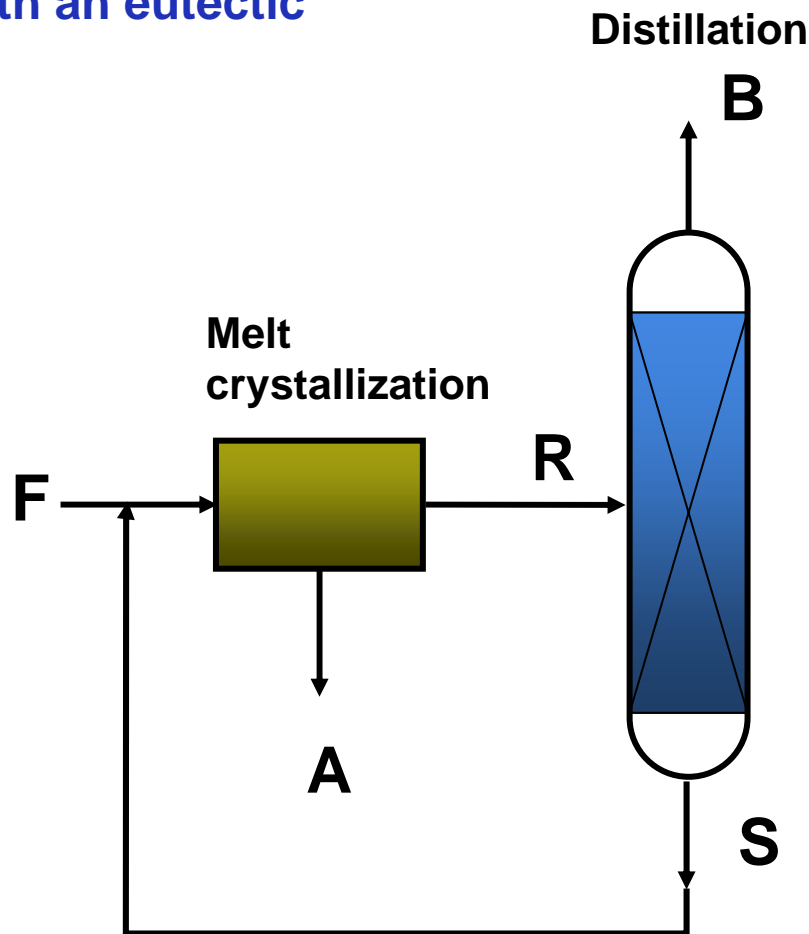
### Separation of a binary mixture with eutectic and azeotrope



