Purification of Electronic-Grade Isopropyl Alcohol

Team Social Distillation

Project Background

- Electronic grade solvents are used at various steps within semi-conductor processing
- Purity of solvents is important for product quality
- Isopropyl alcohol is used in the final step in semi-conductor production in pure form

Project Goals

- Feed to be purified contains organic and metal components
- Goal specified from industry partner is to separate 99.999% pure isopropyl alcohol
- Implement recycle streams for additive solvents to reduce materials costs and chemical waste

Sample	Concentration in Stream	
[-]	[wt %]	[adjusted wt%]
Ethanol	0.034%	0.034%
1-Propanol	0.110%	0.110%
Isopropyl Alcohol	99.651%	99.645%
2-Butanol	0.013%	0.013%
1-Propanol, 2-methyl (Isobutyl alcohol)	0.029%	0.029%
Acetic Acid, 1-methylethyl ester (Isopropyl acetate)	0.164%	0.164%
Water	[redacted]	0.006%

Previous Work

- Rachel Meeuwsen’s Honor’s Thesis has been the foundation of our project
- Primary method of separation is distillation and molecular sieves
- Solvents used and not recycled -- DMSO and cyclopentane
- Final purity of isopropyl alcohol was 99.95%

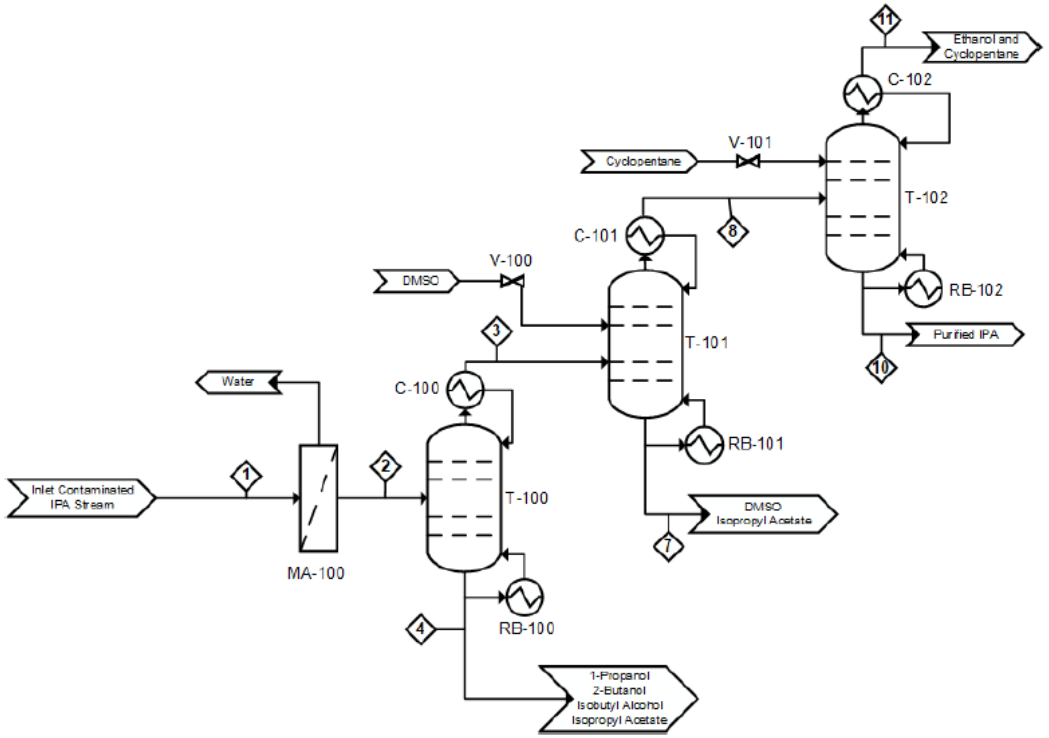


Figure 1: Process flow diagram for the purification of an isopropyl alcohol stream. Process includes a molecular sieve separation with three distillation columns in series following the sieve separation.

