

MEMORANDUM

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RE: DME Recovery and Purification

DATE: 9/6/2017

ATTACHMENTS: Report titled: "Proposal for DME Recovery and Purification"

CH3-Energy facility is developing plans for a dimethyl ether (DME) production plant. This report outlines the preliminary design for the purification of dimethyl ether, given the selective conversion of crude methanol using a DME acid catalyst. This reaction creates multiple impurities that require separation from the DME product. The separation process proposed in this report generates 96.1% DME recovery with a purified methanol recycle stream that recovers 97.3% of unreacted methanol. From these results, we recommend moving forward with the proposed design for DME purification. Further study includes deeper investigation of carbon dioxide separation from DME, additional sensitivity analysis, and more rigorous safety measures when handling methanol storage.

Proposal for DME Recovery and Purification

9/18/17

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This proposal outlines the preliminary design and analysis of a dimethyl ether purification process.

Abstract

This report contains a proposed design for the purification of dimethyl ether (DME) from an effluent stream with various impurities. In general, separation processes can employ various methods for purification including extraction, distillation, and adsorption, among others. Distillation was chosen to be the method of separation in this design, as historically it is the most economical method for liquid separation [1]. Three columns were used to separate DME, resulting in a DME recovery of 96.1% with minimal impurities. A methanol recovery of 97.3% is also achieved, benefiting the environmental and economic impact of the design.

The proposed design is capable of withstanding 2-3 kmol/hr fluctuations for most major feed components, and the distillation columns perform equally well in the midst of $\pm 5^{\circ}\text{C}$ change in the major feed streams.

Alternate designs were considered to improve purity and recovery, such as stripping columns and additional separators, but were found to add complexity and cost with too little benefit.

The UNIQUAC thermodynamic package was used to design this process; additional tests were run using the Peng Robinson and NRTL methods for added security. The process performs sufficiently when the NRTL model is applied, but falls short under Peng Robinson. Peng Robinson results in discontinuities for by-stage temperature and composition profiles, but the accuracy of UNIQUAC and NRTL was still called into question by lower quality fits of experimental data than Peng Robinson.

With the results seen under various thermodynamic models and when compared to alternative designs, the design proposed meets and exceeds design specifications. The recommended next step is to further investigate the failure of the model under the Peng Robinson equation of state, and assess the added costs for adding additional separations units. The proposed DME purification process assumes a specific composition of the reactor effluent, and upon analysis has shown to impact DME recovery and affect the purity of the methanol recycle stream. Therefore, fluctuations in reactor effluent composition needed to be properly analyzed to support or discredit the conclusions about the sensitivity of the system.

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Introduction

The purpose of this report is to present a preliminary design of a dimethyl ether (DME) purification unit for the use at the CH3-Energy facility. Dimethyl ether is economical for use as an aerosol, and has potential future value in diesel and petrol engines, and gas turbines due to its high cetane number[2].

Completed using ASPEN Plus modeling tools, the unit is designed to separate high-purity DME and methanol recycle from the reactor at a rate of 90,000 tonne/day pure methanol consumption. This report is a step toward understanding operating conditions for a reasonably designed purification unit of the plant, and more in depth analysis is required to understand operating costs, safety measures, design sensitivity, and control systems.

This system was designed using the following specifications:

- >95% DME recovery with impurities less than 1 ppm methanol, 50 ppm CO₂ and C₃+C₄ hydrocarbons, and 50 ppm water
- Final product temperature at 30°C for safe handling
- 65 kmol/hr (of 68.71 kmol/hr) methanol recycle recovery at a minimum purity of 95 wt% methanol
- Wastewater temperature at 50°C or less prior to leaving the area

Note that recovery is defined as moles of a component in the desired product stream as a percentage of the moles in the feed stream from the reactor.

Separation unit design highly influences the profitability of DME production because it directly impacts the final DME purity. Another important consideration is the methanol recycle stream, which has the possibility to reduce methanol usage costs by a significant amount due to the limiting equilibrium of the methanol-DME reaction. The reaction is thought to be limited to less than 90% single pass conversion for dry methanol, and water impurities further inhibit equilibrium conditions [3] [4].

Separation consistency is crucial for lowering operating costs, and maintaining high DME reaction conversion. If process constraints for DME and methanol purity are not met, further impurities will be introduced to the process, leading to higher operating costs and limiting DME production. Additionally, if methanol recovery is not met, raw product costs would suffer.

Results

Design results and requirements

The proposed design consists of three distillations columns to separate on the light key-heavy key basis of DME-methanol, methanol-water, and carbon dioxide-DME. The proposed design Block Flow Diagram is **Error! Reference source not found.** The system was designed to operate with a reactor effluent at 350°C, 11 barg with composition listed in Table 1.

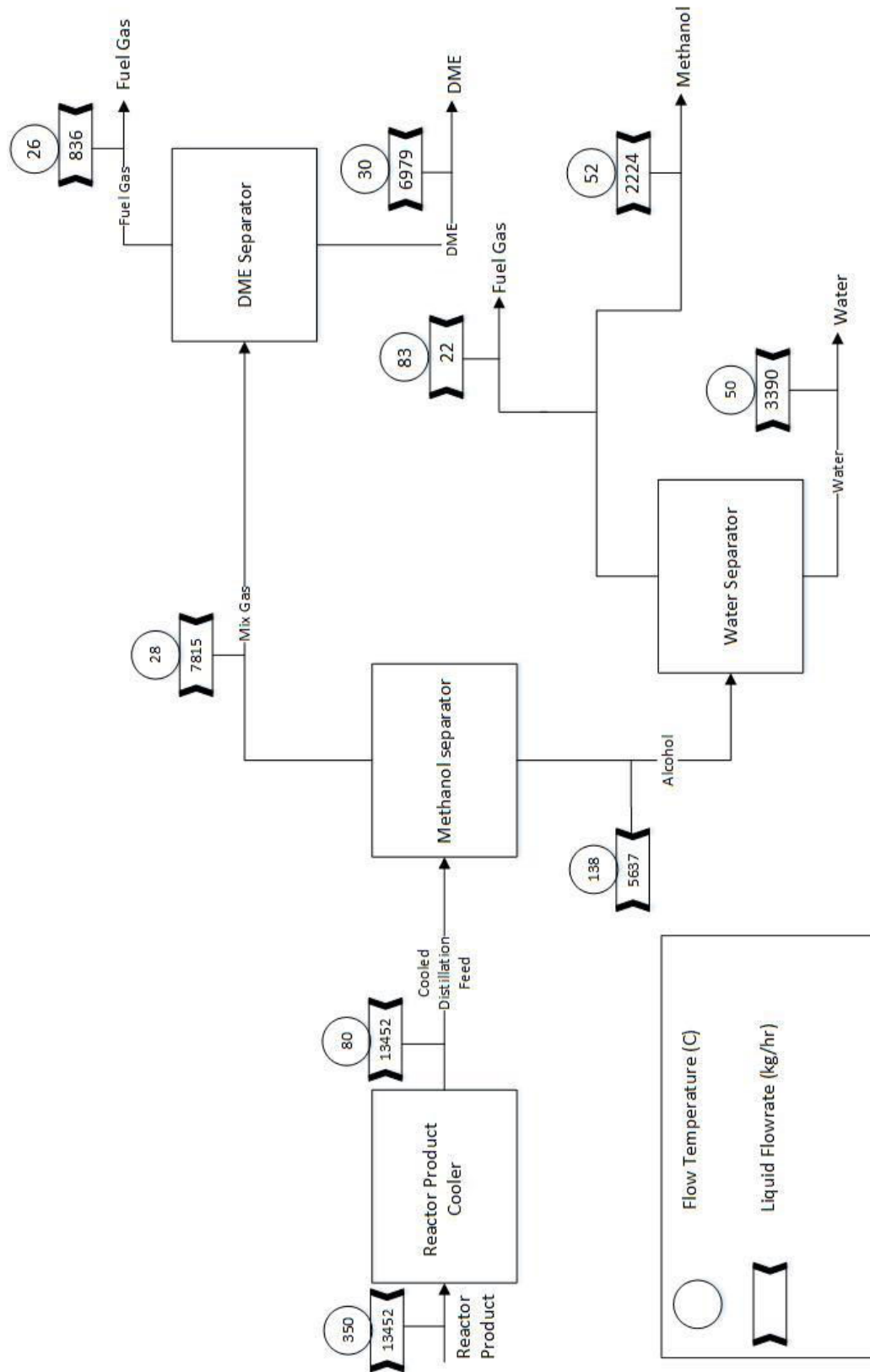


Figure 1: DME separation block flow diagram (BFD) for proposed process design. BFD outlines the three separation units and preparatory processes before separations.

Table 1: Compositions of the system effluent stream which exists the DME reactor. These compositions are assumed based on information provided by the CH3-Energy facility.

Material	Expected flow (Kmol/h)
CO	1.62
CO ₂	12.06
CH ₄	0.01
H ₂	3.04
Ethanol	0.30
Formaldehyde	0.22
Water	187.69
DME	157.72
Methanol	68.71

Full details of expected final stream compositions are documented in “A.2 Stream Summaries” in the Appendix. Table 2 summarizes the process model’s estimates for DME and methanol streams, as compared to design specifications.

Table 2: Design requirements for DME purification compared to ASPEN model results

Stream	Specification	Requirement	Result
DME	% DME recovery	95	96.1
	CO ₂ impurities, ppm	<50	12
	Methanol impurities, ppm	<1	0.26
	Water, ppm	<1	negligible
Methanol recycle	wt % methanol	95	96.3
	recovery, kmol/hr	65	66.9

Process overview

This overview references stream names and conditions in the Process Flow Diagram (PFD), starting on page 9.

The reactor effluent (Stream 1) enters the separations area at an expected rate of approximately 430 kmol/hr, throttled to below 10 bar (atmospheric), then pre-cooled to a temperature of 100°C in the pre-cooler (E-101) prior to prevent stage drying in the first distillation column. The two-phase stream is throttled to 7 bar and fed to the methanol separator (T-101) to separate the light gases and DME from the higher boiling point components. The reboiler for this column (E-103) uses low pressure steam, and the reflux condenser (E-102) uses chilled water.

The bottoms (Stream 6) is sent to the methanol-water cooler (E-104) to prevent flashing at the entrance of the water separator (T-301). The water separator produces a liquid distillate (stream 20) of approximately 96.3wt% methanol at 66.85kmol/hr, which is to be recycled to the reactor. The bottoms, which is wastewater, is cooled to 50°C by the waste water cooler (E-303) and sent to waste treatment.

The reflux drum (V-301) is oversized to act as a temporary storage drum for the methanol during fluctuations of recycle demand. It is to be maintained near atmospheric pressure with a vent to a flare to prevent the accumulation of light gasses in the tank. The reboiler for the column (E-302) uses low pressure steam, and the reflux condenser (E-301) uses cooling water.

The methanol separator distillate (Stream 5) is a mixture of DME and light gases. It is compressed from 7 bar to 23 bar by a centrifugal compressor (C-101), and cooled to 30°C by a cooling water (E-201) and chilled water (E-202) exchangers operating in series. At this point, the stream is expected to be 95% liquid to allow for easier distillation at 20 bar as it enters the DME separator. The distillate is light gases and with a small amount of DME, and is sent out of the area to be used elsewhere. The bottoms, liquid DME product, is cooled to 50°C to prevent flashing downstream by E-205, then depressurized to a lower pressure. Additional cooling in E-203 drops the temperature to 30°C for safe handling. The estimated DME recovery rate of 96.1%. The majority of DME loss is a result of being sent off with the fuel gas during distillation. The reboiler for this column (E-204) uses low pressure steam, and the reflux condenser (E-203) uses chilled water.

Discussion of design specifications continues on page 12.

Process flow diagram

Discussion

Thermodynamic Package Selection

Non-Random Two-Liquid (NRTL), UNIQUAC, Wilson, Soave-Redlich-Kwong (SRK), Peng Robinson (PR), and Ideal thermodynamic packages were all tested as possible best-choices to model our separations system. The NRTL, Wilson, and UNIQUAC liquid-state activity-coefficient models were pursued as none of the components are supercritical, and many of the unit operations are within normal conditions. However, many of the minor components were non-condensable, and the high pressure of the DME separator raised concerns for model accuracy. This led to the selection of the SRK and PR Equation of State models, which are better suited for simple, high pressure, or supercritical systems [1].

The selection for the best thermodynamic package was done by comparison to vapor-liquid equilibrium (VLE) experimental data for each of the three light key-heavy key pairs: Methanol-Water, DME-Methanol, and DME-Carbon Dioxide. Equilibrium data from National Institute of Standards and Technology (NIST) ThermoData Engine (TDE) was selected for temperature and pressure ranges that seemed like reasonable operating conditions at the beginning of the project. DME and carbon dioxide were compared isothermally at two points (15.05°C and 35°C) because it was unknown at the time what operating conditions were expected, although it was known that the temperature needed to be above at 5°C because of utility restrictions. Graphs for each of the comparisons are available in **Error! Reference source not found.** of the Appendix [4] [5] [6].

The SRK and PR models predicted DME-CO₂ and Methanol-DME interactions incredibly well. The DME-CO₂ interactions for NRTL, UNIQUAC and Wilson models all performed fairly well, but overestimated pressure at a given vapor composition. These overestimates were more or less constantly above the true values. These models did not perform incredibly well for calculating interactions at mole fractions up to 0.4 DME in the DME-Methanol trials. NRTL, UNIQUAC, and Wilson packages predicted water-methanol interactions very well, with PR as a close second, followed by SRK and Ideal models.

Under normal consideration, either the SRK or PR models would be chosen as the best choice for system-wide calculations - likely PR, which is considered to be less limited in VLE calculations than SRK [7]. However, ASPEN noted that the cubic equations extrapolated the liquid volume root to complete calculations which can lead to inaccuracies. This was a major concern as VLE calculations are the root of the separations process and discontinuities in calculations could lead to poor results. Because of this, the UNIQUAC Model was determined to be most appropriate for use in developing a working ASPEN model. The NRTL and PR Equation of State were noted as best alternatives and were tested in the

Pressure relief valves are at the entrance of reactor effluent stream 1, and water effluent stream from cooler E-303. The flow streams in this separation process fitted with valves to manipulate the stream flowrate to maintain a desirable separation process condition. Most of the valves were manually controlled hand valves with the exceptions of the valves installed on the reflux drum V-101, V-201, and V-301. The reflux rate from the reflux drums V-101, V-201, and V-301 were controlled by an automatic valve that is fitted with a flow indicator controller. This flow indicator and controller (FIC) would send a signal to an operator in the control room who would then send a pneumatic signal to the valve to move the valve to the desired position. Level indicator controllers (LIC) were installed at the bottom of the distillation towers T-101, T-201, and T-301 to monitor and control the level of the liquid bottom by manipulating the flow of the liquid bottom effluent stream. These indicators and controllers are a

combination of transmitter and controller that works together to control flowrates. Pressure controller PC is used to monitor and control the pressure in the separator column by controlling the flowrate of the cooling water in the condenser to increase the rate of condensation.

