Multi-Scale Approaches for Process Synthesis and Intensification

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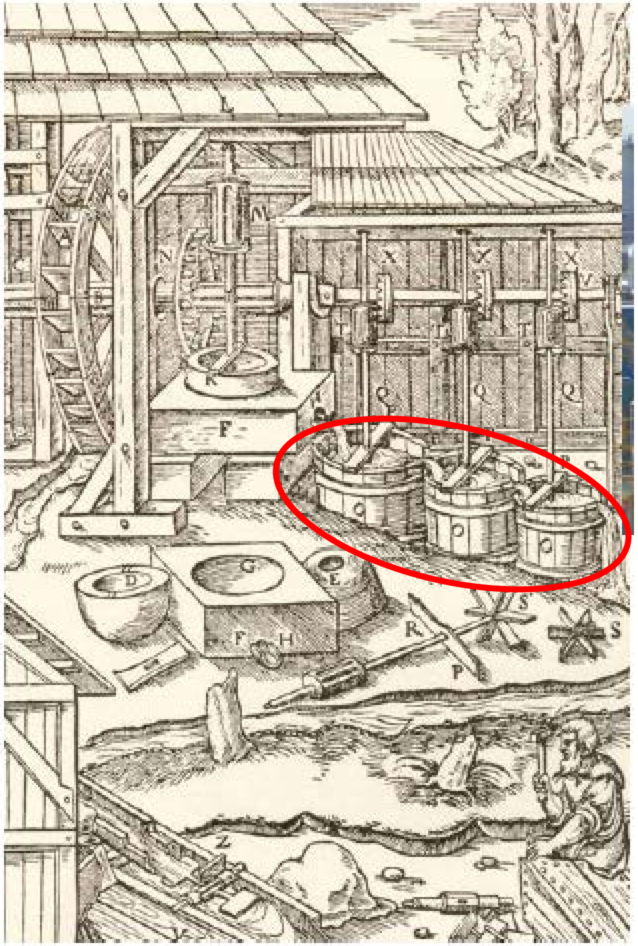
Motivating Questions



G. Agricola, *De Re Metallica*, 1556

Do we have innovative processes to make the many necessary products?

Chemical Process Industry, 2006

Is it possible to achieve improvements in the design of such processes?

COMPUTER AIDED FLOWSHEET DESIGN

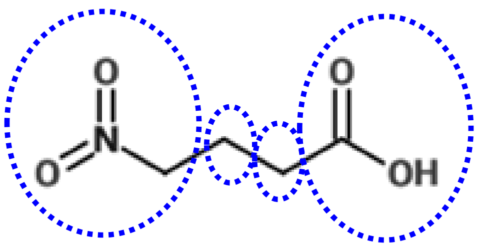
Flowsheet Design

- **Process Synthesis Methods**
  - Heuristic/expert systems
  - Optimization/algorithmic approaches

- **Computer Aided Flowsheet Design (CAFD)**
  - Combines physical insights with algorithmic reverse design approaches
  - Systematic identification of feasible flowsheets at significantly reduced computational expense
  - Based on process group (PG) contribution approach by d'Anterroches and Gani (2005)
  - Inspired by group contribution based methods for Computer Aided Molecular Design (CAMD)

Molecular vs. Process Groups

Molecule



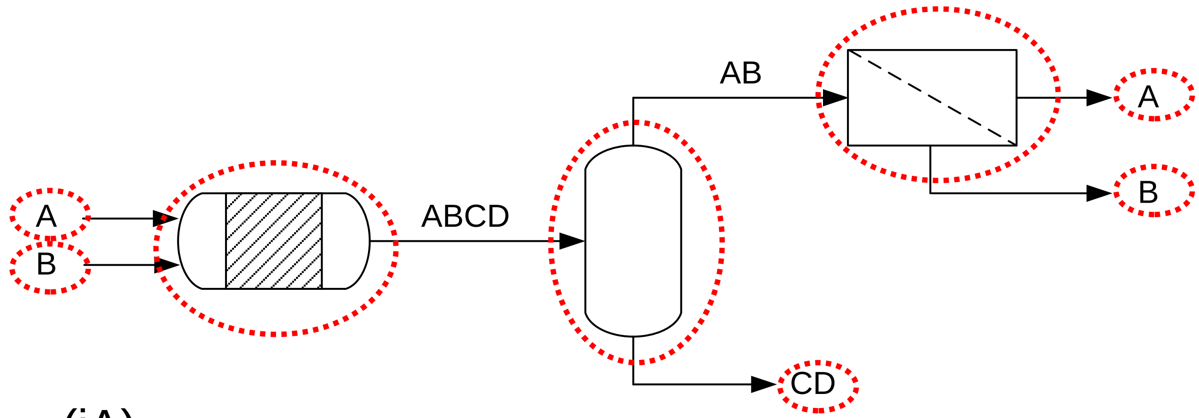
1 CH_2NO_2

2 CH_2

1 COOH

- Generated and represented by functional molecular groups (MGs)
- MGs characterized by atoms and molecular weight

Flowsheet



(iA)
(iB)

(rAB/pABCD)

(AB/CD)

(oCD)

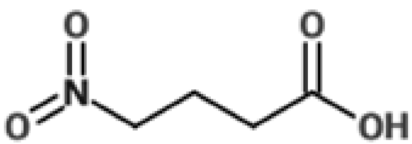
(mA/B)

(oA)
(oB)

- Generated and represented by functional process-groups (PGs)
- PGs characterized by type of unit operation and driving force
- PGs are mass balance independent
- Connections between PGs are component dependent

SMILES vs. SFILES

Molecule



1 CH_2NO_2

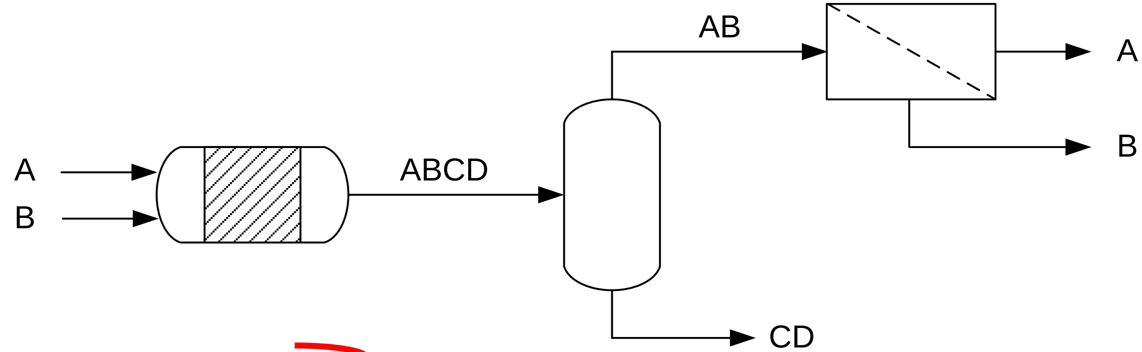
2 CH_2

1 COOH

Simplified
Molecular
Input
Line
Entry
Specification

O=N(=O)CCCC(=O)O

Flowsheet



(iA)
(iB)

(rAB/pABCD)

(AB/CD)

(oCD)

(mA/B)

(oA)
(oB)

Simplified
Flowsheet
Input
Line
Entry
Specification

(iA)(rAB/pABCD)[<(iB)](AB/CD)[oCD)](mA/B)[(oA)](oB)

CAFD Framework

Problem Definition
- Raw materials and desired product specifications
- Identify optimal flowsheet structure and design parameters
- Performance criteria (energy consumption, amount of external agents used, cost, etc.)

Problem Analysis
- Identify process type (reaction/no reaction) and phases (vapor, liquid, and/or solids)
- Determine number of tasks to be performed
- Select process groups to be used

Flowsheet Synthesis
- Initialization of process groups (PGs)
- Generation of feasible flowsheets and corresponding SFILES representation
- Ranking of alternatives using flowsheet property models

Reverse Simulation
- Determine optimal values of design variables for each operation in feasible flowsheet
- Separation related PGs characterized by driving force
- Reaction related PGs characterized by highest attainable reaction point

Verification
- Verify process configuration and operating conditions
- Rigorous simulation or experiments

Example: Flowsheet Design 1:8

- **HDA Process**
  - Raw materials at STP:
    - Hydrogen (95 kmol/hr)
    - Toluene (50 kmol/hr)
    - Methane (5 kmol/hr)
  - Products:
    - Benzene
    - Biphenyl
  - Reactions:
    - $C_6H_5CH_3 + H_2 \rightarrow C_6H_6 + CH_4$ (Conversion: 75%)
    - $2\ C_6H_6 \rightarrow C_6H_5{-}C_6H_5 + H_2$ (Conversion: 5%)

Example: Flowsheet Design 2:8

- **Mixture Analysis**
  - 10 binary pairs
  - No azeotropes found

- **Binary Property Ratios**

Property	Hy-Meth	Hy-Ben	Hy-Tol	Hy-Bi	Me-Ben	Me-Tol	Me-Bi	Be-Tol	Be-Bi	Tol-Bi
bp	5.48	17.32	18.82	25.9	3.16	3.44	4.73	1.09	1.5	1.38
Rad of Gy	3.01	8.1	9.36	13.03	2.69	3.11	4.32	1.16	1.61	1.39
mp	6.5	19.98	12.77	24.54	3.07	1.96	3.78	1.56	1.23	1.92
mv	1.33	3.13	3.73	5.44	2.36	2.81	4.09	1.19	1.74	1.46
Solub Par	1.74	2.82	2.76	2.9	1.61	1.58	1.66	1.02	1.03	1.05
V.d.W Vol	2.7	7.66	9.42	14.51	2.84	3.49	5.38	1.23	1.89	1.54
V.d.w Ar	2.01	4.2	5.19	7.45	2.08	2.58	3.7	1.24	1.78	1.44

Example: Flowsheet Design 3:8

- **Separation Techniques and Process Groups**
  - Identified using rules by Jaksland *et al.* (1995)

