

# Azeotropic Distillation with Aspen Plus® V8.0

## Production of Anhydrous Ethanol Using an Entrainer

### 1. Lesson Objectives

- Design a separation train for anhydrous ethanol production using cyclohexane as an entrainer
- Include recycle of cyclohexane and the azeotropic mixture so that the recovery of ethanol is >99.9 % and the recovery of cyclohexane is nearly 100 %
- Experiment with the Distillation Synthesis tool included in Aspen Plus
- Successfully converge a flowsheet with multiple recycle streams
- Manipulate convergence methods in **RadFrac** to improve convergence

### 2. Prerequisites

- Aspen Plus V8.0
- Understanding of azeotropes
- Experience using **RadFrac** distillation model
- Experience reading ternary diagrams

### 3. Background

Ethanol production via fermentation occurs in water, which must later be separated to make anhydrous ethanol (99.95% ethanol). There is an azeotrope in the ethanol-water system at approximately 95 mol-% ethanol which is a barrier to separation. Cyclohexane is one of the solvents used for the production of anhydrous ethanol for food and pharmaceutical usage. It is used as an entrainer: the ternary mixture forms a ternary azeotrope with a different ethanol concentration, which allows ethanol to enrich in the other stream. The azeotropic liquid is separated to recover the entrainer and ethanol which exits the column in the azeotropic mixture.

**The examples presented are solely intended to illustrate specific concepts and principles. They may not reflect an industrial application or real situation.**

## 4. Problem Statement and Aspen Plus Solution

### Problem Statement

The feed to the separation train is a stream at 100 kmol/hr with 87 mol-% ethanol and 13 mol-% water. Cyclohexane is added to the column, and > 99.95 mol-% ethanol exits the bottom of the column. The distillate is sent to a decanter (described in Thermo-020\_Decanter). The cyclohexane-rich stream is recycled directly to the first column, while the water- and ethanol-rich stream is sent to a second column from which almost-pure water exits in the bottoms. The distillate of the second column is recycled to the first column.

Design the separation train so that the ethanol product stream meets the purity specification, has >99.9 mol-% recovery, and the water effluent stream is essentially pure water.

### Aspen Plus Solution

If you are unfamiliar with how to start Aspen Plus, select components, define methods, or construct a flowsheet, consult **Get Started Guide for New Users of Aspen Plus.pdf** for instructions.

This model is built using a specific path. The order in which things are done is important for successful convergence of the model. Do not reinitialize the run unless asked to, and if steps are skipped or done out of order you may need to be start at the beginning or from a previously saved version.

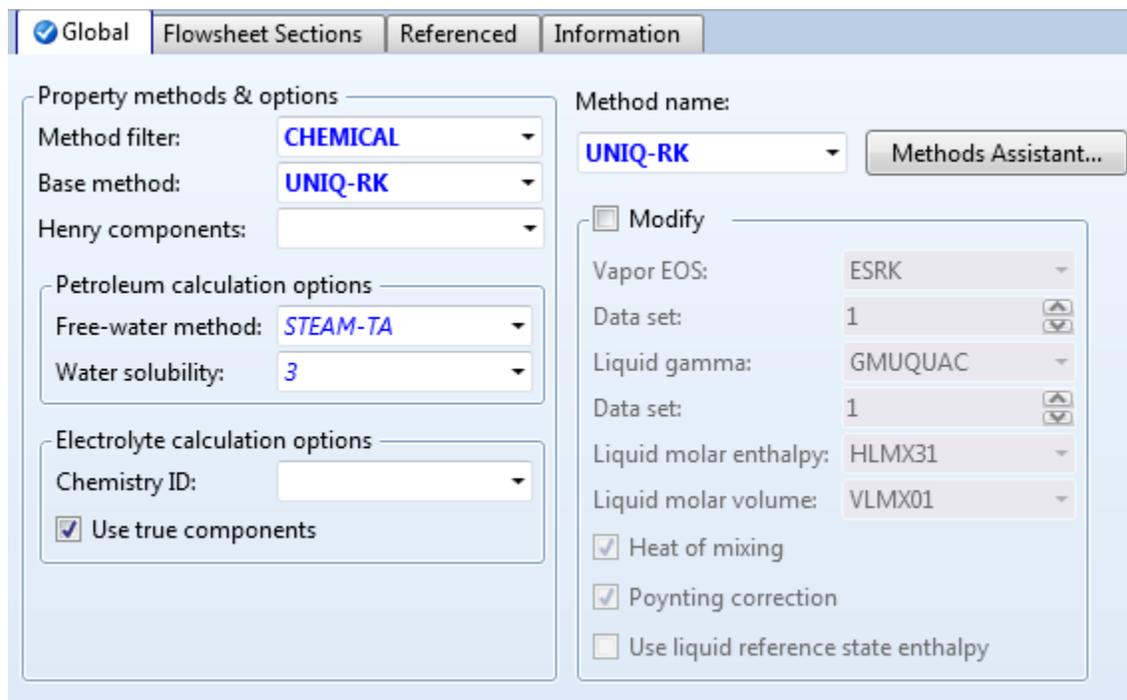
- 4.01. Start a new simulation using the **Blank Simulation** template in Aspen Plus. The **Components | Specification | Selection** sheet is displayed. Enter the components as shown below.