Sensitivity section. These three models - UNIQUAC, NRTL, and PR - facilitated the DME separation process design behind this report.

Process design, condition justifications, and design of equipment

As a general rule for material of construction, aqueous formaldehyde and carbon dioxide are corrosive to carbon steel [8]. These two materials are present through the majority of the process, so it is recommended that material of construction for each major unit operation is some grade of stainless steel or higher.

Safety concerns were one of the most prominent design influences in this process. Where applicable, pressures and temperatures were reduced to prevent damage to persons and property in the event of equipment failure. Since specialized equipment is generally required for pressures greater than 10 bar or temperatures greater than 260°C, avoiding these conditions when possible can also reduce capital investment [9].

Under the safety guidelines, the light gases and DME were removed from water and methanol in the first column. This achieves the following:

- Reduced physical dimensions of downstream distillation columns by splitting flow
- Allows for water-methanol separation at lower temperatures and pressures, facilitating better separation at safer conditions
- Retains higher pressure in the distillate, which reduced the size of the gas compressor downstream

In addition, the operations of the first column are limited by available utility temperature ranges, which is discussed in the next section.

Distillation Columns

Utilization of ASPEN's shortcut distillation method were used to generate initial guesses for number of stages, feed stage, and reflux ratios for each column. Once switching to the rigorous method for distillation calculations, various parameters were adjusted for each column and determined through analyzing by-stage compositions and bisection iteration methods.

Methanol Separator column (T-101) operating conditions were limited by utility availability. The separation between methanol and DME is a separation over a wide temperature range. Low-pressure steam at 9 barg (175 °C) is used in the reboiler and chilled water (5°C) is used in the reflux condenser, and appropriate differential temperatures across the exchangers needed to be maintained [10]. To accommodate this under reasonable fluctuations in operating conditions, the distillate pressure is to be maintained at a pressure of 7 bar.

One area where extreme process conditions could not be avoided was DME Separator T-201 due to the high volatility of the light gases in the feed stream (Stream 13). It was discovered that compressing and

cooling this stream to a vapor fraction of 5% allows for easier removal of carbon dioxide from the final DME stream in the DME Separator. Operating the column at 20 bar allowed the reflux to sit in a temperature range that provides good differential temperature with the chilled water even during times of process fluctuation. The reboiler temperature in this column is the main limiting factor in DME recovery, although sending some DME with the fuel gas by using it as the main reflux component allows for better carbon dioxide release from the DME stream.

The Water Separator column was chosen to operate near atmospheric pressure. Improved safety conditions are a plus, and the lower pressure facilitates better separation of methanol and water systems and reduced the minimum number of stages required.

Column feed stage determination was done through analysis of by-state compositions and temperatures for each column. These data are shown below Figure 2 - Figure 7. Feed stage placement was based on compatible composition of the inlet feed and the stage composition. In general, when minimal composition changes were seen between adjacent stages, it was assumed that those stages were not contributing sufficiently to the separations (save for removal of trace contaminants at the top or bottom of a column), and the feed stage was moved.

The feed stage for the DME Separator is near the top of the column (above Stage 2) to promote the maximum degas of DME, and reflux was adjusted to maintain temperatures appropriate for the chilled water system.

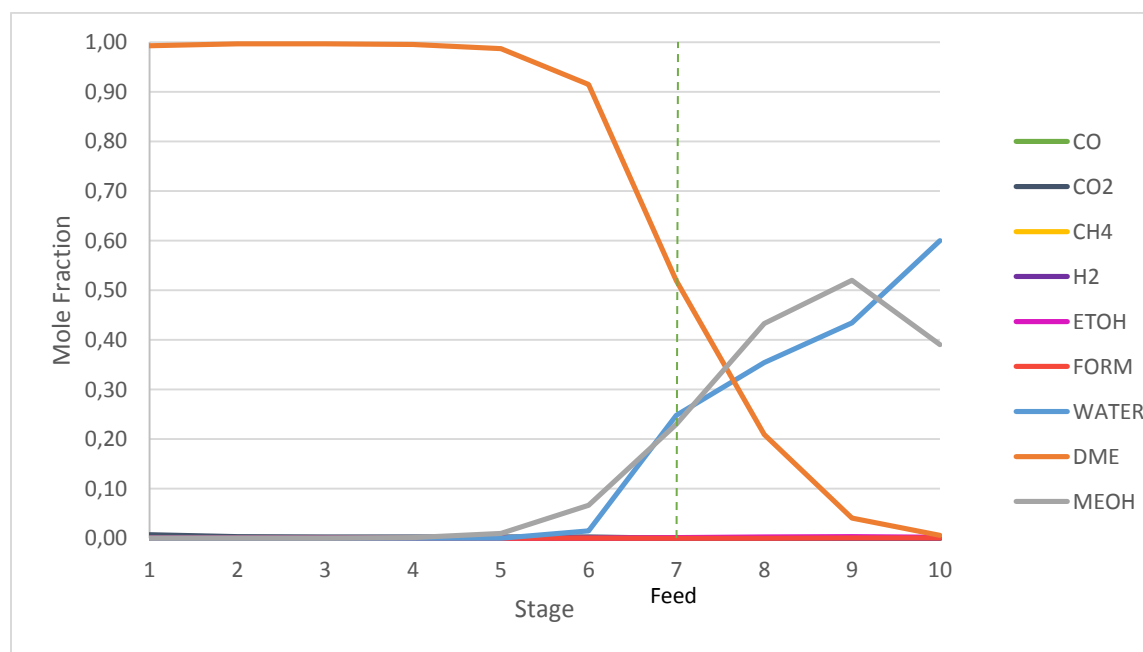


Figure 2: Tray composition by mole fraction for the T-101 Methanol Separator – the first column in the design separation. The light key-heavy key components are DME-Methanol. Inlet feed stage is stage 7.

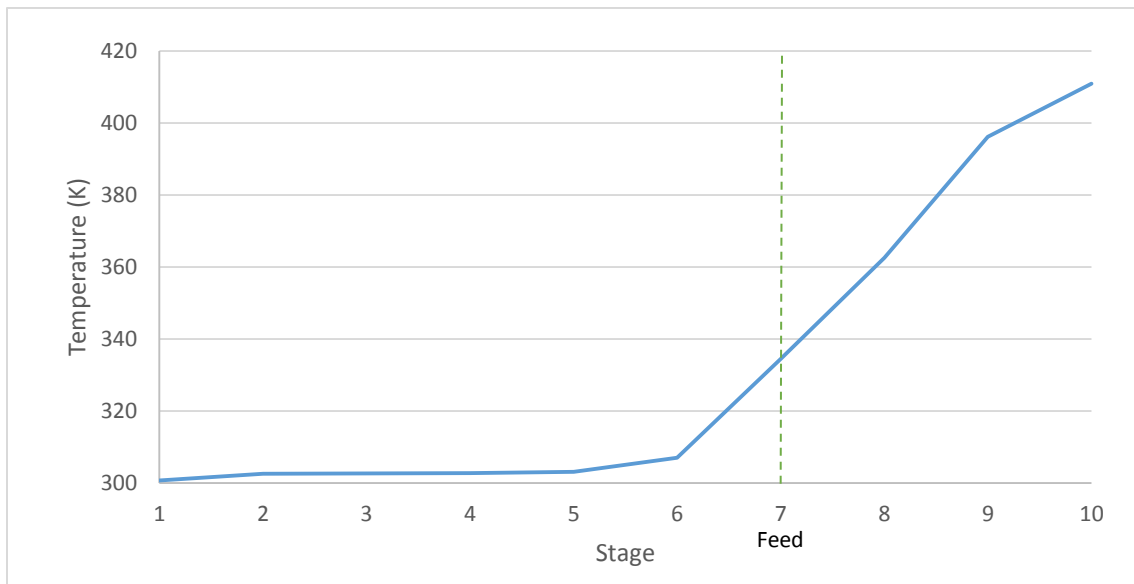


Figure 3: Temperature profile for stages of the T-101 Methanol Separator.

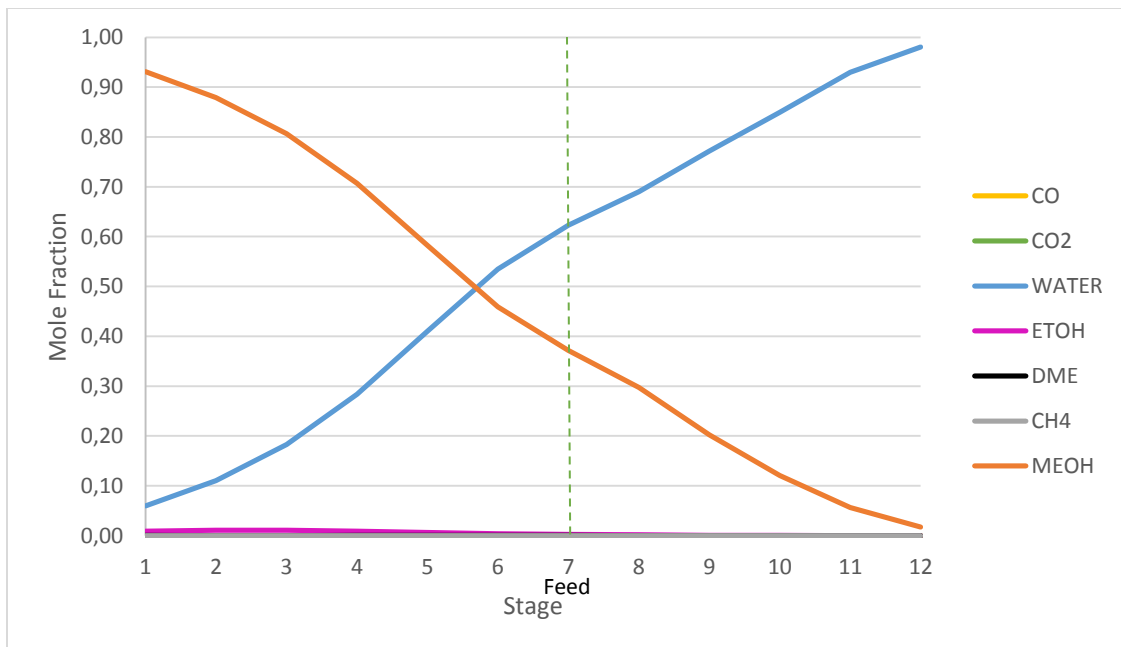


Figure 4: Tray composition by mole fraction for the T-301 Water Separator. The light key-heavy key components are Methanol-water. Inlet feed stage is stage 7.

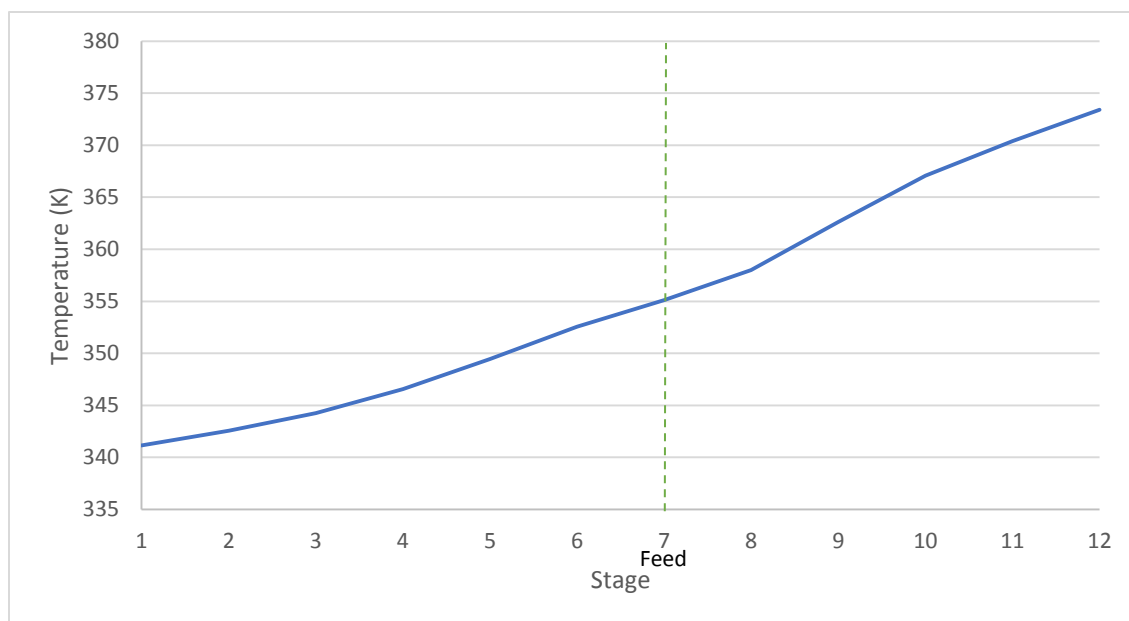


Figure 5: Temperature profile for stages of the T-301 Water Separator.

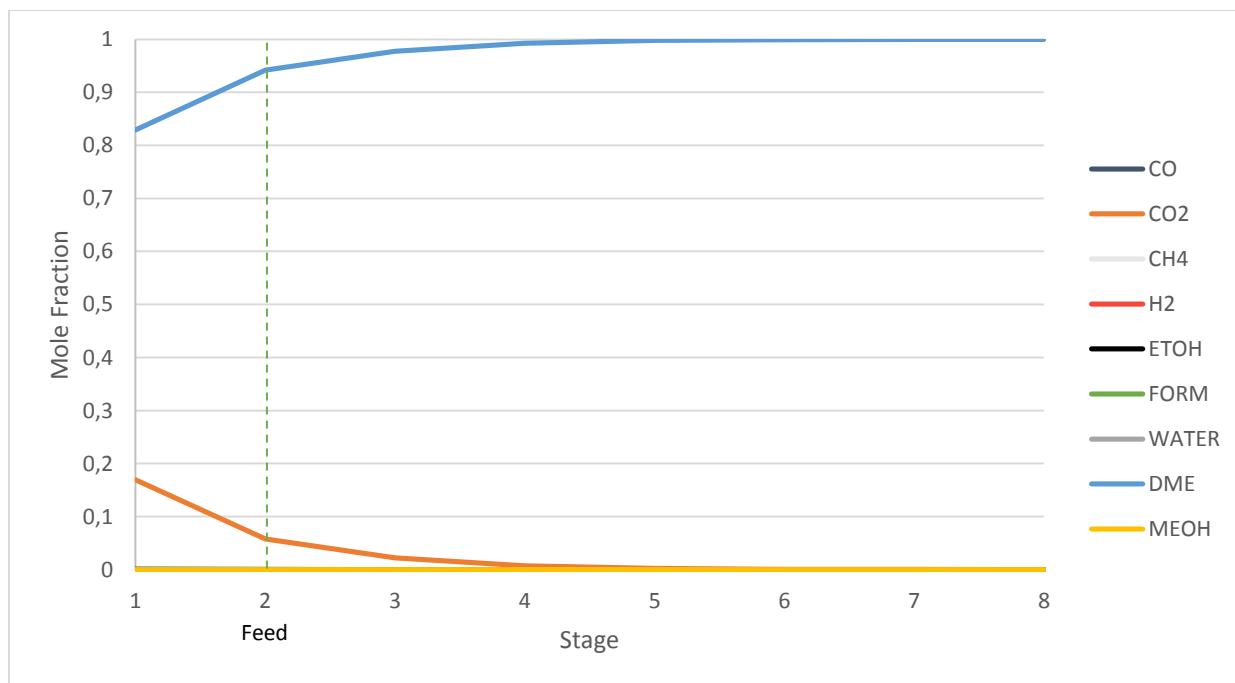


Figure 6: Tray composition by mole fraction for T-201 DME Separator– the final column in the separation process. The light key-heavy key components are carbon dioxide-DME. Inlet feed stage is stage 2.

