COSTELLO

CHEM INNOVATIONS 2010 CONFERENCE & EXPO

Overview of Process Intensification

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Rocky C. Costello, P.E.
http://www.rccostello.con
310-792-5870
rcca@rccostello.com

What is Process Intensification (PI)?

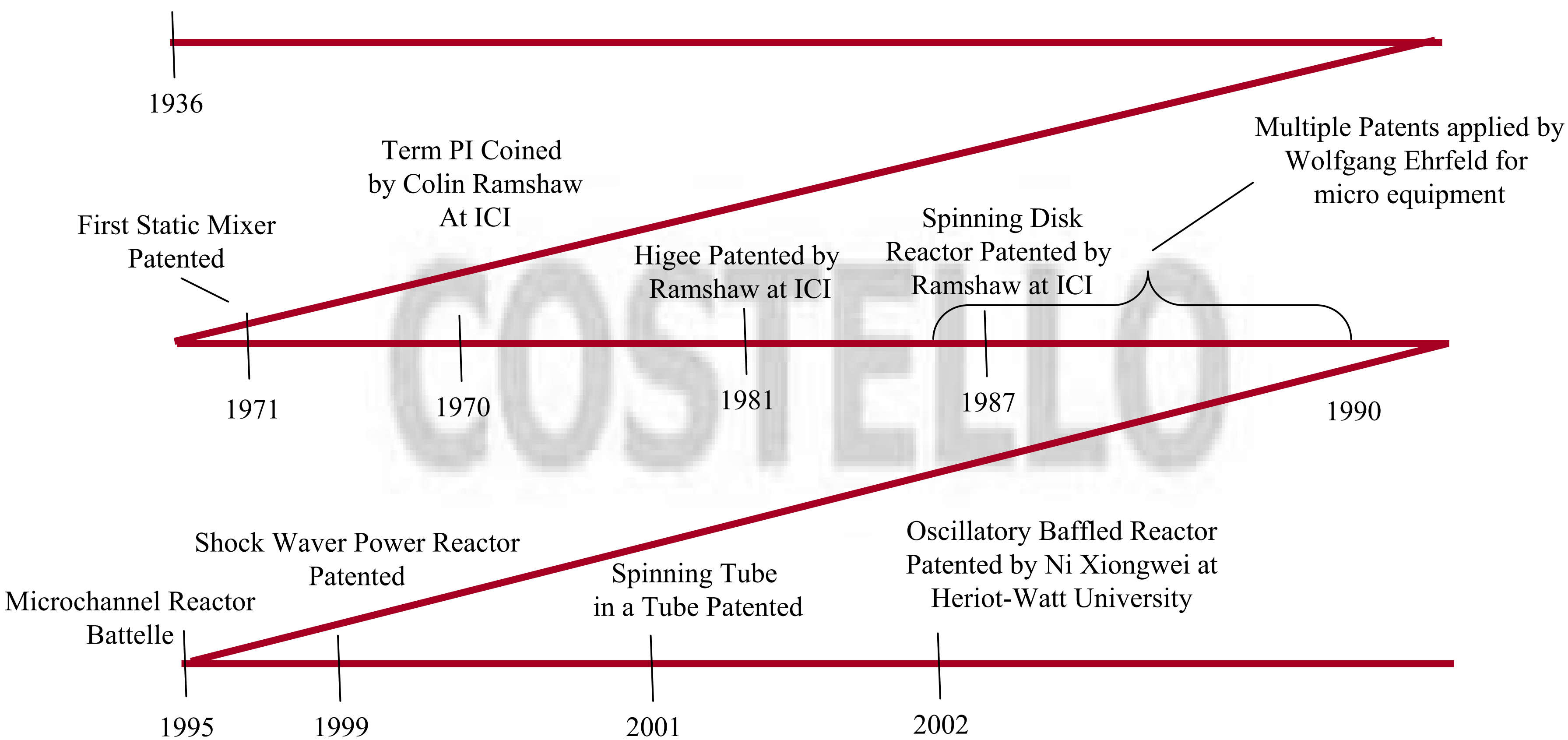
Technologies and Strategies that reduce the physical size of operating units thereby enabling cost reductions

(capital, operating, maintenance)

Why Intensify?

- ❑ ↓ Capital Costs
- ❑ ↓ Operating Costs
- ❑ ↓ Maintenance Costs
- ❑ ↓ Plant Footprint & Profile
- ❑ Facilitate scale up
- ❑ Provide basis for rapid development of products & processes (↓ Time Line)
- ❑ ↑ Safety
- ❑ ↓ Environmental Impact

Time Line



Podbielniak Centrifugal Extractor
1936
First Static Mixer Patented
1971
Term PI Coined by Colin Ramshaw At ICI
1970
Higee Patented by Ramshaw at ICI
1981
Spinning Disk Reactor Patented by Ramshaw at ICI
1987
Multiple Patents applied by Wolfgang Ehrfeld for micro equipment
1990
Microchannel Reactor Battelle
1995
Shock Waver Power Reactor Patented
1999
Spinning Tube in a Tube Patented
2001
Oscillatory Baffled Reactor Patented by Ni Xiongwei at Heriot-Watt University
2002

Equipment Options

Reactors

Distillation

Heat Transfer

Multifunctional Reactors

Other Methods

Reactors

Traditionally,
How Were Reactors Selected?

"Sometimes the choice of equipment was due to expediency and was jelled by tradition; or it may have been an inventor's individual preference, uninhibited by much knowledge of scientific principles."

Source: Reaction Kinetics for Chemical Engineers, Chapter 10, Industrial Reactors, By Stanley M. Walas

Today's Reactor Choice?

- Situation Driven
  (The Chemistry)
- Need Selected
  (Improve product quality)
  (Reduce installed cost)
  (Reduce purification steps)

Reactors Classified by Size

Focus - reactors with moving parts

- Micro - Millimeters
- Meso - Centimeters
- Macro - Meters

Micro Reactors

- Used in laboratories with most being little stirred tank units

- Can be set up in arrays with 100 reactors operating in parallel to test 10 different temperatures on one side of the array versus 10 different molar ratios on the other side of the array for reaction optimization

- May or may not scale up

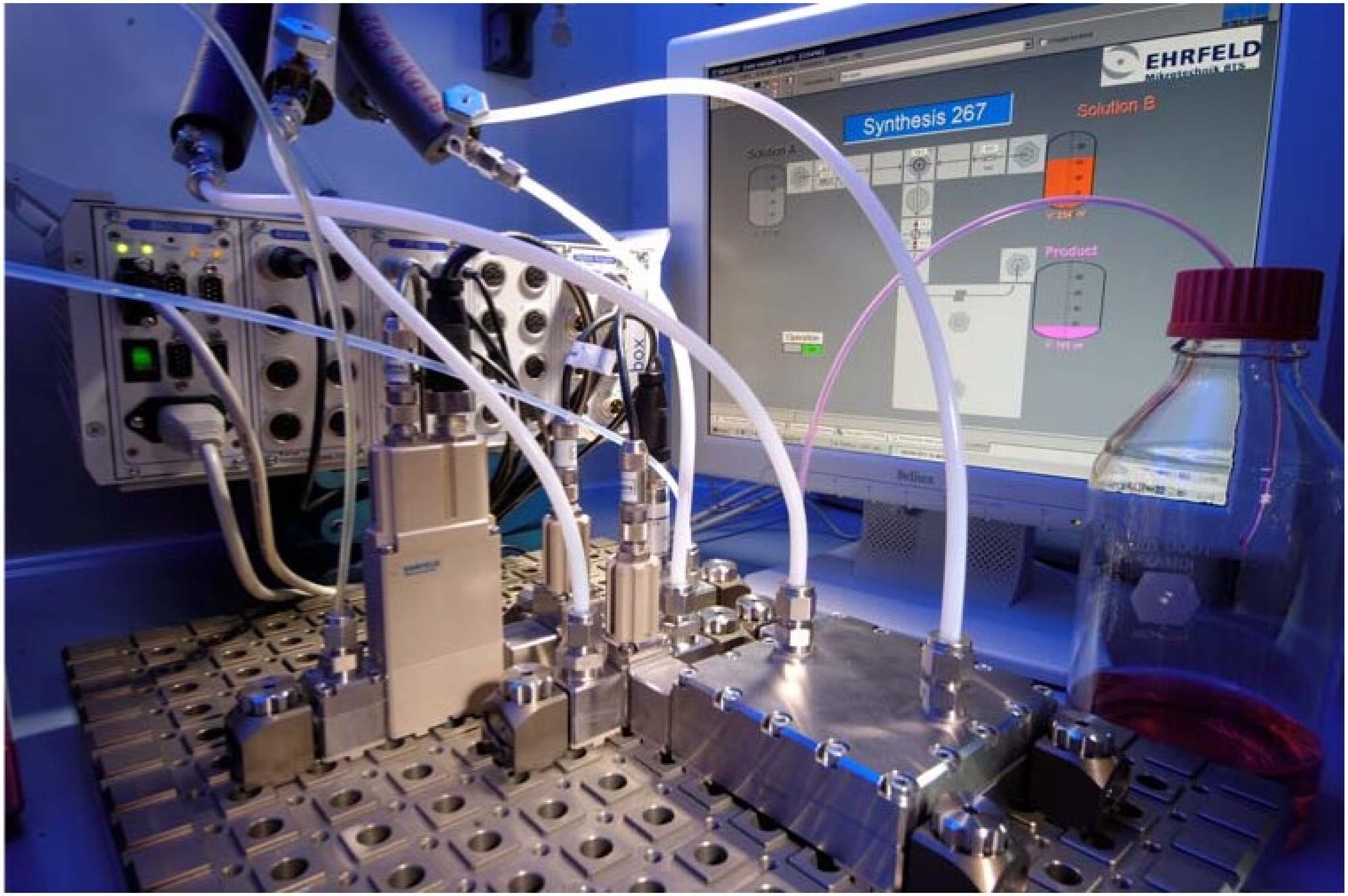
Meso Reactors

- The most commonly used size
- Typically scalable with in the meso scale range
- Wide range of options

Macro Reactors

- None operational to my knowledge

Ehrfeld Modular MicroReaction System



Ehrfeld Modular MicroReaction System

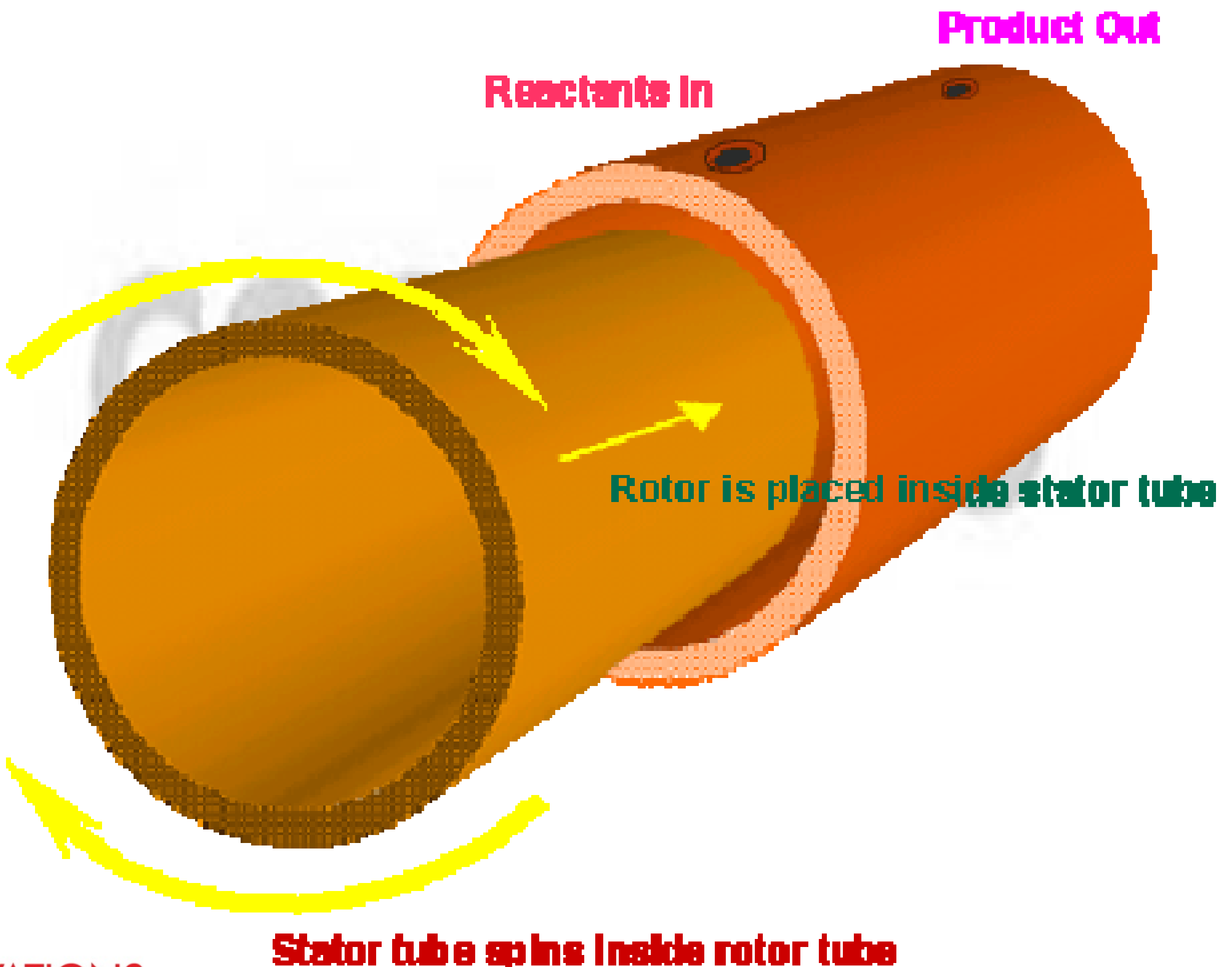
- More than 40 different modules
- Flexible assembly concept
- Easy process control & automation
- Easy Scale-up
- Pressure 0 to 100 bar (other ranges on request)
- Temperature -100 to 200 °C (others ranges on request)
- Materials
  - Stainless Steel 304L & 316L
  - Hastelloy C-276
  - Glass
  - other materials on request

Ehrfeld Modular MicroReaction System

Typical Modules Include:

- Mixers
- Heat Exchangers
- Reactors
- Filters
- Sensors

Kreido STT™



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Shear Rate Calculation

Shear rate is the surface velocity of the rotor divided by the gap size or

$$\frac{\pi D\omega}{d}$$

where: ω is RPS (revolutions per second)
D is the inner rotor diameter, and
d is the annular gap.