Select components:

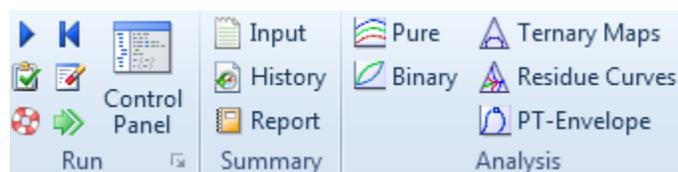
Component ID	Type	Component name	Alias
ETHANOL	Conventional	ETHANOL	C2H6O-2
WATER	Conventional	WATER	H2O
C6H12-1	Conventional	CYCLOHEXANE	C6H12-1

Find    Elec Wizard    User Defined    Reorder    Review

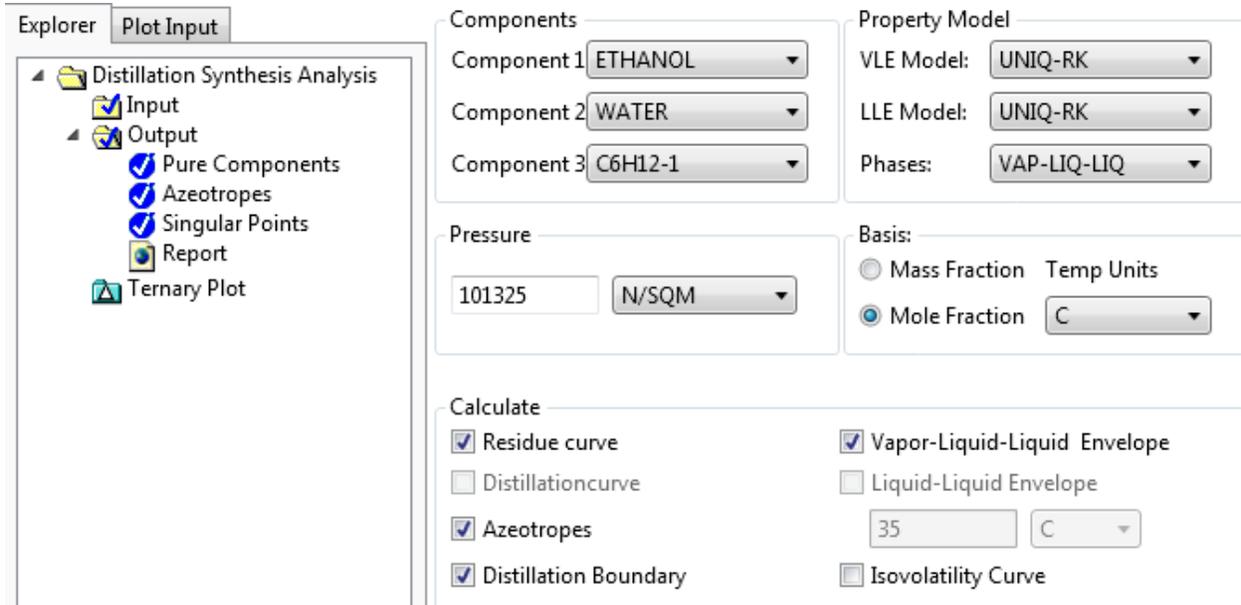
- 4.02. Click on the **Methods** button in the **Navigate** group under the **Home** tab of the ribbon. Change the **Method filter** to **CHEMICAL** and select **UNIQ-RK** as the **Base method**.