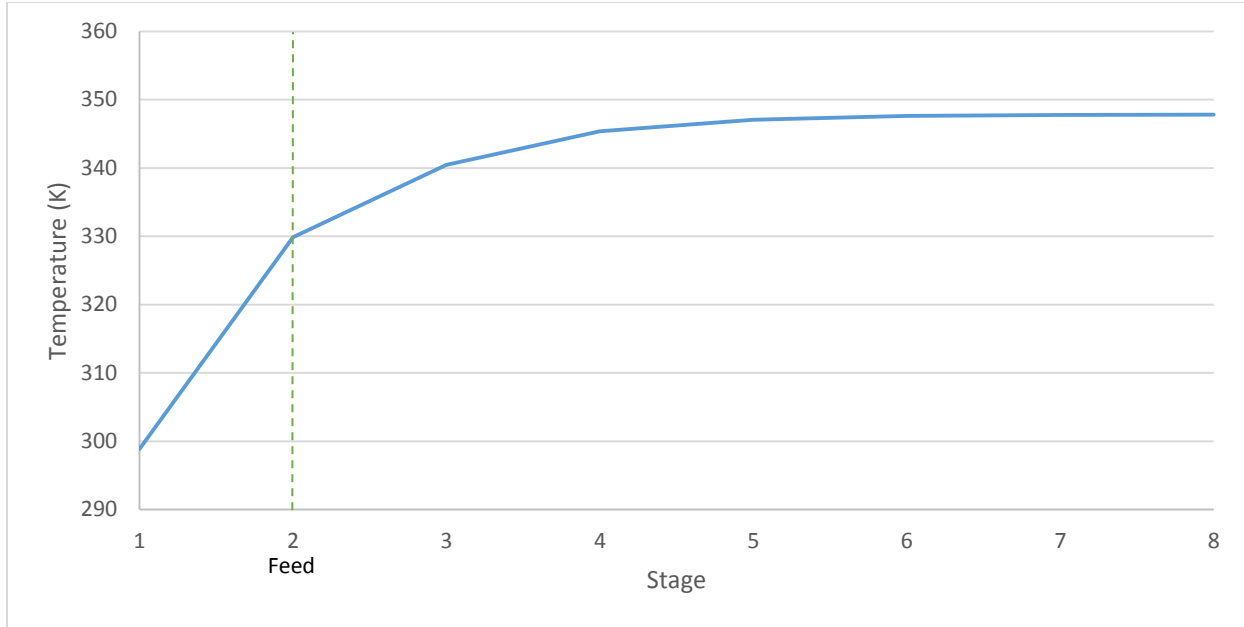


Figure 7: Temperature profile for stages of the T-201 DME Separator.

Calculations for real-world column size was based off ASPEN's modeled number of stages by assuming an average stage efficiency of 70% for a conservative estimate [11]. A 10% oversize was added to the final number of stages as a safety margin. Column height was set with stage spacing of 2 ft, 4 ft of vapor space, and 6 ft of bottoms space to provide appropriate head for the bottoms pumps. The total expected height for each column then follows the outline below:

$$Height = \frac{\# \text{ equilibrium stages}}{0.7} * 2(ft) + 10(ft) \quad 1$$

Tower diameter was calculated using Equation 3 taken from column sizing heuristics, using the column volumetric flow rate and gas velocity Equation 2 [1].

$$F_s = u\rho^{1/2} \quad 2$$

Where:

F_s = vapor factor

u = gas velocity

ρ_u = gas density

$$D_{tower} = \left[\frac{4v}{\pi u} \right]^{1/2} \quad 3$$

Where :

v = volumetric flow rate

Tower pressure drop was calculated using heuristics leading to Equation 4 below:

$$\Delta P_{tower} = \frac{N_{actual}}{\Delta P_{tray}} \quad 4$$

The final expected column sizes are shown below in Table 3.

Table 3: DME separation column sizing and tray details

	MEOH/DME	DME/CO2	MEOH/H2O
Modeled number of stages	10	12	8
Expected number of stages	16	19	13
Column height (m)	12.8	14.6	11.0
Column diameter (m)	0.76	0.52	0.82
Pressure drop (bar)	0.11	0.13	0.09
Modeled feed stage	7	2	7
Expected feed stage	11	3	11

Heat exchangers

For heat exchanger sizing calculations, Equation 5 was used to estimate heat exchanger areas [12]. The nonideality factor, f , accounts for imperfect countercurrent flow in shell and tube exchangers due to the flow tortuosity from baffles. The factor was assumed to be 0.85 in shell-and-tube exchangers and 1 in plate-and-frame exchangers.

$$Q = fUA\Delta T_{lm} \quad 5$$

Where:

Q : rate of heat transfer

f : nonideality factor for countercurrent flow

U : overall heat transfer coefficient

A : heat exchange area

ΔT_{lm} : log-mean temperature difference

The log-mean temperature difference is defined by Equation 6 - 8.

$$\Delta T_{lm} = \frac{\Delta T_1 - \Delta T_2}{\ln\left(\frac{\Delta T_1}{\Delta T_2}\right)} \quad 6$$

$$\Delta T_1 = (T_{hot,in} - T_{cold,out}) \quad 7$$

$$\Delta T_2 = (T_{hot,out} - T_{cold,in}) \quad 8$$

Overall heat transfer coefficients were estimated by the general guidelines outlined by Table 4 [13].

Table 4: Estimated heat transfer coefficients for separate systems [13]

Utility	Process side	Overall heat transfer coefficient (J/m ² s K)	
		Lower limit	Upper limit
Water	Water	900	1000
	Alcohols	600	700
	condensing alcohol	800	1100
	condensing organic solvents (w/ inert gas)	200	700
	condensing water	1200	1400
	non-condensing gases	90	120
	low viscosity organics	550	650
Steam	Water	1200	1400
	Alcohols	800	1000
	Boiling alcohols	1200	1400
	Light hydrocarbons	1200	1400

To generate a heat transfer coefficient for multi-component streams, a weighted molar average of the stream's major components and Table 4 was used. In each case, the major components do not make up the entirety of the stream, non –major components were assumed to be non-condensable gases; Equation 9 describes this.

$$U_{mixed} = \sum_i U_i x_i \quad 9$$

Where:

x_i is the stream mole fraction of some component i .

For sizing in each of the exchangers, the lower limit of the heat transfer ranges in

Table 4 were selected to give a conservative estimate on exchanger size. Additionally, a 20% oversize for exchange area was added. Each of the distillation columns was chosen to be kettle type, and all condensers or exchangers that dealt with phase change were chosen to be shell and tube. Orientation of the exchangers depended on outlet conditions: if condensed liquids needed to be separated from gases, the exchanger is horizontal with the shell side being the process side to let liquids drain from the accumulation area. If the flow does not need to be separated but there is partial condensation, such as in the feed stream cooler, the process side was the tube-side. Plate and frame style was chosen for all liquid-liquid exchangers to conserve space. Final area for each exchanger and comments on design choices are outlined in A.2 Stream Summaries

Stream Table for Unit 100

	Units	1	4	5	6	10
Description						

From			PRE-COOL	OH-COL	OH-COL	COMP-1
To		B1	OH-COL	COMP-1	H2O-COOL	IS-COOL
Temperature	C	3.50E+02	8.00E+01	2.75E+01	1.38E+02	8.31E+01
Pressure	bar	1.10E+01	1.00E+01	7.00E+00	7.00E+00	2.10E+01
Molar Density	mol/cc	2.12E-04	1.50E-03	2.80E-04	3.50E-02	7.09E-04
Mass Density	kg/cum	6.62E+00	4.66E+01	1.26E+01	7.65E+02	3.19E+01
Molar Vapor Fraction		1.00E+00	2.18E-01	1.00E+00	0.00E+00	1.00E+00
Mole Flows	kmol/hr	4.31E+02	4.31E+02	1.74E+02	2.58E+02	1.74E+02
CO	kmol/hr	1.62E+00	1.62E+00	1.62E+00	2.10E-12	1.62E+00
CO2	kmol/hr	1.21E+01	1.21E+01	1.21E+01	1.11E-06	1.21E+01
CH4	kmol/hr	1.00E-02	1.00E-02	1.00E-02	9.26E-13	1.00E-02
H2	kmol/hr	3.04E+00	3.04E+00	3.04E+00	4.84E-17	3.04E+00
ETOH	kmol/hr	3.00E-01	3.00E-01	2.89E-12	3.00E-01	2.89E-12
FORM	kmol/hr	2.20E-01	2.20E-01	1.25E-04	2.20E-01	1.25E-04
WATER	kmol/hr	1.88E+02	1.88E+02	3.66E-11	1.88E+02	3.66E-11
DME	kmol/hr	1.58E+02	1.58E+02	1.57E+02	7.20E-01	1.57E+02
MEOH	kmol/hr	6.87E+01	6.87E+01	5.56E-05	6.87E+01	5.56E-05
Mole Fractions						
CO		3.76E-03	3.76E-03	9.32E-03	8.16E-15	9.32E-03
CO2		2.80E-02	2.80E-02	6.94E-02	4.32E-09	6.94E-02
CH4		2.32E-05	2.32E-05	5.76E-05	3.59E-15	5.76E-05
H2		7.05E-03	7.05E-03	1.75E-02	1.88E-19	1.75E-02
ETOH		6.95E-04	6.95E-04	1.66E-14	1.16E-03	1.66E-14
FORM		5.10E-04	5.10E-04	7.18E-07	8.53E-04	7.18E-07
WATER		4.35E-01	4.35E-01	2.11E-13	7.28E-01	2.11E-13
DME		3.66E-01	3.66E-01	9.04E-01	2.80E-03	9.04E-01
MEOH		1.59E-01	1.59E-01	3.20E-07	2.67E-01	3.20E-07
Mass Flows	kg/hr	1.35E+04	1.35E+04	7.82E+03	5.64E+03	7.82E+03
CO	kg/hr	4.54E+01	4.54E+01	4.54E+01	5.89E-11	4.54E+01
CO2	kg/hr	5.31E+02	5.31E+02	5.31E+02	4.90E-05	5.31E+02
CH4	kg/hr	1.60E-01	1.60E-01	1.60E-01	1.49E-11	1.60E-01
H2	kg/hr	6.13E+00	6.13E+00	6.13E+00	9.75E-17	6.13E+00
ETOH	kg/hr	1.38E+01	1.38E+01	1.33E-10	1.38E+01	1.33E-10
FORM	kg/hr	6.61E+00	6.61E+00	3.74E-03	6.60E+00	3.74E-03
WATER	kg/hr	3.38E+03	3.38E+03	6.59E-10	3.38E+03	6.59E-10
DME	kg/hr	7.27E+03	7.27E+03	7.23E+03	3.32E+01	7.23E+03
MEOH	kg/hr	2.20E+03	2.20E+03	1.78E-03	2.20E+03	1.78E-03
Mass Fractions						
CO		3.37E-03	3.37E-03	5.81E-03	1.04E-14	5.81E-03
CO2		3.95E-02	3.95E-02	6.79E-02	8.69E-09	6.79E-02
CH4		1.19E-05	1.19E-05	2.05E-05	2.64E-15	2.05E-05

H2		4.56E-04	4.56E-04	7.84E-04	1.73E-20	7.84E-04
ETOH		1.03E-03	1.03E-03	1.70E-14	2.45E-03	1.70E-14
FORM		4.91E-04	4.91E-04	4.79E-07	1.17E-03	4.79E-07
WATER		2.51E-01	2.51E-01	8.44E-14	6.00E-01	8.44E-14
DME		5.40E-01	5.40E-01	9.25E-01	5.89E-03	9.25E-01
MEOH		1.64E-01	1.64E-01	2.28E-07	3.91E-01	2.28E-07
Volume Flow	cum/hr	2.03E+03	2.88E+02	6.20E+02	7.36E+00	2.45E+02

Stream Summary Table for Unit 200

	Units	11	13	FUELGAS(18)	7
Description					
From		IS-COOL	B5	DME-CO2	DME-CO2
To		B5	DME-CO2		B3
Temperature	C	5.00E+01	3.00E+01	2.57E+01	7.47E+01
Pressure	bar	2.00E+01	2.00E+01	2.00E+01	2.00E+01
Molar Density	mol/cc	4.47E-03	7.82E-03	8.05E-04	1.22E-02
Mass Density	kg/cum	2.01E+02	3.52E+02	3.03E+01	5.62E+02
Molar Vapor Fraction		1.18E-01	4.88E-02	1.00E+00	0.00E+00
Mole Flows	kmol/hr	1.74E+02	1.74E+02	2.22E+01	1.51E+02
CO	kmol/hr	1.62E+00	1.62E+00	1.62E+00	8.46E-12
CO2	kmol/hr	1.21E+01	1.21E+01	1.21E+01	1.92E-03
CH4	kmol/hr	1.00E-02	1.00E-02	1.00E-02	3.66E-11
H2	kmol/hr	3.04E+00	3.04E+00	3.04E+00	4.33E-19
ETOH	kmol/hr	2.89E-12	2.89E-12	3.47E-17	2.89E-12
FORM	kmol/hr	1.25E-04	1.25E-04	2.61E-06	1.22E-04
WATER	kmol/hr	3.66E-11	3.66E-11	1.80E-16	3.66E-11
DME	kmol/hr	1.57E+02	1.57E+02	5.51E+00	1.51E+02
MEOH	kmol/hr	5.56E-05	5.56E-05	1.79E-08	5.56E-05
Mole Fractions					
CO		9.32E-03	9.32E-03	7.29E-02	5.58E-14
CO2		6.94E-02	6.94E-02	5.42E-01	1.27E-05
CH4		5.76E-05	5.76E-05	4.50E-04	2.42E-13
H2		1.75E-02	1.75E-02	1.37E-01	2.86E-21
ETOH		1.66E-14	1.66E-14	1.56E-18	1.91E-14
FORM		7.18E-07	7.18E-07	1.17E-07	8.06E-07
WATER		2.11E-13	2.11E-13	8.09E-18	2.42E-13
DME		9.04E-01	9.04E-01	2.48E-01	1.00E+00
MEOH		3.20E-07	3.20E-07	8.04E-10	3.67E-07
Mass Flows	kg/hr	7.82E+03	7.82E+03	8.36E+02	6.98E+03
CO	kg/hr	4.54E+01	4.54E+01	4.54E+01	2.37E-10
CO2	kg/hr	5.31E+02	5.31E+02	5.31E+02	8.46E-02
CH4	kg/hr	1.60E-01	1.60E-01	1.60E-01	5.88E-10
H2	kg/hr	6.13E+00	6.13E+00	6.13E+00	8.74E-19