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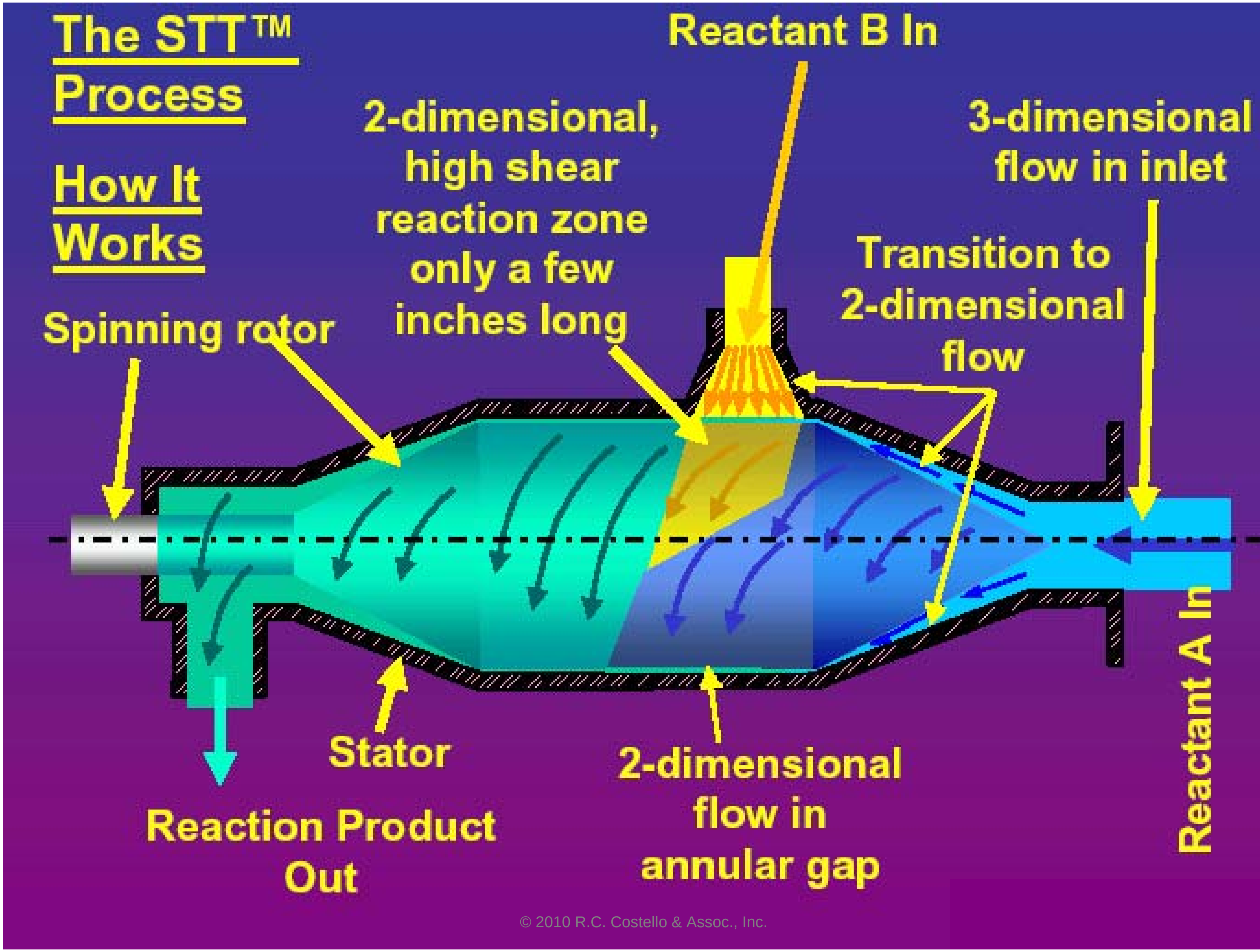
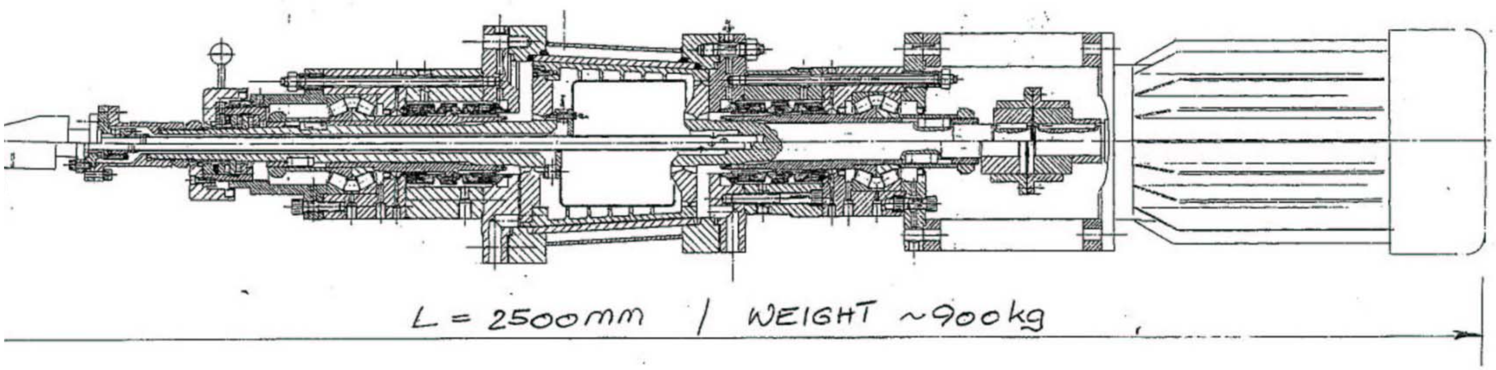


The STT™ Process
How It Works
Reactant B In
2-dimensional, high shear reaction zone only a few inches long
3-dimensional flow in inlet
Transition to 2-dimensional flow
Spinning rotor
Stator
2-dimensional flow in annular gap
Reaction Product Out
Reactant A In

REACTOR 0.2m², 3000 U/min

GIG KARASEK
system solutions for evaporation and biopharma



REQUIRED ACCESSOIRES:

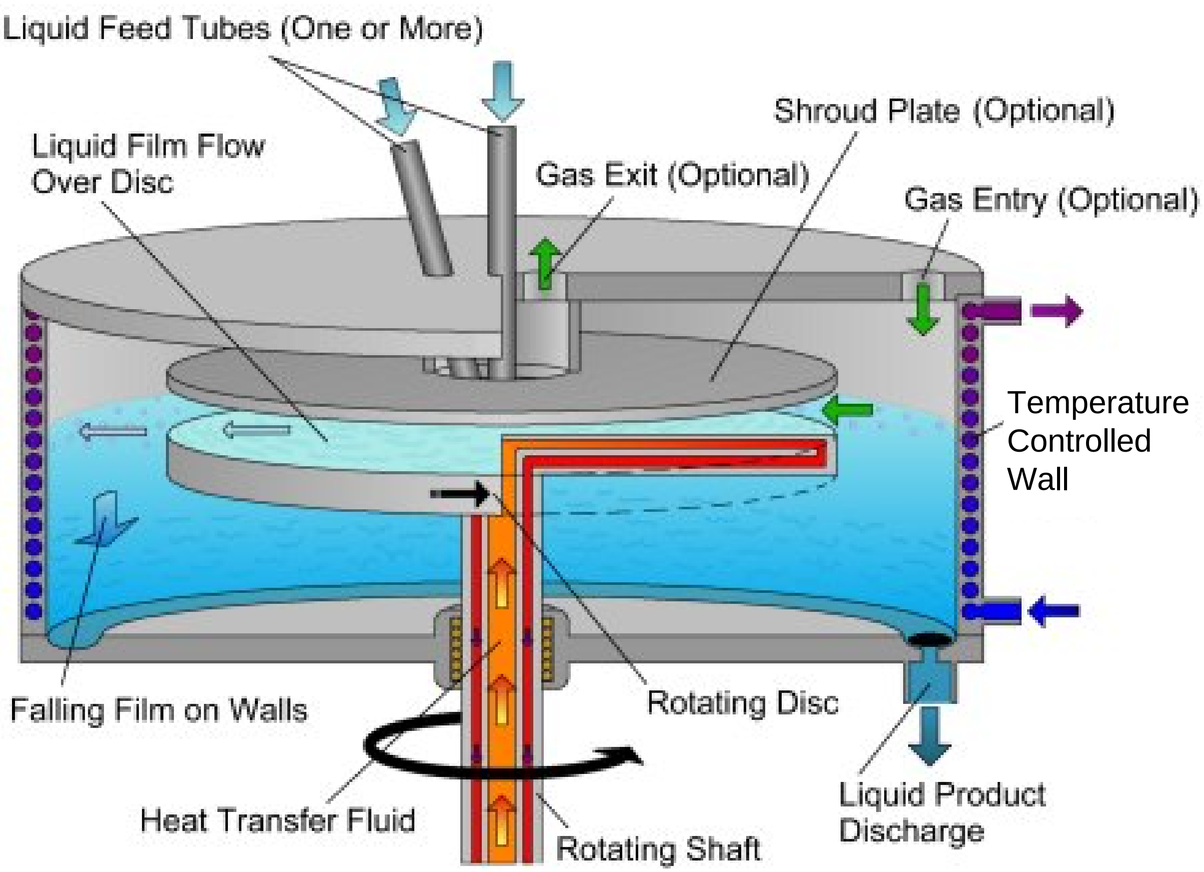
1) HEATING / COOLING THERMALOIL UNIT FOR JACKET, max. 300°C, 6barg
2) HEATING / COOLING WATER UNIT FOR ROTOR, max 150°C, 6barg
3) SEALING LIQUID UNIT FOR DOUBLE MECHANICAL SEAL, TWO CIRCUITS, max 150°C, max 80barg
4) OIL CIRCULATION SYSTEM FOR LUBRICATION AND COOLING
5) VALVES AND SAFETY DEVICES FOR PRODUCT
6) FREQUENCY CONTROL FOR MAINDRIVE 22KW
7) PROCESS CONTROL SYSTEM – SIMATIC

GIG Karasek
Thin Film Conceptual Reactor

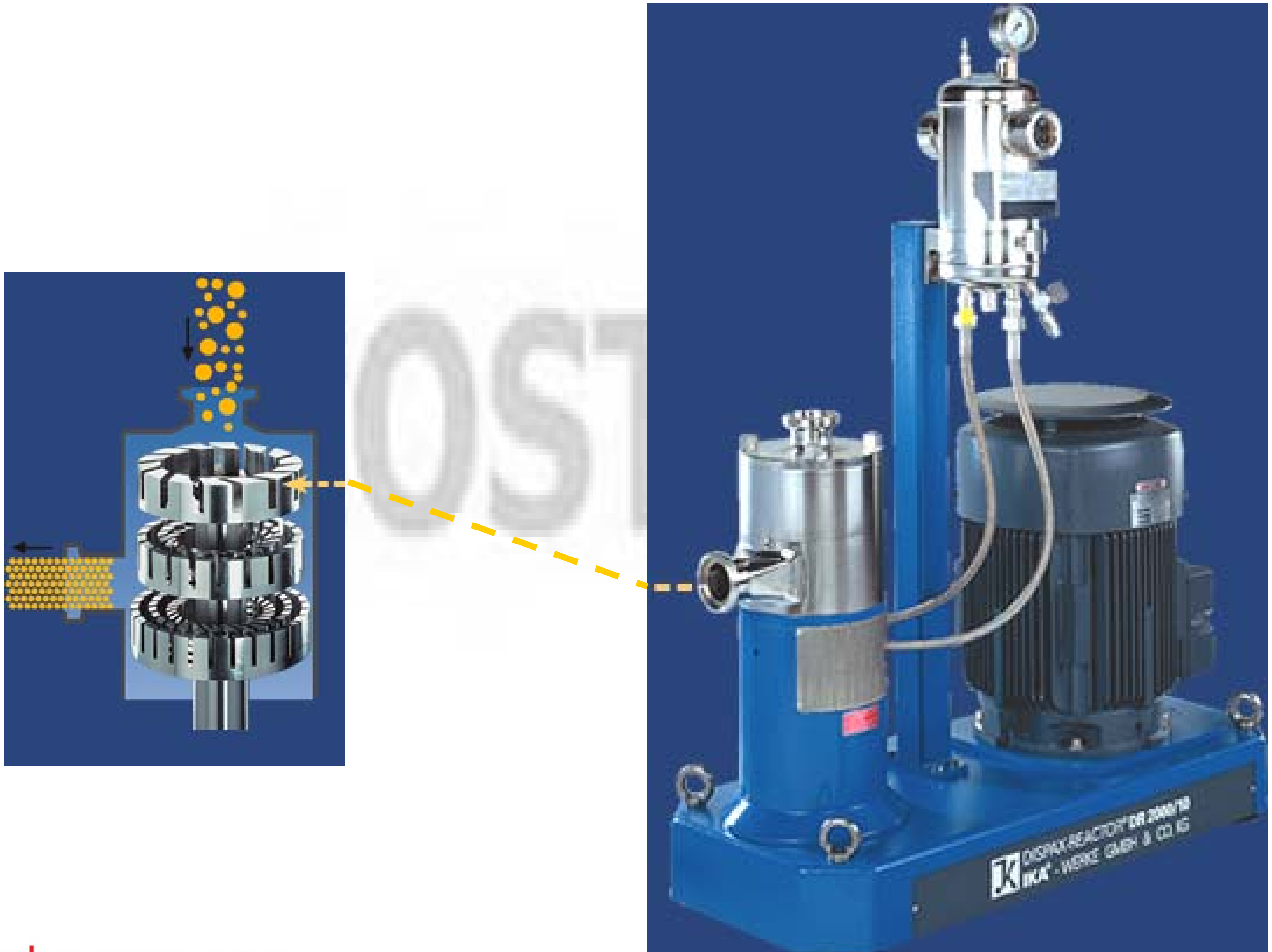
Features of the GIG Karaseck Conceptual Thin Film Reactor

- Variable speed
- Variable gap
- Cooling on the rotor
- Cooling on the stator
- Mechanical Seals
- Experience with thin film evaporators

Protensive Spinning Disk

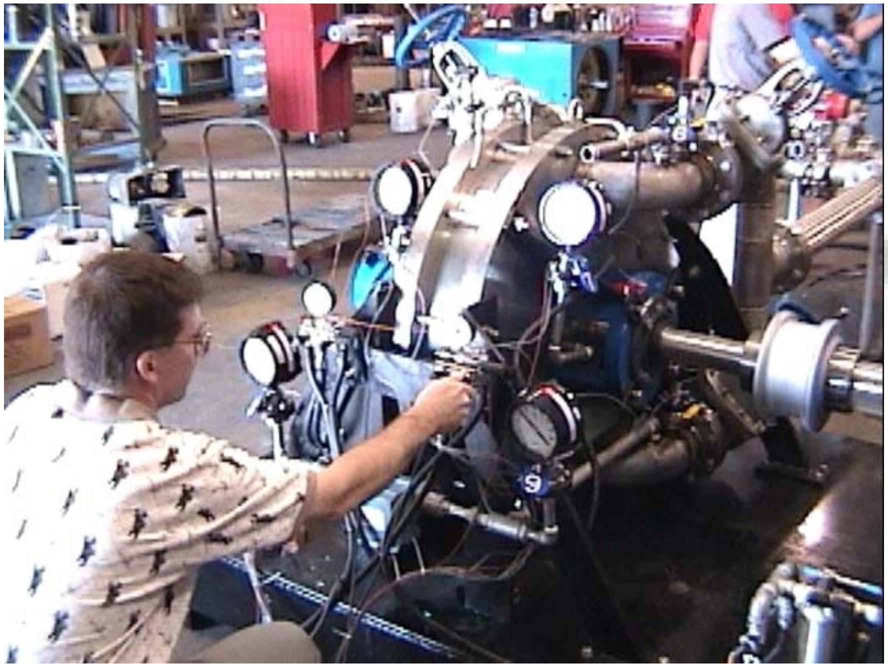


IKA Dispax



Hydrodynamics

ShockWave Power Reactor



The ShockWave Power reactor works by taking a fluid into the machine housing, where it is passed over the generator's spinning cylinder. The specific geometry of the holes in the cylinder, clearance between the cylinder, and the housing and rotational speed create pressure differences within the liquid where tiny bubbles form and collapse. These collapsing bubbles generate shock waves that are used to heat, concentrate and mix.