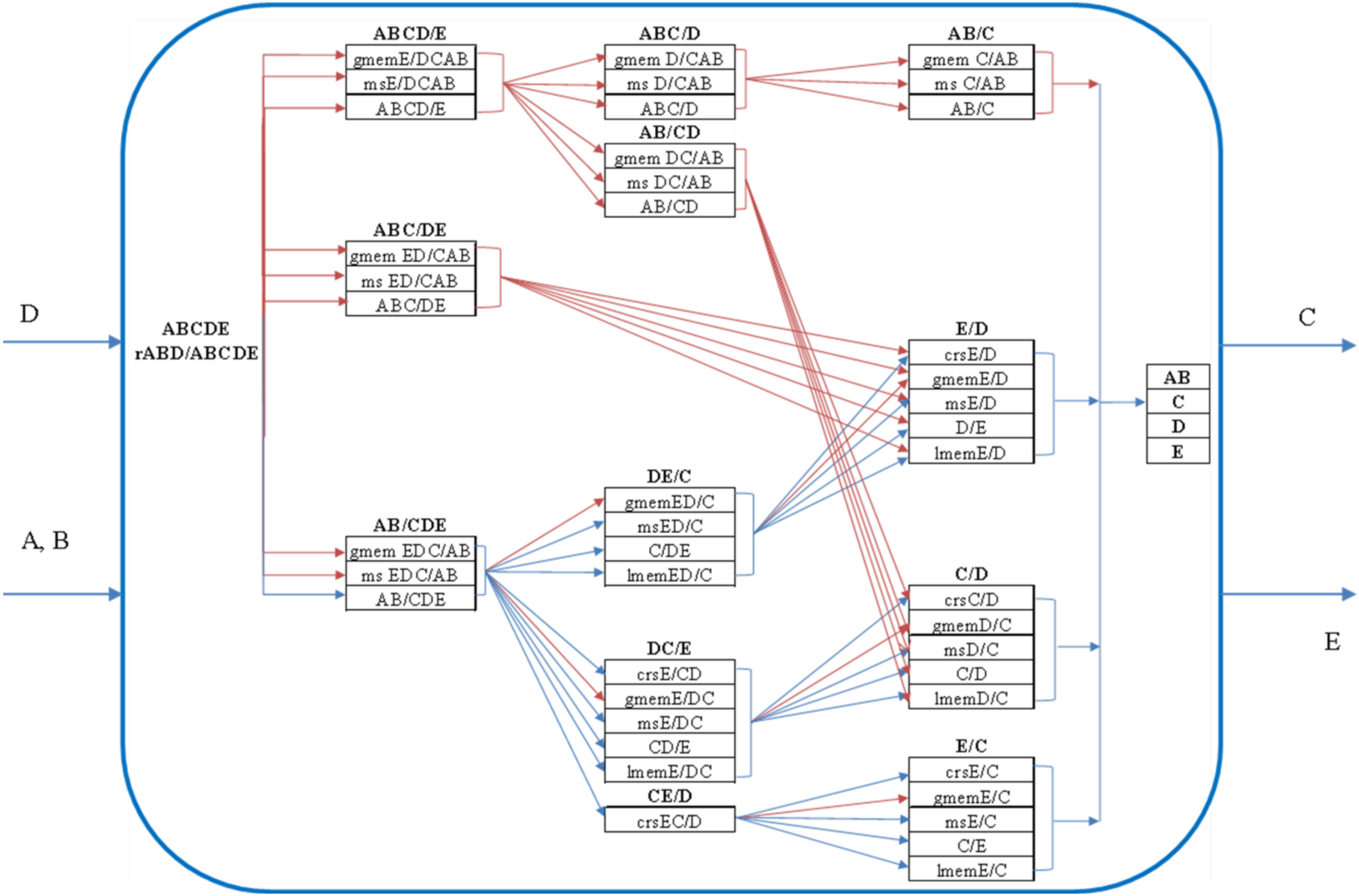
Sepration Techniques			
gmemE/DCAB	gmem D/CAB	gmem C/AB	crs E/D
ms E/DCAB	ms D/CAB	ms C/AB	gmemE/D
ABCD/E	ABC/D	AB/C	ms E/D
gmem ED/CAB	gmem DC/AB	gmemED/C	D/E
ms ED/CAB	ms DC/AB	ms ED/C	lmemE/D
AB/CD	C/DE	crs C/D	gmemD/C
crs E/C	gmemE/C	ms E/C	C/D
ABC/DE	gmem EDC/AB	ms EDC/AB	AB/CDE
crs E/CD	gmemE/DC	ms E/DC	lmemE/DC
lmemE/C	crs EC/D	lmemED/C	ms D/C
lmemD/C	C/E		

Legends
gmem- gas separation membranes
lmem - liquid separation membranes
ms - molecular sieve separation
crs- crystalization
distillation
A - Hydrogen
B-Methane
C-Benzene
D-Toluene
E-Biphenyl

Example: Flowsheet Design 4:8

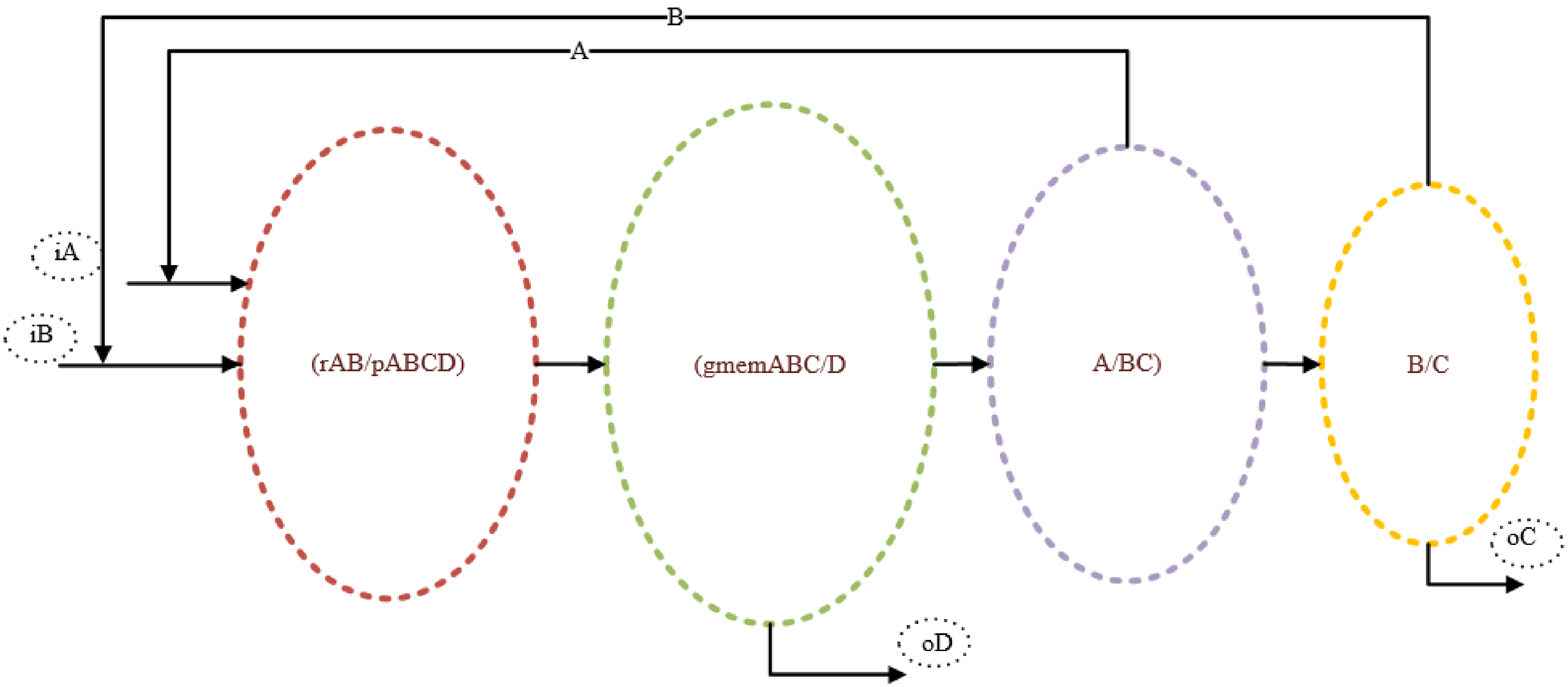
- **Generation of Flowsheets**
  - Total number of PGs: 42 + 2 inlet streams + 1 reactor
  - Total number of combinations: (42!)/(42-3)! = 68880
  - Infeasible combinations discarded by structural optimization
  - Total feasible flowsheet combinations: 271

Example: Flowsheet Design 5:8



Example: Flowsheet Design 6:8

- **Generation of SFILES**



(iA)(rAB/pABCD)<1<2[<(iB)](gmemABC/D)[(oD)](A/BC)1(B/C)2(oC)

Example: Flowsheet Design 6:8

- ## Generation of SFILES

Sno	SFILES
1	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](C/DE)[(oC)](crsE/D)[(oD)2](oE)
2	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](C/DE)[(oC)](D/E)[(oD)2](oE)
3	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](C/DE)[(oC)](msE/D)[(oD)2](oE)
4	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](lmemED/C)[(oC)](crsE/D)[(oD)2](oE)
5	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](C/DE)[(oC)](lmemE/D)[(oD)2](oE)
6	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](lmemED/C)[(oC)](D/E)[(oD)2](oE)
7	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](msED/C)[(oC)](crsE/D)[(oD)2](oE)
8	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](lmemED/C)[(oC)](msE/D)[(oD)2](oE)
9	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](lmemED/C)[(oC)](lmemE/D)[(oD)2](oE)
..	………………………………………………………………..
263	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](msED/C)[(oC)](msE/D)[(oD)2](oE)
264	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](msED/C)[(oC)](lmemE/D)[(oD)2](oE)
265	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](crsE/CD)[(oE)](C/D)[(oD)2](oC)
266	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](CD/E)[(oE)](C/D)[(oD)2](oC)
267	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](crsE/CD)[(oE)](lmemD/C)[(oD)2](oC)
268	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](crsE/CD)[(oE)](msD/C)[(oD)2](oC)
269	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](CD/E)[(oE)](lmemD/C)[(oD)2](oC)
270	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](lmemE/DC)[(oE)](C/D)[(oD)2](oC)
271	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)1](msE/DC)[(oE)](C/D)[(oD)2](oC)