ETOH	kg/hr	1.33E-10	1.33E-10	1.60E-15	1.33E-10
FORM	kg/hr	3.74E-03	3.74E-03	7.84E-05	3.67E-03
WATER	kg/hr	6.59E-10	6.59E-10	3.24E-15	6.59E-10
DME	kg/hr	7.23E+03	7.23E+03	2.54E+02	6.98E+03
MEOH	kg/hr	1.78E-03	1.78E-03	5.73E-07	1.78E-03
Mass Fractions					
CO		5.81E-03	5.81E-03	5.43E-02	3.39E-14
CO2		6.79E-02	6.79E-02	6.35E-01	1.21E-05
CH4		2.05E-05	2.05E-05	1.92E-04	8.42E-14
H2		7.84E-04	7.84E-04	7.33E-03	1.25E-22
ETOH		1.70E-14	1.70E-14	1.91E-18	1.91E-14
FORM		4.79E-07	4.79E-07	9.38E-08	5.25E-07
WATER		8.44E-14	8.44E-14	3.87E-18	9.45E-14
DME		9.25E-01	9.25E-01	3.04E-01	1.00E+00
MEOH		2.28E-07	2.28E-07	6.85E-10	2.55E-07
Volume Flow	cum/hr	3.89E+01	2.22E+01	2.76E+01	1.24E+01

Stream Summary Table for Unit 200 (continued)

	Units	14	15	DME(19)
Description				
From		PRE-COOL	B9	B8
To		OH-COL	B8	
Temperature	C	8.00E+01	4.82E+01	3.00E+01
Pressure	bar	1.00E+01	1.10E+01	8.00E+00
Molar Density	mol/cc	1.50E-03	8.94E-03	1.40E-02
Mass Density	kg/cum	4.66E+01	4.12E+02	6.46E+02
Molar Vapor Fraction		2.18E-01	1.57E-02	0.00E+00
Mole Flows	kmol/hr	4.31E+02	1.51E+02	1.51E+02
CO	kmol/hr	1.62E+00	8.46E-12	8.46E-12
CO2	kmol/hr	1.21E+01	1.92E-03	1.92E-03
CH4	kmol/hr	1.00E-02	3.66E-11	3.66E-11
H2	kmol/hr	3.04E+00	4.33E-19	4.33E-19
ETOH	kmol/hr	3.00E-01	2.89E-12	2.89E-12
FORM	kmol/hr	2.20E-01	1.22E-04	1.22E-04
WATER	kmol/hr	1.88E+02	3.66E-11	3.66E-11
DME	kmol/hr	1.58E+02	1.51E+02	1.51E+02
MEOH	kmol/hr	6.87E+01	5.56E-05	5.56E-05
Mole Fractions				
CO		3.76E-03	5.58E-14	5.58E-14
CO2		2.80E-02	1.27E-05	1.27E-05
CH4		2.32E-05	2.42E-13	2.42E-13
H2		7.05E-03	2.86E-21	2.86E-21
ETOH		6.95E-04	1.91E-14	1.91E-14

FORM		5.10E-04	8.06E-07	8.06E-07
WATER		4.35E-01	2.42E-13	2.42E-13
DME		3.66E-01	1.00E+00	1.00E+00
MEOH		1.59E-01	3.67E-07	3.67E-07
Mass Flows	kg/hr	1.35E+04	6.98E+03	6.98E+03
CO	kg/hr	4.54E+01	2.37E-10	2.37E-10
CO2	kg/hr	5.31E+02	8.46E-02	8.46E-02
CH4	kg/hr	1.60E-01	5.88E-10	5.88E-10
H2	kg/hr	6.13E+00	8.74E-19	8.74E-19
ETOH	kg/hr	1.38E+01	1.33E-10	1.33E-10
FORM	kg/hr	6.61E+00	3.67E-03	3.67E-03
WATER	kg/hr	3.38E+03	6.59E-10	6.59E-10
DME	kg/hr	7.27E+03	6.98E+03	6.98E+03
MEOH	kg/hr	2.20E+03	1.78E-03	1.78E-03
Mass Fractions				
CO		3.37E-03	3.39E-14	3.39E-14
CO2		3.95E-02	1.21E-05	1.21E-05
CH4		1.19E-05	8.42E-14	8.42E-14
H2		4.56E-04	1.25E-22	1.25E-22
ETOH		1.03E-03	1.91E-14	1.91E-14
FORM		4.91E-04	5.25E-07	5.25E-07
WATER		2.51E-01	9.45E-14	9.45E-14
DME		5.40E-01	1.00E+00	1.00E+00
MEOH		1.64E-01	2.55E-07	2.55E-07
Volume Flow	cum/hr	2.88E+02	1.69E+01	1.08E+01

Stream Table for Unit 300

	Units	21(toH2OCol)	Fuel(23)	BULKMEOH(20)
Description				
From		H2O-COOL	MEOH-H2O	B6
To		MEOH-H2O		
Temperature	C	8.00E+01	5.15E+01	1.27E+00
Pressure	bar	8.00E+00	1.10E+00	5.00E+00
Molar Density	mol/cc	3.82E-02	4.07E-05	2.60E-02
Mass Density	kg/cum	8.36E+02	1.58E+00	8.23E+02
Molar Vapor Fraction		0.00E+00	1.00E+00	0.00E+00
Mole Flows	kmol/hr	2.58E+02	5.81E-01	7.03E+01
CO	kmol/hr	2.10E-12	1.87E-12	2.32E-13
CO2	kmol/hr	1.11E-06	5.05E-07	6.08E-07
CH4	kmol/hr	9.26E-13	7.23E-13	2.03E-13
H2	kmol/hr	4.84E-17	0.00E+00	0.00E+00
ETOH	kmol/hr	3.00E-01	7.58E-04	2.99E-01
FORM	kmol/hr	2.20E-01	2.21E-09	6.23E-06

WATER	kmol/hr	1.88E+02	4.18E-03	2.68E+00
DME	kmol/hr	7.20E-01	2.77E-01	4.43E-01
MEOH	kmol/hr	6.87E+01	2.99E-01	6.68E+01
Mole Fractions				
CO		8.16E-15	3.22E-12	3.30E-15
CO2		4.32E-09	8.69E-07	8.66E-09
CH4		3.59E-15	1.24E-12	2.88E-15
H2		1.88E-19	0.00E+00	0.00E+00
ETOH		1.16E-03	1.30E-03	4.25E-03
FORM		8.53E-04	3.80E-09	8.87E-08
WATER		7.28E-01	7.19E-03	3.81E-02
DME		2.80E-03	4.77E-01	6.30E-03
MEOH		2.67E-01	5.14E-01	9.51E-01
Mass Flows	kg/hr	5.64E+03	2.25E+01	2.22E+03
CO	kg/hr	5.89E-11	5.24E-11	6.49E-12
CO2	kg/hr	4.90E-05	2.22E-05	2.68E-05
CH4	kg/hr	1.49E-11	1.16E-11	3.25E-12
H2	kg/hr	9.75E-17	0.00E+00	0.00E+00
ETOH	kg/hr	1.38E+01	3.49E-02	1.38E+01
FORM	kg/hr	6.60E+00	6.63E-08	1.87E-04
WATER	kg/hr	3.38E+03	7.53E-02	4.83E+01
DME	kg/hr	3.32E+01	1.28E+01	2.04E+01
MEOH	kg/hr	2.20E+03	9.58E+00	2.14E+03
Mass Fractions				
CO		1.04E-14	2.33E-12	2.92E-15
CO2		8.69E-09	9.89E-07	1.20E-08
CH4		2.64E-15	5.16E-13	1.46E-15
H2		1.73E-20	0.00E+00	0.00E+00
ETOH		2.45E-03	1.55E-03	6.19E-03
FORM		1.17E-03	2.95E-09	8.41E-08
WATER		6.00E-01	3.35E-03	2.17E-02
DME		5.89E-03	5.69E-01	9.17E-03
MEOH		3.91E-01	4.26E-01	9.63E-01
Volume Flow	cum/hr	6.74E+00	1.43E+01	2.70E+00

Stream Table for Unit 300 (continued)

	Units	8	9	WASTE H2O(22)
Description				
From		MEOH-H2O	B7	B2
To		B7	B2	
Temperature	C	1.00E+02	5.01E+01	5.01E+01
Pressure	bar	1.10E+00	5.00E+00	5.00E+00
Molar Density	mol/cc	5.03E-02	5.32E-02	5.32E-02
Mass Density	kg/cum	9.13E+02	9.65E+02	9.65E+02
Molar Vapor		0.00E+00	0.00E+00	0.00E+00

Fraction				
Mole Flows	kmol/hr	1.87E+02	1.87E+02	1.87E+02
CO	kmol/hr	7.14E-34	7.14E-34	7.14E-34
CO2	kmol/hr	2.71E-22	2.71E-22	2.71E-22
CH4	kmol/hr	6.48E-32	6.48E-32	6.48E-32
H2	kmol/hr	0.00E+00	0.00E+00	0.00E+00
ETOH	kmol/hr	5.19E-04	5.19E-04	5.19E-04
FORM	kmol/hr	2.20E-01	2.20E-01	2.20E-01
WATER	kmol/hr	1.85E+02	1.85E+02	1.85E+02
DME	kmol/hr	5.98E-11	5.98E-11	5.98E-11
MEOH	kmol/hr	1.56E+00	1.56E+00	1.56E+00
Mole Fractions				
CO		3.82E-36	3.82E-36	3.82E-36
CO2		1.45E-24	1.45E-24	1.45E-24
CH4		3.47E-34	3.47E-34	3.47E-34
H2		0.00E+00	0.00E+00	0.00E+00
ETOH		2.78E-06	2.78E-06	2.78E-06
FORM		1.18E-03	1.18E-03	1.18E-03
WATER		9.90E-01	9.90E-01	9.90E-01
DME		3.20E-13	3.20E-13	3.20E-13
MEOH		8.36E-03	8.36E-03	8.36E-03
Mass Flows	kg/hr	3.39E+03	3.39E+03	3.39E+03
CO	kg/hr	2.00E-32	2.00E-32	2.00E-32
CO2	kg/hr	1.19E-20	1.19E-20	1.19E-20
CH4	kg/hr	1.04E-30	1.04E-30	1.04E-30
H2	kg/hr	0.00E+00	0.00E+00	0.00E+00
ETOH	kg/hr	2.39E-02	2.39E-02	2.39E-02
FORM	kg/hr	6.60E+00	6.60E+00	6.60E+00
WATER	kg/hr	3.33E+03	3.33E+03	3.33E+03
DME	kg/hr	2.75E-09	2.75E-09	2.75E-09
MEOH	kg/hr	5.01E+01	5.01E+01	5.01E+01
Mass Fractions				
CO		5.90E-36	5.90E-36	5.90E-36
CO2		3.52E-24	3.52E-24	3.52E-24
CH4		3.07E-34	3.07E-34	3.07E-34
H2		0.00E+00	0.00E+00	0.00E+00
ETOH		7.06E-06	7.06E-06	7.06E-06
FORM		1.95E-03	1.95E-03	1.95E-03
WATER		9.83E-01	9.83E-01	9.83E-01
DME		8.12E-13	8.12E-13	8.12E-13
MEOH		1.48E-02	1.48E-02	1.48E-02
Volume Flow	cum/hr	3.71E+00	3.51E+00	3.51E+00

A.3 Equipment list in the Appendix.

Pumps and compressors

The main gas compressor in the system is a centrifugal type, and sized to an operating pressure of 23 bar, with a 3 bar pressure difference between the compressor outlet and the DME Separator column. The modeled pressure difference should account for overestimated pressure losses from piping and process equipment. Under this design, a compressor efficiency of 70% (for both mechanical and isentropic) was assumed. By including the overdesign in the model, ASPEN was able to calculate the power requirements with greater accuracy than would have been possible with manual calculations. The final power requirement estimate is 155 kW.

Feed pumps were also assumed to operate at 70% efficiency, and each one is a centrifugal pump. To account for unexpected head losses in the system, each pump was oversized by 25% [11].

Pump sizes were based on Equation 10 [12].

$$P = 1.67F * \frac{\Delta P}{\varepsilon} \quad 10$$

Where

P : Power requirement, kw

F : Volumetric flow rate, m³/hr

ΔP : Differential pressure across the pump, bar

ε : Pump efficiency

Volumetric flow rates are available in the ASPEN stream summaries but differential pressures are estimated from densities.

$$\Delta P = 1 \text{ bar} + \rho g \Delta h \quad 11$$

Where

ρ : Fluid density

g : Gravitational constant, 9.8 m/s²

Δh : Difference in fluid height between on inlet and outlet sides

Under this equation, an additional 1 bar differential pressure was added to the calculation to cover losses from general process equipment, control valves, and in the case of reflux pumps, the differential pressure between the reflux tank and the feed stage. Additionally, it was assumed that the fluid level in the reflux drum was roughly equal to the fluid level in the bottoms space of the distillation column. Differential height, then, is calculated from the number of stages times the tray spacing, written in Equation 12.

$$\Delta h = (\text{Total number of stages}) * 2 \text{ ft} \quad 12$$

Reflux drums

All reflux drums are designed to be horizontal, and drums V-101 for the T-101 Methanol Separator and V-201 for the T-201 DME Separator were designed to have a residence time of five minutes for column reflux when half full [11]. The total volume calculation for these were based on the fluid density and reflux rate (kmol/hr) estimated by ASPEN and under the assumption that the reflux was nearly all DME as it is the main component for both columns.

The distillate receiver V-301 for the T-301 Water Separator has a much larger size. At half full it has a residency of 20 minutes for the reflux because it also acts as a storage tank for the liquid methanol distillate to account for front end fluctuations in methanol demand. This vessel is meant to operate slightly above atmospheric with a vent to a flare so that non-condensable materials do not create a backpressure in the drum during operation. Sizing for this drum follows the same principles as the others, save for the fluid is now methanol and the residence time is increased.