Research for Azeotropic Distillation

- Alternative chemical additives for azeotropic distillation processes
  - Ethylene Glycol for the removal of Isopropyl Acetate as an alternative for DMSO

    US Patent 4,826,576
  - Methyl ethyl ketone, 2,4-Dimethyl Pentane, 2-Pyrrolidinone for the removal of ethanol as alternatives for Cyclopentane, all chemicals create a solution with relative volatility of 1.45

    US Patent 5,338,411
- Parameters for evaluating chemical performance
  - Removal efficiency
  - Recyclability
  - Flowrate requirements and overall cost
  - Environmental impact and waste treatment requirements

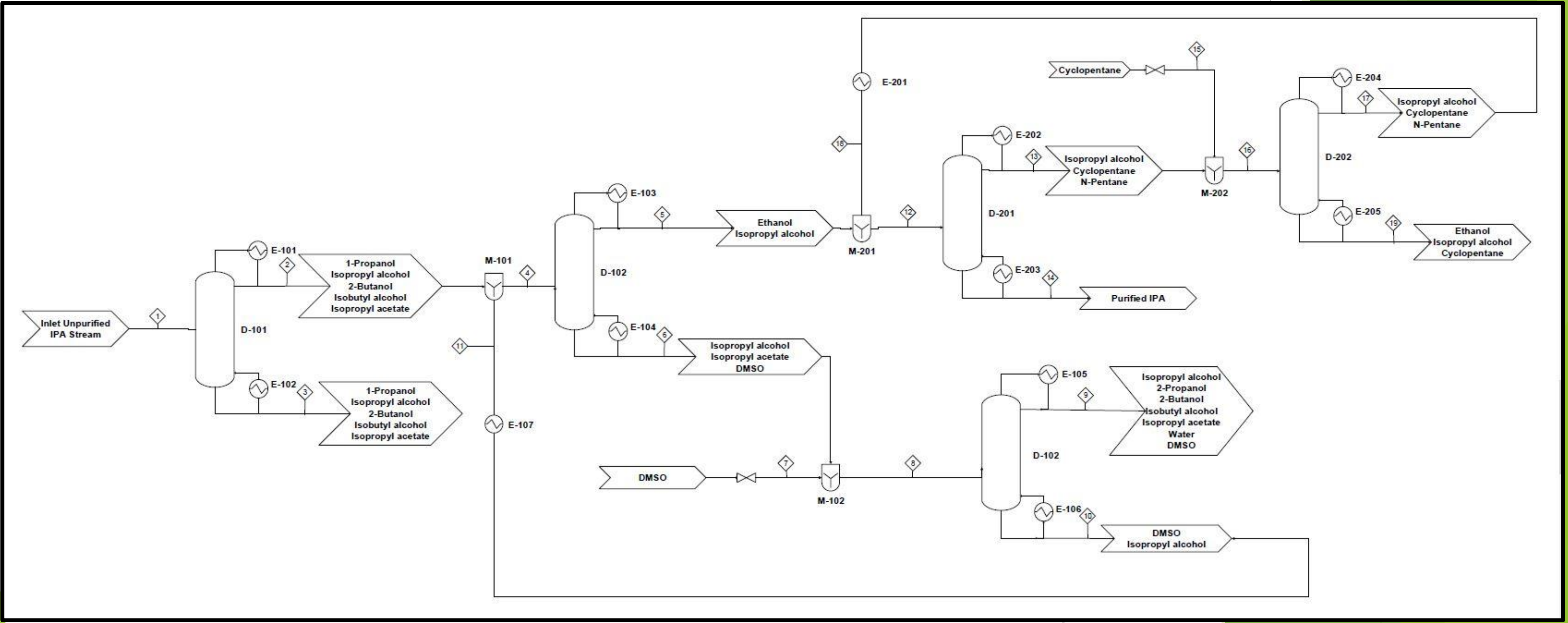
TABLE 2

Effective Azeotropic Distillation Agents For Separating Ethanol From Isopropanol

Compounds	Relative Volatility
None	1.14
Methyl formate	1.2
t-Butyl methyl ether	1.2
Isopropyl ether	1.25
Methyl isopropyl ketone	1.3
2,2-Dimeth oxy propane	1.2
Ethyl formate	1.35
t-Amyl methyl ether	1.3
Methyl propionate	1.35
2,3-Butanedione	1.2
1,3-Dioxolane	1.4
2-Pyrrolidinone	1.45
Propyl formate	1.25
Acetone	1.20
Methyl ethyl ketone	1.45
Hexane	1.4
Cyclopentane	1.45
Cyclohexane	1.35
Hexene-1	1.4
2,4-Dimethyl pentane	1.45