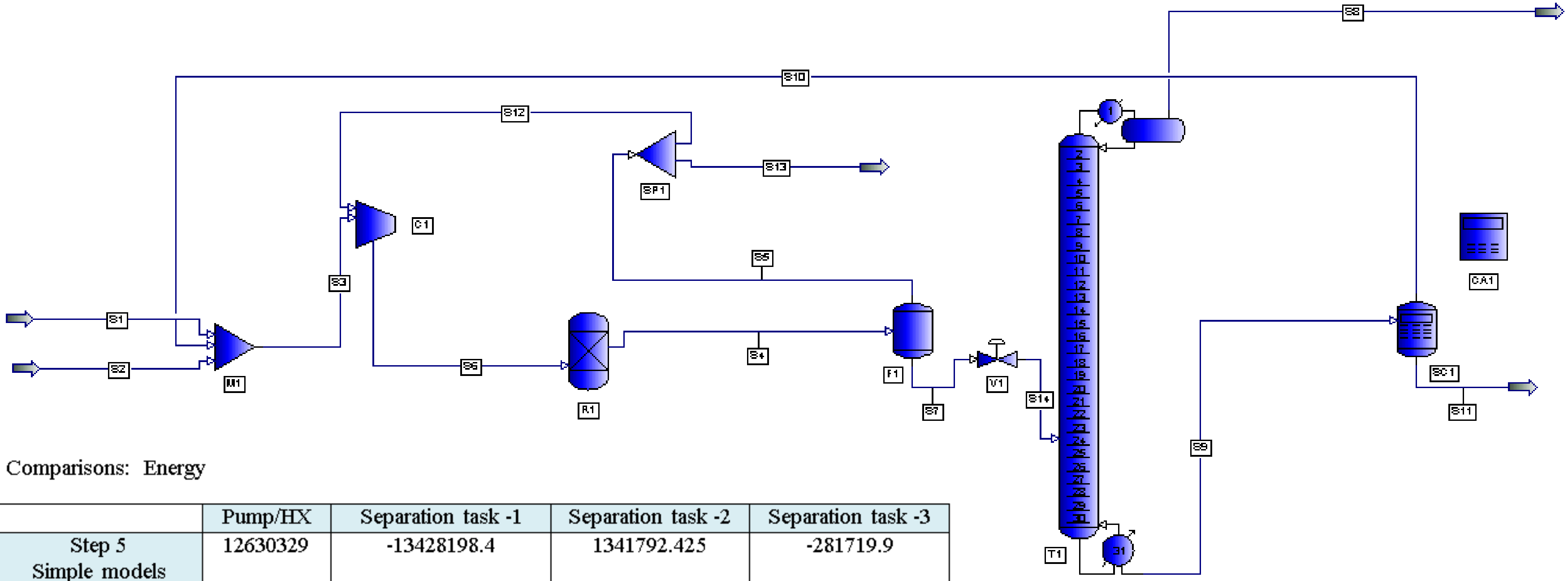
Example: Flowsheet Design 7:8

- **Ranking of Flowsheets**
  - Option #2 is the standard industrial process for producing benzene from toluene by the HDA process
  - Option #1 can be seen to perform better than the industry standard
    - Same product purity
    - Higher atom efficiency
    - Increased recovery
    - Reduced energy consumption

Ranking	Process Alternatives (SFILES)	Energy	Atom efficency	Purity	Recovery
1	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)]1(C/DE)[(oC)](crsE/D)[(oD)2](oE)	2.71E+07	81.57	99.95	49.92
2	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)]1(C/DE)[(oC)](D/E)[(oD)2](oE)	2.76E+07	81.52	99.95	49.90
3	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)]1(C/DE)[(oC)](msE/D)[(oD)2](oE)	2.69E+07	79.90	99.95	48.90
4	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)]1(lmemED/C)[(oC)](crsE/D)[(oD)2](oE)	2.69E+07	80.22	98.29	49.10
5	(iAB)(rAD/ABCDE)<1<2[(<iD)](AB/CDE)[(oAB)]1(C/DE)[(oC)](lmemE/D)[(oD)2](oE)	2.78E+07	80.73	99.95	49.41

Example: Flowsheet Design 8:8

- **Flowsheet Verification**



Comparisons: Energy

	Pump/HX	Separation task -1	Separation task -2	Separation task -3
Step 5 Simple models	12630329	-13428198.4	1341792.425	-281719.9
Step 6&7 Rigorous simulation	13709044.4	-13617700	1831200	-135630

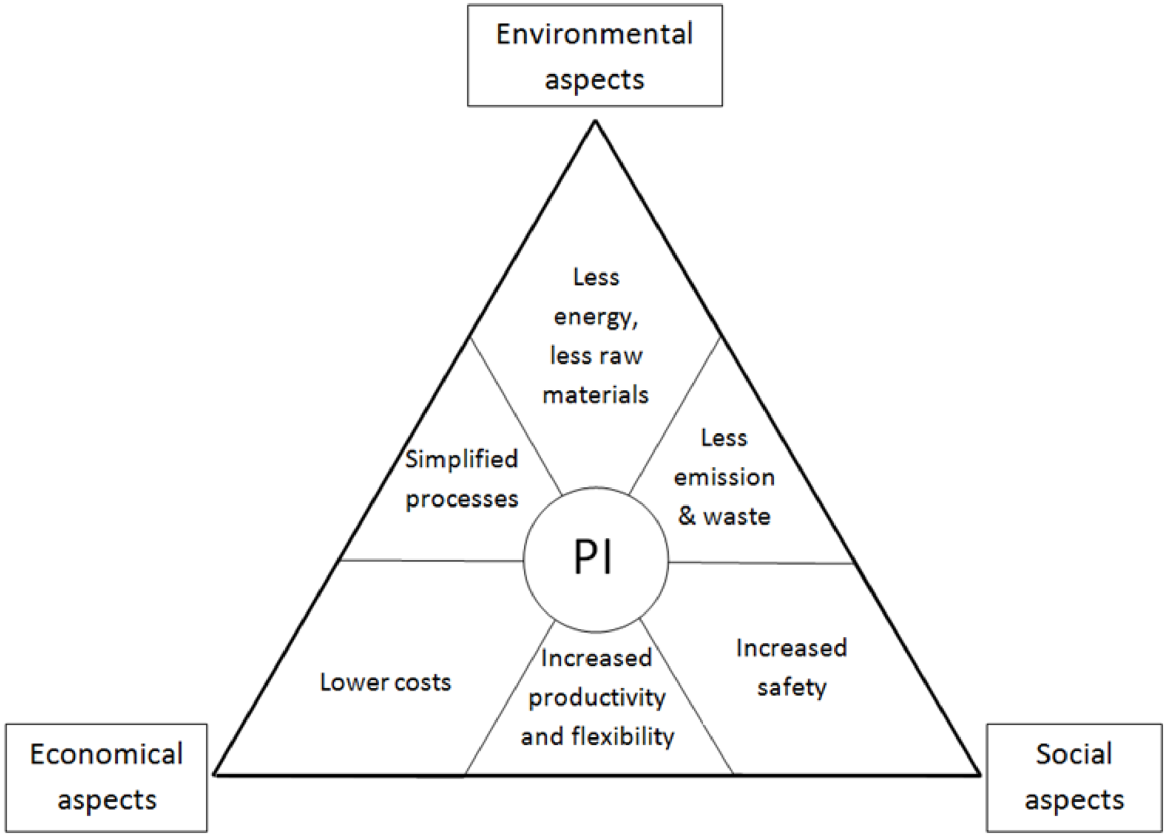
Recovery and purity

	Benzene Production (kmol/hr)	Bip Production (kmol/hr)
Step 5 Simple models	49.56	1.3175
Step 6&7 Rigorous simulation	46.975	1.334

PHENOMENA BASED PROCESS DESIGN & INTENSIFICATION

Process Intensification

- **Integration of unit operations**
- **Integration of functions**
- **Integration of phenomena**
- **Targeted enhancement of phenomenon in operation**



Lutze, Gani & Woodley, Chem Eng Process 49, 547-558, (2010)

Successful Applications of PI

PI Principle	PI technology	Case	Improvements
1) Integration of unit operations	Reactive distillation	Methyl-acetate[2]	↑ conversion, purification, profit ↓ process steps, energy, costs
2) Integration of functions	HEX reactor	Hydroformylation of cyclo-dodecatriene[3]	↑ heat and mass transfer, ↑ selectivity, productivity, ↓ residence time
3) Integration of phenomena	Oscillatory flow reactor	Ester hydrolysis[4]	↑ yield ↓ size, residence time
4) Targeted enhancement of a phenomenon	Micro-structured catalytic wall reactors	Phthalic anhydride from o-xylene[5]	↑ conversion (≈100%) ↓ size, process steps

[2] Sundmacher & Kienle: Reactive Distillation: Status and Future Directions, Wiley-VCH (2003).

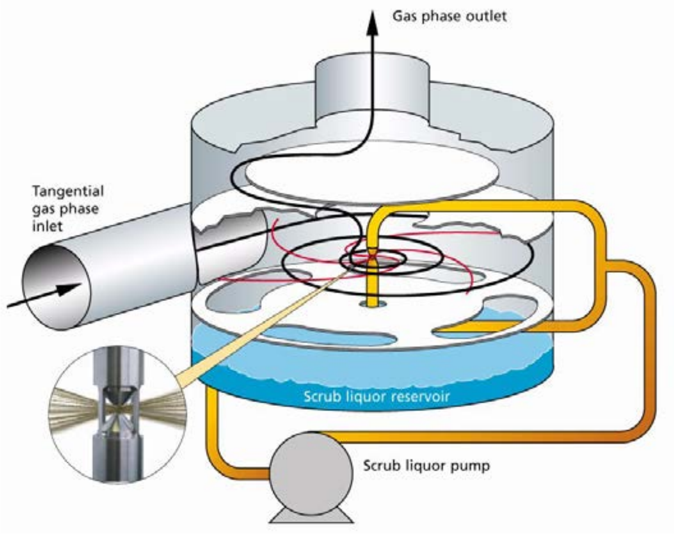
[3] Enache et al., Catalysis Today 128(1-2), 18-25 (2007).