The conversion from reflux rate to drum size is in Equation 13.

$$\text{Drum volume} = \text{Reflux rate} * \text{Molecular wt} / \text{Density} \quad 13$$

The drums are all assumed to be cylinders with a length: diameter ratio of 3 [11].

Controls

Pressure relief valves are at the entrance of reactor effluent stream 1, and water effluent stream from cooler E-303. The flow streams in this separation process fitted with valves to manipulate the stream flowrate to maintain a desirable separation process condition. Most of the valves were manually controlled hand valves with the exceptions of the valves installed on the reflux drum V-101, V-201, and V-301. The reflux rate from the reflux drums V-101, V-201, and V-301 were controlled by an automatic valve that is fitted with a flow indicator controller. This flow indicator and controller (FIC) would send a signal to an operator in the control room who would then send a pneumatic signal to the valve to move the valve to the desired position. Level indicator controllers (LIC) were installed at the bottom of the distillation towers T-101, T-201, and T-301 to monitor and control the level of the liquid bottom by manipulating the flow of the liquid bottom effluent stream. These indicators and controllers are a combination of transmitter and controller that works together to control flowrates. Pressure controller PC is used to monitor and control the pressure in the separator column by controlling the flowrate of the cooling water in the condenser to increase the rate of condensation.

Sensitivity analysis

The UNIQUAC thermodynamic package was determined to be the most appropriate for estimating system-wide interactions. To gain additional insight on separation conditions, the design was also tested using the NRTL and PR methods. As stated before, PR can result inaccurate errors during distillation processes. However, because of its close fit experimental data and the elevated pressure of the DME Separator, this method was tested anyway. Final process results for each thermodynamic package is outlined in Table 5 below.

Table 5: Results of DME and methanol recovery for the UNIQUAC, NRTL, and Peng Robinson thermodynamic models

Package	DME Product Stream			Methanol Recovery Stream	
	% Recovery	PPM Carbon components	PPM Methanol	% recovery	wt %
UNIQUAC	96.1	12	0.26	97.3	96.3
NRTL	96.1	6	0.43	97.2	96.2

Peng Robinson	95.4	5540	0.43	98.1	96.5
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Both the UNIQUAC and NRTL models suggested that the final process streams fit well within specifications for purity and recovery. The Peng Robinson model only fell out of spec on carbon impurities, but the result was severely different than the other two models. Unfortunately, discontinuities in temperature and vapor/liquid fractions was seen in the distillation columns; this brings the reliability of the model into question.

To understand sensitivity of the system under the UNIQUAC model and process fluctuations, results were assed when major feed components (DME, carbon dioxide, methanol, and water) were varied by steps of 1 kmol/hr. These simulations were done with an additional stage added to the original number to account for the margin of safety set into a physical system. Upper and lower estimates for each component flow is listed in

Table 6 where the process can still results in in-specification products.

Table 6: Expected upper and lower flow rates for major feed components that produce in-spec products

Component	Lower	Expected (kmol/hr)	Upper
Carbon Dioxide	11	12.06	13
DME	157	157.72	159
Methanol	68	68.71	70
Water	186	187.69	189

The system does handle some fluctuations but not major ones. In each case, methanol impurities in the DME product and the required weight fraction for recycled methanol were the limiting factors of variation for the process. Impurity of methanol in DME product jumped very quickly, from being in spec for one flow rate but having impurities in the thousands of ppm out of spec for a change in flow rate of just 1 kmol/hr. It is thought that the high variation is from the calculation method in which ASPEN determines stream compositions. In each column, the distillate flow rate is estimated as a fraction of the feed. That is, if a small amount of DME is removed from the system and replaced with unreacted methanol, that methanol is expected to show up mostly in the Methanol Separator distillate. Regardless, if this is the only cause for the high fluctuations in stream composition, it is worth studying further.

Finally, the model was ran when temperature streams feeding into each distillation column were modified one at a time. The temperatures were either increased or decreased by 5°C, which are expected to be the bounds on the process's exchangers during normal operation, shown in Table 7.

Table 7: ASPEN model results for stream temperature deviations of +/-5°C

Change	Streams impacted	DME Product Stream			Methanol Recycle	
		% Recovery	PPM Carbons	PPM Methanol	% recovery	Wt %
+5°C	4	96.1	12	0.33	97.3	96.3
	13, 21	96.1	19	0.26	97.3	96.3
	4, 13, 21	96.0	19	0.33	97.3	96.3
-5°C	4	96.1	12	0.20	97.3	96.3
	13, 21	96.1	8	0.26	97.3	96.3

In general, the process can sustain product streams well within spec despite reasonable fluctuations in temperatures feeding to the distillation columns. The component that fluctuates the most with feed temperatures was the carbon impurities in the DME product. The area's exchangers could be tweaked to save on utilities, but the way they are designed now should provide protection against even system wide fluctuations.

Of particular economic concern is the operating conditions of the distillation towers. Maintaining high temperatures

Materials of construction

Because of the presence of formaldehyde and the likely formation of carbonic acid, all major equipment is recommended to be made of stainless steel or higher grade metals. Formaldehyde and carbonic acid both present corrosion concerns for carbon steel [14]. Stainless is the first choice because of its resistance and availability.

Utility usage and economic impacts

Besides recovery of methanol and DME, utility usage is the next biggest economic concern for operations. Total area utility usage is listed in Table 8 in terms of overall use and in terms of per kg DME product, which will make conversion to dollars much easier. For a complete breakdown of utility usage per piece of equipment, see the Utility Summary table in the Appendix.

Table 8: Estimated area utility usage compared with the amount of DME produced.

Utility	Rate (kg/hr)	Use per kg of DME (kg/kg)
chilled water	309000	43.2
cooling water	725000	106.4
9 bar steam	7000	1.0

Utility	Rate (kW)	Use per kg of DME (kW/kg)
electricity	173.5	0.026

The values shown for water and steam usage are a direct result of the ASPEN plus model. The true values can be expected to be slightly higher because Table 8 does not account for general heat losses to the environment. Electricity usage is a summation of the area's pumps and main compressor's final estimated power requirements; recall that this takes into account overdesign and efficiencies. Utility usage, namely steam and cooling water usage, can be reduced by implementing a heat recovery system on the reactor effluent stream. This is discussed in further details in the next section.

Depending on the pressure in the column, cooler water flow for condenser increases or decreases with fluctuation of the feed flow rate to accommodate for the pressure. That is, losses in cooling ability prior to feeding or jumps in reactor effluent temperature will result in higher cooling usage. Electricity for compression depends on the flow out of the reflux drum V-101. The amount in vapor will be most dominantly impacted by the amount of light gases coming from the reactor.

Of particular economic interest is the operating conditions of each of the distillation columns. Maintaining high temperature and pressure conditions can be extremely pricey, however the discussion of column operating conditions outlines the limiting ranges for operations well. Other interests, such as column size, were considered when determining the reflux ratios of the columns. Increasing reflux demands more cooling capacity in the condenser but reduces the number of stages needed.

Economics were considered especially in cooling operations. By implementing exchangers in series, cooling water can achieve the bulk amount of cooling, which saves on chilled water usage. However, capital investment and installation costs will be increased for purchasing multiple coolers.

Alternative designs

Several alternative designs were considered, particularly to deal with separation of the DME from the fuel gas and carbon dioxide. Initially, stripping columns were considered using either the recovered methanol or wastewater. The goal of this design was to eliminate the need for a high pressure distillation column and save on utility costs. However, this design was limited by poor recovery (or very high solute flows) and added complexity in removing and liquefying the DME from the solute flow, which offset any predicted savings in utilities.

A second design considered was to compress the DME Separator feed to a lower pressure and use a column with a lower number of stages. To improve purity, a fraction of the final DME product (roughly 60%) was to be recycled to the column to be given a second chance to degas. Incredibly pure DME product was produced, however the level of purity was not necessarily needed and came with an added costs. Additionally, start-up for this process would have been overly complicated. During start up, the final DME product would have been out of specification in terms of purity, and storing the DME for long periods until steady-state was reached was unreasonable.

To remove the start-up issues, the placement of a secondary DME column was considered after the DME Separator column. In the secondary column, a highly pure bottoms stream of DME would have been

sent as product while a mixture of CO₂ and DME would be returned to the primary column to keep recovery of DME high. Again, this design came with added operating costs, plus additional equipment. The added costs ultimately led to deciding against adding this column.

Instead of a secondary distillation column, a flash drum was considered, which would have multiple benefits. First, there is not operating cost for a flash drum, unlike the distillation column. Second, the DME stream pressure dropping to 6 bar would reduce the temperature to under 30°C. This means that the modeled throttling and cooling process would be eliminated, saving costs on utilities. Finally, it would act as a wide spot in the line for processes downline if excess DME was generated. The downside to this design is that a much larger compressor would be needed to feed the DME separation column, in addition to making the column itself larger. This design did not significantly impact DME recovery, so the addition of a flash drum was not implemented, but it is something to consider if purity concerns arise in the future.

This process is only a preliminary design for the separations unit, and several improvements can be made for more detailed designs. One of the most obvious improvements can be made with heat integration of the system. The process feed cooler has a duty of over 4 Gcal/hour which can be recovered and used elsewhere, mainly in the E-204 DME Reboiler. One idea is to install a closed loop hot water system and tank in the plant. The hot water tank could produce a reasonable amount of hot water (95°C) that can be used though the rest of the unit and other parts of the plant if necessary. This installation would reduce both steam and cooling water dependencies by a major amount.

This idea was not implemented because it was not deemed necessary for a preliminary design. Also, implementation and design of a hot water system ultimately depends on requests for adjacent areas in the plant to see if they agree that it would be beneficial. This information was not available during the design process, but can be surveyed and implemented in future steps.

Safety

The proposed design for DME separation manages multiple hazardous materials, which are listed along with potential hazards in Table 9.

Table 9: List of major hazards for materials used in the DME separation process.

	Flammable	Carcinogenic	Corrosive	Asphyxiant
Carbon Monoxide	x			
Methanol	x			
Carbon Dioxide	x			x
Methane	x			x
Dimethyl Ether (DME)	x			x

Ethanol	x		
Formaldehyde	x	x	x

Carbon monoxide, methanol, carbon dioxide, methane, dimethyl ether, ethanol and formaldehyde are flammable in the presence of sparks and open flames, and can lead to explosions if released in vapor form.

The process location is also important to note – if there are other nearby plants that contribute nitrogen dioxide to the atmosphere, a potential methyl nitrite hazard (which is fatal if inhaled) can be created if during a methanol release [14].

Formaldehyde is a known carcinogen, and exposure can lead to organ damage, reproductive disorders and death. It is also miscible in water and corrosive to carbon steel in its liquid phase, which prompted the avoidance of carbon steel when choosing process materials [8].

Hazardous process conditions exist in the DME separation process due to high pressure and high temperature conditions. The process reactor effluent at 350 °C and DME and fuel gas stream at 20 bar increase the overall safety risk of the design.

General recommendations for safety measures in this process include implementing relief valves to combat high pressure release situations, and isolation valves for safe shutdowns. Due to the high flammability of the materials, all relief systems should be sent to an appropriate knockdown drum. Vapors not caught by the drum should be sent to a flare to prevent localized concentrations of explosive gases and vapors. Avoiding ignition sources around the process is a logical step, and we recommend making the area intrinsically safe. Incorporating insulation for high temperature streams and vessels is needed to minimize potential hazards for workers. If materials are to be stored, they should be in secure, well ventilated areas, and protected from sunlight [15].

Environmental Concerns

The production of dimethyl ether (DME) from crude methanol creates various impurities such as carbon monoxide, carbon dioxide, methane, formaldehyde, and ethanol. These materials pose multiple environmental concerns, but the relatively small quantities facilitate manageable conditions from an environmental perspective.

Materials generated such as carbon monoxide, carbon dioxide, ethanol and methane all contribute to atmospheric greenhouse gasses, affecting ozone concentrations and further advancing global climate change [14]. Ethanol and methanol, if released, contribute to smog but will naturally biodegrade, and are removed from the atmosphere with rain. Dimethyl ether and formaldehyde also degrade in the atmosphere within 36 and 45 hours respectively [8]. The main environmental concerns for materials in DME production are contributing to environmental greenhouse gasses; the use of a flare will greatly counteract these pollutants.

Environmental impact could be improved by researching the DME acid catalyst reaction, and optimizing the process reactor. This optimization will lead to improved source reduction as less impurities would be generated, leading to a smaller number of hazardous materials to manage.

Conclusion and recommendations

The preliminary design of the separations unit exceeds design specifications and performs well under multiple thermodynamic packages. Therefore, we believe there is sufficient support for this process design as a reasonable approach for DME purification. The next step in this process development is to examine the discontinuities in under the Peng Robinson model and determine the accuracy of that model's results. While the NRTL and UNIQUAC packages noted the process to be well within spec, these models did not fit experimental data as well as the Peng Robinson model. Once this is addressed, the following step would be to determine the expected fluctuations in reactor effluent temperature and composition.

The sensitivity analysis raised concerns that fluctuations in reactor effluent composition may lead to high methanol impurities in the final DME product. There is some skepticism in these results, as discussed, but they are none the less important to address with further study. If these two next steps still suggest issues with DME production, then additional margins of safety for separations units should be considered or the re-consideration of the alternative designs may be necessary.

Another area of concern warranting further study is the ventilation of the methanol storage drum. Vent sizing and methanol losses from the venting need to be rigorously analyzed. Besides the obvious product losses, the ventilation of methanol may introduce environmental and safety concerns that are not worth entertaining. If ventilation or flaring are precarious, a possible alternative to ventilation is the compression of this stream and mixing with fuel gas for other uses.