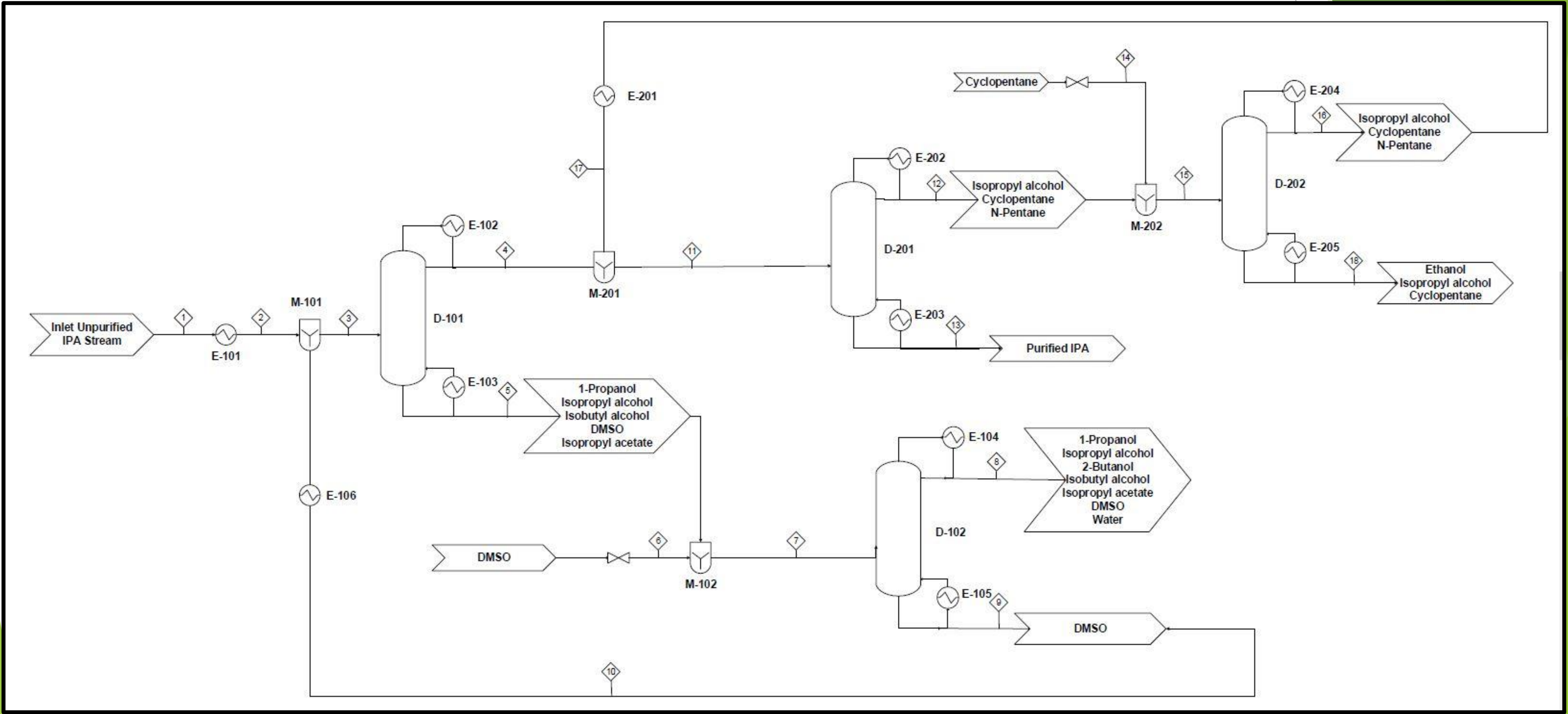
PFD

Original Column Design with recycle/purification loops



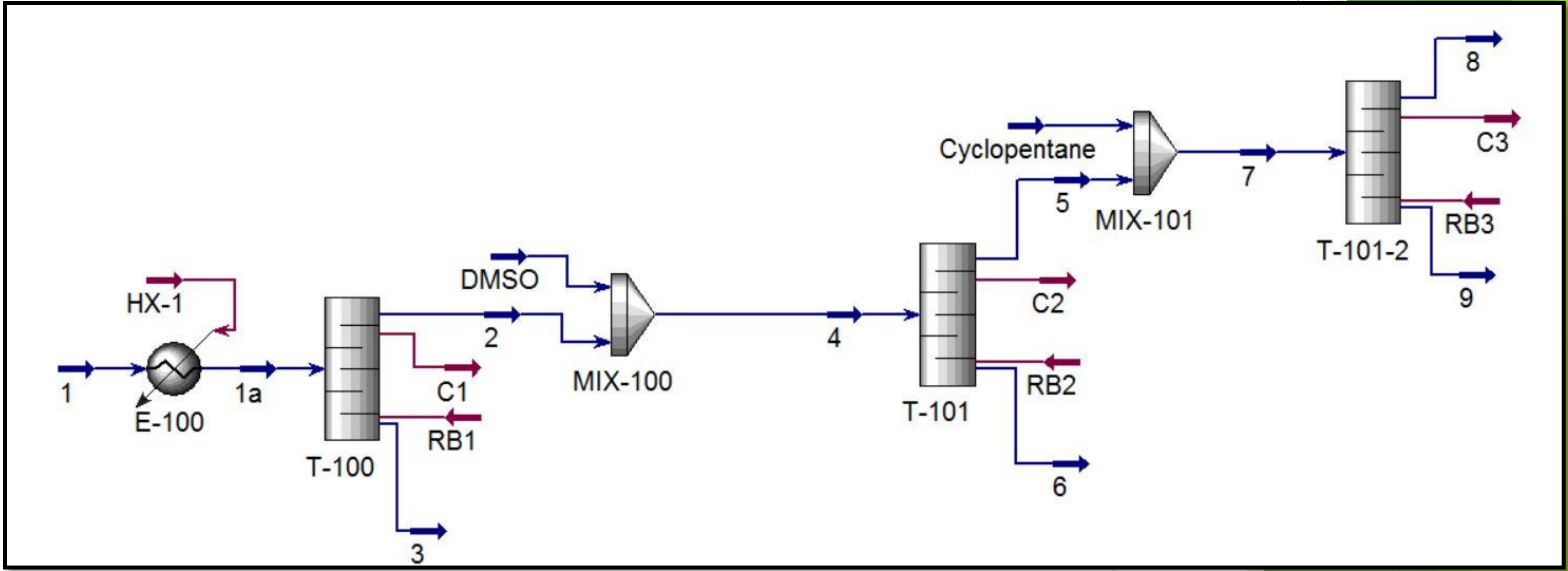
PFD

Two Column Design with recycle/purification loops