### Separation of a binary mixture with an eutectic



A x B



### KEY APPLICATIONS

- (ultra) high purities
- color / odor removal
- isomer separation

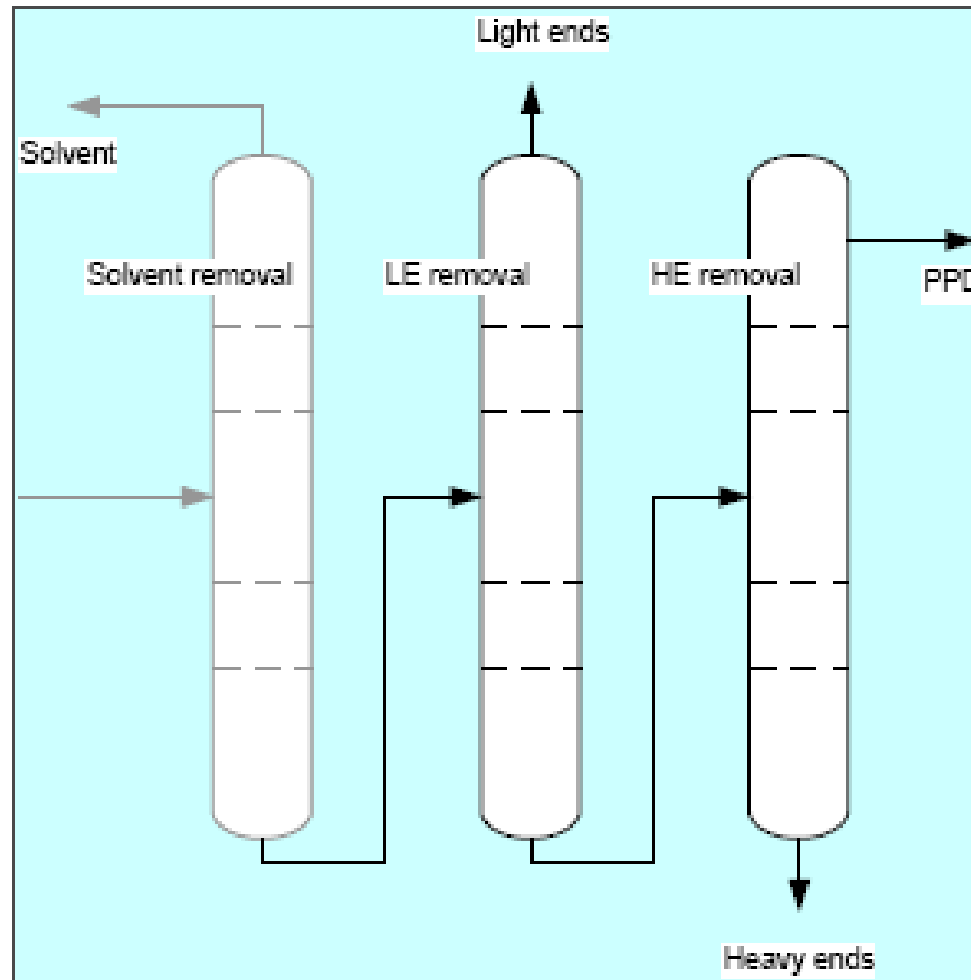
### APPLICATION EXAMPLES

- Dichlorobenzene Isomer Separation
- Nitrochlorobenzene Isomer Separation
- Nitrotoluene Isomer Separation
- Chlorotoluene Isomer Separation
- Meta-Xylene Purification with Ethylbenzene as byproduct
- MDI Isomer Separation and Purification

- Bisphenol A Purification
- Caprolactam Purification
- DMT Purification
- Trioxane
- Monochloroacetic Acid
- High Purity Benzoic Acid
- Glacial Acrylic Acid
- Glacial Lactide
- Naphthalene Purification
- Para-tert. Butylphenol Purification
- Anthracene/Carbazole Separation and Purification