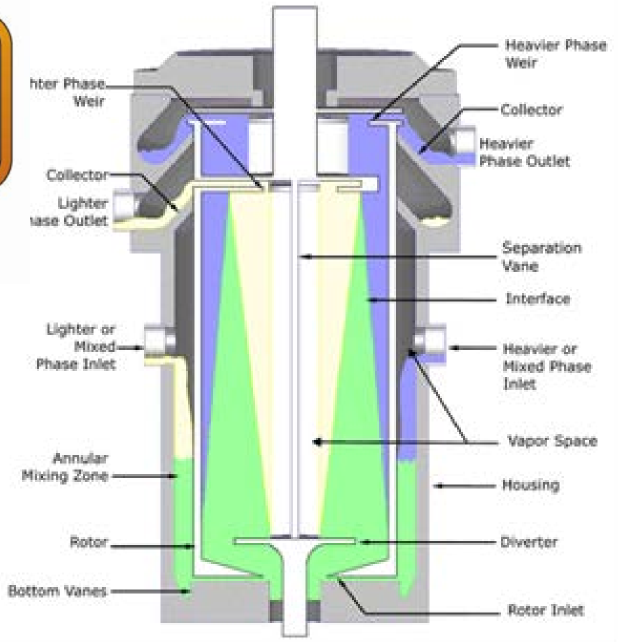
[4] Anxionnaz et al., Chem Eng Process 47(12), 2029-2050 (2008).

[5] Becht et al., Chem Eng Process 48(1), 329-332 (2009).

Examples of PI Equipment

They are mutants with special powers (functions)!





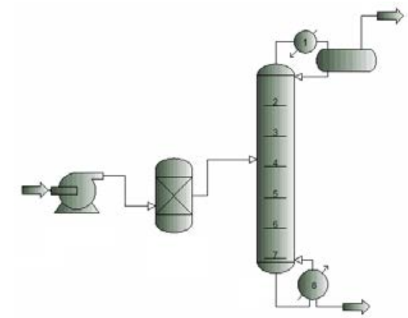
Different Scales for New Units

1. How, when and where to intensify a process using existing PI technologies for the needed improvement?

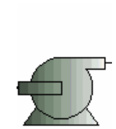
2. How to systematically design new (tailor-made) PI equipment for processes needing improvement?

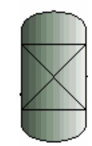
3. Decide on the building block.

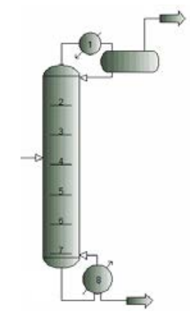
Processes



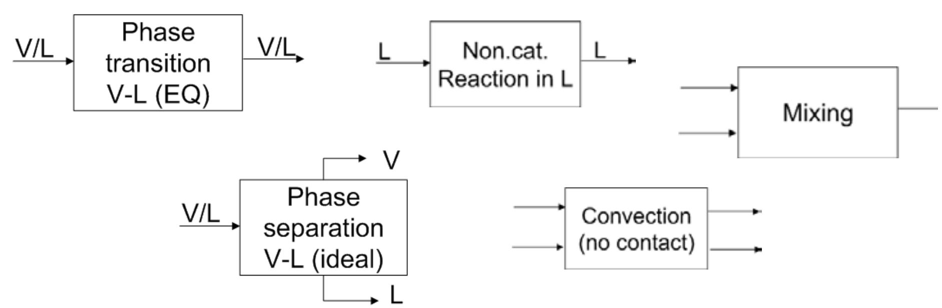
Unit operations







Phenomena



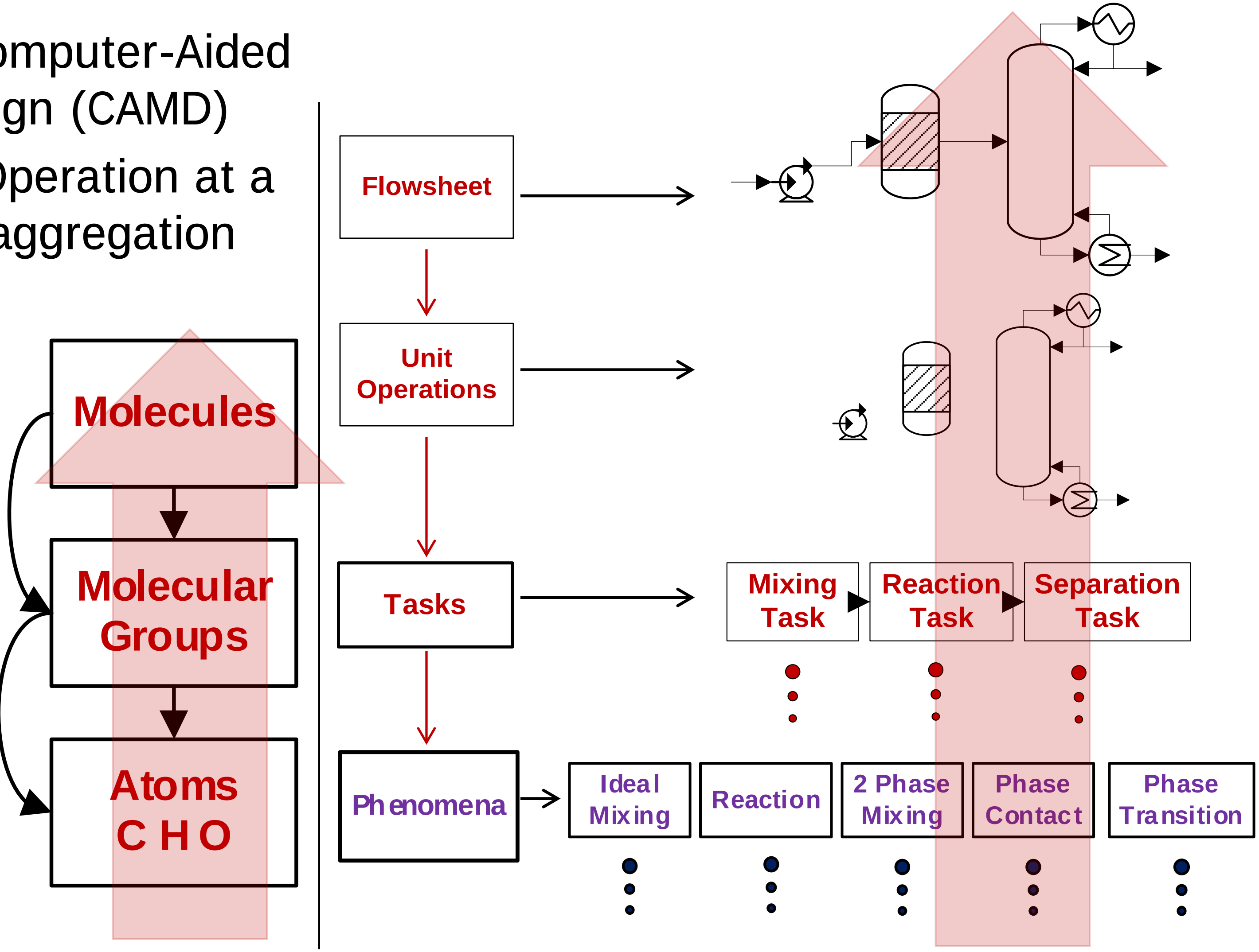
Molecular vs. Flowsheet Design

Comparison to Computer-Aided Molecular Design (CAMD)

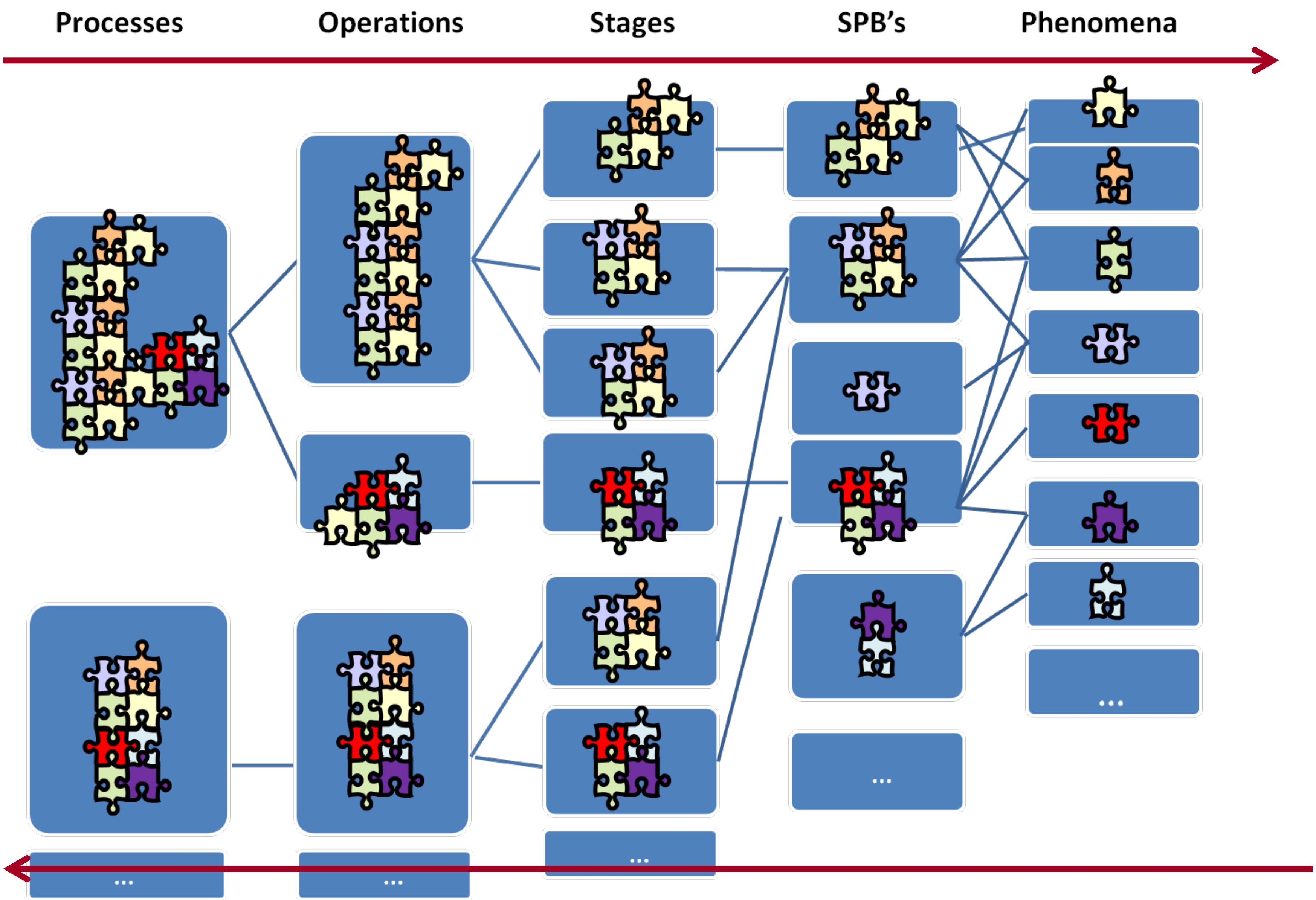
Key concept: Operation at a lower level of aggregation