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Appendix A

A.1 Thermodynamic model comparisons

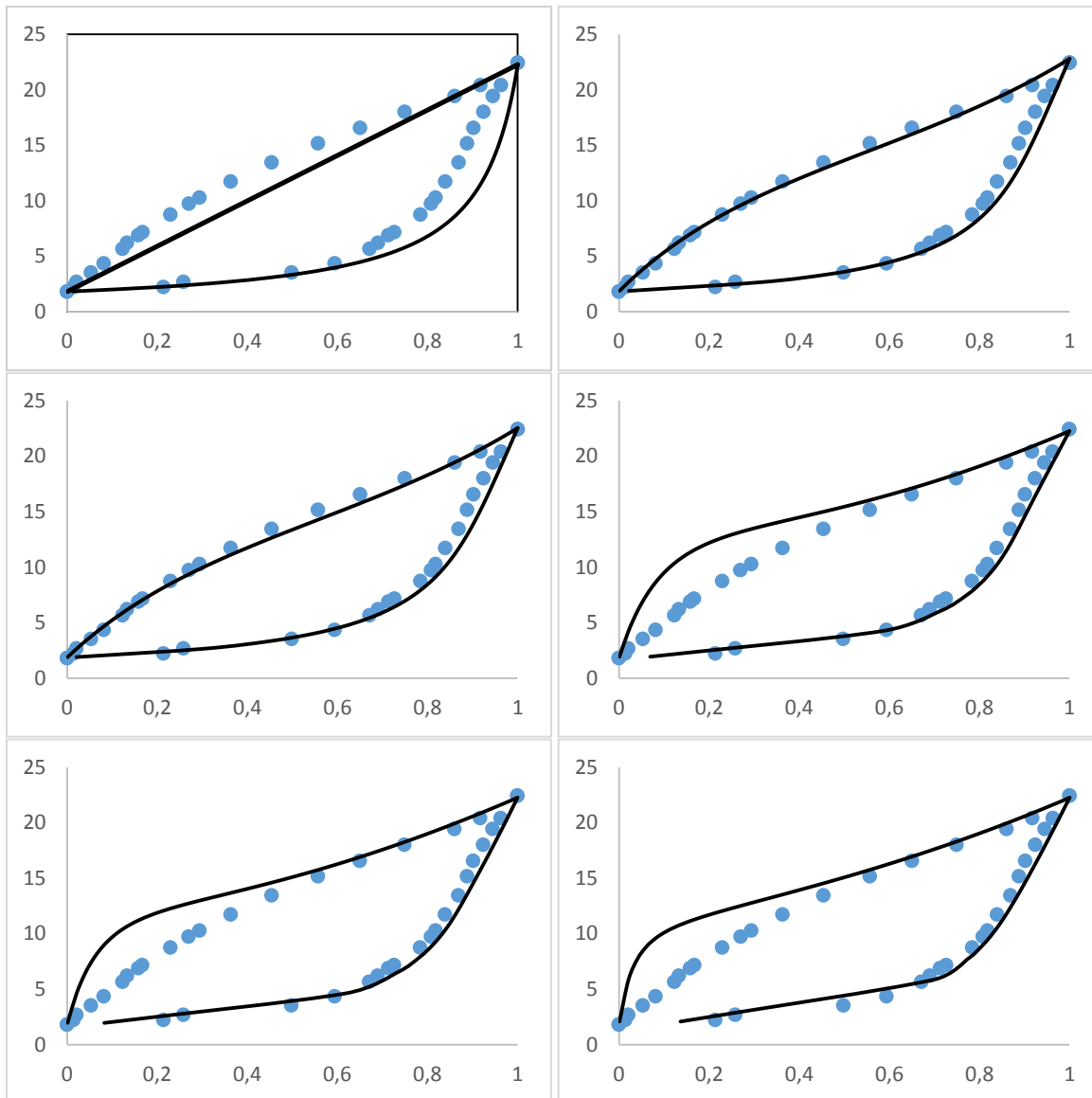


Figure 8: Comparison of VLE experimental data (dots) vs various thermodynamic ASPEN results (lines) for a methanol-DME system at 353 K. From left to right: (row 1) Ideal, SRK, (row 2) Peng Robinson, NRLT, (row 3) UNIQUAC, Wilson. **X-axis:** mol fraction DME **Y-axis:** Pressure (bar) [4]

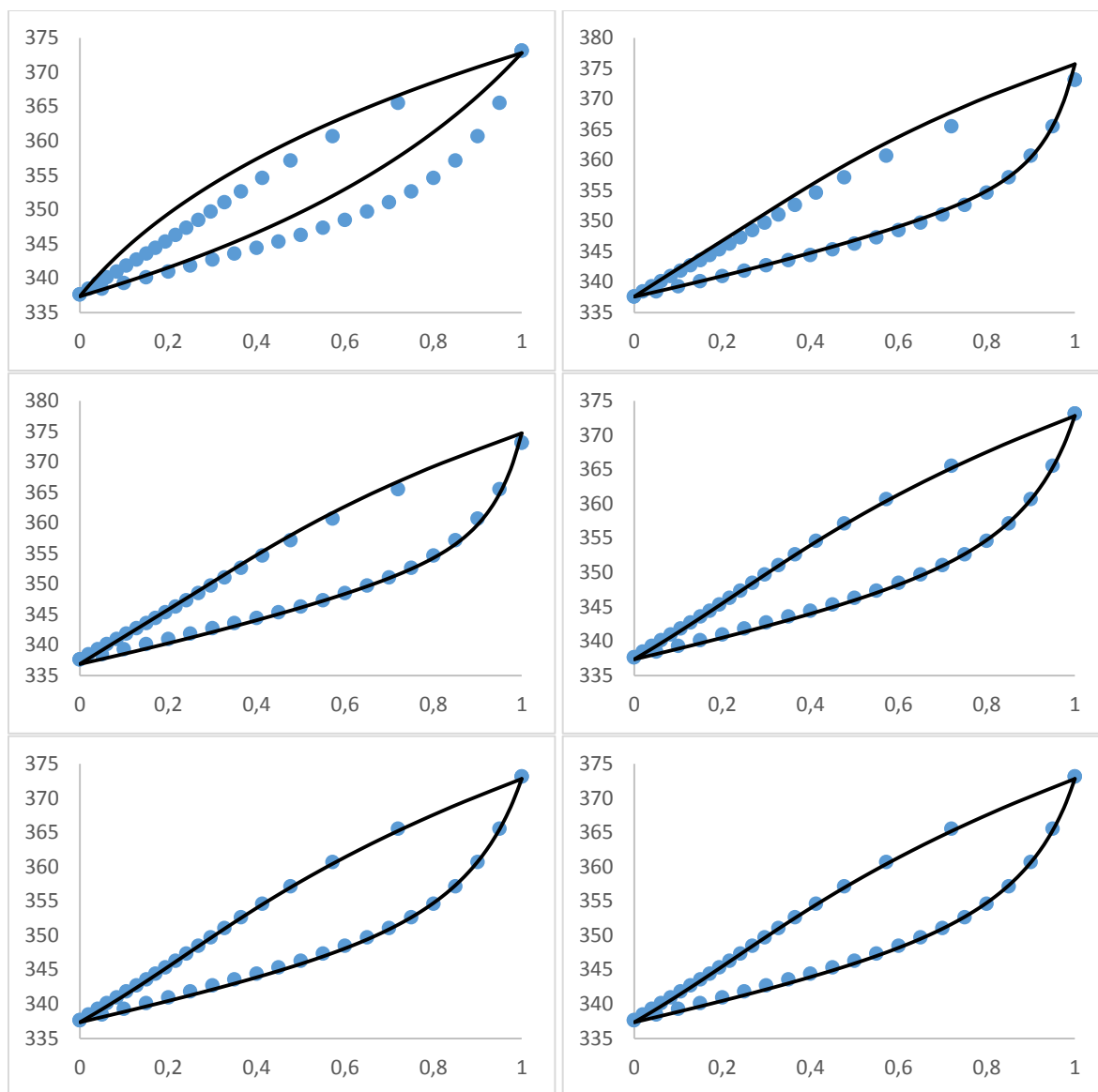


Figure 9: Comparison of VLE experimental data (dots) vs various thermodynamic ASPEN results (lines) for a methanol-water system at 2 bar. From left to right: (row 1) Ideal, SRK, (row 2) Peng Robinson, NRTL, (row 3) UNIQUAC, Wilson. X-axis: mol fraction water Y-axis: temperature, Kelvin [6]

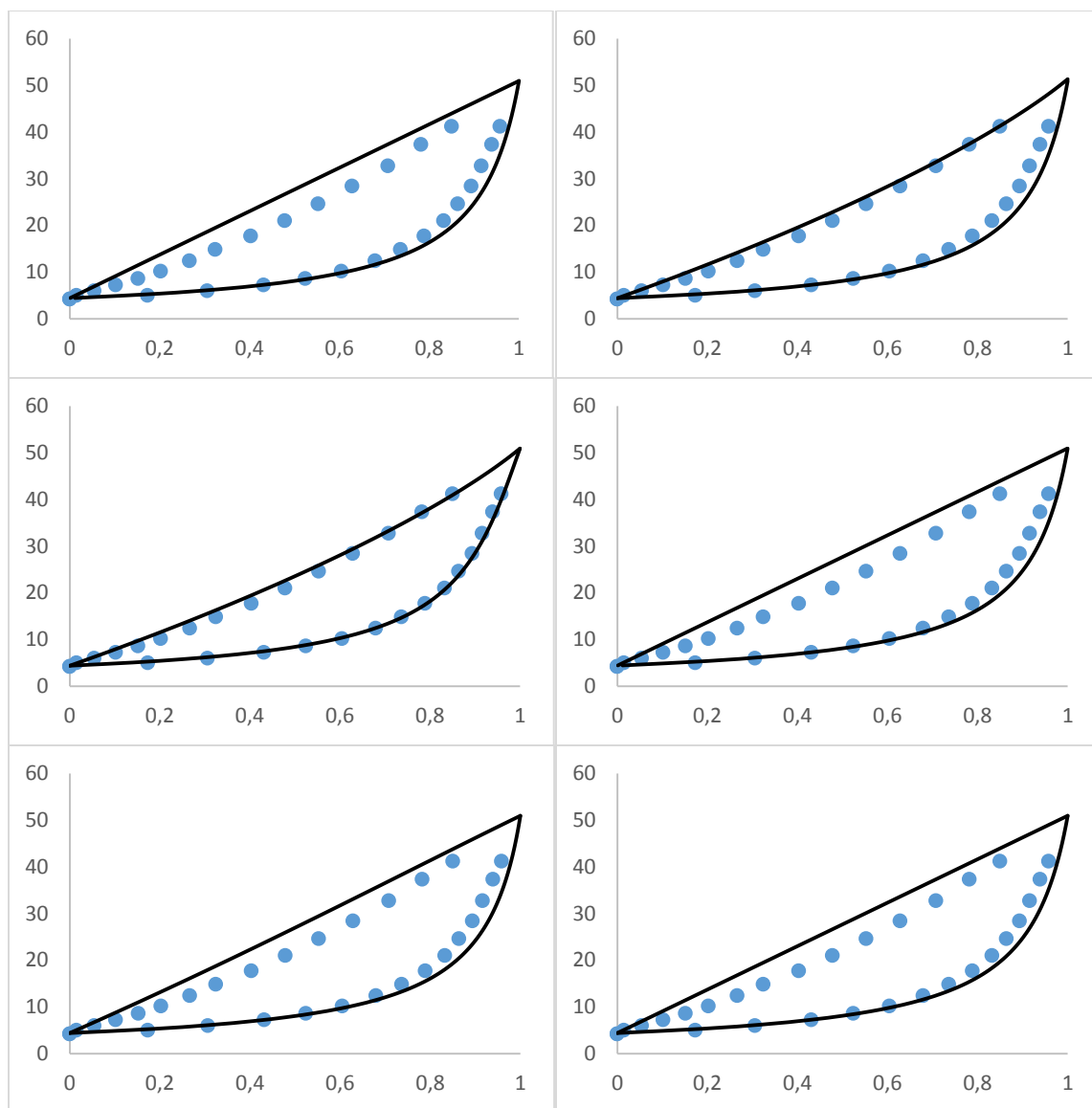


Figure 10: Comparison of VLE experimental data (dots) vs various thermodynamic ASPEN results (lines) for a DME-carbon dioxide system at 15.05 °C. From left to right: (row 1) Ideal, SRK, (row 2) Peng Robinson, NRLT, (row 3) UNIQUAC, Wilson. X-axis: mol fraction carbon dioxide Y-axis: Pressure (bar) [5]

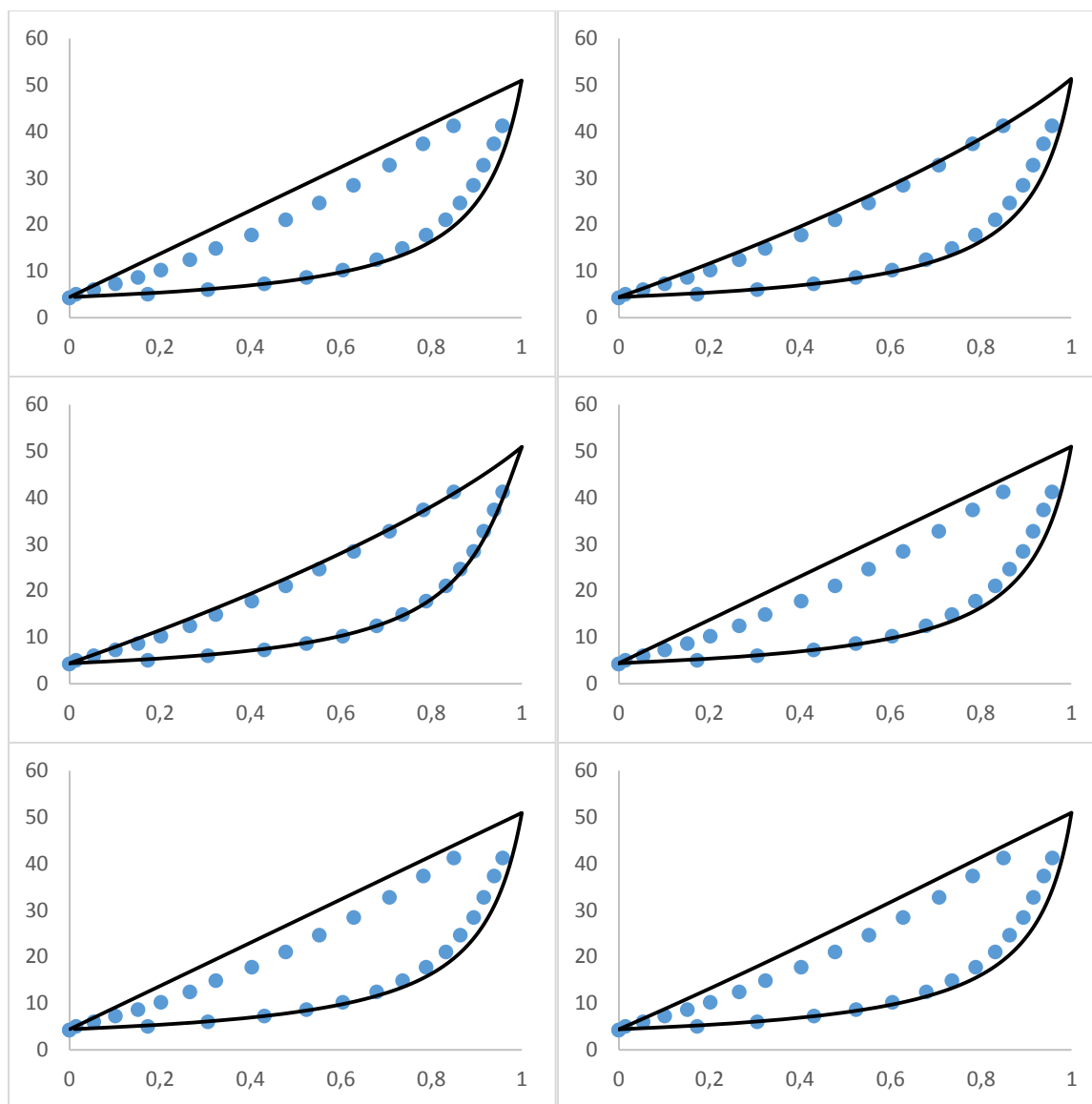


Figure 11: Comparison of VLE experimental data (dots) vs various thermodynamic ASPEN results (lines) for a DME-carbon dioxide system at 35 °C. From left to right: (row 1) Ideal, SRK, (row 2) Peng Robinson, NRLT, (row 3) UNIQUAC, Wilson. **X-axis:** mol fraction carbon dioxide **Y-axis:** Pressure (bar) [5]