# Hybrid processes Crystallization and Distillation

## Application example

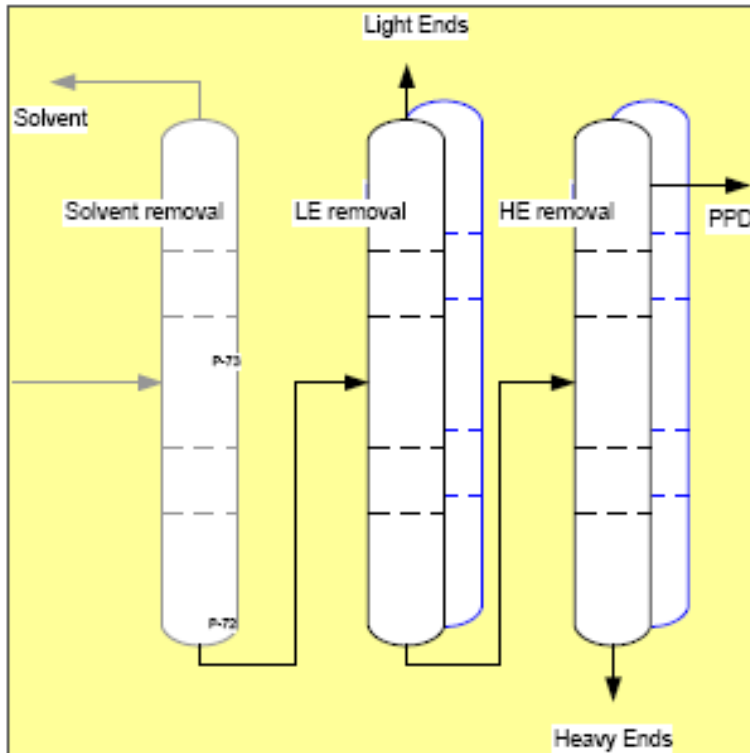


Lay-out before extension

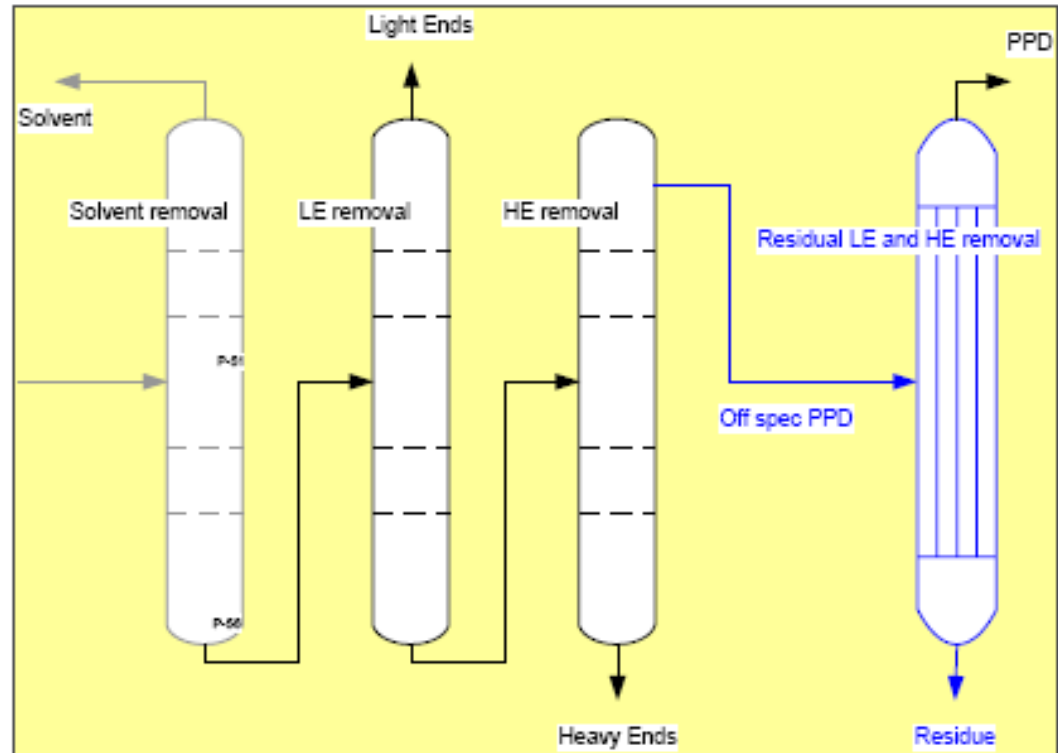
# Hybrid processes Crystallization and Distillation

## Application example

Conventional option



Hybrid option



## Expansion scenario's

### DISTILLATION

- general applicable (if volatile)
- proven technology
- engineering correlations available
- known technology at customer
- lab & pilot units available

### However

- high operating temperature (boiling point >> degradation)
- energy intensive
- limited purity (VLE depending)
- isomer separation difficult
- close-boilers difficult to separate

### CRYSTALLIZATION

- applicable for product
  - proven, but 'innovative' technology
  - confirmation pilot tests needed
  - new technology for customer
  - lab & pilot units available
- 
- lower operating temperature (melting-point >> no side reactions)
  - lower energy consumption
  - high purity (limited by eutectic point)
  - no problem with isomer, close boilers or azeotropic separation