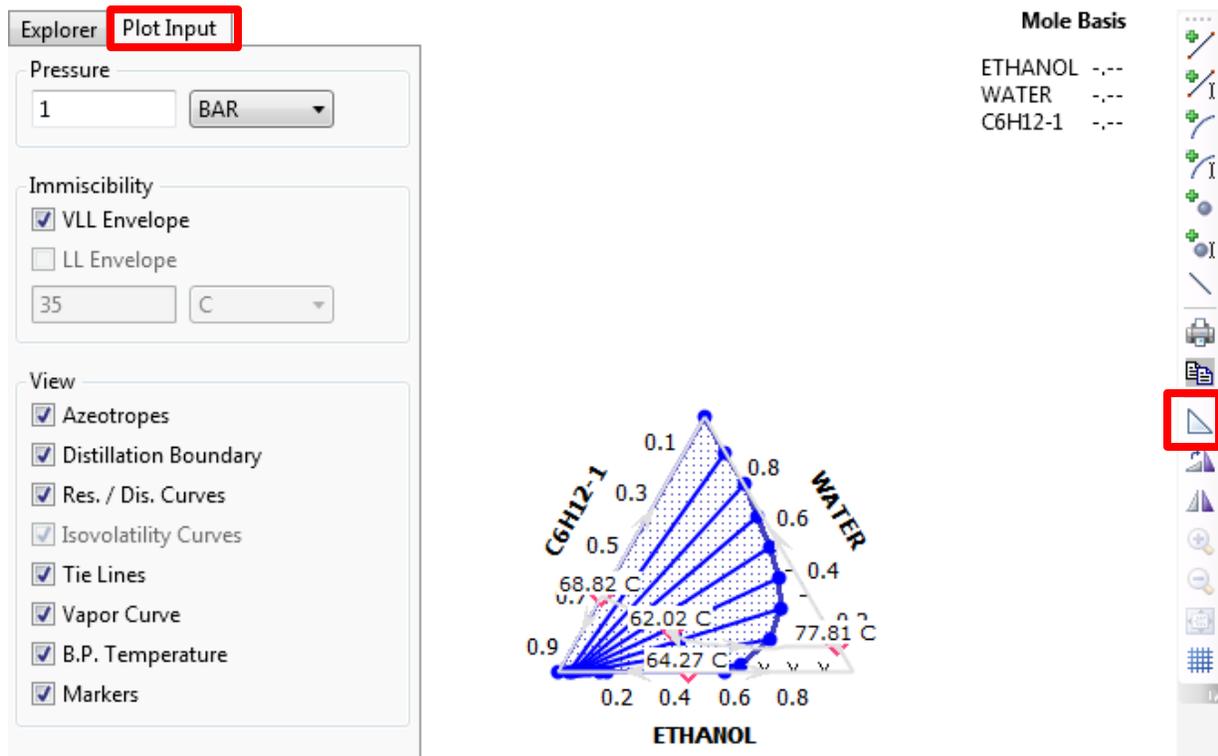
- 4.03. Press the **F4** key. Note that the **Methods | Parameters | Binary Interaction | UNIQ-1 | Input** sheet is displayed and binary parameters are filled automatically.
- 4.04. Click on the **Analysis | Residue Curves** button in the **Home** tab of the ribbon.