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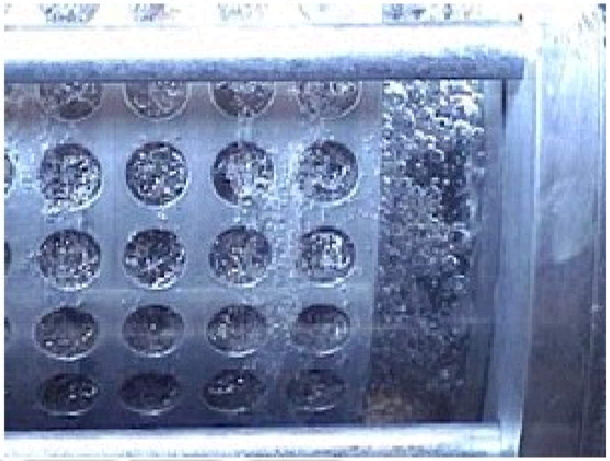
Hydrodynamics

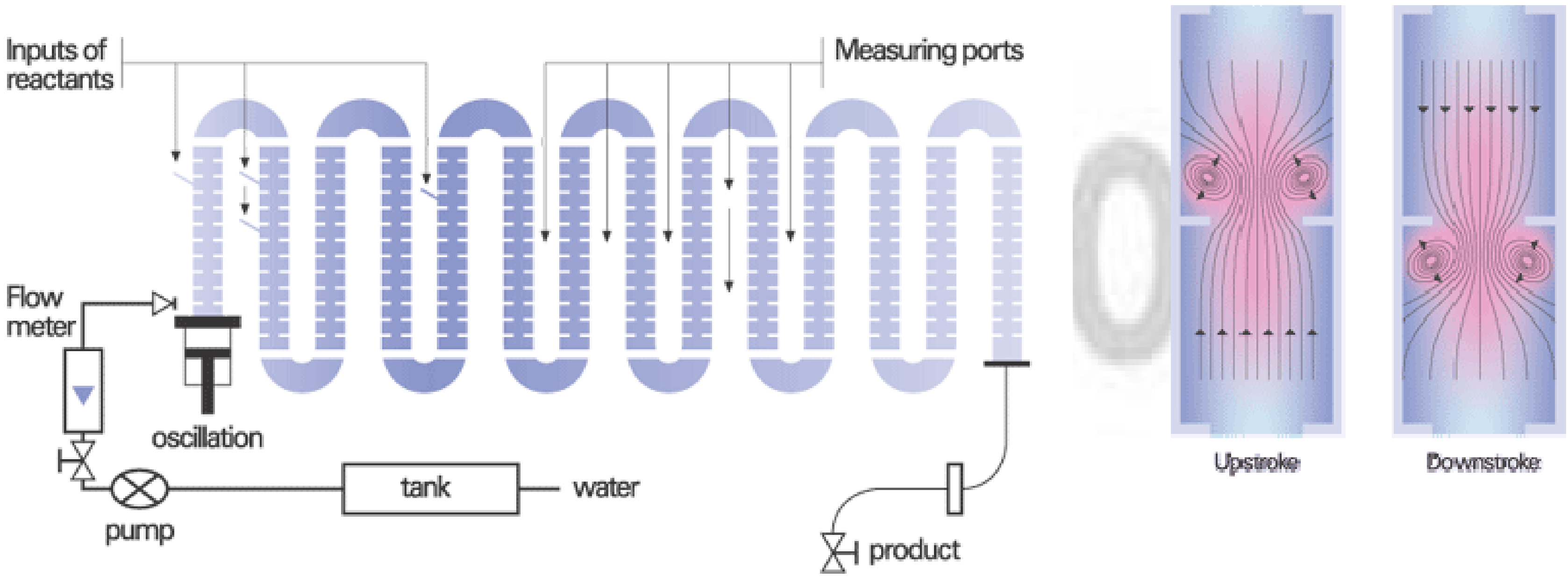
ShockWave Power Reactor

Mixing capabilities of the SPR at slow speeds. →



← Mixing at higher speeds

Continuous Oscillatory Baffled Reactor (COBR)



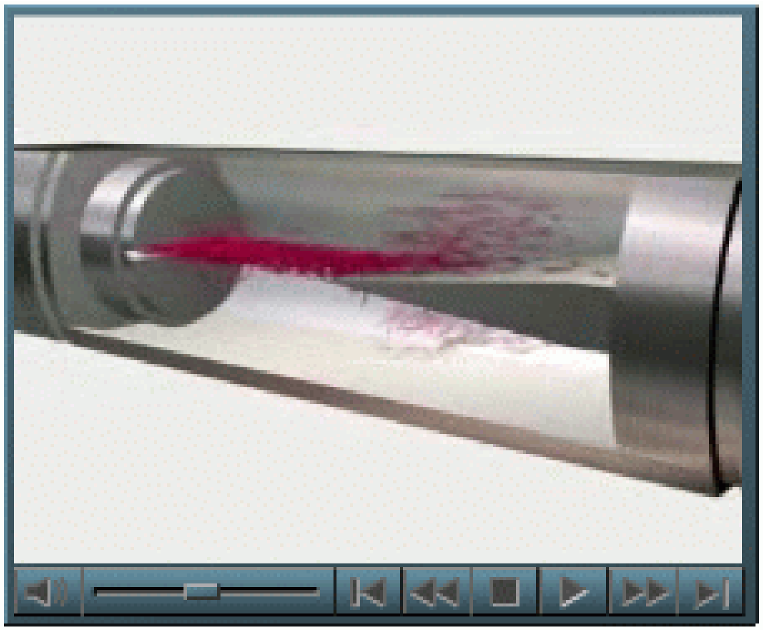
Continuous Oscillatory Baffled Reactor (COBR)

The basis of the technology is a tubular reactor with the presence of annular-baffles. The figure shows the mixing mechanism in a baffled cell . If a liquid is pushed up through the tube, eddies will be created around the baffles, enabling significant radial motion Likewise on a down stroke, eddies will be created on the opposite side and the intensity of eddy generation and cessation can precisely be controlled thus very effective mixing is created

Continuous Oscillatory Baffled Reactor (COBR)

- Fewer side reactions
- Less losses through unnecessary changeovers and clean out
- Less out-of-spec product

Sonic Engineering - Sonalator



The Sonolator is an in-line, continuous, high pressure homogenizer that subjects fluids to high pressure, extreme acceleration and ultrasonic cavitation by forcing the material through an engineered Orifice. The material is forced by a PD pump through the Orifice and is accelerated to greater than 300 ft/sec. The fluid cavitates as vortices of material are violently spun off in the opposite direction of the flow.

Jacketed Static Mixer

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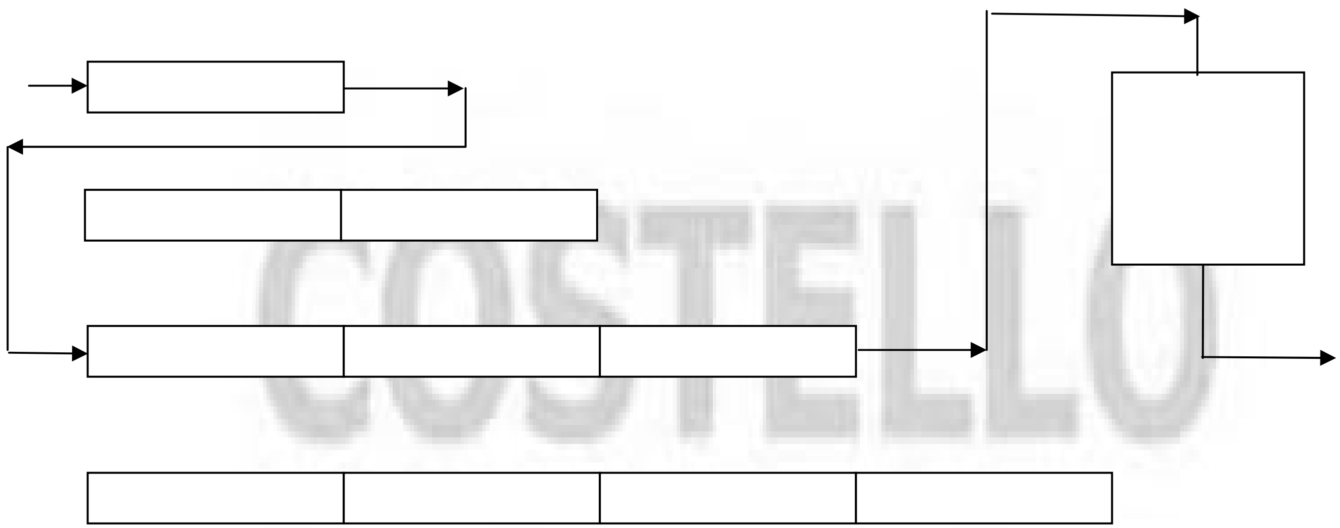
Static Mixer
Bench Scale Test Unit



1, 2, 3 and 4 foot static mixers that can be valved in any combination up to 10 feet.

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Jacketed Static Reactor

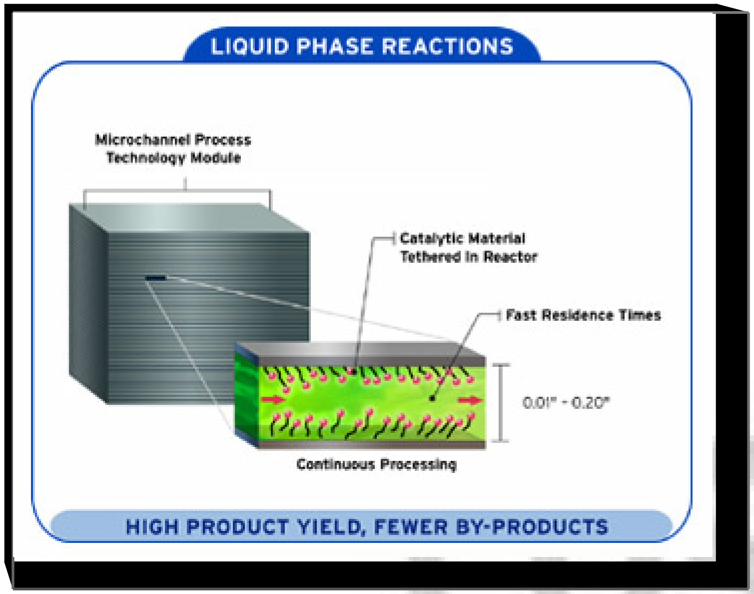
- Low cost
- Enhances Reaction Rates
- Performs better than plug flow reactor
- Jacketed for heat removal
- Scales up

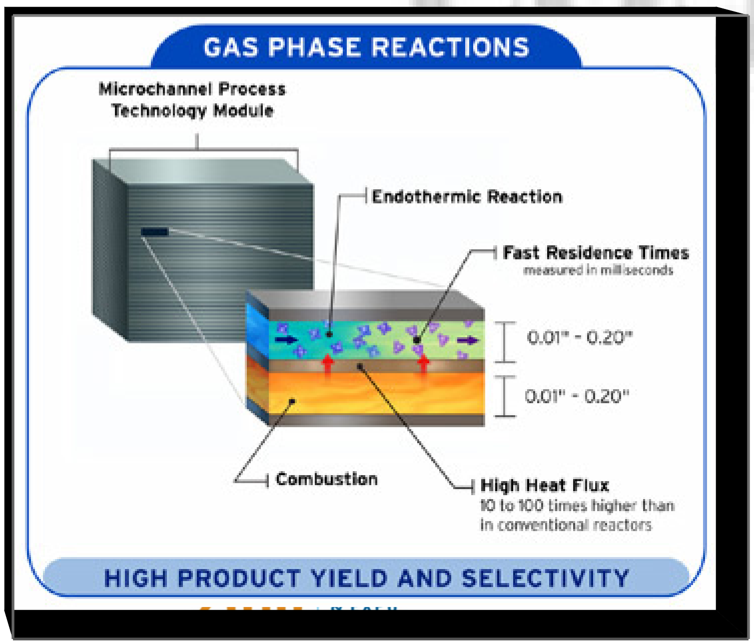
Microchannel Reactors

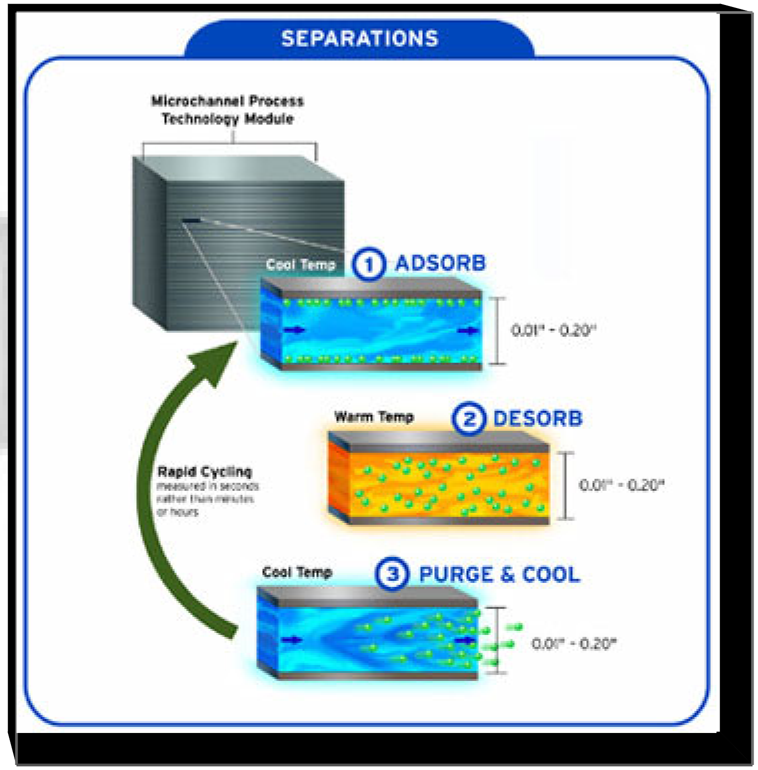
Two or more distinct fluid streams moving in the same capillary at low Reynolds number . . . do not develop turbulence at the interface between them, or at the interface with the capillary walls. The only mechanism for mixing the components of such a stream is diffusion across the interface.