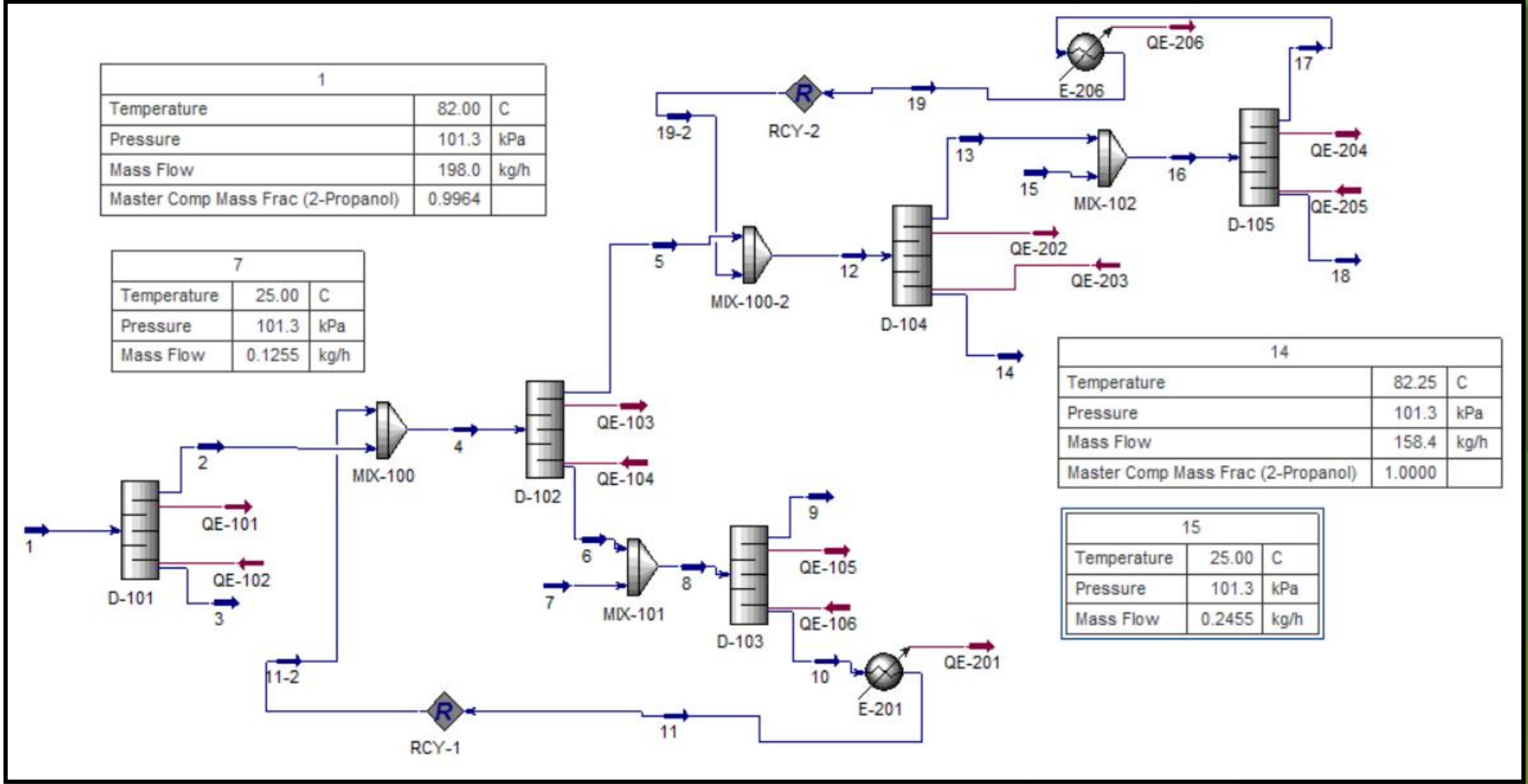
HYSYS Modeling

Original Design- Completed by Rachel Meeuwsen



HYSYS Modeling

Original Design with purification/recycle loops



1		
Temperature	82.00	C