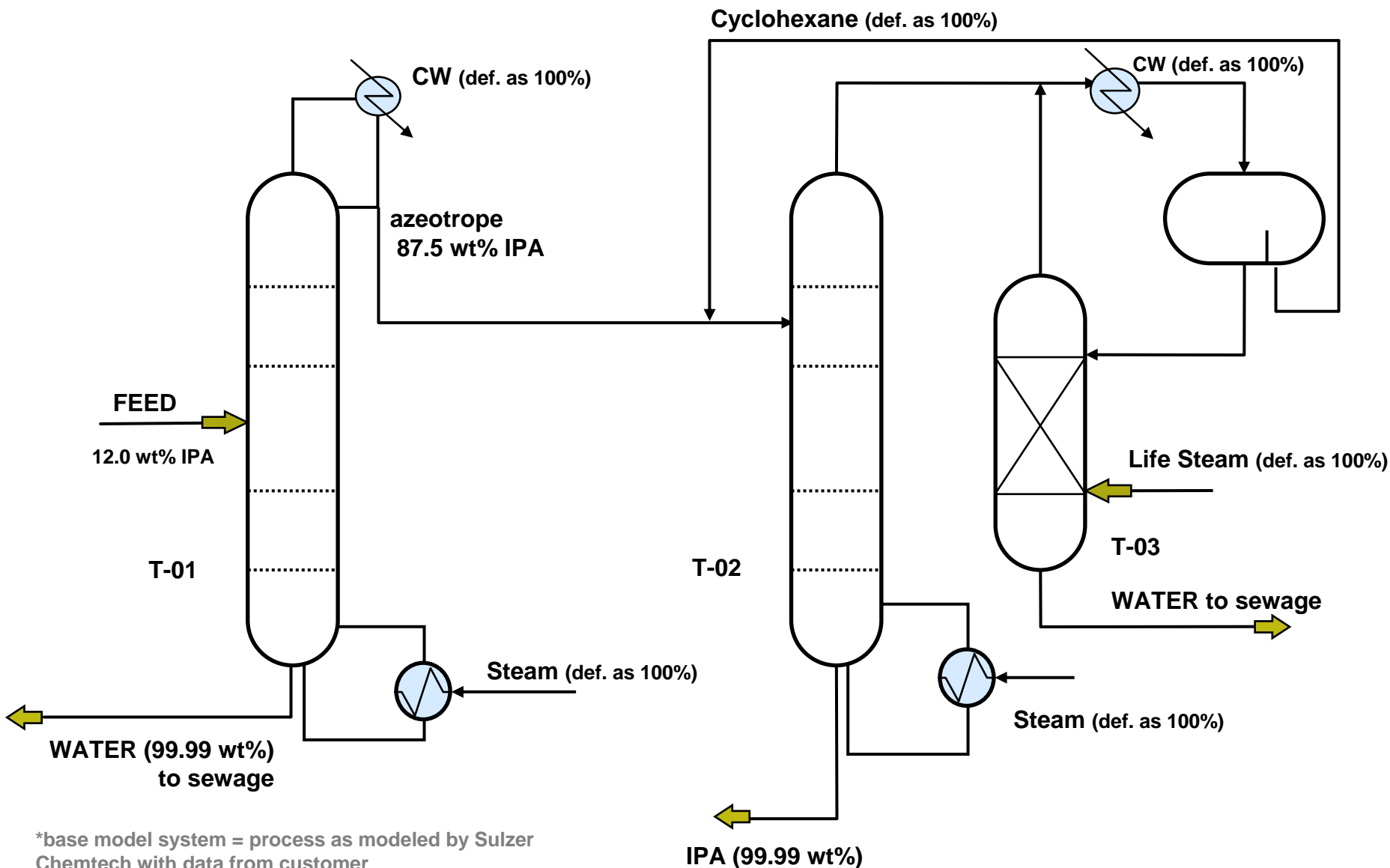


### Summary:

- Yield for crystallization of impure mixtures is poor due to phase diagram limitation of the product system
- Crystallization from impure mixtures requires multistage operation that leads to larger equipment and higher energy demand
- Distillation can, at relatively low energy demand, separate the bulk of impurities easily
- Combining the strong points leads to an ideal basis for a hybrid solution and a lower investment @ lower energy consumption
- **RESULT : HYBRID OPTION CHOSEN AND INSTALLED**