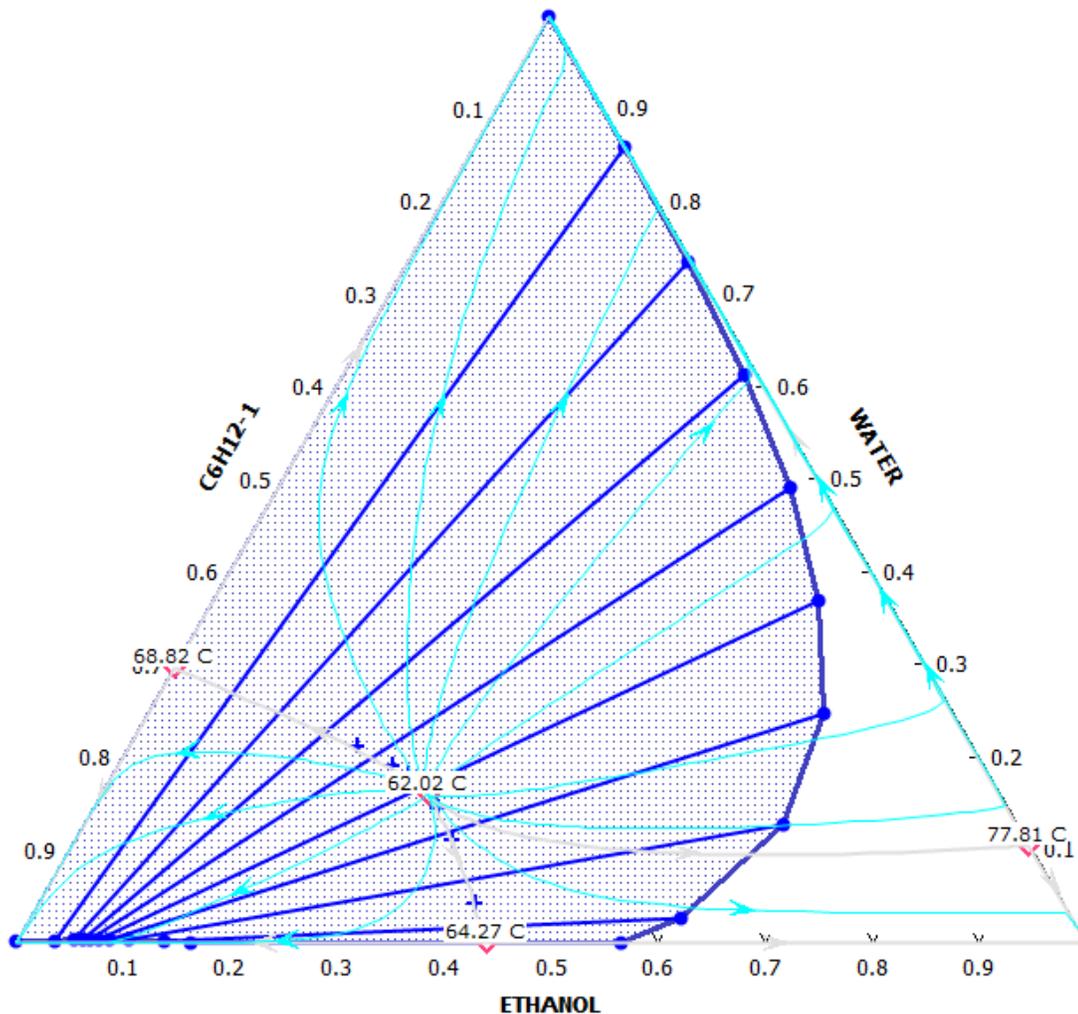
- 4.05. In the popup dialog box, click the **Use Distillation Synthesis ternary maps** button.
- 4.06. Ensure that the components are in the same order so that the axes match. Ensure that the **Phases** field is **VAP-LIQ-LIQ** so that the model is accurate.



- 4.07. Change the **Pressure** to **1 bar**, as this will be the pressure for the columns. Click on the **Plot Input** sheet (it may take 8-15 seconds for this sheet to be displayed) and select the **Switch Coordinates** option to change the format of the graph.