Pressure	101.3	kPa
Mass Flow	198.0	kg/h
Master Comp Mass Frac (2-Propanol)	0.9964	

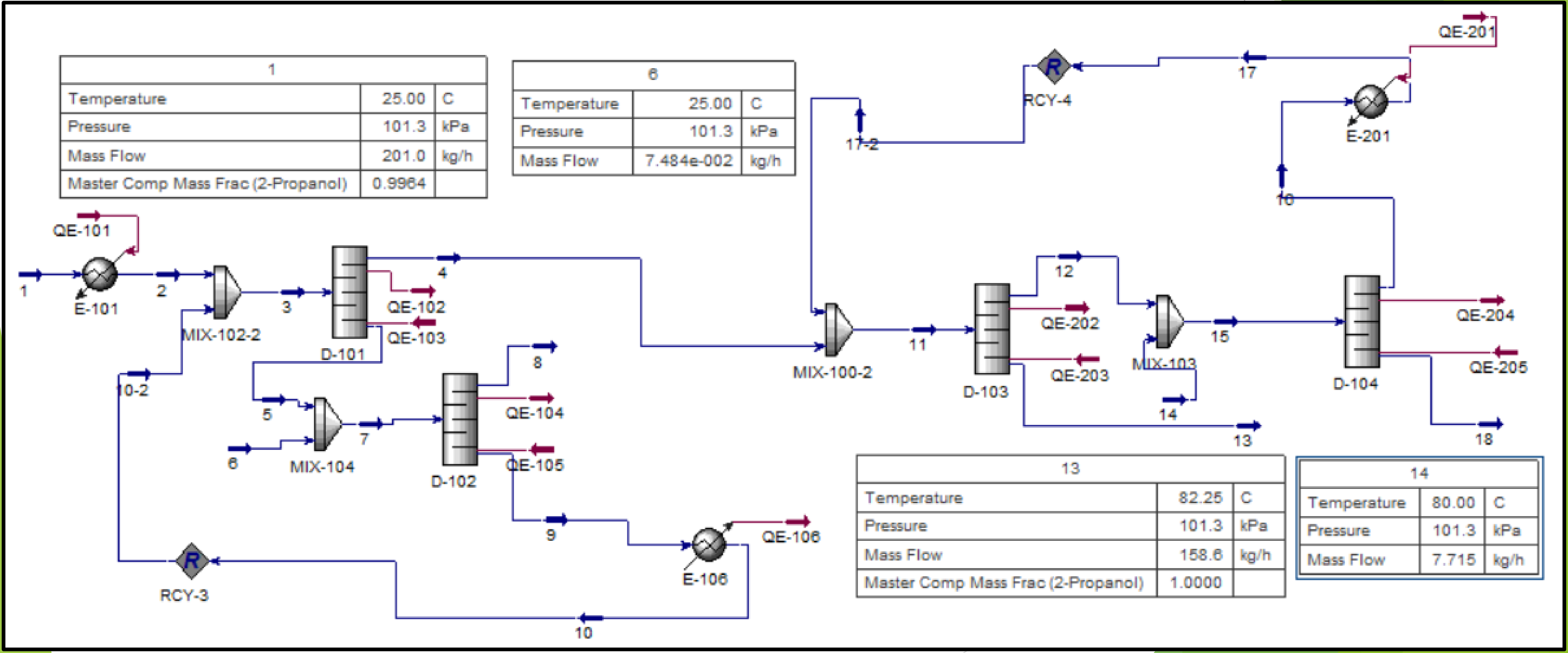
7		
Temperature	25.00	C
Pressure	101.3	kPa
Mass Flow	0.1255	kg/h