A.2 Stream Summaries

Stream Table for Unit 100

	Units	1	4	5	6	10
Description						
From			PRE-COOL	OH-COL	OH-COL	COMP-1
To		B1	OH-COL	COMP-1	H2O-COOL	IS-COOL
Temperature	C	3.50E+02	8.00E+01	2.75E+01	1.38E+02	8.31E+01
Pressure	bar	1.10E+01	1.00E+01	7.00E+00	7.00E+00	2.10E+01
Molar Density	mol/cc	2.12E-04	1.50E-03	2.80E-04	3.50E-02	7.09E-04
Mass Density	kg/cum	6.62E+00	4.66E+01	1.26E+01	7.65E+02	3.19E+01
Molar Vapor Fraction		1.00E+00	2.18E-01	1.00E+00	0.00E+00	1.00E+00
Mole Flows	kmol/hr	4.31E+02	4.31E+02	1.74E+02	2.58E+02	1.74E+02
CO	kmol/hr	1.62E+00	1.62E+00	1.62E+00	2.10E-12	1.62E+00
CO2	kmol/hr	1.21E+01	1.21E+01	1.21E+01	1.11E-06	1.21E+01
CH4	kmol/hr	1.00E-02	1.00E-02	1.00E-02	9.26E-13	1.00E-02
H2	kmol/hr	3.04E+00	3.04E+00	3.04E+00	4.84E-17	3.04E+00
ETOH	kmol/hr	3.00E-01	3.00E-01	2.89E-12	3.00E-01	2.89E-12
FORM	kmol/hr	2.20E-01	2.20E-01	1.25E-04	2.20E-01	1.25E-04
WATER	kmol/hr	1.88E+02	1.88E+02	3.66E-11	1.88E+02	3.66E-11
DME	kmol/hr	1.58E+02	1.58E+02	1.57E+02	7.20E-01	1.57E+02
MEOH	kmol/hr	6.87E+01	6.87E+01	5.56E-05	6.87E+01	5.56E-05
Mole Fractions						
CO		3.76E-03	3.76E-03	9.32E-03	8.16E-15	9.32E-03
CO2		2.80E-02	2.80E-02	6.94E-02	4.32E-09	6.94E-02
CH4		2.32E-05	2.32E-05	5.76E-05	3.59E-15	5.76E-05
H2		7.05E-03	7.05E-03	1.75E-02	1.88E-19	1.75E-02
ETOH		6.95E-04	6.95E-04	1.66E-14	1.16E-03	1.66E-14
FORM		5.10E-04	5.10E-04	7.18E-07	8.53E-04	7.18E-07
WATER		4.35E-01	4.35E-01	2.11E-13	7.28E-01	2.11E-13
DME		3.66E-01	3.66E-01	9.04E-01	2.80E-03	9.04E-01
MEOH		1.59E-01	1.59E-01	3.20E-07	2.67E-01	3.20E-07
Mass Flows	kg/hr	1.35E+04	1.35E+04	7.82E+03	5.64E+03	7.82E+03
CO	kg/hr	4.54E+01	4.54E+01	4.54E+01	5.89E-11	4.54E+01
CO2	kg/hr	5.31E+02	5.31E+02	5.31E+02	4.90E-05	5.31E+02
CH4	kg/hr	1.60E-01	1.60E-01	1.60E-01	1.49E-11	1.60E-01
H2	kg/hr	6.13E+00	6.13E+00	6.13E+00	9.75E-17	6.13E+00
ETOH	kg/hr	1.38E+01	1.38E+01	1.33E-10	1.38E+01	1.33E-10
FORM	kg/hr	6.61E+00	6.61E+00	3.74E-03	6.60E+00	3.74E-03
WATER	kg/hr	3.38E+03	3.38E+03	6.59E-10	3.38E+03	6.59E-10
DME	kg/hr	7.27E+03	7.27E+03	7.23E+03	3.32E+01	7.23E+03

MEOH	kg/hr	2.20E+03	2.20E+03	1.78E-03	2.20E+03	1.78E-03
Mass Fractions						
CO		3.37E-03	3.37E-03	5.81E-03	1.04E-14	5.81E-03
CO2		3.95E-02	3.95E-02	6.79E-02	8.69E-09	6.79E-02
CH4		1.19E-05	1.19E-05	2.05E-05	2.64E-15	2.05E-05
H2		4.56E-04	4.56E-04	7.84E-04	1.73E-20	7.84E-04
ETOH		1.03E-03	1.03E-03	1.70E-14	2.45E-03	1.70E-14
FORM		4.91E-04	4.91E-04	4.79E-07	1.17E-03	4.79E-07
WATER		2.51E-01	2.51E-01	8.44E-14	6.00E-01	8.44E-14
DME		5.40E-01	5.40E-01	9.25E-01	5.89E-03	9.25E-01
MEOH		1.64E-01	1.64E-01	2.28E-07	3.91E-01	2.28E-07
Volume Flow	cum/hr	2.03E+03	2.88E+02	6.20E+02	7.36E+00	2.45E+02

Stream Summary Table for Unit 200

	Units	11	13	FUELGAS(18)	7
Description					
From		IS-COOL	B5	DME-CO2	DME-CO2
To		B5	DME-CO2		B3
Temperature	C	5.00E+01	3.00E+01	2.57E+01	7.47E+01
Pressure	bar	2.00E+01	2.00E+01	2.00E+01	2.00E+01
Molar Density	mol/cc	4.47E-03	7.82E-03	8.05E-04	1.22E-02
Mass Density	kg/cum	2.01E+02	3.52E+02	3.03E+01	5.62E+02
Molar Vapor Fraction		1.18E-01	4.88E-02	1.00E+00	0.00E+00
Mole Flows	kmol/hr	1.74E+02	1.74E+02	2.22E+01	1.51E+02
CO	kmol/hr	1.62E+00	1.62E+00	1.62E+00	8.46E-12
CO2	kmol/hr	1.21E+01	1.21E+01	1.21E+01	1.92E-03
CH4	kmol/hr	1.00E-02	1.00E-02	1.00E-02	3.66E-11
H2	kmol/hr	3.04E+00	3.04E+00	3.04E+00	4.33E-19
ETOH	kmol/hr	2.89E-12	2.89E-12	3.47E-17	2.89E-12
FORM	kmol/hr	1.25E-04	1.25E-04	2.61E-06	1.22E-04
WATER	kmol/hr	3.66E-11	3.66E-11	1.80E-16	3.66E-11
DME	kmol/hr	1.57E+02	1.57E+02	5.51E+00	1.51E+02
MEOH	kmol/hr	5.56E-05	5.56E-05	1.79E-08	5.56E-05
Mole Fractions					
CO		9.32E-03	9.32E-03	7.29E-02	5.58E-14
CO2		6.94E-02	6.94E-02	5.42E-01	1.27E-05
CH4		5.76E-05	5.76E-05	4.50E-04	2.42E-13
H2		1.75E-02	1.75E-02	1.37E-01	2.86E-21
ETOH		1.66E-14	1.66E-14	1.56E-18	1.91E-14
FORM		7.18E-07	7.18E-07	1.17E-07	8.06E-07
WATER		2.11E-13	2.11E-13	8.09E-18	2.42E-13
DME		9.04E-01	9.04E-01	2.48E-01	1.00E+00

MEOH		3.20E-07	3.20E-07	8.04E-10	3.67E-07
Mass Flows	kg/hr	7.82E+03	7.82E+03	8.36E+02	6.98E+03
CO	kg/hr	4.54E+01	4.54E+01	4.54E+01	2.37E-10
CO2	kg/hr	5.31E+02	5.31E+02	5.31E+02	8.46E-02
CH4	kg/hr	1.60E-01	1.60E-01	1.60E-01	5.88E-10
H2	kg/hr	6.13E+00	6.13E+00	6.13E+00	8.74E-19
ETOH	kg/hr	1.33E-10	1.33E-10	1.60E-15	1.33E-10
FORM	kg/hr	3.74E-03	3.74E-03	7.84E-05	3.67E-03
WATER	kg/hr	6.59E-10	6.59E-10	3.24E-15	6.59E-10
DME	kg/hr	7.23E+03	7.23E+03	2.54E+02	6.98E+03
MEOH	kg/hr	1.78E-03	1.78E-03	5.73E-07	1.78E-03
Mass Fractions					
CO		5.81E-03	5.81E-03	5.43E-02	3.39E-14
CO2		6.79E-02	6.79E-02	6.35E-01	1.21E-05
CH4		2.05E-05	2.05E-05	1.92E-04	8.42E-14
H2		7.84E-04	7.84E-04	7.33E-03	1.25E-22
ETOH		1.70E-14	1.70E-14	1.91E-18	1.91E-14
FORM		4.79E-07	4.79E-07	9.38E-08	5.25E-07
WATER		8.44E-14	8.44E-14	3.87E-18	9.45E-14
DME		9.25E-01	9.25E-01	3.04E-01	1.00E+00
MEOH		2.28E-07	2.28E-07	6.85E-10	2.55E-07
Volume Flow	cum/hr	3.89E+01	2.22E+01	2.76E+01	1.24E+01

Stream Summary Table for Unit 200 (continued)

	Units	14	15	DME(19)
Description				
From		PRE-COOL	B9	B8
To		OH-COL	B8	
Temperature	C	8.00E+01	4.82E+01	3.00E+01
Pressure	bar	1.00E+01	1.10E+01	8.00E+00
Molar Density	mol/cc	1.50E-03	8.94E-03	1.40E-02
Mass Density	kg/cum	4.66E+01	4.12E+02	6.46E+02
Molar Vapor Fraction		2.18E-01	1.57E-02	0.00E+00
Mole Flows	kmol/hr	4.31E+02	1.51E+02	1.51E+02
CO	kmol/hr	1.62E+00	8.46E-12	8.46E-12
CO2	kmol/hr	1.21E+01	1.92E-03	1.92E-03
CH4	kmol/hr	1.00E-02	3.66E-11	3.66E-11
H2	kmol/hr	3.04E+00	4.33E-19	4.33E-19
ETOH	kmol/hr	3.00E-01	2.89E-12	2.89E-12
FORM	kmol/hr	2.20E-01	1.22E-04	1.22E-04
WATER	kmol/hr	1.88E+02	3.66E-11	3.66E-11
DME	kmol/hr	1.58E+02	1.51E+02	1.51E+02
MEOH	kmol/hr	6.87E+01	5.56E-05	5.56E-05

Mole Fractions				
CO		3.76E-03	5.58E-14	5.58E-14
CO2		2.80E-02	1.27E-05	1.27E-05
CH4		2.32E-05	2.42E-13	2.42E-13
H2		7.05E-03	2.86E-21	2.86E-21
ETOH		6.95E-04	1.91E-14	1.91E-14
FORM		5.10E-04	8.06E-07	8.06E-07
WATER		4.35E-01	2.42E-13	2.42E-13
DME		3.66E-01	1.00E+00	1.00E+00
MEOH		1.59E-01	3.67E-07	3.67E-07
Mass Flows	kg/hr	1.35E+04	6.98E+03	6.98E+03
CO	kg/hr	4.54E+01	2.37E-10	2.37E-10
CO2	kg/hr	5.31E+02	8.46E-02	8.46E-02
CH4	kg/hr	1.60E-01	5.88E-10	5.88E-10
H2	kg/hr	6.13E+00	8.74E-19	8.74E-19
ETOH	kg/hr	1.38E+01	1.33E-10	1.33E-10
FORM	kg/hr	6.61E+00	3.67E-03	3.67E-03
WATER	kg/hr	3.38E+03	6.59E-10	6.59E-10
DME	kg/hr	7.27E+03	6.98E+03	6.98E+03
MEOH	kg/hr	2.20E+03	1.78E-03	1.78E-03
Mass Fractions				
CO		3.37E-03	3.39E-14	3.39E-14
CO2		3.95E-02	1.21E-05	1.21E-05
CH4		1.19E-05	8.42E-14	8.42E-14
H2		4.56E-04	1.25E-22	1.25E-22
ETOH		1.03E-03	1.91E-14	1.91E-14
FORM		4.91E-04	5.25E-07	5.25E-07
WATER		2.51E-01	9.45E-14	9.45E-14
DME		5.40E-01	1.00E+00	1.00E+00
MEOH		1.64E-01	2.55E-07	2.55E-07
Volume Flow	cum/hr	2.88E+02	1.69E+01	1.08E+01

Stream Table for Unit 300

	Units	21(toH2OCool)	Fuel(23)	BULKMEOH(20)
Description				
From		H2O-COOL	MEOH-H2O	B6
To		MEOH-H2O		
Temperature	C	8.00E+01	5.15E+01	1.27E+00
Pressure	bar	8.00E+00	1.10E+00	5.00E+00
Molar Density	mol/cc	3.82E-02	4.07E-05	2.60E-02
Mass Density	kg/cum	8.36E+02	1.58E+00	8.23E+02
Molar Vapor Fraction		0.00E+00	1.00E+00	0.00E+00
Mole Flows	kmol/hr	2.58E+02	5.81E-01	7.03E+01