Chemical & Engineering News, July 5, 1999; **Science**, July 2, 1999

Velocys Microchannel Reactors





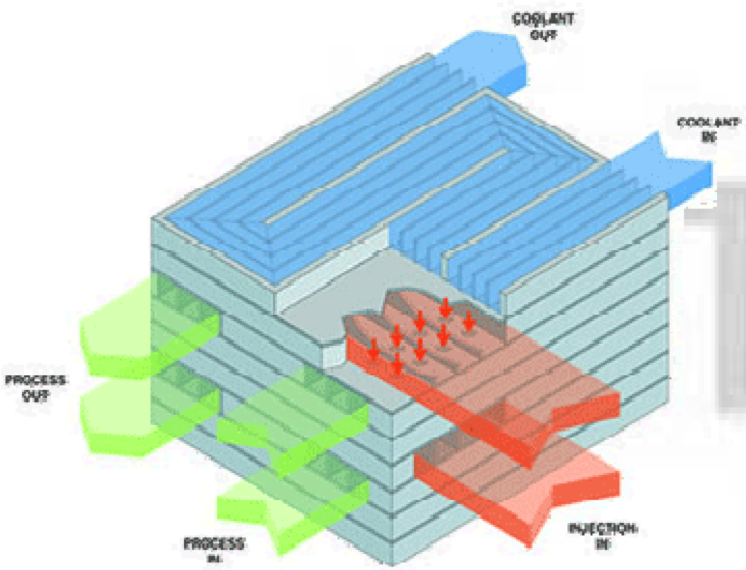


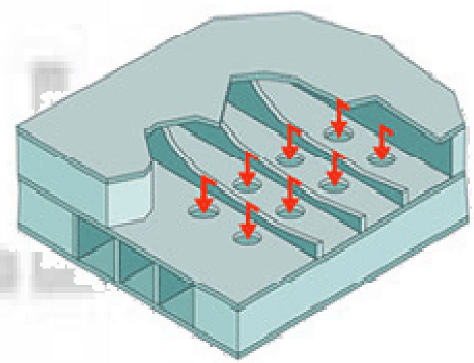
Velocys Features

- ↑ product yield & energy efficiency by improving heat & mass transfer performance
- ↓ capital costs
- Enhance catalyst productivity
- Cost-effective debottlenecking & expansion
- Substantially reduce pollutant emissions
- Create new products by enabling optimal processing conditions not possible with conventional hardware

Chart Energy & Chemicals

A macrochannel reactor and/ or heat exchanger





http://www.chart-ind.com/app_ec_reactortech.cfm

Reaction System Sizing

Kinetic Rate Expression

- ❑ 1st order reversible
- ❑ 2nd order reversible
- ❑ 1st order irreversible
- ❑ 2nd order irreversible

Reactor Equation

- ❑ Batch
- ❑ CSTR
- ❑ Plug Flow

Reactor Equations

- Batch $t = \int_0^X \frac{dX}{-r_A V}$
- $V = \frac{F_{A0} X}{-r_A}$ CSTR
- Plug Flow $V = F_{A0} \int_0^X \frac{dX}{-r_A}$
- Static Mixer – None
- SPR Reactor – None
- Microchannel Reactor – None

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Where Are We Regarding Reactor Design & Scale Up?

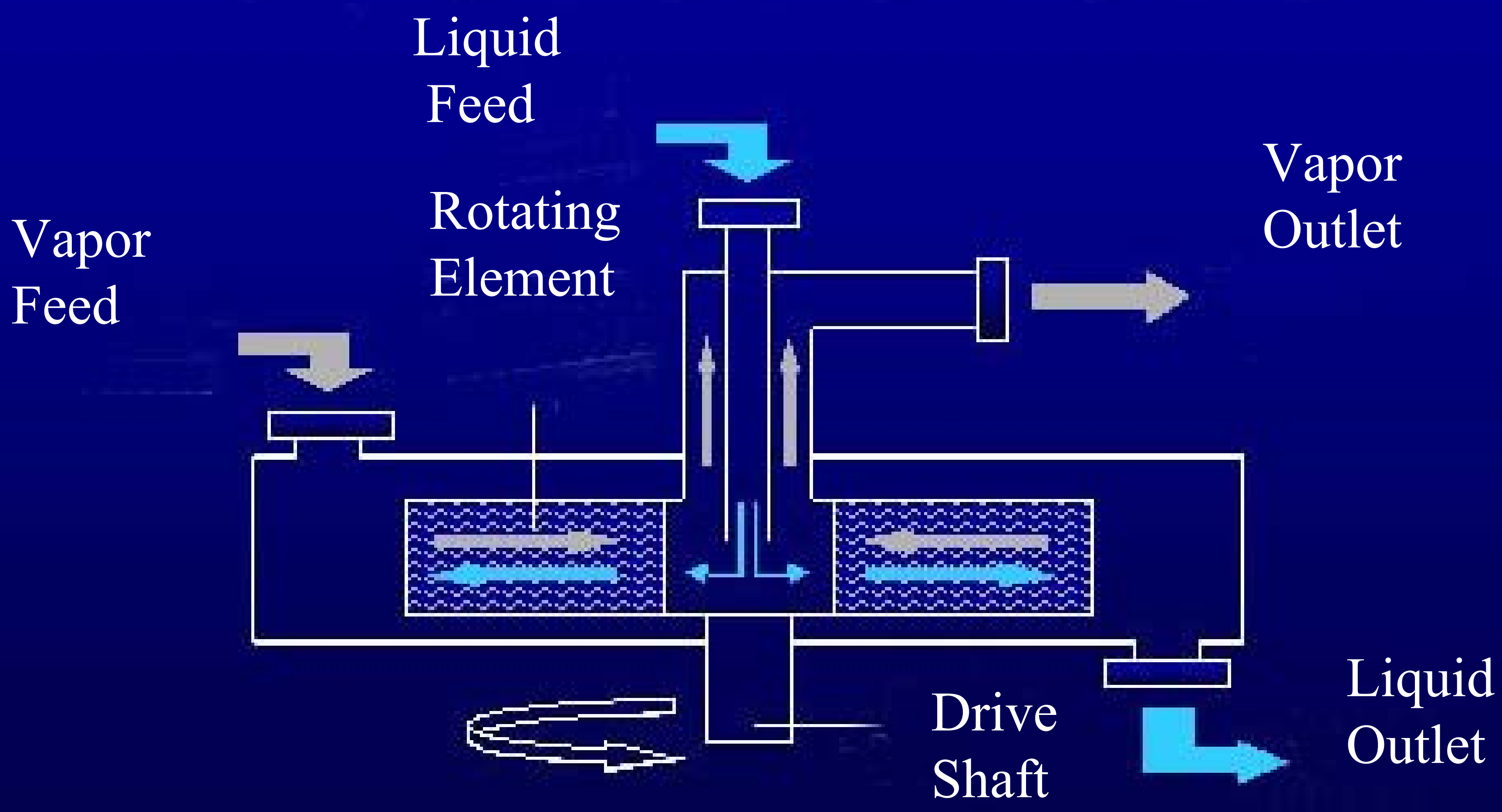
- ✓ Information void
- ✓ Bench Scale testing is crucial

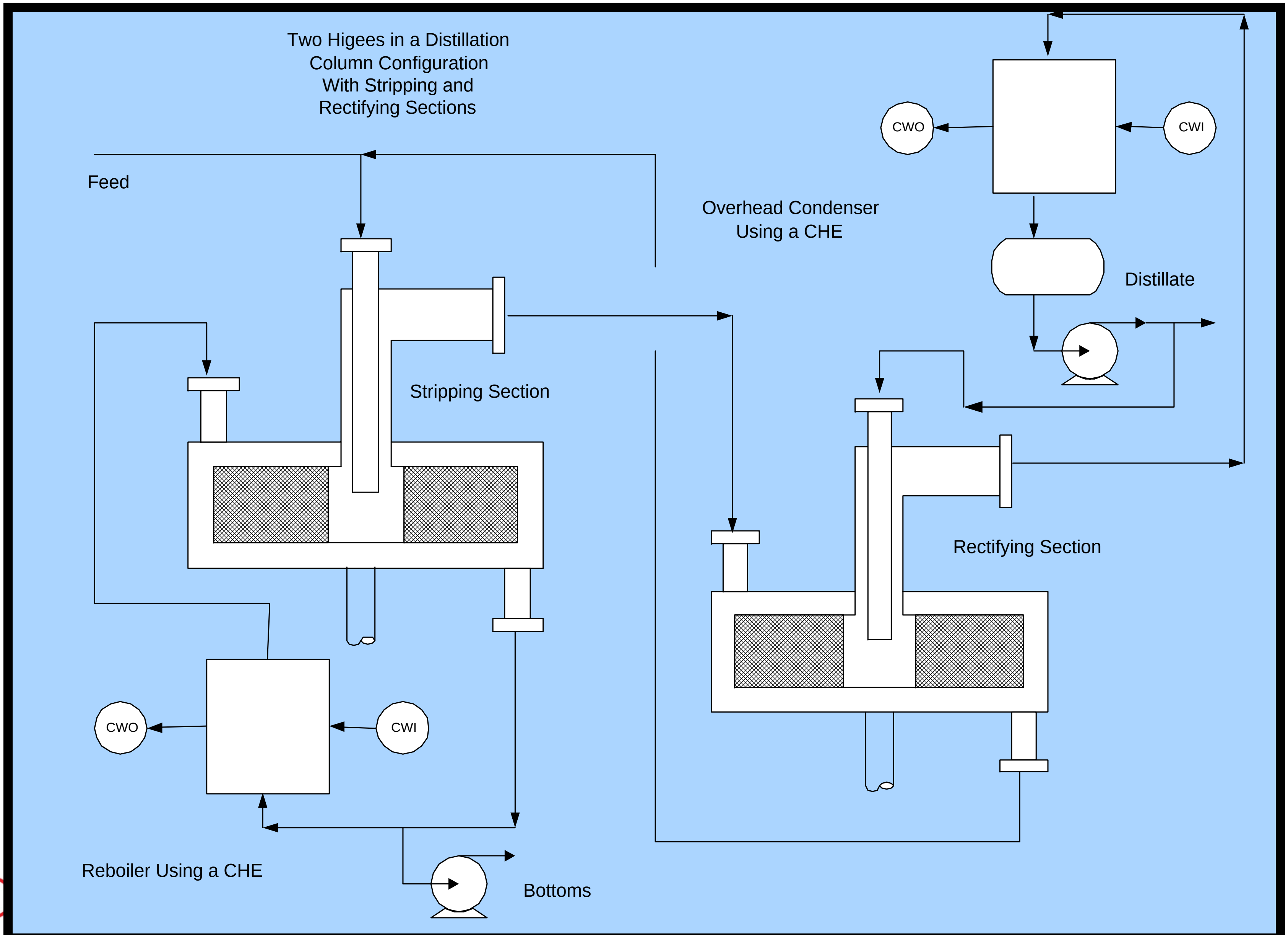
What can we expect?

✓Reduction in residence time compared to CSTR or plug flow reactor
✓Sometimes side reactions are minimized
✓Rarely side reactions are amplified

Distillation

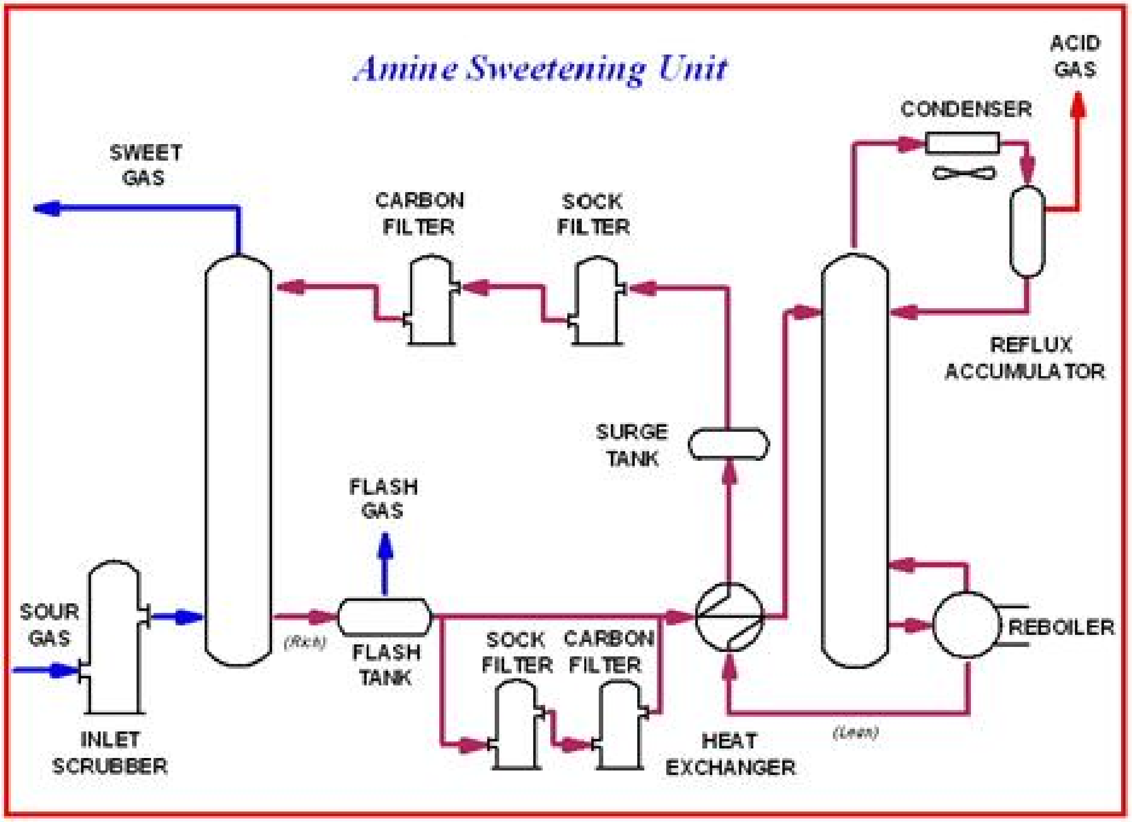
Higee - High Gravity Rotating Contactor





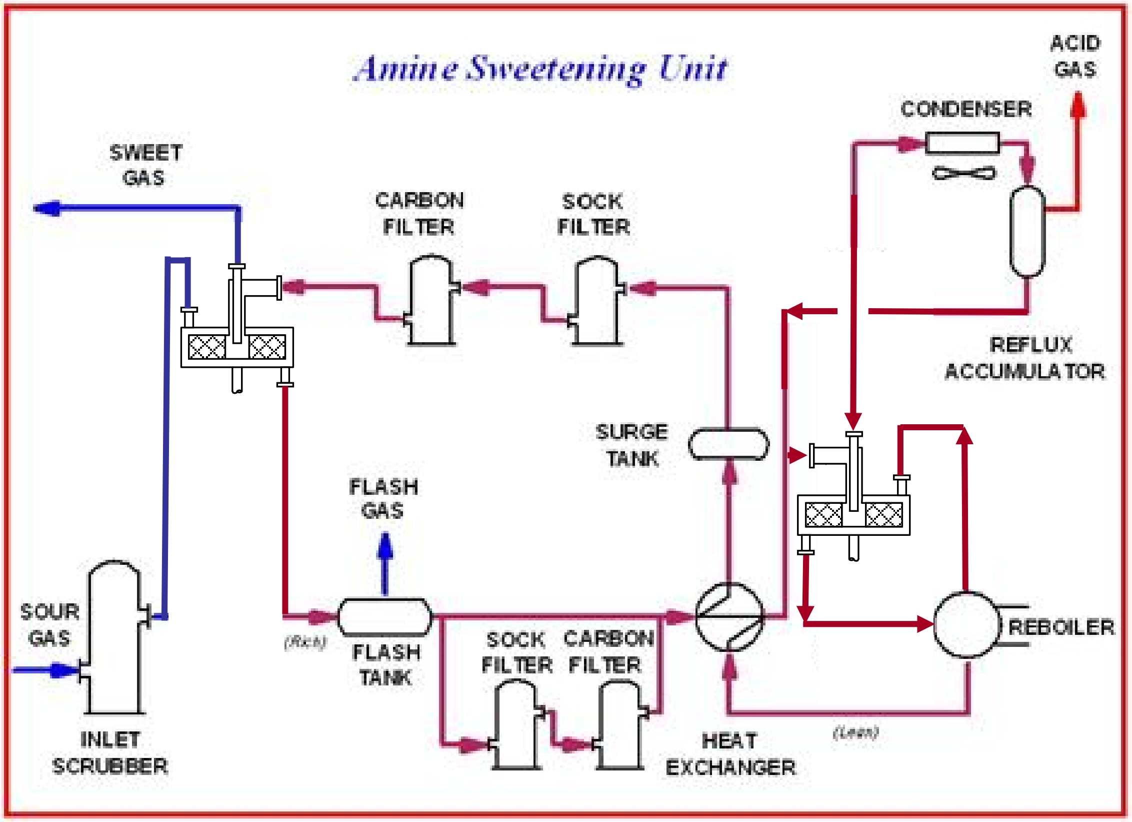
Two Higees in a Distillation
Column Configuration
With Stripping and
Rectifying Sections
Feed
CWO
CWI
Overhead Condenser
Using a CHE
Distillate
Stripping Section
Rectifying Section
CWO
CWI
Reboiler Using a CHE
Bottoms