# Debottlenecking (Base Case\*)

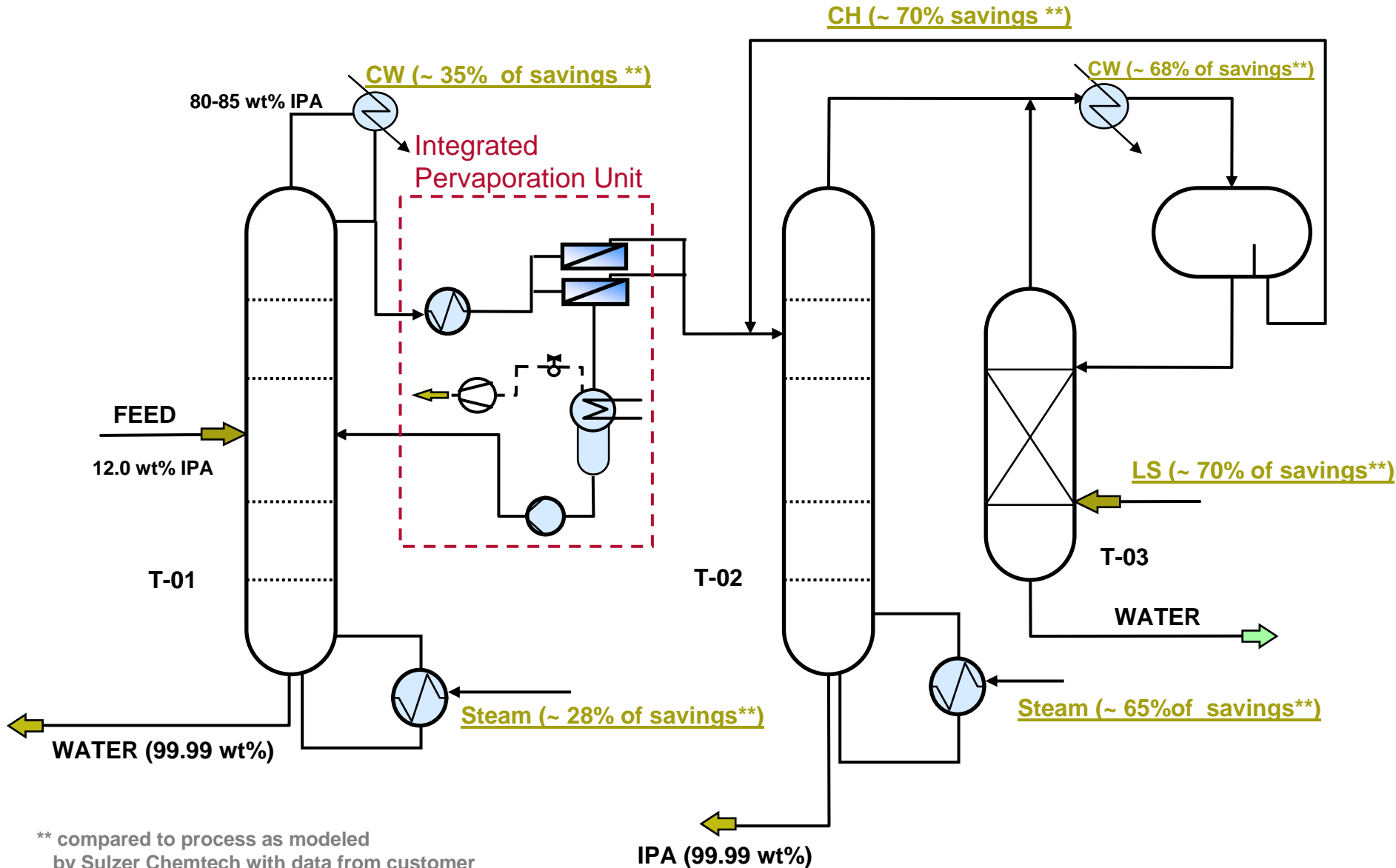
## Isopropanol Entrainer (azeotropic) Distillation