Lower Level of Aggregation

Next Lower Level of Aggregation



Flowsheet to Phenomena



Phenomena Building Blocks

Most chemical processes can be represented by different combinations of the following 9 phenomena referred to as phenomena building blocks (**PBBs**) *just as atoms are the building blocks of groups*

Mixing (M)

Two phase mixing (2phM)

Phase Contact (PC)

Phase Transition (PT)

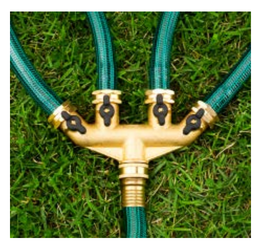
Phase Separation (PS)

Heating (H)

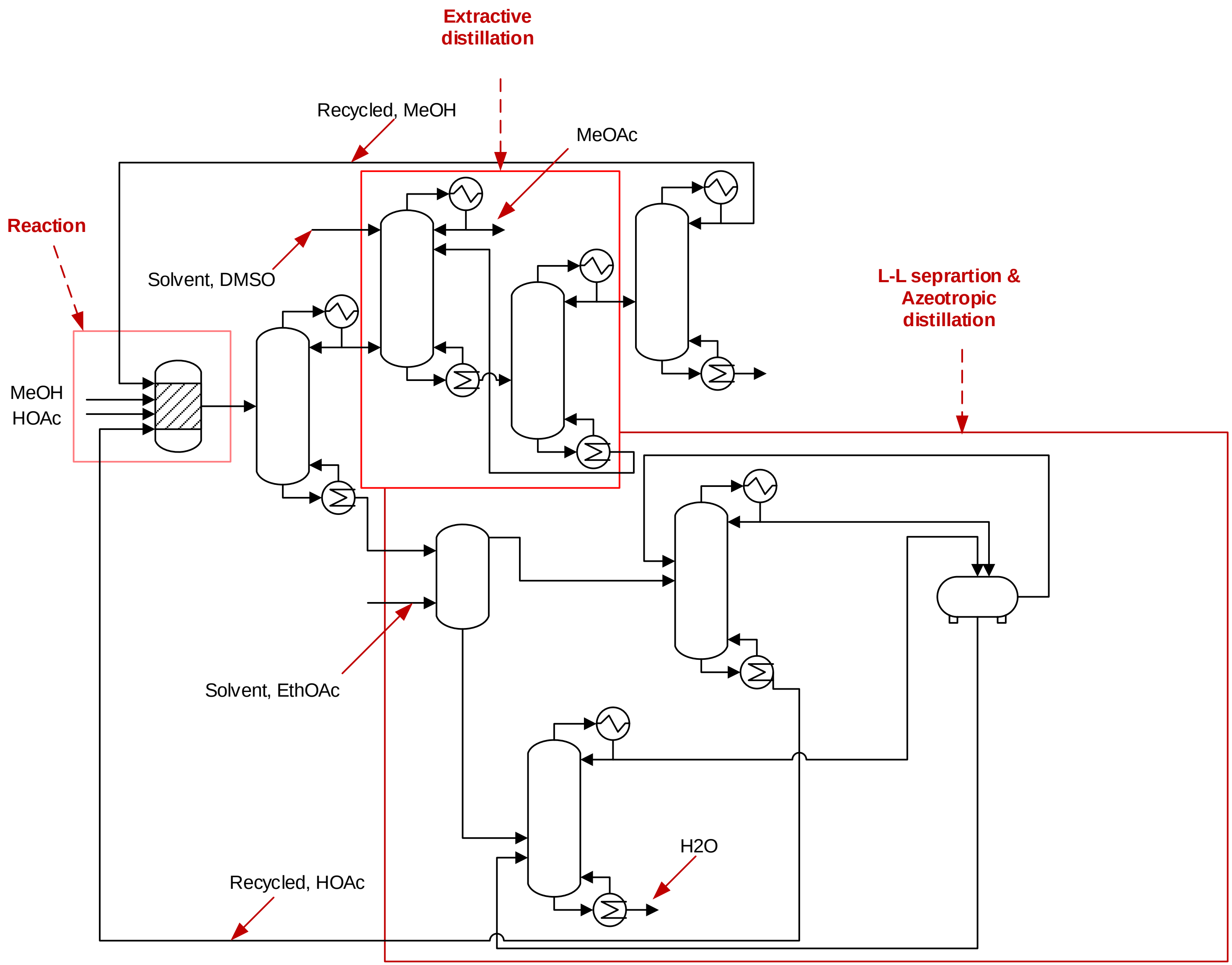
Cooling (C)

Reaction (R)

Dividing (D)



Example: MeOAc Production

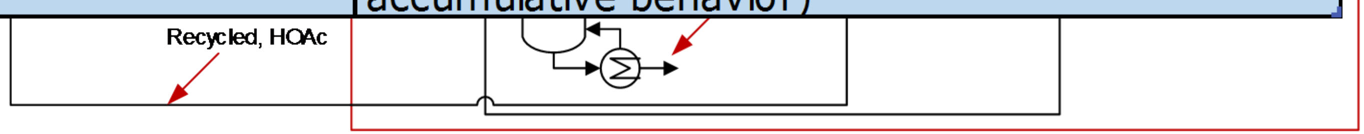


J. J Siirola (2011)

Example: MeOAc Production

Extractive distillation

Compounds Present	Bottlenecks
HOAc	**Reaction conditions** (high energy consumption)
	Recovery + recycle (high energy consumption)
MeOH	**Reaction conditions** (high energy consumption)
	Recovery + recycle (high energy consumption)
	MeOH in the product stream (high material loss)
MeOAc	-
H_2O	**Removal** (Highest energy consumption)
DMSO (solvent)	**High amount used** (recovery → high energy consumption)
EtOAc (solvent)	**Inefficient separations** (material loss + accumulative behavior)

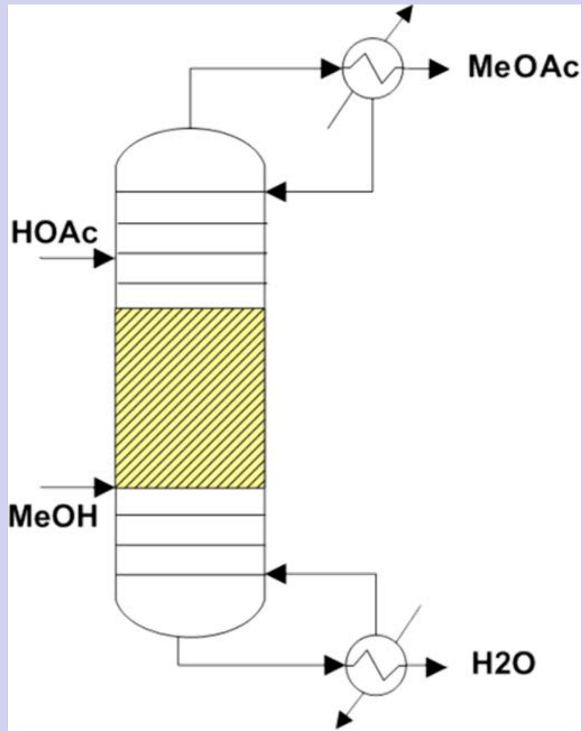


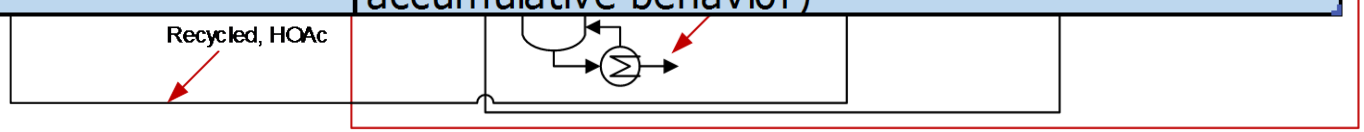
J. J Siirola (2011)