Before PI



Amine Sweetening Unit
ACID GAS
CONDENSER
SWEET GAS
CARBON FILTER
SOCK FILTER
REFLUX ACCUMULATOR
SURGE TANK
FLASH GAS
SOUR GAS
(Rich)
FLASH TANK
SOCK FILTER
CARBON FILTER
REBOILER
INLET SCRUBBER
HEAT EXCHANGER
(Lean)

After PI



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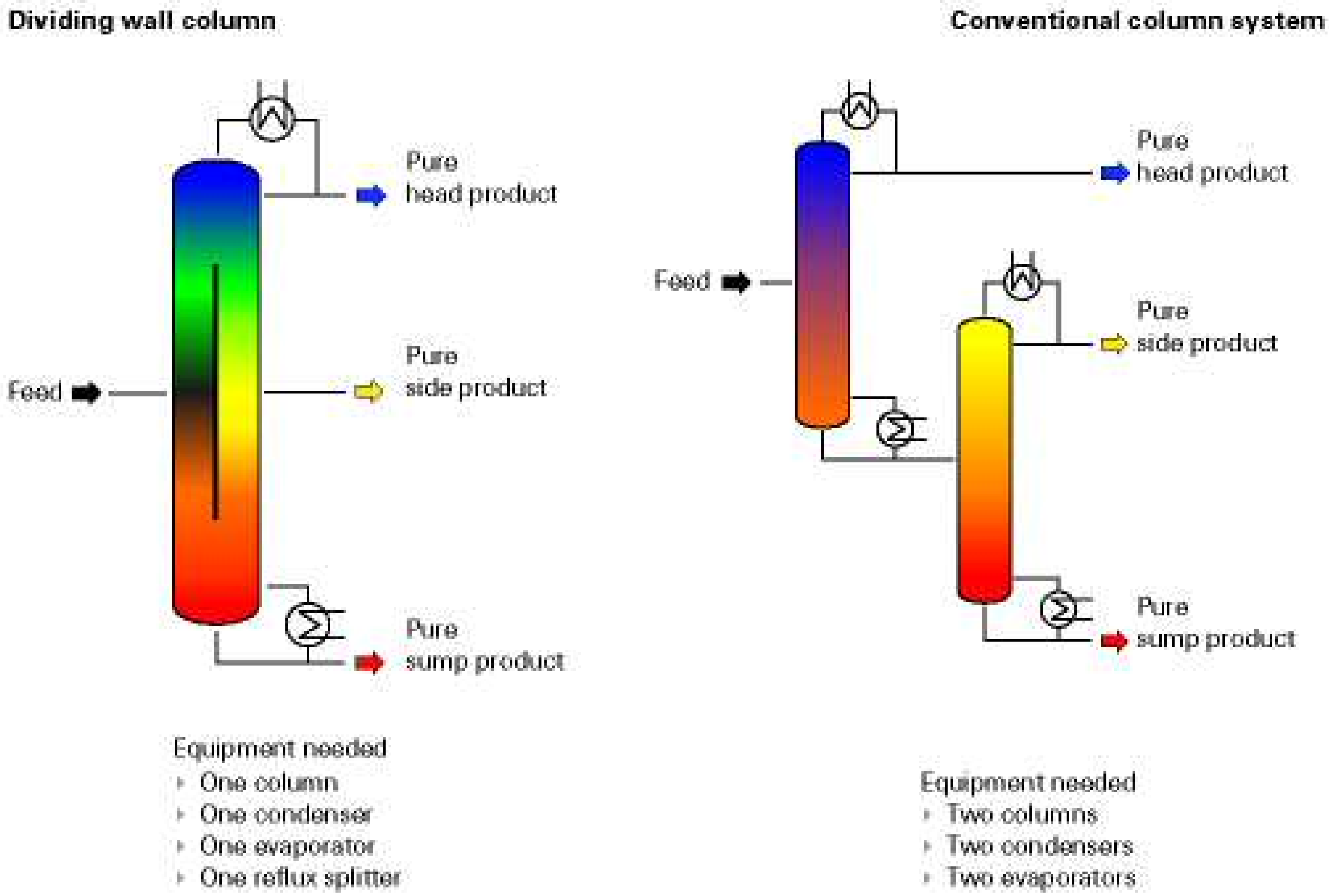
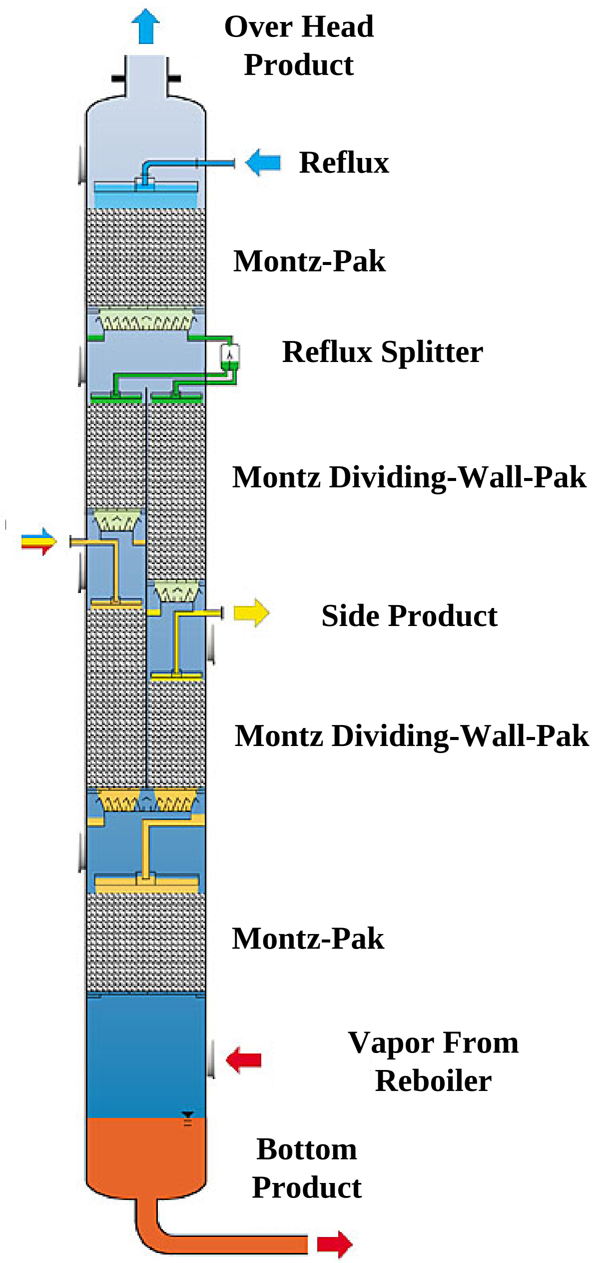
Divided Wall Column

- One divided-wall column can replace two or more conventional columns.
- The divided-wall column offers capital, energy and plot area savings compared to the conventional column configurations.
- The divided-wall column is applicable to some ternary separations where the mid boiling component is desired in high purity along with high purity lighter and heavier boiling products.
- It is estimated over 35 columns are in service.
- Used commercially so far in niche applications in fine chemicals, petrochemical, gas separation and refining industries

Divided Wall Column



Divided Wall Column



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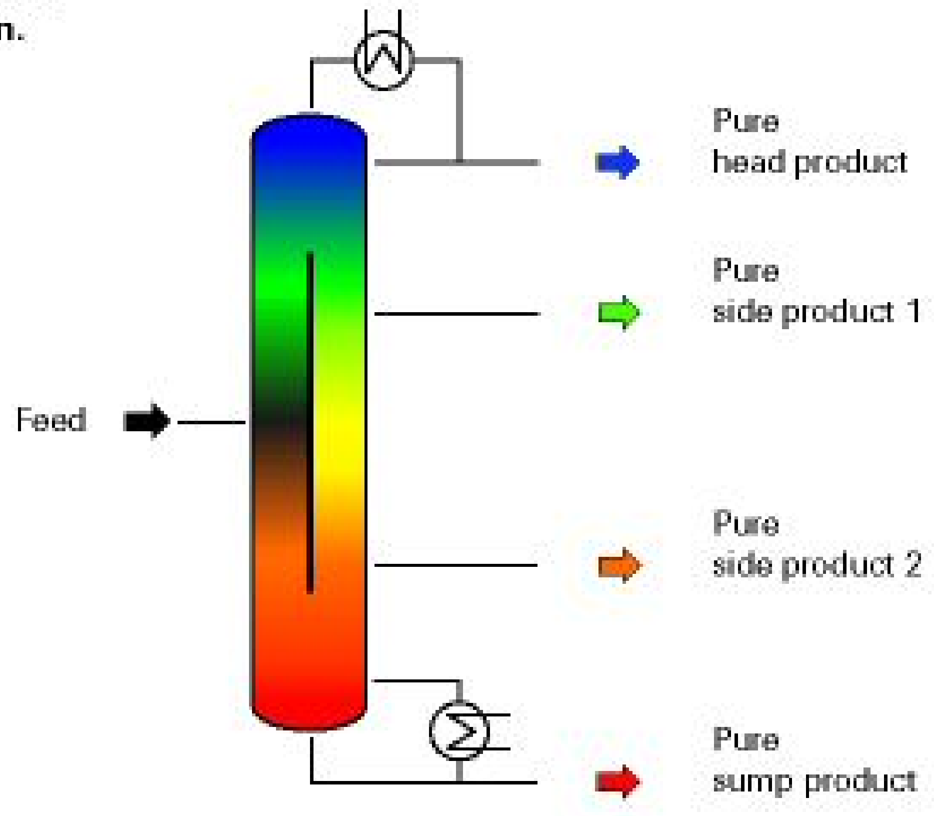
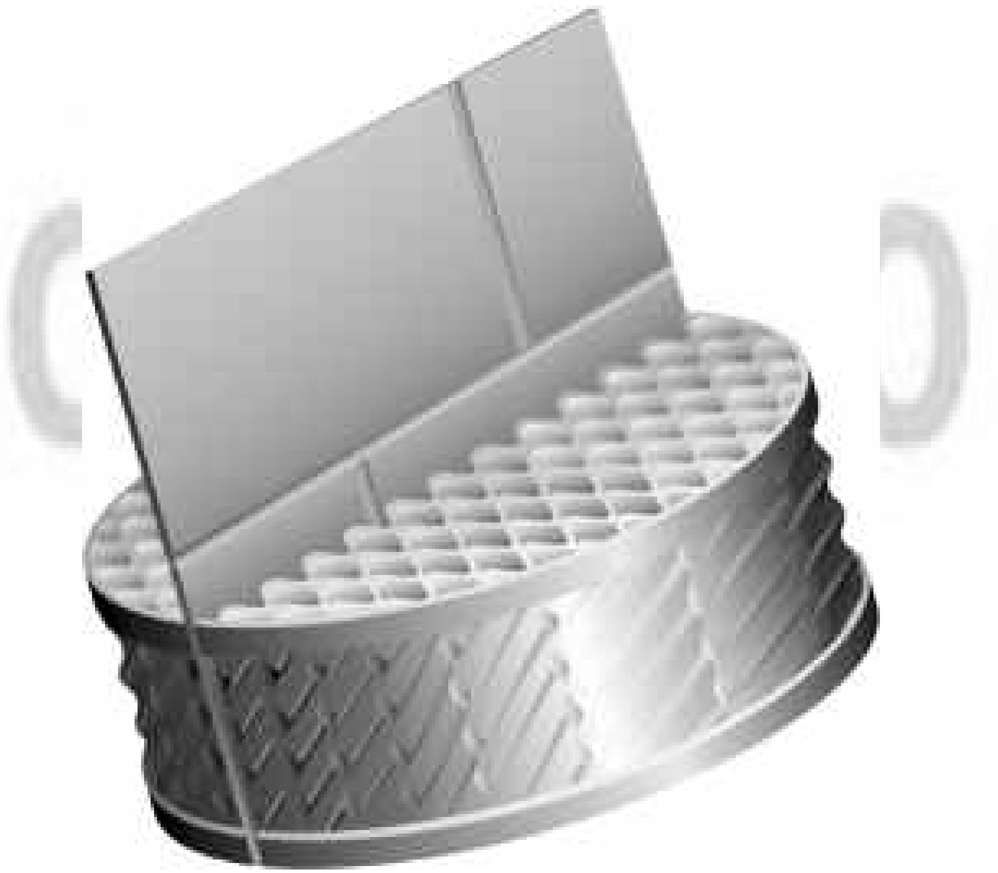
Divided Wall Column

4-component mixtures can also be separated into pure fractions in a single dividing wall column.