\*base model system = process as modeled by Sulzer Chemtech with data from customer  
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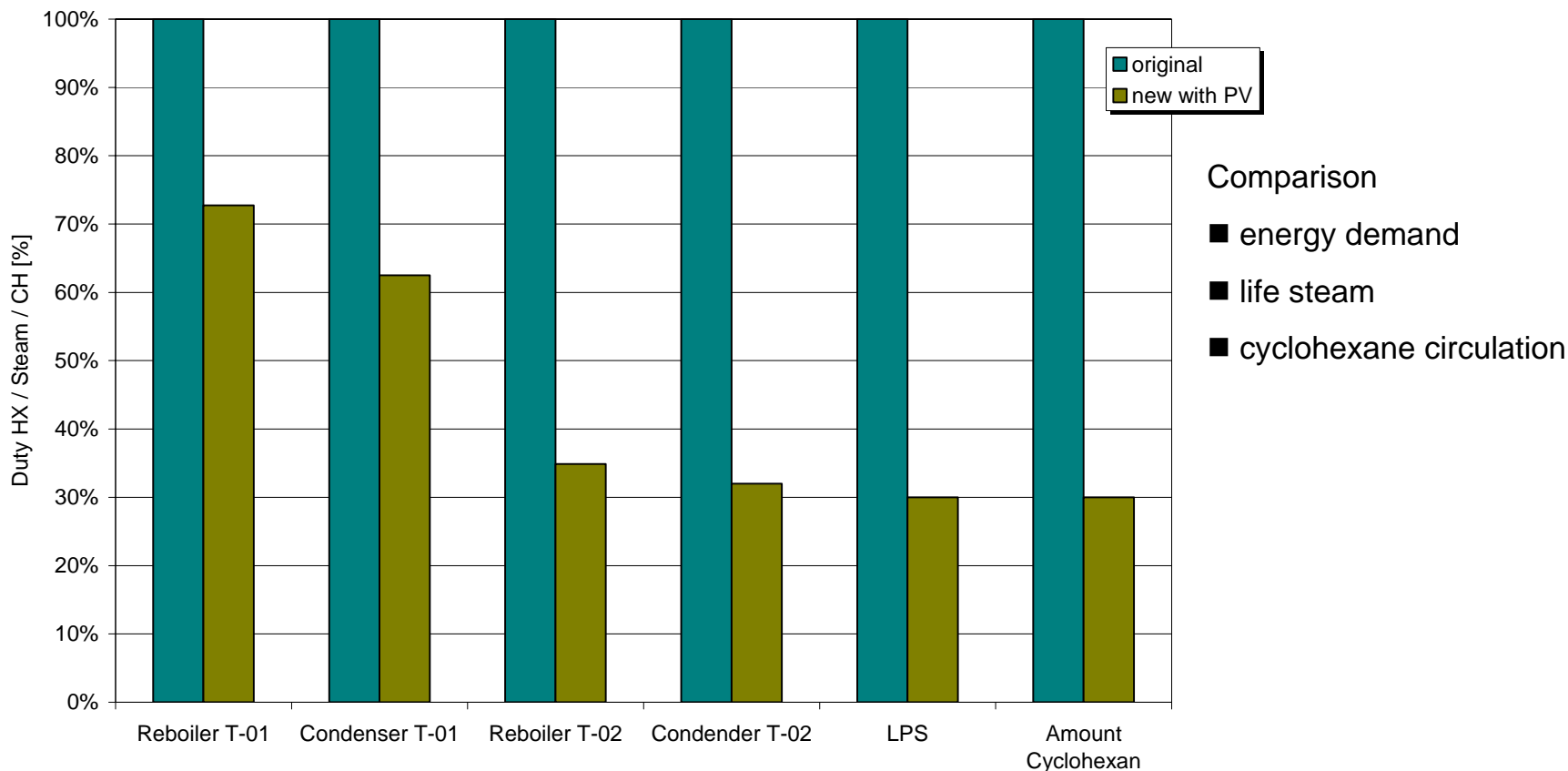
# Energy Efficient Hybrid System

(existing setup with low investment integrated PERVAP™)