14		
Temperature	82.25	C
Pressure	101.3	kPa
Mass Flow	158.4	kg/h
Master Comp Mass Frac (2-Propanol)	1.0000	

15		
Temperature	25.00	C
Pressure	101.3	kPa
Mass Flow	0.2455	kg/h

HYSYS Modeling

Two Column Design with recycle/purification loops



1		
Temperature	25.00	C
Pressure	101.3	kPa
Mass Flow	201.0	kg/h
Master Comp Mass Frac (2-Propanol)	0.9964	

6		
Temperature	25.00	C
Pressure	101.3	kPa
Mass Flow	7.484e-002	kg/h

13		
Temperature	82.25	C
Pressure	101.3	kPa
Mass Flow	158.6	kg/h
Master Comp Mass Frac (2-Propanol)	1.0000	

14		
Temperature	80.00	C
Pressure	101.3	kPa
Mass Flow	7.715	kg/h

Metal Particle Removal

- Initial metal concentrations:

Type of Metal	Concentration (ppb)
Aluminum	0.025
Barium	0.0025
Calcium	0.025
Chromium	0.005
Copper	0.005
Magnesium	0.028
Manganese	0.005
Potassium	0.052
Sodium	0.61
Titanium	0.005
Zinc	0.01

Most Critical:
- Copper
- Chromium

Critical:
- Sodium
- Potassium
- Calcium

Least Critical:
- Aluminum
- Magnesium

These concentrations are so low that they will not be an issue in the final product.

Metal Particle Removal

Options for removal:

- Ion Exchange
  - Resin can be regenerated
  - Not effective at removal
  - Can incur high costs over time