Divided Wall Column

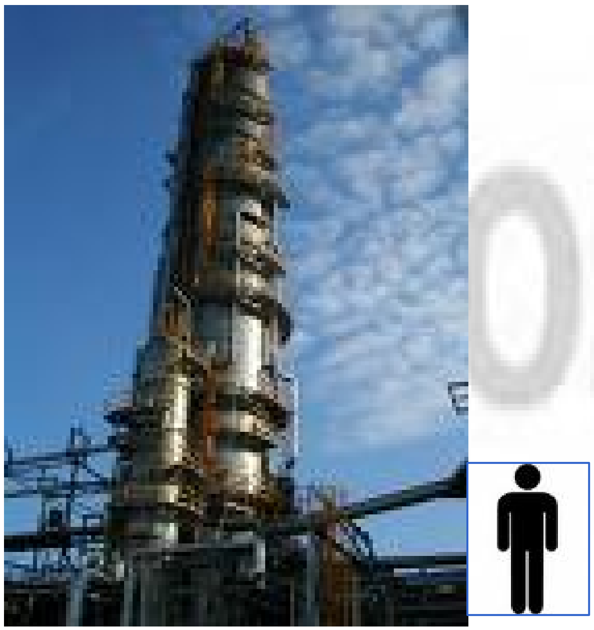


Divided Wall Column

Reflux Splitter

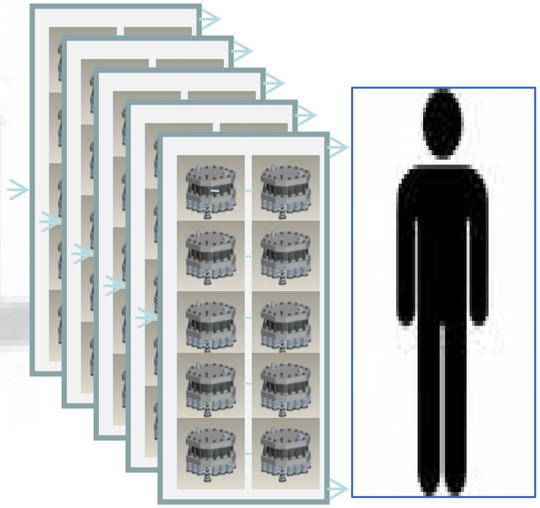
The iPod for distillation

Today's Distillation



fluXXion HEC:

High Efficiency Contactor



Diameter 0.5-6 m
Height 5-100 m
Weight 1-100 ton

100 times smaller

Modular system
Microsieve stacks
Box system

Fast, Flexible and Safe

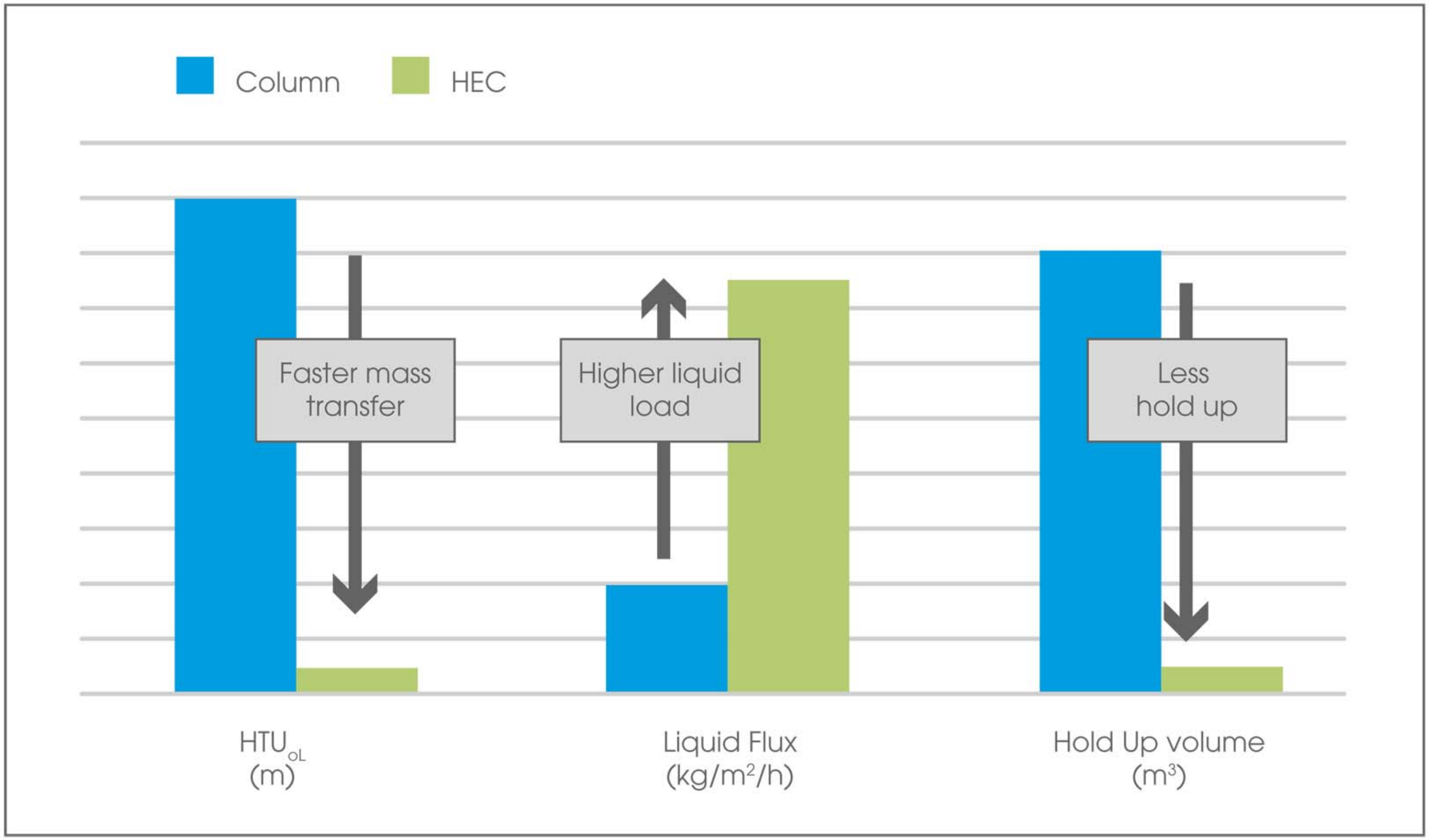
Today's Distillation

- High energy loss
- Separation inside column, heat recovery outside column
- Inflexible system
- Large dimensions
- High installation cost
- Well proven system

fluxxion

- Integrated heat and mass transfer
  - 100-1000x faster
  - 20-40% energy savings
- High flexibility
  - Capacity in operation
  - Modularity
  - Purity
  - Products
- Small dimensions
  - Easy to place anywhere
- Enhanced safety
  - small liquid hold up

Small, Flexible, Safe



- Stable operation (large pressure differential)
- No moving parts
- Individual module/stack temperature control possible
- High pressure operation possible (put module in autoclave!)

- Hazardous materials & pathways
- capacity & purity flexibility
- volume/height restricted situations
- low gas volume stripping

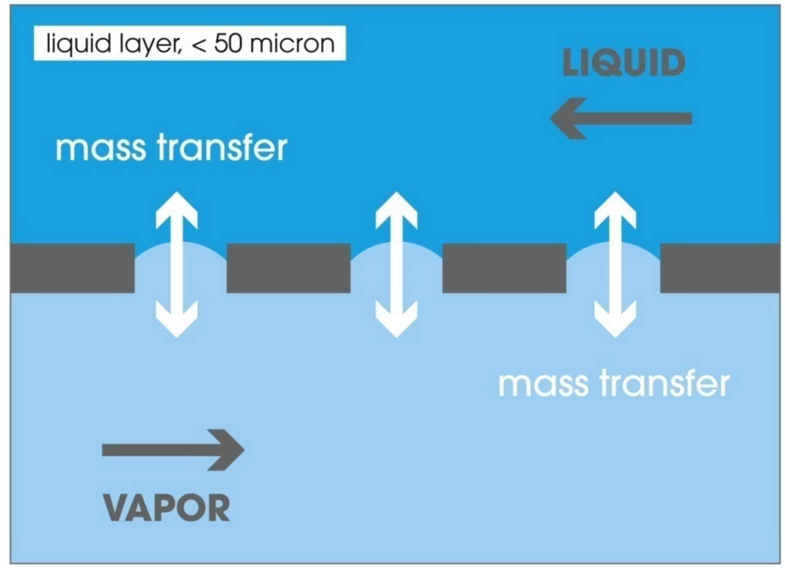
Proven Science

UNIQUE MICROSIEVE TECHNOLOGY

15 cm diameter

0,7 mm thick

High volume production



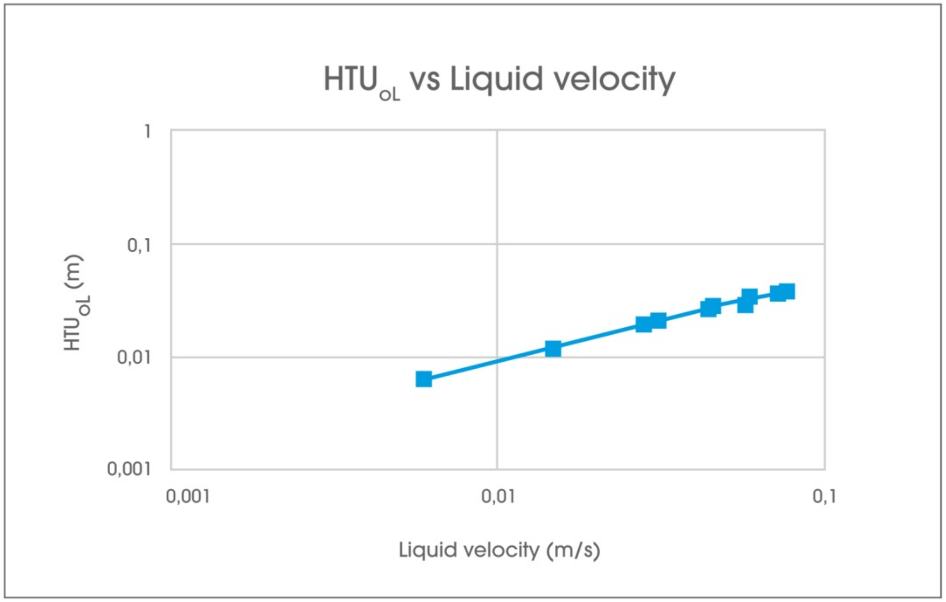
5 billion identical pores

0,45 micron diameter

1 micron membrane thickness

PROOF: ultra short HTU_{OL}

Stripping MTBE from water by nitrogen at ambient conditions was used as a test system for performance evaluation purposes. Inlet concentration of MTBE , different flow rates for liquid and gas were varied in a wide range. The best efficiency was achieved in all cases at lowest liquid flow rate, and tended to decrease progressively with increasing liquid flow rate, i.e. decreasing residence time. Gas flow rate exhibited practically no effect on separation, indicating that this system also can be considered as fully controlled by liquid side mass transfer resistance. Since the value of the overall liquid side based volumetric mass transfer coefficient was almost constant, the overall height of liquid side transfer unit (HTUoL) increased nearly proportionally and ranged 0.5-4 cm (!). The corresponding heights equivalent to a theoretical plate (HETP values) were 2 to 10 cm (!). These numbers indicate a high mass transfer efficiency, an order of magnitude above that experienced with common types and sizes of corrugated sheet structured packing in similar applications.



Energy Reduction Alternatives in the Fuel Ethanol Industry

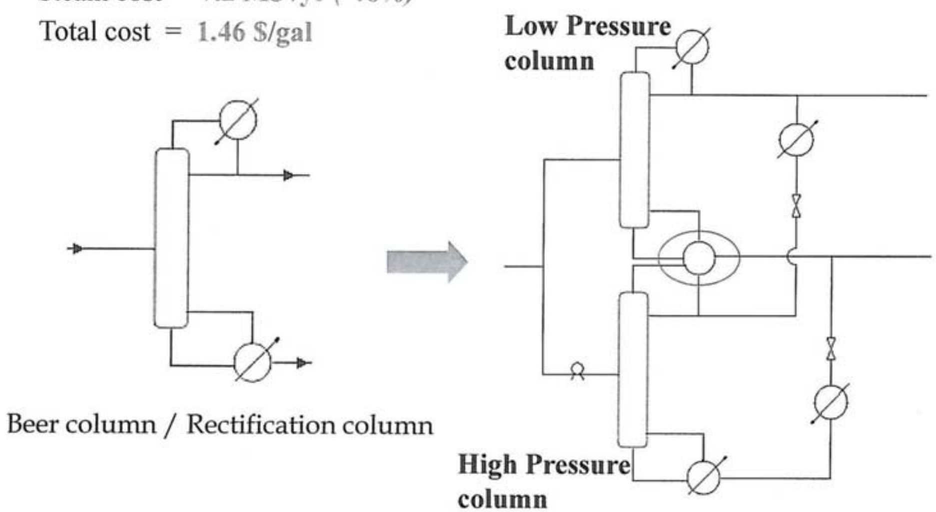
Use of Multieffect columns to replace the distillation columns Can reduce the energy costs by $.05/ gallon

Multi Effect Distillation

More Energy Reduction Alternatives

- Use of Multieffect columns to replace the distillation columns

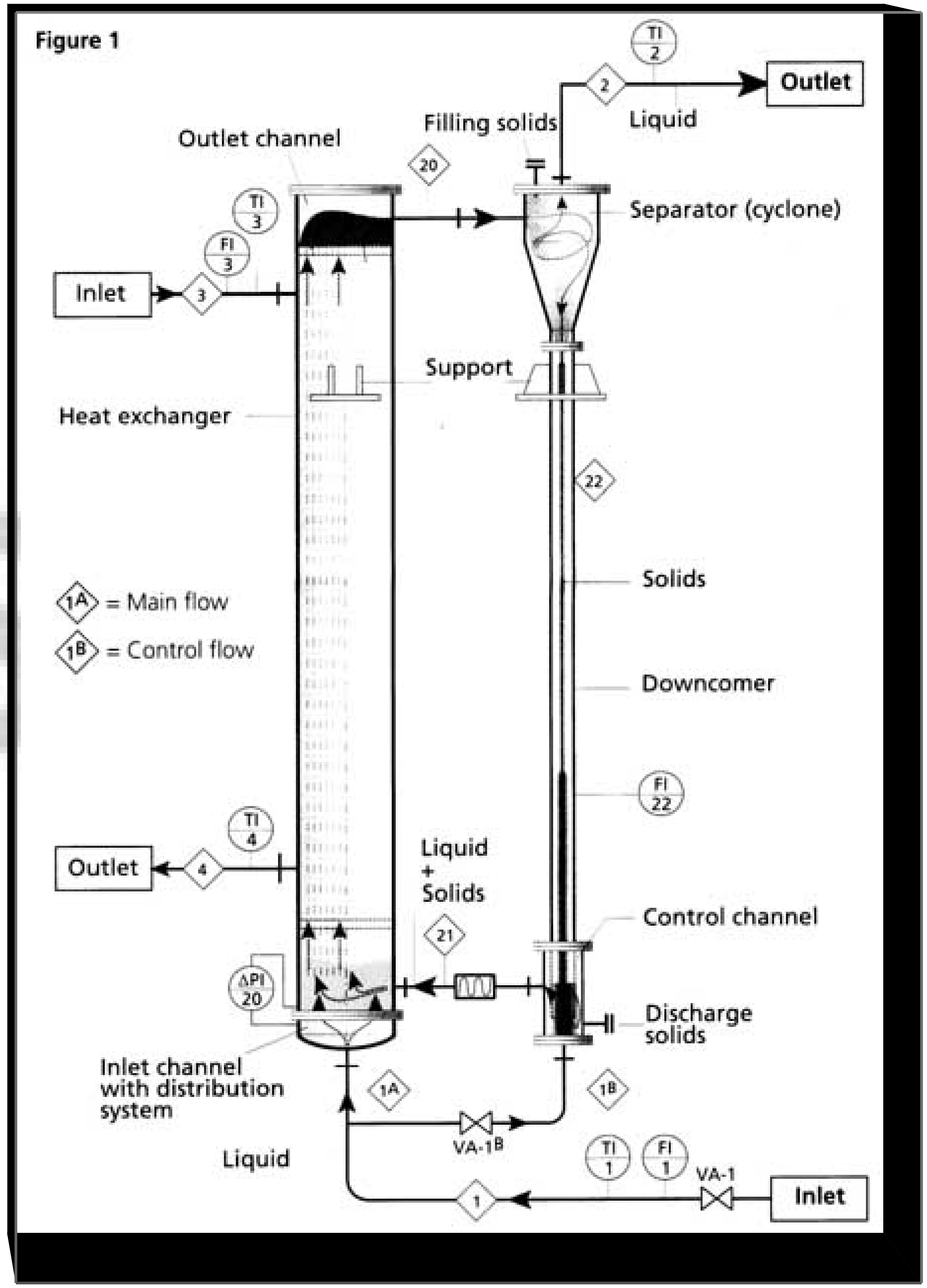
Total equipment cost = 47.5 M$
Steam cost = 7.2 M$ /yr *(-48%)*
Total cost = 1.46 $/gal