CO	kmol/hr	2.10E-12	1.87E-12	2.32E-13
CO2	kmol/hr	1.11E-06	5.05E-07	6.08E-07
CH4	kmol/hr	9.26E-13	7.23E-13	2.03E-13
H2	kmol/hr	4.84E-17	0.00E+00	0.00E+00
ETOH	kmol/hr	3.00E-01	7.58E-04	2.99E-01
FORM	kmol/hr	2.20E-01	2.21E-09	6.23E-06
WATER	kmol/hr	1.88E+02	4.18E-03	2.68E+00
DME	kmol/hr	7.20E-01	2.77E-01	4.43E-01
MEOH	kmol/hr	6.87E+01	2.99E-01	6.68E+01
Mole Fractions				
CO		8.16E-15	3.22E-12	3.30E-15
CO2		4.32E-09	8.69E-07	8.66E-09
CH4		3.59E-15	1.24E-12	2.88E-15
H2		1.88E-19	0.00E+00	0.00E+00
ETOH		1.16E-03	1.30E-03	4.25E-03
FORM		8.53E-04	3.80E-09	8.87E-08
WATER		7.28E-01	7.19E-03	3.81E-02
DME		2.80E-03	4.77E-01	6.30E-03
MEOH		2.67E-01	5.14E-01	9.51E-01
Mass Flows				
CO	kg/hr	5.64E+03	2.25E+01	2.22E+03
CO2	kg/hr	5.89E-11	5.24E-11	6.49E-12
CO2	kg/hr	4.90E-05	2.22E-05	2.68E-05
CH4	kg/hr	1.49E-11	1.16E-11	3.25E-12
H2	kg/hr	9.75E-17	0.00E+00	0.00E+00
ETOH	kg/hr	1.38E+01	3.49E-02	1.38E+01
FORM	kg/hr	6.60E+00	6.63E-08	1.87E-04
WATER	kg/hr	3.38E+03	7.53E-02	4.83E+01
DME	kg/hr	3.32E+01	1.28E+01	2.04E+01
MEOH	kg/hr	2.20E+03	9.58E+00	2.14E+03
Mass Fractions				
CO		1.04E-14	2.33E-12	2.92E-15
CO2		8.69E-09	9.89E-07	1.20E-08
CH4		2.64E-15	5.16E-13	1.46E-15
H2		1.73E-20	0.00E+00	0.00E+00
ETOH		2.45E-03	1.55E-03	6.19E-03
FORM		1.17E-03	2.95E-09	8.41E-08
WATER		6.00E-01	3.35E-03	2.17E-02
DME		5.89E-03	5.69E-01	9.17E-03
MEOH		3.91E-01	4.26E-01	9.63E-01
Volume Flow	cum/hr	6.74E+00	1.43E+01	2.70E+00

Stream Table for Unit 300 (continued)

	Units	8	9	WASTEH2O(22)
Description				
From		MEOH-H2O	B7	B2

To		B7	B2	
Temperature	C	1.00E+02	5.01E+01	5.01E+01
Pressure	bar	1.10E+00	5.00E+00	5.00E+00
Molar Density	mol/cc	5.03E-02	5.32E-02	5.32E-02
Mass Density	kg/cum	9.13E+02	9.65E+02	9.65E+02
Molar Vapor Fraction		0.00E+00	0.00E+00	0.00E+00
Mole Flows	kmol/hr	1.87E+02	1.87E+02	1.87E+02
CO	kmol/hr	7.14E-34	7.14E-34	7.14E-34
CO2	kmol/hr	2.71E-22	2.71E-22	2.71E-22
CH4	kmol/hr	6.48E-32	6.48E-32	6.48E-32
H2	kmol/hr	0.00E+00	0.00E+00	0.00E+00
ETOH	kmol/hr	5.19E-04	5.19E-04	5.19E-04
FORM	kmol/hr	2.20E-01	2.20E-01	2.20E-01
WATER	kmol/hr	1.85E+02	1.85E+02	1.85E+02
DME	kmol/hr	5.98E-11	5.98E-11	5.98E-11
MEOH	kmol/hr	1.56E+00	1.56E+00	1.56E+00
Mole Fractions				
CO		3.82E-36	3.82E-36	3.82E-36
CO2		1.45E-24	1.45E-24	1.45E-24
CH4		3.47E-34	3.47E-34	3.47E-34
H2		0.00E+00	0.00E+00	0.00E+00
ETOH		2.78E-06	2.78E-06	2.78E-06
FORM		1.18E-03	1.18E-03	1.18E-03
WATER		9.90E-01	9.90E-01	9.90E-01
DME		3.20E-13	3.20E-13	3.20E-13
MEOH		8.36E-03	8.36E-03	8.36E-03
Mass Flows	kg/hr	3.39E+03	3.39E+03	3.39E+03
CO	kg/hr	2.00E-32	2.00E-32	2.00E-32
CO2	kg/hr	1.19E-20	1.19E-20	1.19E-20
CH4	kg/hr	1.04E-30	1.04E-30	1.04E-30
H2	kg/hr	0.00E+00	0.00E+00	0.00E+00
ETOH	kg/hr	2.39E-02	2.39E-02	2.39E-02
FORM	kg/hr	6.60E+00	6.60E+00	6.60E+00
WATER	kg/hr	3.33E+03	3.33E+03	3.33E+03
DME	kg/hr	2.75E-09	2.75E-09	2.75E-09
MEOH	kg/hr	5.01E+01	5.01E+01	5.01E+01
Mass Fractions				
CO		5.90E-36	5.90E-36	5.90E-36
CO2		3.52E-24	3.52E-24	3.52E-24
CH4		3.07E-34	3.07E-34	3.07E-34
H2		0.00E+00	0.00E+00	0.00E+00
ETOH		7.06E-06	7.06E-06	7.06E-06
FORM		1.95E-03	1.95E-03	1.95E-03
WATER		9.83E-01	9.83E-01	9.83E-01
DME		8.12E-13	8.12E-13	8.12E-13

MEOH		1.48E-02	1.48E-02	1.48E-02
Volume Flow	cum/hr	3.71E+00	3.51E+00	3.51E+00

A.3 Equipment list

Utility Summary Table for Unit 100

E-101	E-102	E-103	E-104
CTW	CW	LPS	CTW
433517.81	230544.17	3239.50	36180.97

Major Equipment Summary for Unit 100

Compressors
C-101 Carbon steel W=132kW 70% efficiency

Heat Exchangers	
E-101 A=71.m ² Floating head, stainless steel, shell-and-tube design Process stream in tube Q=18076.27MJ/hr	E-104 A= 7.0m ² Floating head, stainless steel, plate and frame Q=1508.63 MJ/hr
E-102 A= 543.3m ² Floating head, stainless steel, shell-and-tube design Process stream in shell Q=4829.66MJ/hr	
E-103 A= 57.6m ² Floating head, stainless steel, kettle Process stream in shell Q=6568.97 MJ/hr	

Pumps
P-101 A/B Centrifugal/electric drive Carbon steel Power=51.2 kW 70% efficient

Towers

T-101
Stainless Steel
22 SS sieve trays plus reboiler and condenser
2-ft tray spacing
Column height =12.8m
Diameter=0.76m

Vessels

V-101
Horizontal
Carbon steel
Length=3.28m
Diameter=1.09m

Utility Summary Table for Unit 200

E-201	E-202	E-203	E-204	E-205	E-206
CTW	CW	CW	LPS	CTW	CW
67936.3543	29943.1075	27830.4715	837.094941		

Major Equipment Summary for Unit 200

Heat Exchangers	
E-201 A= 237m² Floating head, stainless steel, shell-and-tube design Process stream in tube Q= 2823.72 MJ/hr	E-204 A= 5.5m² Floating head, stainless steel, kettle reboiler Process stream in shell Q= 1697.44 MJ/hr
E-202 A= 43.7m² Floating head, stainless steel, shell-and-tube design Process stream in tube Q= 627.28 MJ/hr	E-205 A= 20.0m² Floating head, stainless steel, shell-and-tube design Q=605.53MJ/hr
E-203 A= 119.8m² Floating head, stainless steel, shell-and-tube design Process stream in shell Q= 583.02 MJ/hr	E-206 A= 9.5m² Floating head, stainless steel, shell-and-tube design Q=434.14MJ/hr

Pumps

P-201 A/B
Centrifugal/electric drive

Carbon steel
Power=66.0 kW
70% efficient

Towers

T-201
Stainless Steel
22 SS sieve trays plus reboiler and condenser
2-ft tray spacing
Column height = 14.63m
Diameter=0.52m

Vessels

V-201
Horizontal
Carbon steel
Length=1.45m
Diameter=0.48m

Utility Summary Table for Unit 300

E-301	E-302	E-303
CW	LPS	CTW
155085.567	2898.86189	17899.4687

Major Equipment Summary for Unit 300

Heat Exchangers

E-301
A= 16.8m²
Floating head, stainless steel, shell-and-tube design
Process stream in shell
Q= 391.73 MJ/hr

E-302
A= 25.5m²
Floating head, stainless steel, kettle reboiler
Process stream in shell
Q= 5878.23 MJ/hr

E-303
A= 9.0m²
Floating head, Stainless Steel, Plate and Frame
Q= 746.35 MJ/hr

Pumps

P-301 A/B
Centrifugal/electric drive

Carbon steel

Power=10.7kW

70% efficient

P-302 A/B

Centrifugal/electric drive

Carbon steel

Power=13.5kW

70% efficient

P-303 A/B

Centrifugal/electric drive

Carbon steel

Power=32.2kW

70% efficient

Towers

T-301

Stainless Steel

22 SS sieve trays plus reboiler and condenser

2-ft tray spacing

Column height =10.97m

Diameter=0.76m

Vessels

V-301

Horizontal

Carbon steel

Length=2.80 m

Diameter=0.93m

A.4 Sample calculations for equipment sizing

A.4.1 Distillation column height and diameter sizing: DME-Methanol column

From Table 3, the expected number of real stages for this tower is 16. Additionally, there is 6 ft of bottoms space and 4 ft of vapor space added to the column. According to Equation 1, the column height is then

$$height = 16 * 2ft + 4ft + 6ft = 42ft$$

Column diameter and pressure drop are calculated using the following equations

$$F_s = u\rho^{1/2}$$
$$D_{tower} = \left[\frac{4v}{\pi u} \right]^{1/2}$$
$$\Delta P_{tower} = \frac{N_{actual}}{\Delta P_{tray}}$$

Where

F_s = vapor factor = 1.2-1.5 m/s

u = gas velocity [m/s]

ρ_u = gas density [kg/m³]

v = volumetric flow rate [m³/s]

N_{actual} = expected number of stages

ΔP_{tray} = tray pressure drop

	MEOH/DME	DME/CO2	MEOH/H2O
Vapor Factor	1.5	1.5	1.5
Gas velocity (m/s)	0.16	0.10	0.75
Gas density (kg/m3)	14.5	22.4	3.010
Volumetric flow rate (m3/s)	0.070	0.020	0.400
Number of trays	16	19	13
Tray pressure drop (bar)	0.007	0.007	0.007
Diameter (m)	0.760	0.520	0.820
Tower Pressure Drop (bar)	0.110	0.130	0.090

A.4.2 Heat Exchanger sizing: Reactor effluent pre-cooler

The reactor feed cooler is a shell-and-tube style cooler with countercurrent flow. Because of the partial condensation of the stream, the shell side was set to hold the process fluid. The process and utility streams have the inlet/outlet temperatures of 350/80°C and 35/45°C respectively. The log mean temperature differences are shown as:

$$\Delta T_1 = (T_{hot,in} - T_{cold,out}) = 350 - 45 = 305 \text{ }^{\circ}\text{C}$$

$$\Delta T_2 = (T_{hot,out} - T_{cold,in}) = 80 - 35 = 45 \text{ }^{\circ}\text{C}$$

$$\Delta T_{lm} = \frac{\Delta T_1 - \Delta T_2}{\ln\left(\frac{\Delta T_1}{\Delta T_2}\right)} = \frac{305 - 45}{\ln(305 - 45)} = 135.87^{\circ}\text{C}$$

The compositions and expected heat transfer coefficient for each is specified below:

the product side

Component	Mole fraction	U value (J/m ² s °C)
Water (condensing)	.435	1200
DME (condensing)	.366	200
Methanol + Ethanol (condensing)	.159	800
Carbon dioxide	.028	90
other	.012	90

Taking a weighted molar sum:

$$U = (.435 * 1200) + (.366 * 200) + (.159 * 800) + (.028 * 90) + (.012 * 90)$$
$$= 726 \frac{J}{m^2 \text{ s } ^{\circ}\text{C}}$$

From the ASPEN model summary, the heat duty for this exchanger is 4.320 Gcal/hr or 5.02*10⁶ J/s. The non-ideality factor used for shell-and-tube exchangers was assumed to be 0.85.

Area is solved for area and multiplied by a 20% oversize factor:

$$Area = \frac{Q}{fU\Delta T_{lm}} * (1 + oversize) = \frac{5.02 * 10^6}{.85 * 726 * 135.86} * (1.2) = 71.9 \text{ m}^2$$

A.4.3 Pump sizing: DME-Methanol P-201 A/B reflux pump

From the ASPEN model, the estimated reflux rate is 260.6 kmol/hr. The reflux was assumed to have the same molecular weight and density as pure DME (646.4 kg/m³), since the reflux is 99.9+% DME [source: aspen]. The number of stages for this column is 16 with a feed stage of 7. So the differential in fluid level across the pump is:

$$\Delta h = (16 \text{ stages}) * 2 \text{ ft} = 32 \text{ ft or } 9.73 \text{ m}$$

Calculating the head requirement for this:

$$\Delta P = 1 \text{ bar} + \rho g \Delta h = 1 + \frac{646.4 \text{ kg}}{\text{m}^3} * \frac{9.8 \text{ m}}{\text{s}^2} * 9.73 \text{ m} * \frac{1 \text{ bar}}{10^5 \text{ Pa}} = 1.62 \text{ bar}$$

Using the molar flow from ASPEN, the reflux is converted to volumetric flow

$$260.6 \frac{\text{kmol}}{\text{hr}} * 46 \frac{\text{kg}}{\text{kmol DME}} * \frac{1 \text{ m}^3}{646.4 \text{ kg}} = 18.55 \frac{\text{m}^3}{\text{hr}}$$

The minimum power requirement for this pump is then:

$$P = 1.67 F * \frac{\Delta P}{\varepsilon} = 1.67 * 18.55 \frac{\text{m}^3}{\text{hr}} * \frac{1.62 \text{ bar}}{.7} = 71.7 \text{ kw}$$

With a 25% oversize, the estimated value is then 89.5 Kw, or 120 hP.

A.4.4 Reflux drum sizing: DME-MEOH reflux drum

From A.4.3, the reflux flow rate was shown to be 18.55 m³/hr. The DME-MEOH reflux drum was given a 5 minute residence time for a half-full vessel. That is

$$\text{volume half full} = 18.55 \frac{\text{m}^3}{\text{hr}} * 5 \text{ min} * \frac{1 \text{ hr}}{60 \text{ min}} = 1.55 \text{ m}^3$$

The full volume then is twice this value, or 3.09 m³ or 817 gallons.

With a length to diameter ratio of 3, the volume of the cylindrical drum is solved for from its volume equation:

$$V = \pi * \frac{(\text{diameter})^2}{4} * \text{length} = \frac{3\pi}{4} * \text{diameter}^2$$
$$\text{diameter} = \left(\text{Volume} * \frac{4}{3\pi} \right)^{\frac{1}{3}} = \left(3.09 * \frac{4}{3 * \pi} \right)^{\frac{1}{3}} = 1.09 \text{ m}$$
$$\text{length} = 3 * \text{diamter} = 3.27 \text{ m}$$

A.5 Aspen Data

A.5.1 Aspen Flowsheet

A.5.2 Aspen Stream Summary

A.5.3 Aspen Equipment Summary