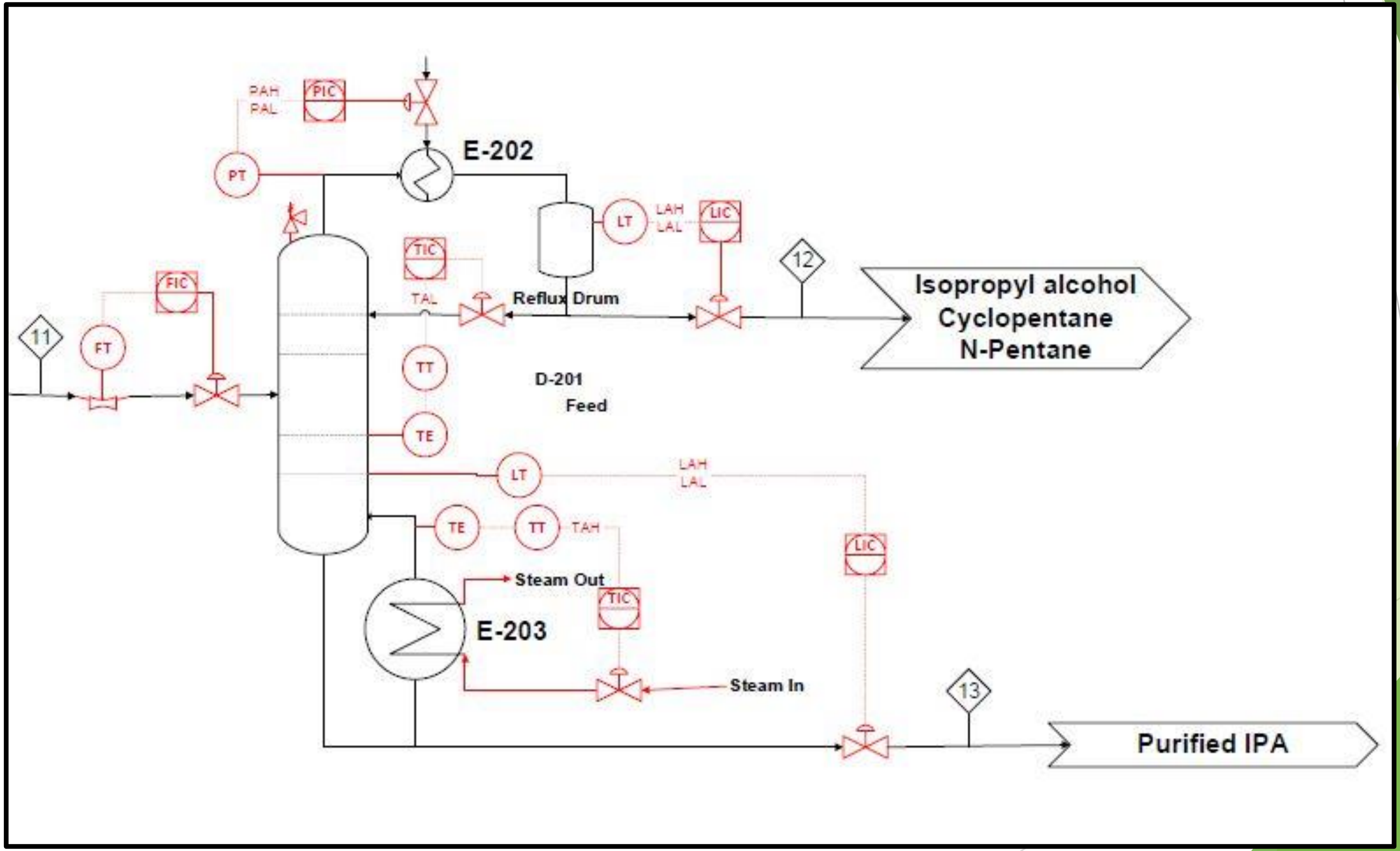
- Membranes/Reverse Osmosis
  - Can be used for removal of metal and other organics
  - Require lower flow rates
  - Lower future costs (less maintenance)

- Electrolysis
  - Is effective at removal and requires minimal maintenance
  - Difficult to control
  - Can have high capital costs and unknown future costs

Safety and environmental considerations

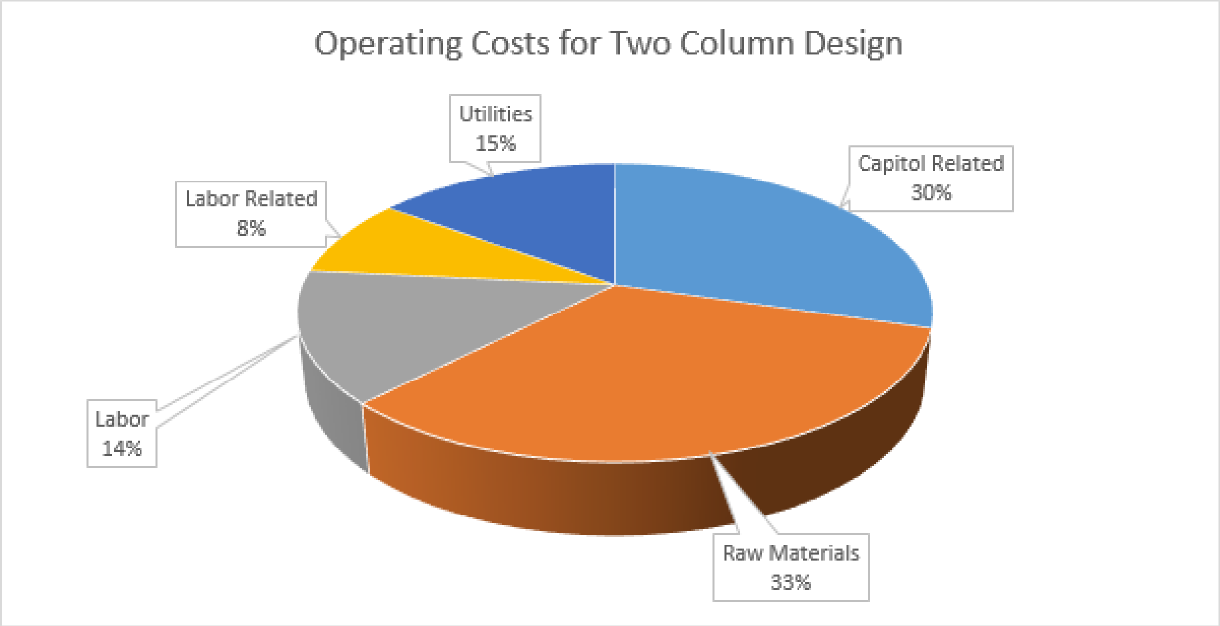
- Low metal concentrations are hard to detect and small concentrations but over lifetime of the plant could have large environmental impact.
- Waste streams from purification of solvents can be sent to waste treatment company.
- Haz Op was conducted to examine possible risks related to the operation of this plant.
  - Loss of cooling in tower condenser
  - Excess heating in column reboiler
  - Excess or loss of flow in and out of column
- Majority of safety features aimed at preventing pressure buildup in column which could result in vessel rupture and explosion