Heat Transfer

Klarex Technology

Self Cleaning
Heat Exchanger



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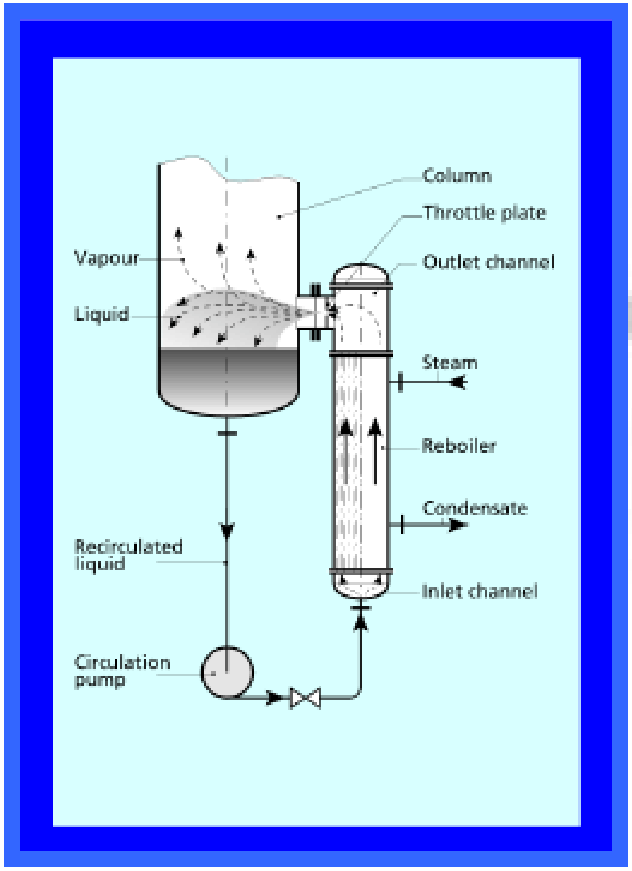
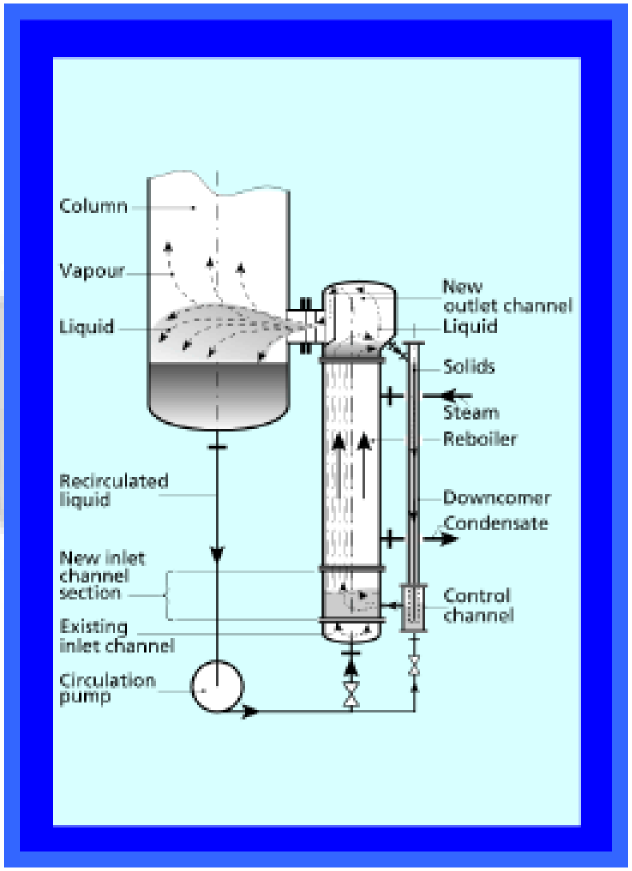
Modified Reboiler



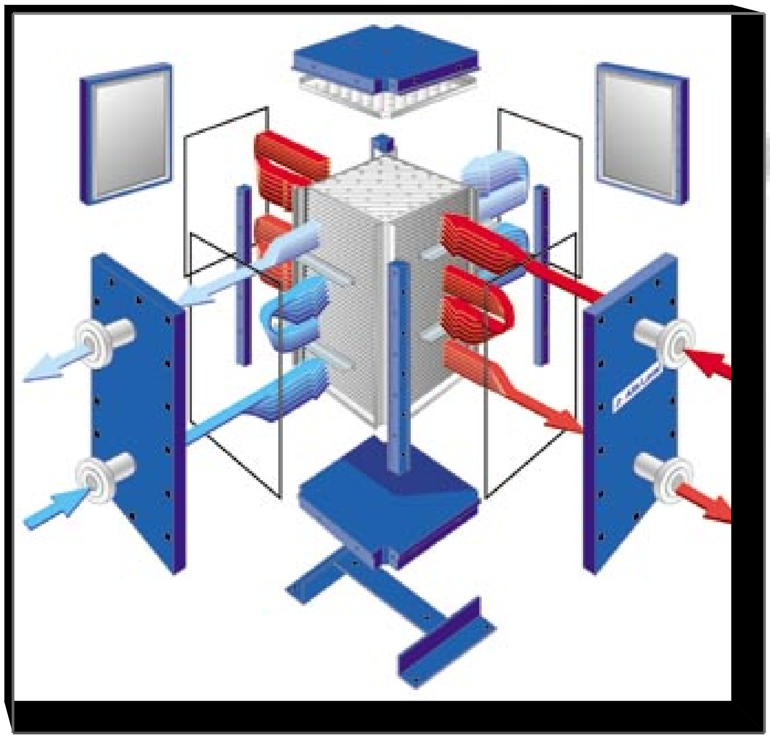


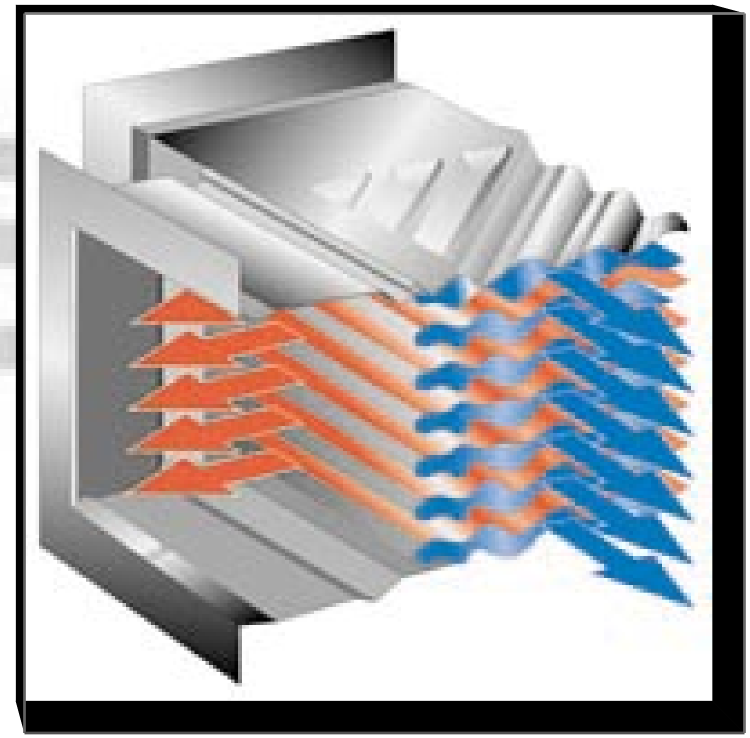
	Self Cleaning Heat Exchanger	Conventional Heat Exchanger
Heat Transfer Service	49,514 ft^2	258,334 ft^2
Pumping Power	1,126 hp	2,815 hp
Number of cleanings per year	0	12

Alfa Laval Compact Heat Exchangers

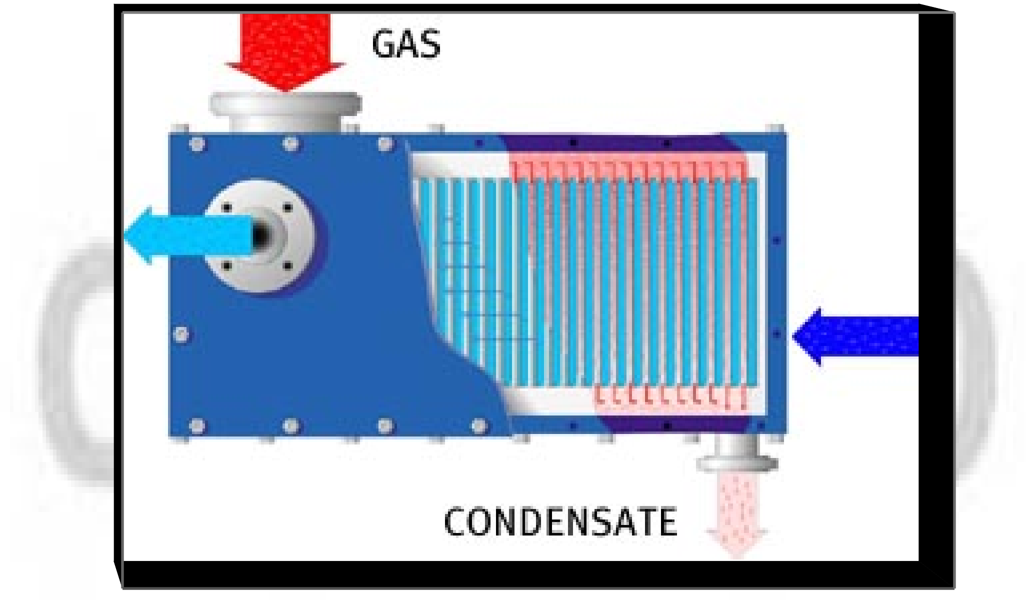
Cube Shaped Compact Heat Exchangers (CHEs) with high square foot areas per cubic foot volume

Cut Away Views





Condenser for Use with Inerts



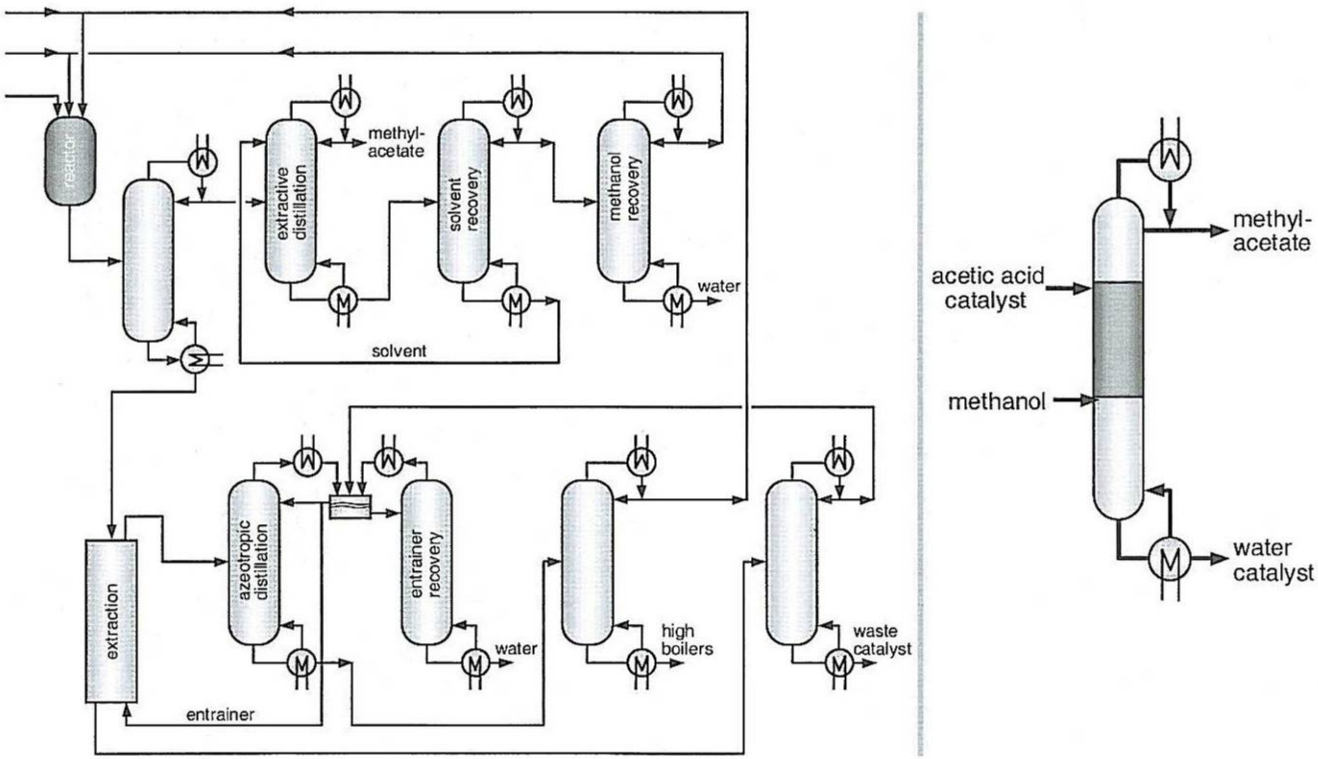
For duties with inerts, special pass configurations allow gas-liquid separation inside the Compabloc® eliminating the need for a separator vessel.