Example: MeOAc Production

Extractive distillation

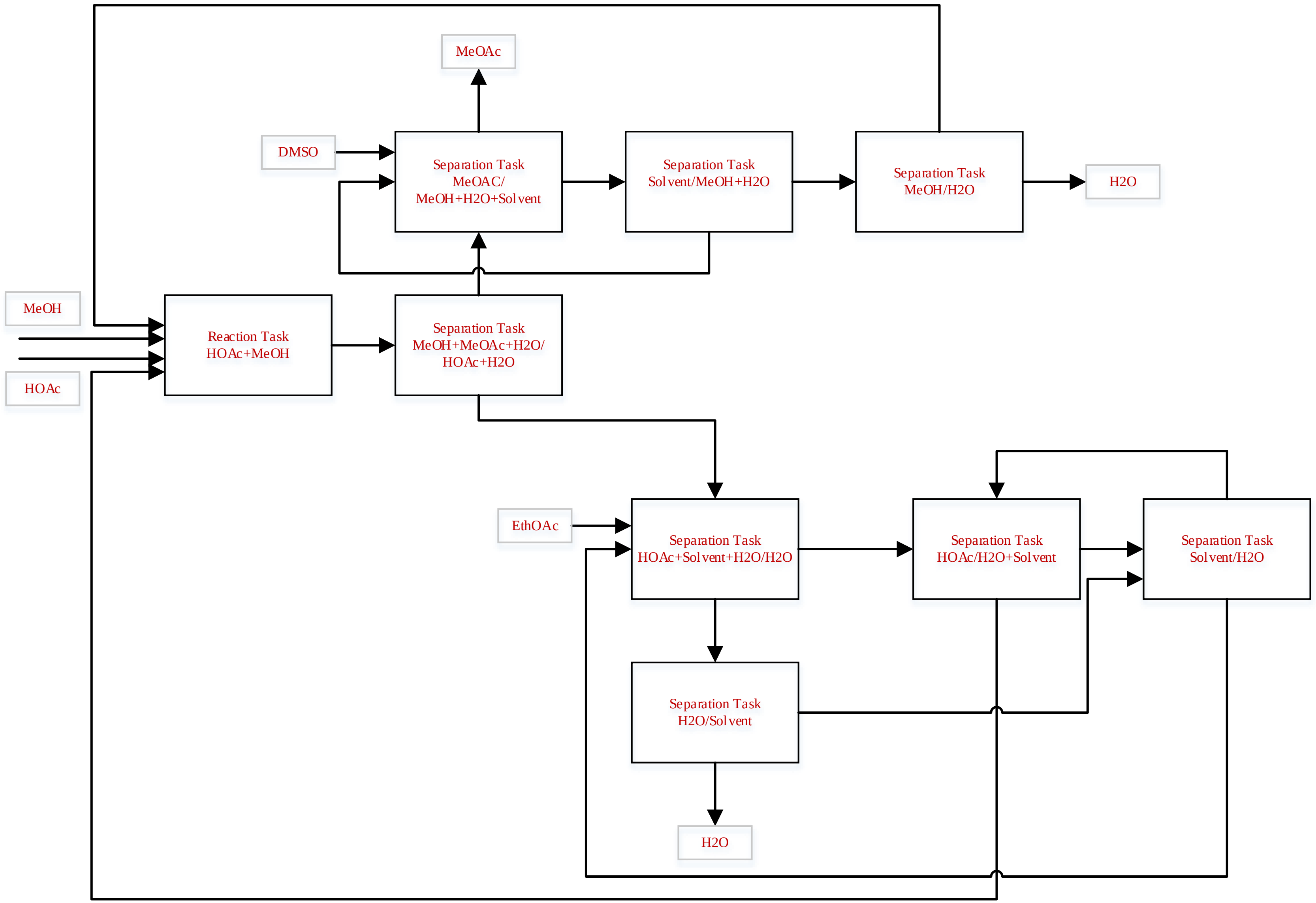
Compounds Present	Bottlenecks
HOAc	…gy consumption)
	…gy consumption)
MeOH	…gy consumption)
	…gy consumption)
	…high material
MeOAc	
H2O	…umption)
DMSO (solvent)	… high energy
EtOAc (solvent)	…al loss + accumulative behavior)



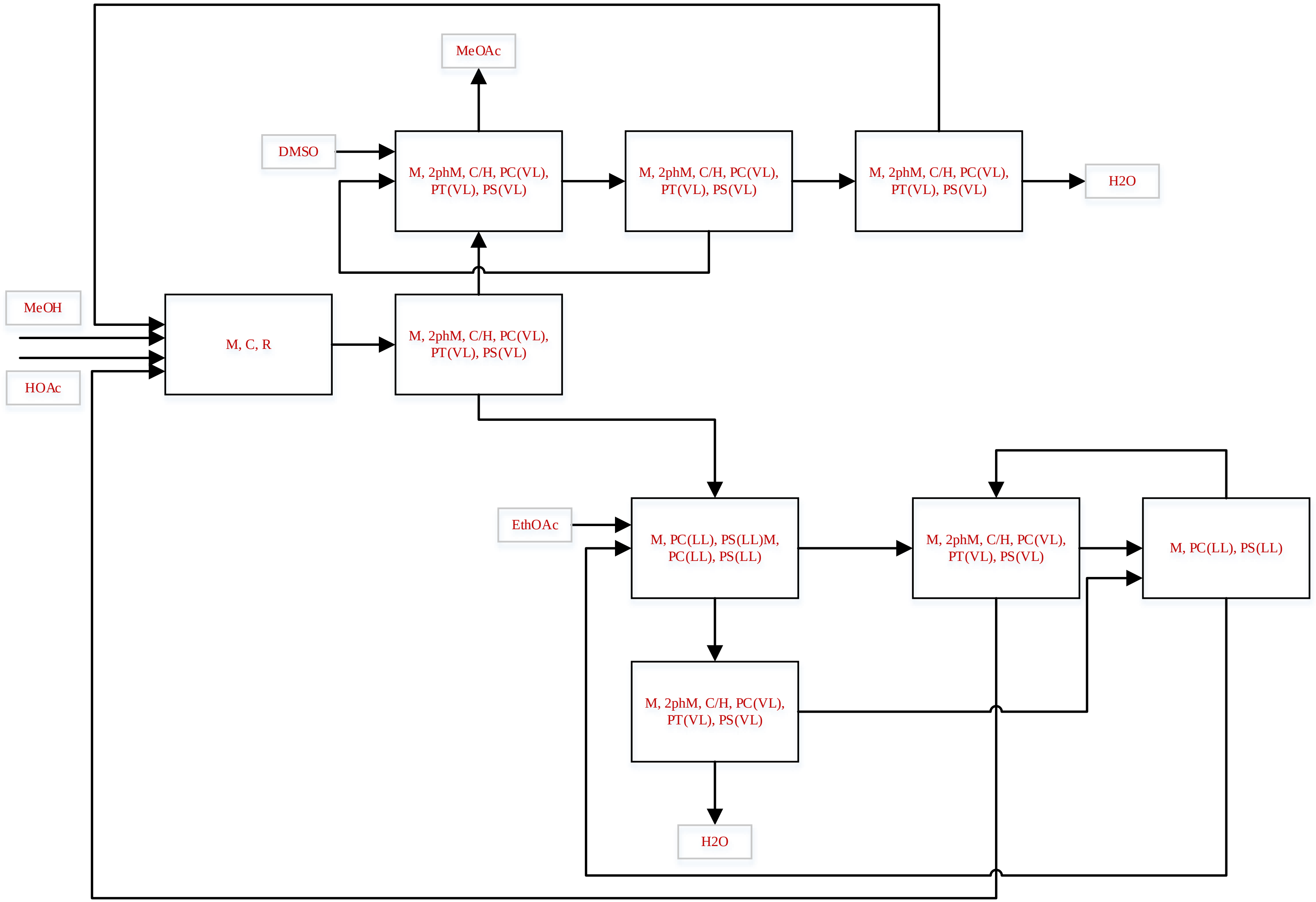


J. J Siirola (2011)

Task Identification



Phenomena Identification



Combination of Phenomena

R, M_I, M_T, M_R M_V, 2phM, PC(V-L), PT(V-L), PT(P:V-L), PS (V-L), D, H, C

13 in total

Reduced from 4017→58 using connectivity rules

Connectivity Rules:

1. **H+C should not exist in the same SPB**
2. **PC phenomena exists together with PT phenomena**
3. **SPB can contain simultaneous R and separation**

SPB	Interconnection Phenomena	In	Out
SPB.1	M	1..n(L)	1(L)
SPB2	M=R	1..n(L)	1(L)
SPB.7	M=R=2phM=PC=PT(VL)	1..n(L,VL)	1(V/L)
SPB.8	M=R=2phM=PC=PT(VL)=PS(VL)	1..n(L,VL)	2(V;L)
SPB.9	M=R=2phM=PC=PT(PVL)=PS(VL)	1..n(L,VL)	2(V;L)
SPB.58	D	1(L;VL,V)	1..n(L;V; VL)

SPB	Interconnection Phenomena	In	Out
	~~M=R=H=C~~	~~1..n(L)~~	~~1(L)~~

SPB	Interconnection Phenomena	In	Out
SPB.7	M=R=2phM=PC=PT(VL)	1..n(L,VL)	1(V/L)

SPB	Interconnection Phenomena	In	Out
SPB.8	M=R=2phM=PC=PT(VL)=PS(VL)	1..n(L,VL)	2(V;L)
SPB.9	M=R=2phM=PC=PT(PVL)=PS(VL)	1..n(L,VL)	2(V;L)