\*\* compared to process as modeled by Sulzer Chemtech with data from customer

## Utility overview

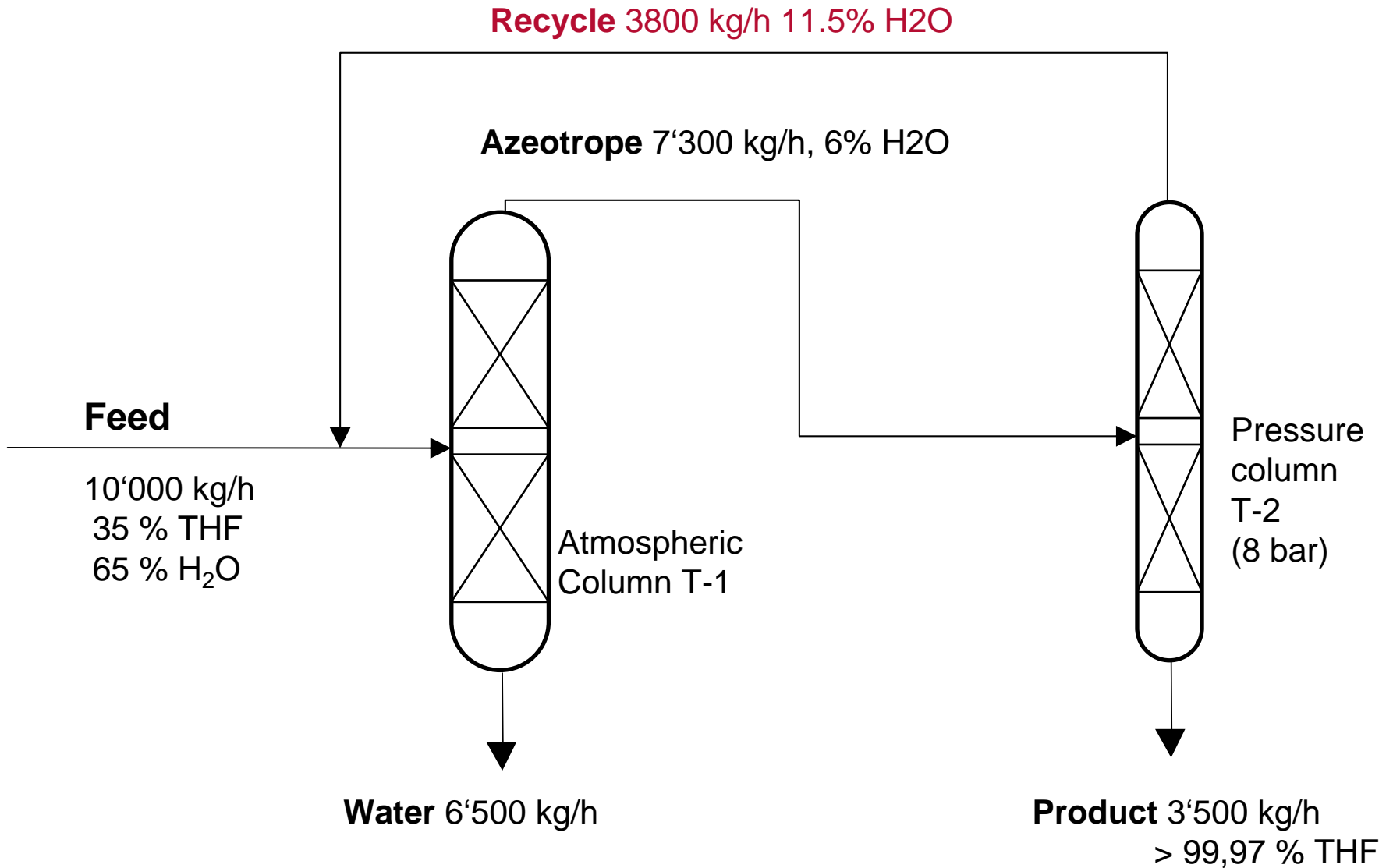


Utility costs (per ton product)	Cost Base		original	with integrated PV
Steam (average)	25	€/ton	100 %	55 %
Cooling Water	0,8	€/m <sup>3</sup>	100 %	49 %
<b>Total</b>			<b>100 %</b>	<b>52 %</b>

- Entrainer volume can be reduced by more than 60 %
- Capacity increase up to 35 % possible
- Energy savings of 120 € per ton/hr of product:
- Annual operation costs PV: ~200'000 Euro/yr
- Simple payback for investment of complete pervaporation system: 2 – 3 years including site engineering, civil engineering & works and pervaporation skid