Multifunctional Reactors

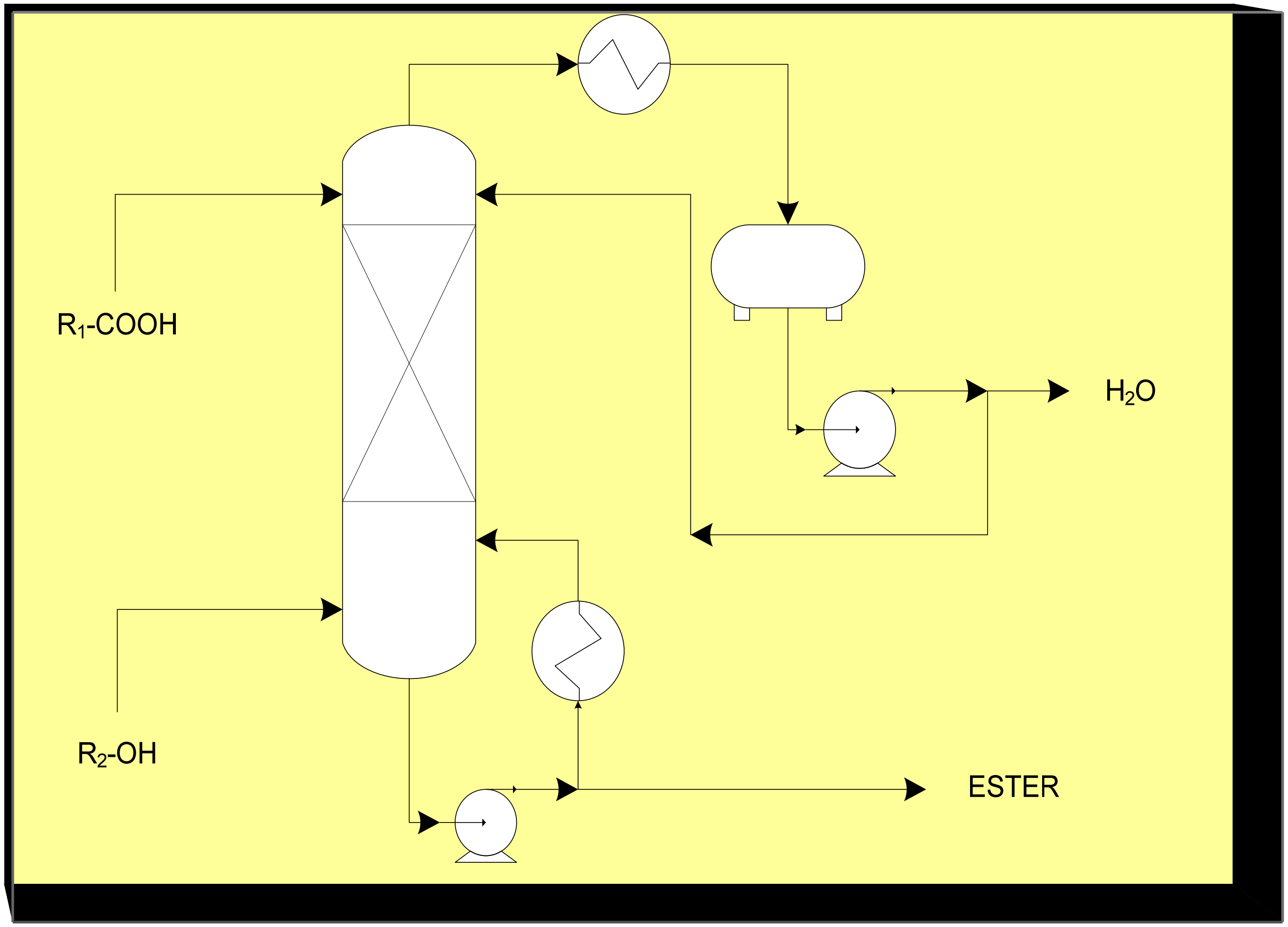
- **In House Technologies**
- **Commercially Available**

Reactive Distillation

Production of methyl acetate at Eastman Kodak: without / with reactive distillation



Reactive Distillation



R_1-COOH + R_2-OH → Ester + H_2O

R_2-OH is more volatile than R_1-COOH

Reactive Distillation Column Internals

- **Random packing**
- **Trays with high weirs**
- **Structured packing**

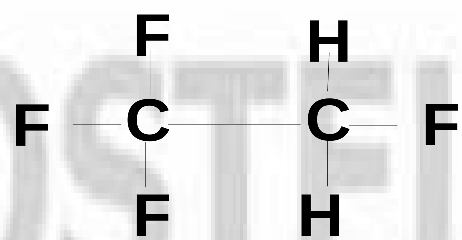
Reactive Distillation Catalysts

- On Trays Sewn into Pillows
- Coating on Random Packing
- Coating Structured Packing

Other Methods

TFE Extraction Technology

1,1,1,2 TETRAFLUORO ETHANE

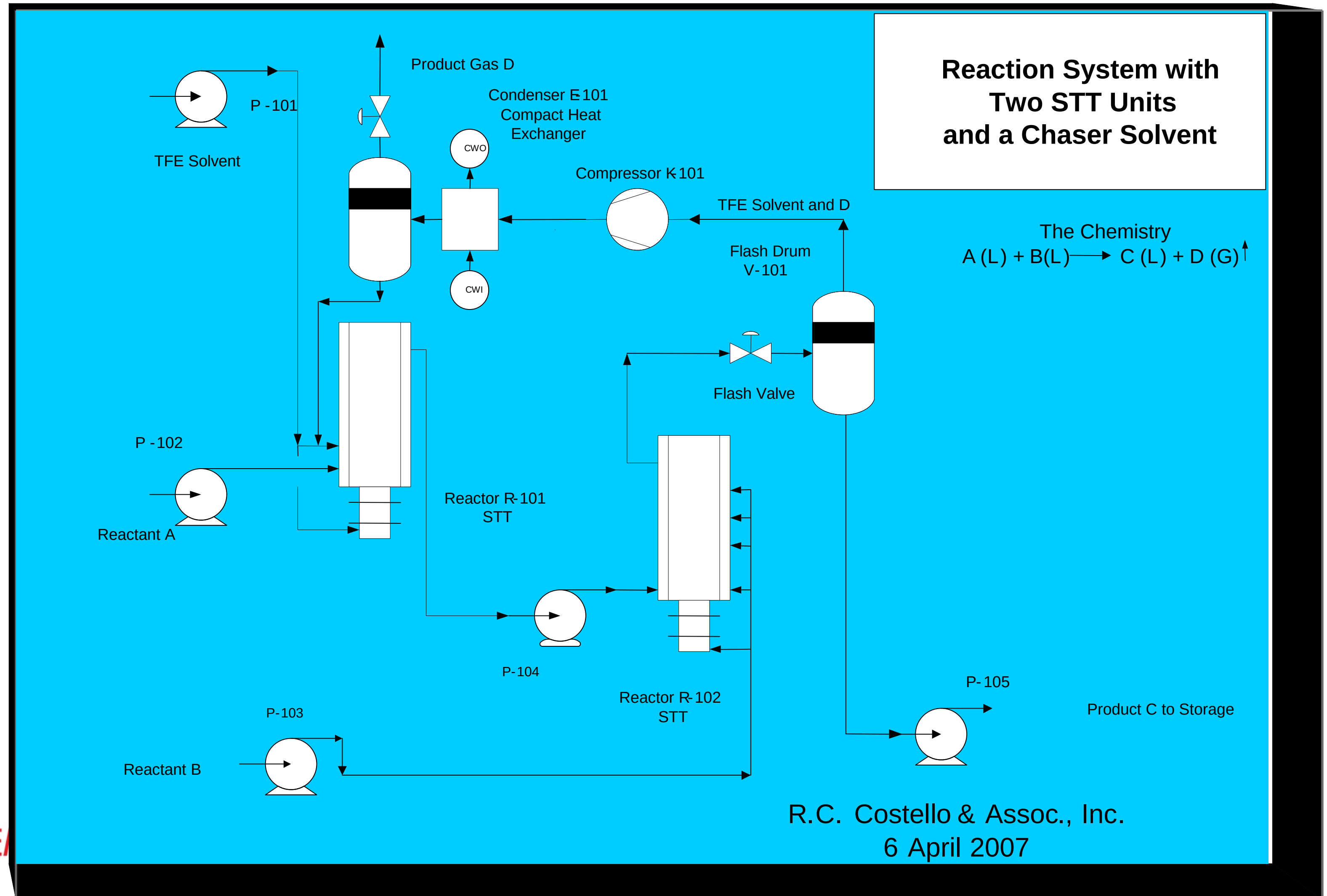


MW	**102**
BP	**-26.5 °C (-15.7°F)**
DENSITY	**1.2 @ 5 bar, 20 °C**
	(72.5 psia/ 68 °F)
SUPERCRITICAL POINT	**42 bar/98 °C**
	(608 psia/ 208.4 °F)

Advantages of 1,1,1,2 – Tetrafluoroethane & its Mixtures

- **ODORLESS & COLORLESS LIQUIDS**
- **LIQUIFY AT 5 BAR PRESSURE, AT AMBIENT TEMPERATURE**
- **RECYCLABLE**
- **LEAVE NO RESIDUES**
- **CHEMICALLY INERT**
- **NON FLAMMABLE**
- **NON CORROSIVE**
- **NON TOXIC**
- **NEUTRAL pH**
- **ENVIRONMENTALLY FRIENDLY**
- **APPROVED FOR USE IN FOOD PROCESSING & FDA FOR MDIs**

Continuous Reaction Systems



Continuous Reaction Systems With Nanoparticle Catalysts

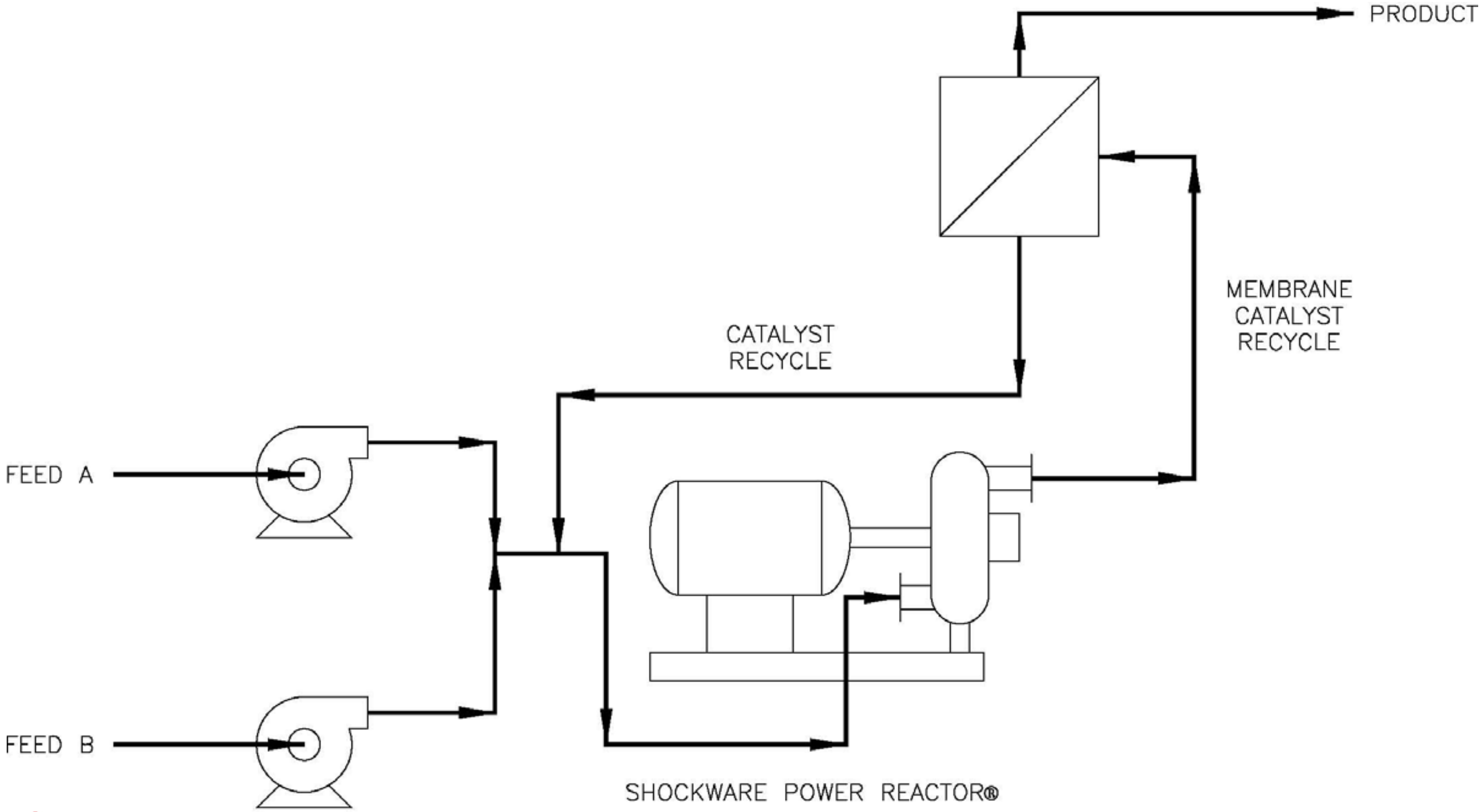
- A **nanoparticle** (or **nanopowder** or **nanocluster** or **nanocrystal**) is a microscopic particle with at least one dimension less than 100 nm.

- Filtration doesn't work for separation of **nanoparticles** from fluids. Bag filters are effective down to 0.5 microns. (1 micron = 1000 nm)

- Surface area of the material dominates the properties in lieu of the bulk properties. Think of a nanoparticle as a molecule.

- Surface area per mg of material is enormous and since catalysis is a surface phenomenon then nanoparticles are ideal

Continuous Reaction System With Nanoparticle Catalyst



CHEM|INNOVATIONS 2010 CONFERENCE & EXPO

Pall Filter

CHEM 2010 & EXPO

Pall Filter

•Two-step flue gas cleaning processes consisting of a particle filter with a subsequent low-dust SCR (Selective Catalytic Reduction) catalyst unit or of a high dust SCR catalyst unit

•The catalytic filter is a hot gas filter equipped with catalytic filter elements and designed for a catalyst operating temperature of 300°C. The catalytic filter elements are catalytically activated ceramic hot gas filter elements consisting of SiC, which provide a highly efficient particle separation by means of a fine filtering outer membrane.

•The catalytic activation of the elements is accomplished by impregnation of the 10 mm thick porous filter element wall with a SCR catalyst with the composition $TiO2$-V_2O_5-WO_3.

Why Intensify?

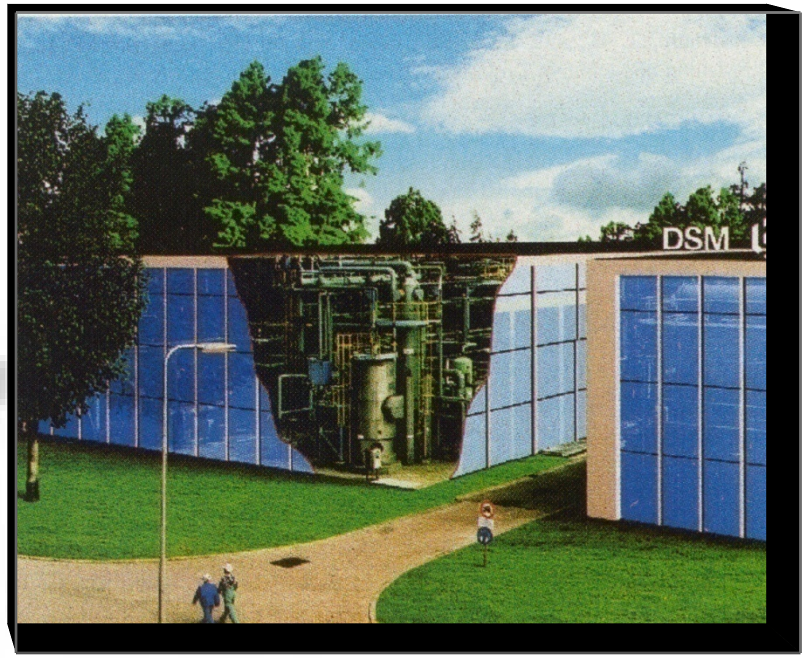
- ↓ Capital Costs
- ↓ Operating Costs
- ↓ Maintenance Costs
- ↓ Plant Footprint & Profile
- Facilitate scale up
- Provide basis for rapid development of products & processes (↓ Time Line)
- ↑ Safety
- ↓ Environmental Impact

What Processes Are Candidates for PI?

- New chemistries.
- Existing old plants where the number of unit ops can be reduced. If your considering a rebuild where a distillation step can be eliminated.
- Existing plants with a severe downtime issue.
- Market requires Improving purity of your product.

Before vs. After PI

Before



After

Source: Chemical Engineering Progress (CEP) Magazine, January 2000.