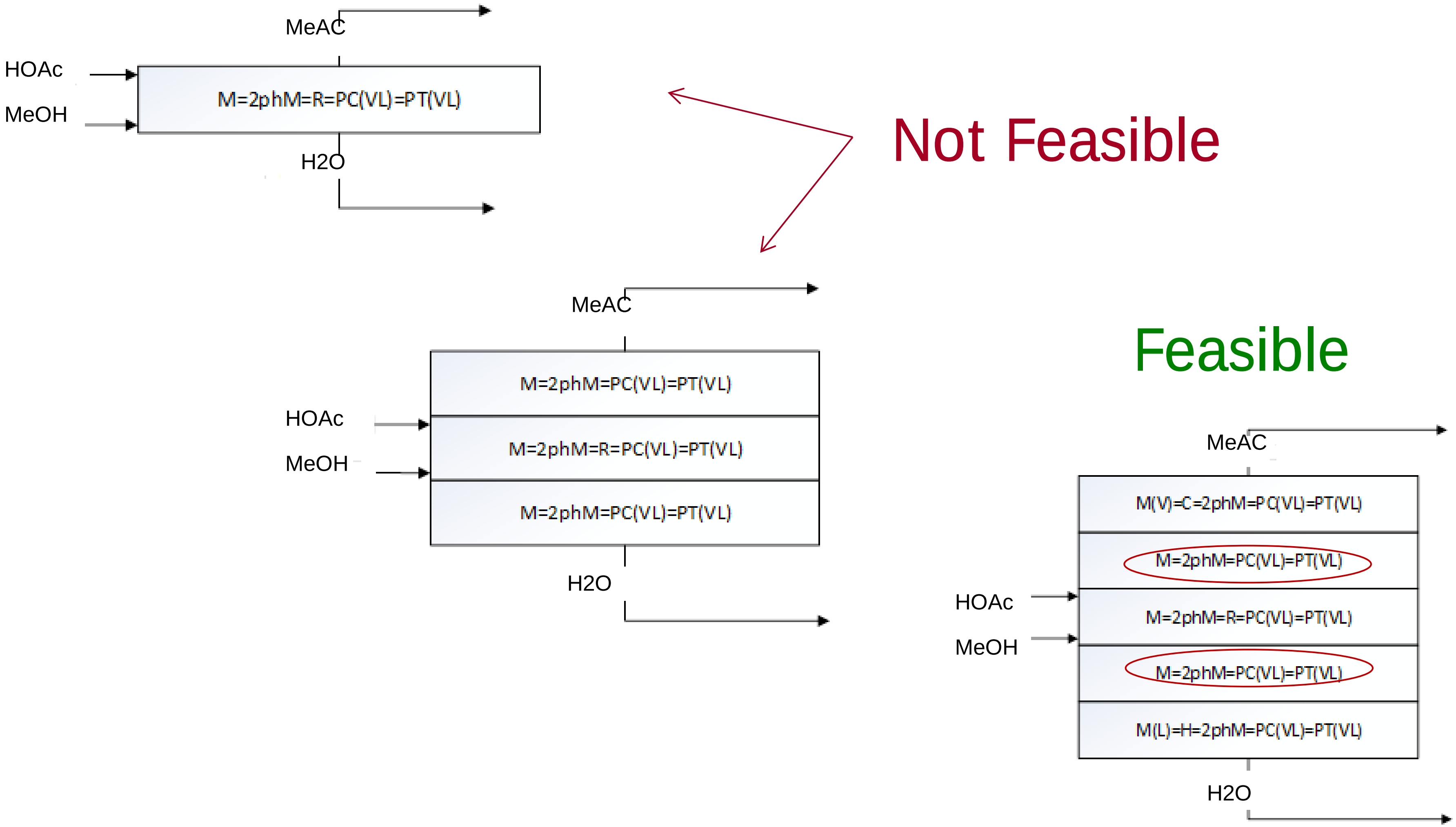
Lutze et al. (2013)

Interconnection Phenomena

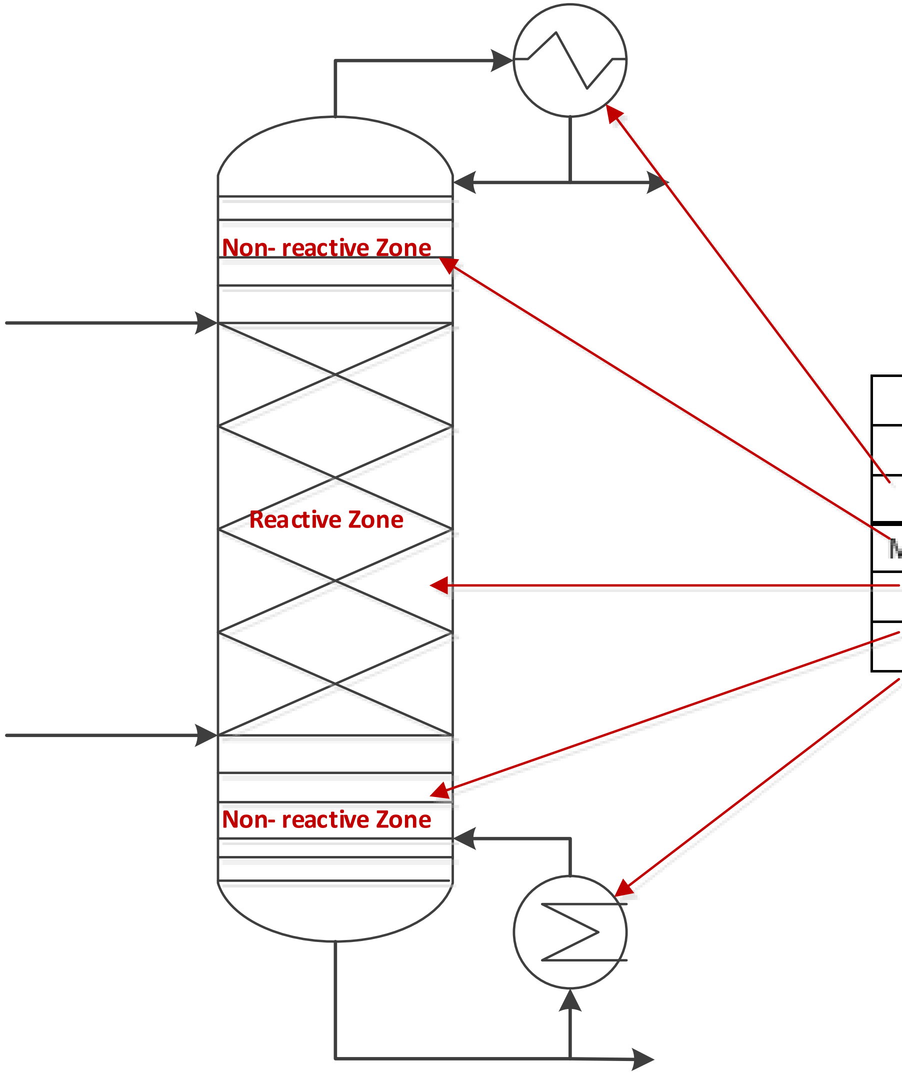
SPB	Interconnection Phenomena	In	Out
1	M	1..n(L)	1(L)
2	M=R	1..n(L)	1(L)
3	M=H	1..n(L)	1(L)
4	M=C	1..n(L)	1(L)
5	M=R=H	1..n(L)	1(L)
6	M=R=C	1..n(L)	1(L)
7	M=R=2phM=PC(VL)=PT(VL)	1..n(L,VL)	1(V/L)
8	M=R=2phM=PC(VL)=PT(P:VL)	1..n(L,VL)	1(V/L)
9	M=R=2phM=PC(LL)=PT(P:LL)	1..n(L,)	1(L)
10	M=R=2phM=PC(VL)=PT(P:VL)=PS(VL)	1..n(L,VL)	2(V;L)
11	M=R=2phM=PC(VL)=PT(VL)=PS(VL)	1..n(L,VL)	2(V;L)
12	M=R=2phM=PC(LL)=PT(P:LL)=PS(LL)	1..n(L,VL)	2(V;L)
13	M=C=2phM=PC(VL)=PT(VL)	1..n(L,VL)	1(VL)
14	M=H=2phM=PC(VL)=PT(VL)	1..n(L,VL)	1(VL)
15	M=H=R=2phM=PC(VL)=PT(P:VL)	1..n(L,VL)	1(VL)
16	M=C=R=2phM=PC(VL)=PT(P:VL)	1..n(L,VL)	1(VL)
17	M=H=R=2phM=PC(LL)=PT(P:LL)	1..n(L,VL)	1(VL)
18	M=C=R=2phM=PC(LL)=PT(P:LL)	1..n(L,VL)	1(VL)
19	M=C=R=2phM=PC(VL)=PT(P:VL)=PS(VL)	1..n(L,VL)	2(V;L)
20	M=H=R=2phM=PC(VL)=PT(P:VL)=PS(VL)	1..n(L,VL)	2(V;L)
21	M=H=R=2phM=PC(VL)=PT(VL)=PS(VL)	1..n(L,VL)	2(V;L)
22	M=C=R=2phM=PC(VL)=PT(VL)=PS(VL)	1..n(L,VL)	2(V;L)
23	M=H=R=2phM=PC(LL)=PT(P:LL)=PS(LL)	1..n(L,VL)	2(L;L)
24	M=C=R=2phM=PC(LL)=PT(P:LL)=PS(LL)	1..n(L,VL)	2(L;L)
25	D	1(L;VL,V)	1..n(L;V;VL)