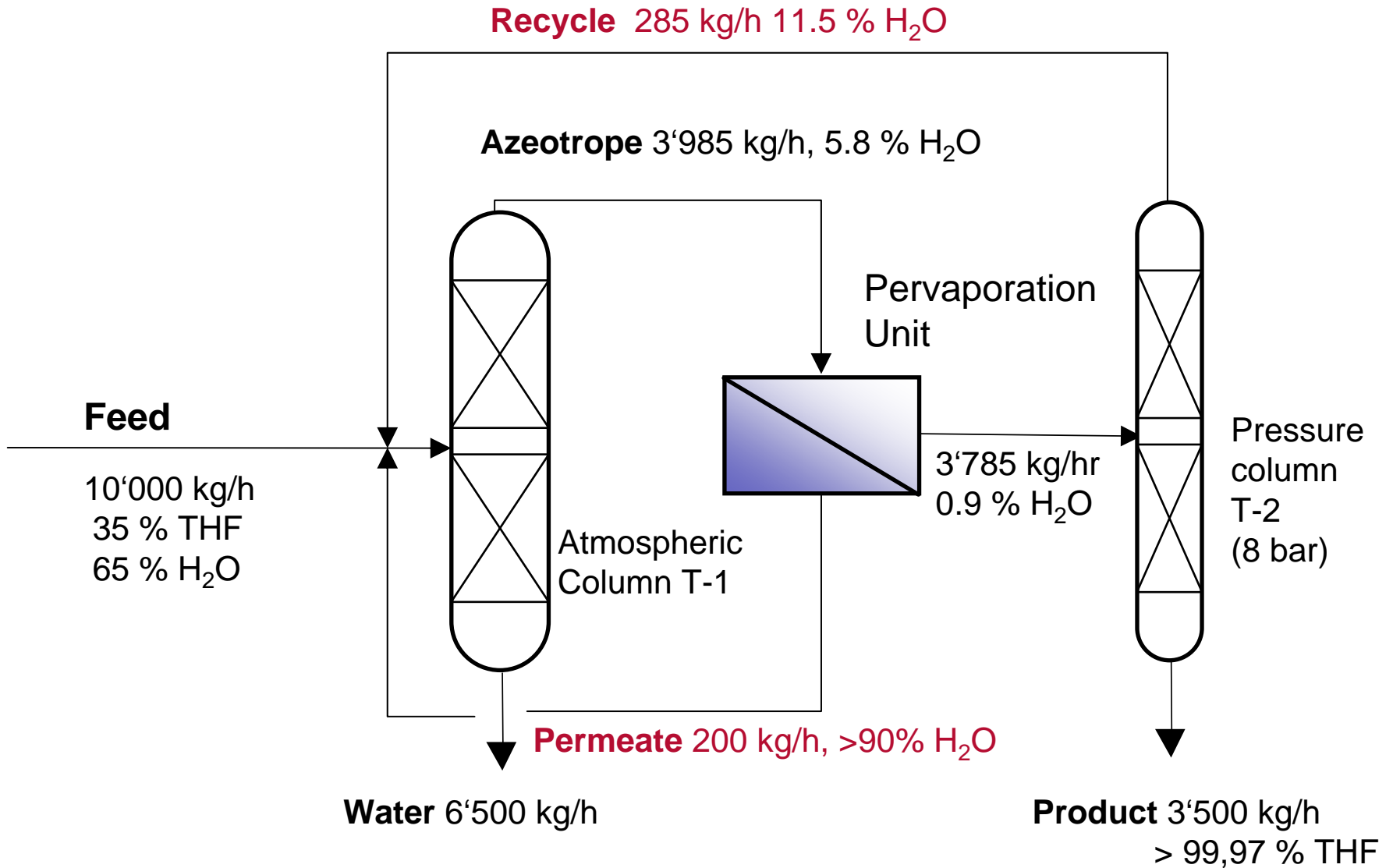
# Tetrahydrofuran (THF)

## Base Case : Pressure Swing Distillation



# Tetrahydrofuran (THF)

## Pressure Swing Distillation with Pervaporation Unit



# Tetrahydrofuran (THF)

## Comparison Heat Balances

### Without PV

Steam 4 bara	MW	t/hr
Reboiler T-1	1.78	3.01
Reboiler T-2	1.22	2.05
<b>Total steam</b>	<b>3.00</b>	<b>5.06</b>

### With PV

Steam 4 bara	MW	t/hr
Reboiler T-1	1.18	1.99
Reboiler T-2	0.30	0.51
PV Stage	0.20	0.34
<b>Total steam</b>	<b>1.68</b>	<b>2.84</b>

Cooling water 10 °C Δ T	MW	m3/hr
Cond. T-1	1.33	114
Cond. T-2.	0.91	78
<b>Total CW</b>	<b>2.24</b>	<b>192</b>

Cooling water 10 °C Δ T	MW	m3/hr
Cond. T-1	0.58	50
Cond. T-2.	0.21	18
Perm. Cond.	0.14	12
<b>Total CW</b>	<b>0.93</b>	<b>80</b>

Production 28.000 t/year > 99.97 wt% THF

- Decrease energy consumption or increase capacity with pervaporation
- Debottleneck and save energy within the same step



# Thank you for your attention !

## Questions ?

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**SULZER**

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Process Technology

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separation technology solutions with guaranteed performance**