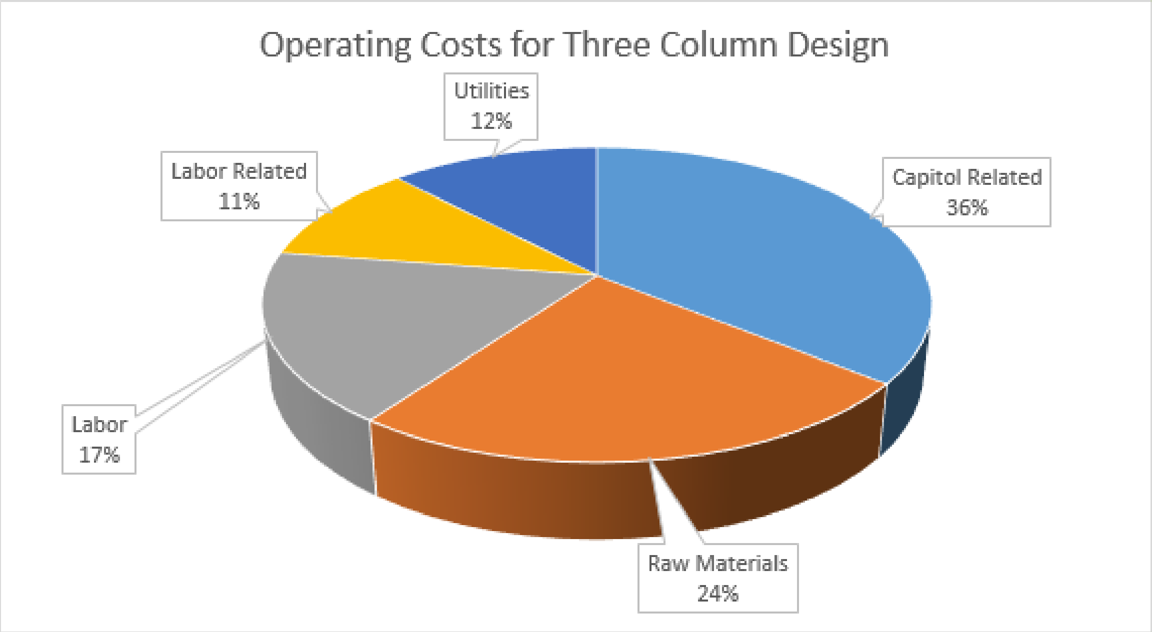
PID Control



PAH
PAL
PIC
PT
E-202
LT
LAH
LAL
LIC
TIC
FIC
TAL
Reflux Drum
12
Isopropyl alcohol
Cyclopentane
N-Pentane
11
FT
TT
D-201
Feed
TE
LT
LAH
LAL
TE
TT
TAH
LIC
Steam Out
TIC
E-203
Steam In
13
Purified IPA

Cost Comparison





	Two Column	Three Column
Capital Investment	$4.82M	$7.11M
Total Reoccurring costs	$4.22 M/year	$6.69 M/year
Sales Price	$5.00/L	$5.00/L
Payback (years)	1.08	2.03

Summary

- Final design consists of two main azeotropic distillation columns and two towers for solvent purification
- DMSO and Cyclopentane as solvents
- Overall recovery of 79%
- Initial investment of $4.82M and recurring costs of $4.22 M/year
- Payback time of only 1.08 years