Combine Phenomena

- **Potential New Operations**

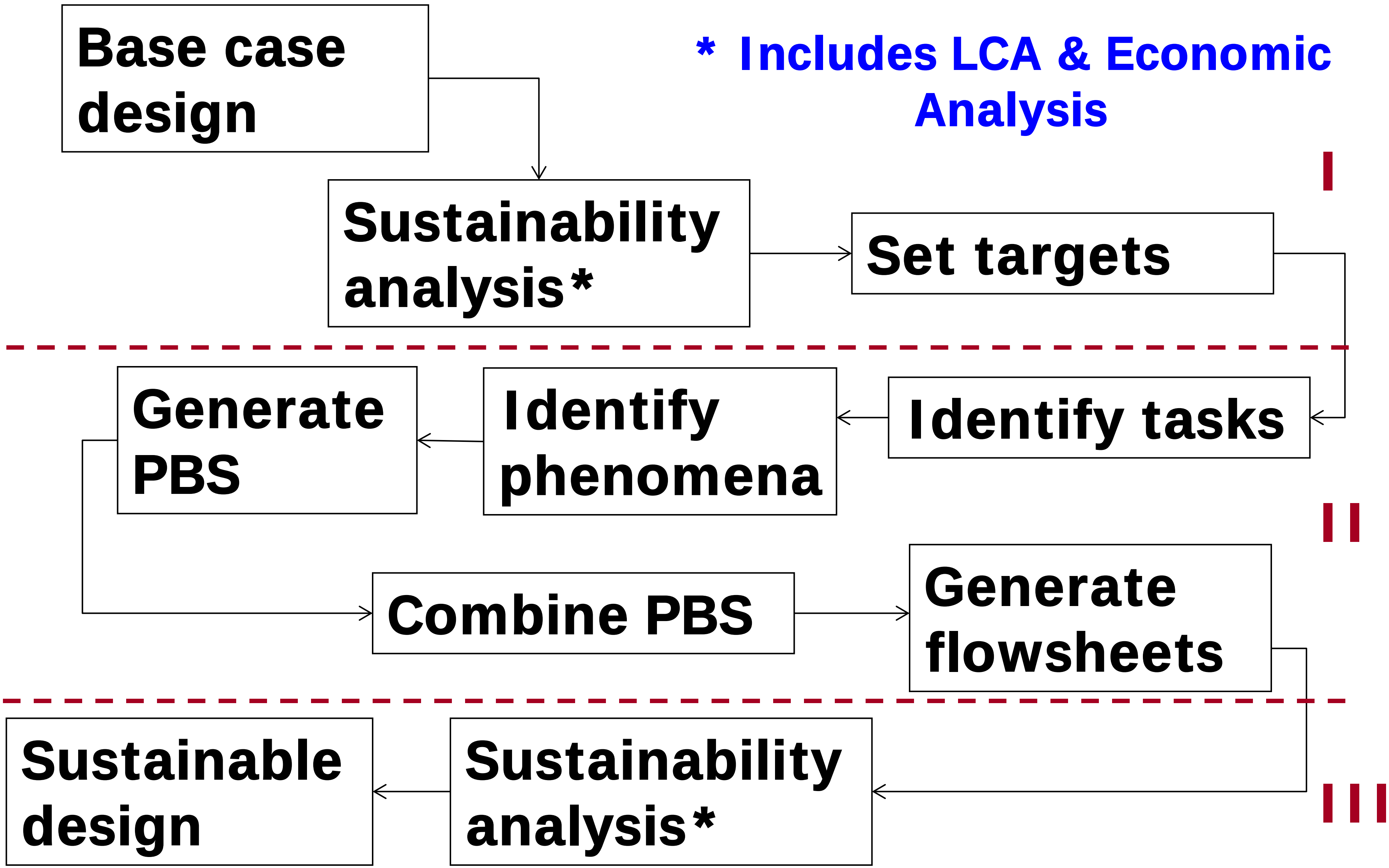


Innovative Solution



Operation
M(V)=C=2phM=PC(VL)=PT(VL)
M(V-L)=2phM=PC(VL)=PT(VL)
M(V-L)=R=2phM=PC(VL)=PT(VL)=PS(VL)
M(V-L)=2phM=PC(VL)=PT(VL)
M(L)=H=2phM=PC(VL)=PT(VL)

Conceptual Framework



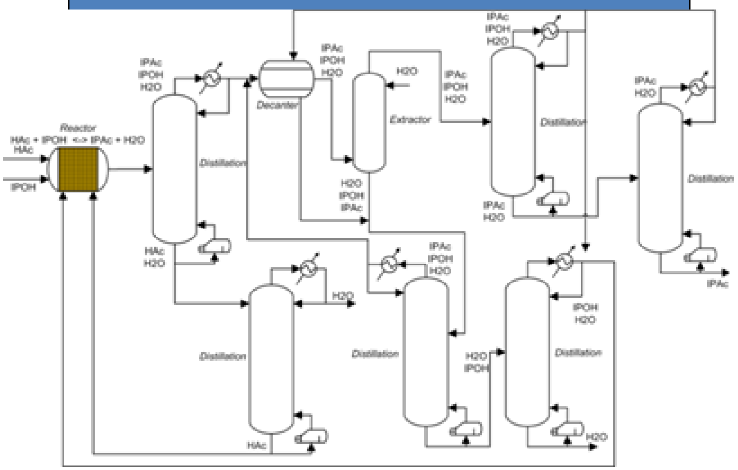
Problems Solved

- **Operations as Building Blocks**
  - Production of H_2O_2
  - Production of HMF
  - Production of Bioethanol
  - Brine Workup

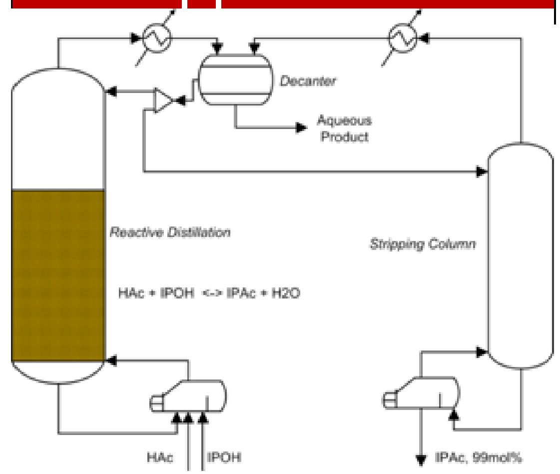
- **Phenomena as Building Blocks**
  - Production of isopropyl-acetate, methyl acetate
  - Separation of H_2O_2/H_2O
  - Production of Biodiesel

Isopropyl Acetate Production

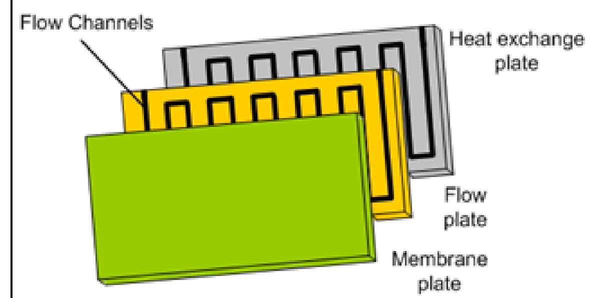
Base-Case-Design

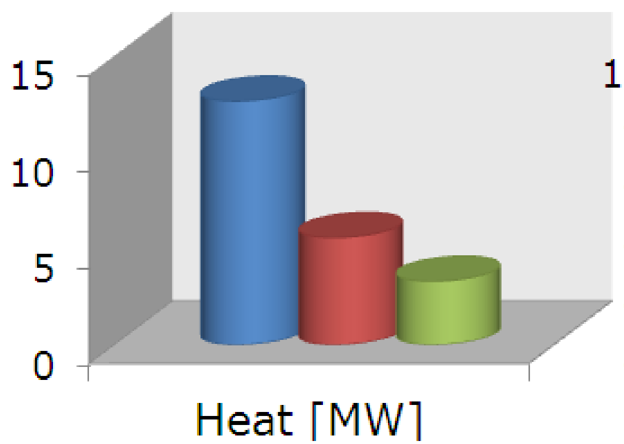


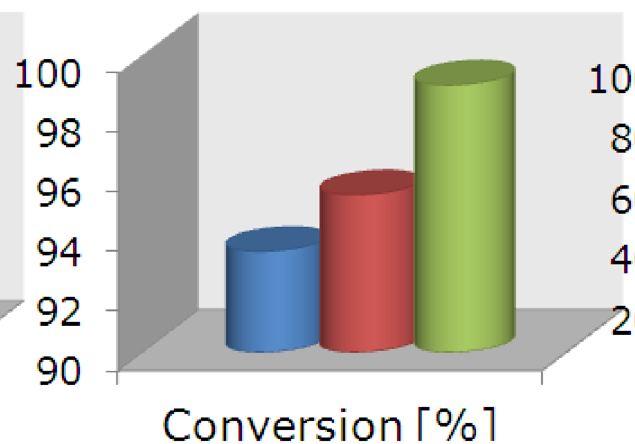
PI Unit-Operational Approach

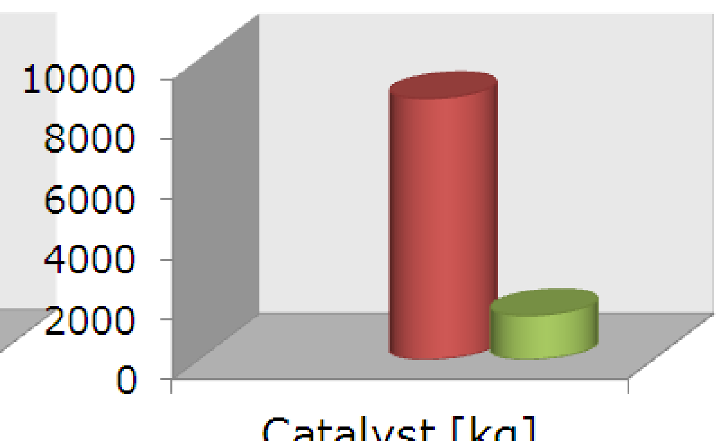


PI Phenomena-Based Approach









Invitation to Visit Denmark

International Scientific Comittee

Representatives of the PSE/ESCAPE communities from Europe, Asia, the Americas and Africa have been invited to join this committee.

Name	Institution	Country
G. Henning	Universidad Nacional del Litoral	Argentina
S. Diaz	PLAPIQUI	Argentina
M. Narodoslawsky	Graz University of Technology	Austria
F. Logist	University of Leuven	Belgium
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J. Zhao	Tsinghua University	China
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S. Zhang	Chinese Academy of Sciences	China
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K. Gernaey	DTU Chemical Engineering	Denmark
J. K. Huusom	DTU Chemical Engineering	Denmark
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B.-G. Rong	University of Southern Denmark	Denmark
B. Sarup	Alfa Laval Copenhagen	Denmark
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T. Majozi	University of Pretoria	South Africa
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A. Espuña	Universidad Politecnica de Catalunya	Spain
F. Marechal	École Polytechnique Fédérale de Lausanne	Swiss

International Scientific Comittee

(Continued)

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I-L. Chien	National Taiwan University	Taiwan
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C. Floudas	Princeton University	USA
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M. El-Halwagi	Texas A&M University	USA
L. Achenie	Virginia Polytechnic Institute and State Univ.	USA
J. Siirola	Carnegie Mellon University/Purdue University	USA
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M. K. Burka	NSF	USA
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PSE2015/ ESCAPE25

31 May - 4 June 2015 Copenhagen
PSE-2015 / ESCAPE-25 Joint Event

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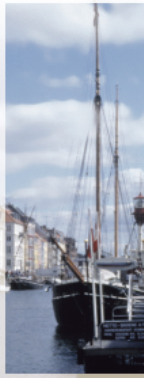
DK-2300 Copenhagen S



Technical University of Denmark DTU