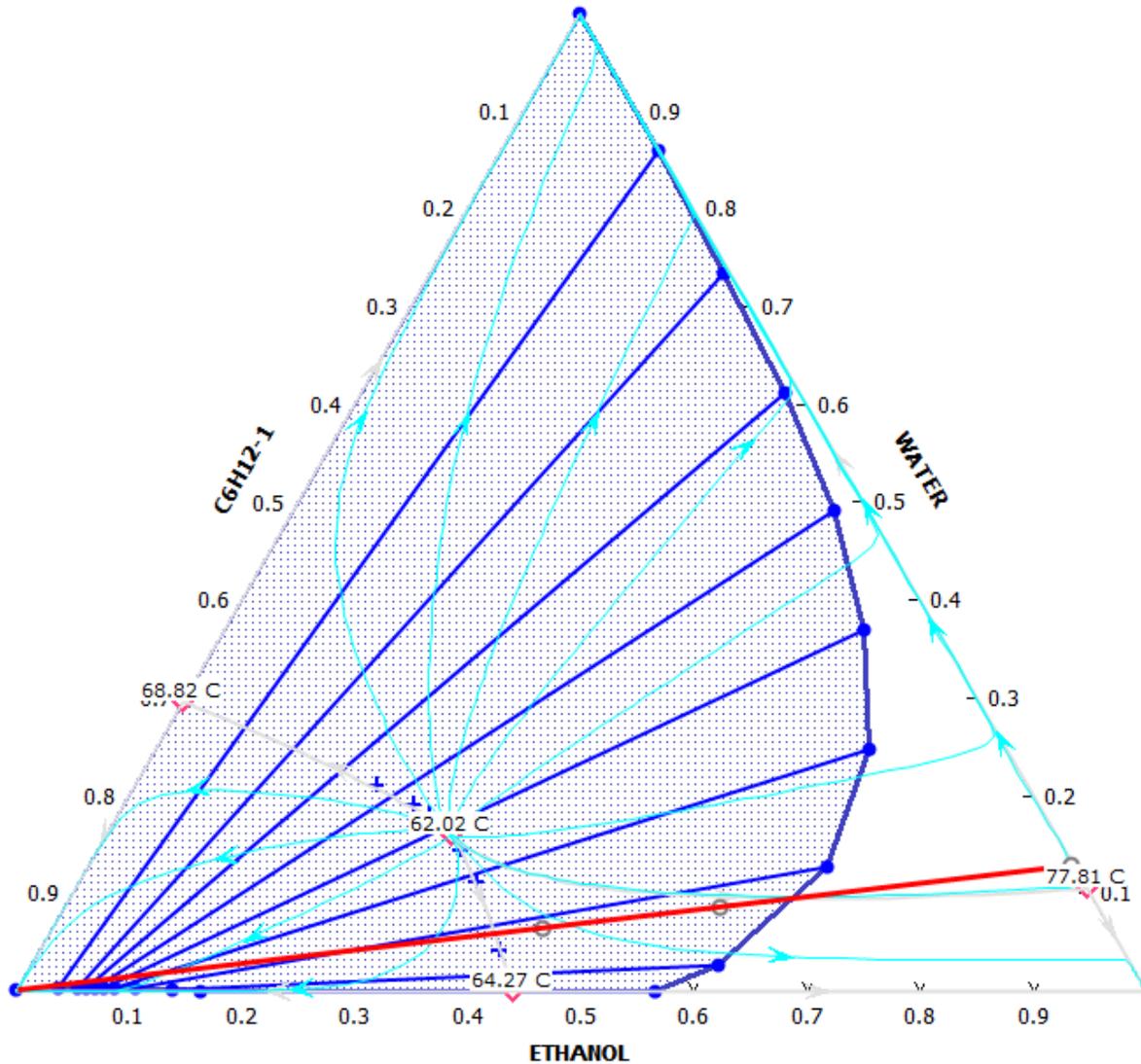


- 4.08. You can increase the size of the graph by dragging a corner of the **Aspen Plus** window. For help on reading ternary diagrams, refer to **Thermo-016\_Ternary\_Maps**. The gray lines are the boundaries for the distillation regions. The arrows point towards higher boiling point mixtures. Red diamonds are azeotropes. The dark blue line is the LLE envelope. The slightly lighter blue lines are tie lines for the LLE split. Select the **Add Curve** button and click the plot to add residue curves at specific points in diagram. These curves will be light blue.



- 4.09. All residue curves point towards the vertices, which represent pure components. A distillation column will enrich a single component in the bottoms, and the enriched component depends on which distillation region the feed composition is in. We plan to enrich ethanol from the bottom of the first column.

- 4.10. Use the **Add Marker By Value** button to put a marker at the feed composition (**0.87, 0.13, and 0.0**, respectively). Use the **Draw Line** button to draw a line between the marker you just made and the 100% cyclohexane vertex. A combination of a pure cyclohexane stream and the feed stream will have a composition somewhere along this line, depending on the ratio of the flow rates. A section of this line goes through the region which enriches ethanol in the bottoms. Calculate the composition of adding 50 kmol/hr of cyclohexane to the feed stream and adding 100 kmol/hr of cyclohexane to the feed stream. Remember, the feed stream has a flow rate of 100 kmole/hr. Use the **Add Marker By Value** button to put markers at these locations.



- 4.11. Both points are within the correct distillation region, but the 100 kmol/hr addition marker is farther in the region. Because of this, the initial feed of solvent to the column will be 100 kmol/hr. Use this tool throughout the simulation construction to understand what the decanter or distillation columns are doing. Now, close the **Distillation Synthesis** window.
- 4.12. Switch to the **Simulation** environment by clicking the **Simulation** bar in the navigation pane. In the navigation pane, go to the **Set up | Report Options | Stream** sheet. Check the **Mole** and **Mass** boxes in the **Fraction basis** frame. This changes the default information shown in stream results which makes it easier to determine what section of the ternary map the stream falls in. This information will also be used later on when closing recycle loops.

General | Flowsheet | Block | **Stream** | Property | ADA

Generate a standard stream report     Include stream descriptions

Items to be included in stream report

<b>Flow basis</b> <input checked="" type="checkbox"/> Mole <input type="checkbox"/> Mass <input type="checkbox"/> Std.liq.volume	<b>Fraction basis</b> <input checked="" type="checkbox"/> Mole <input checked="" type="checkbox"/> Mass <input type="checkbox"/> Std.liq.volume	<b>Stream format</b> TFF: <i>FULL</i> <input checked="" type="radio"/> Standard (80 characters) <input type="radio"/> Wide (132 characters) <input checked="" type="checkbox"/> Sort streams alphanumerically
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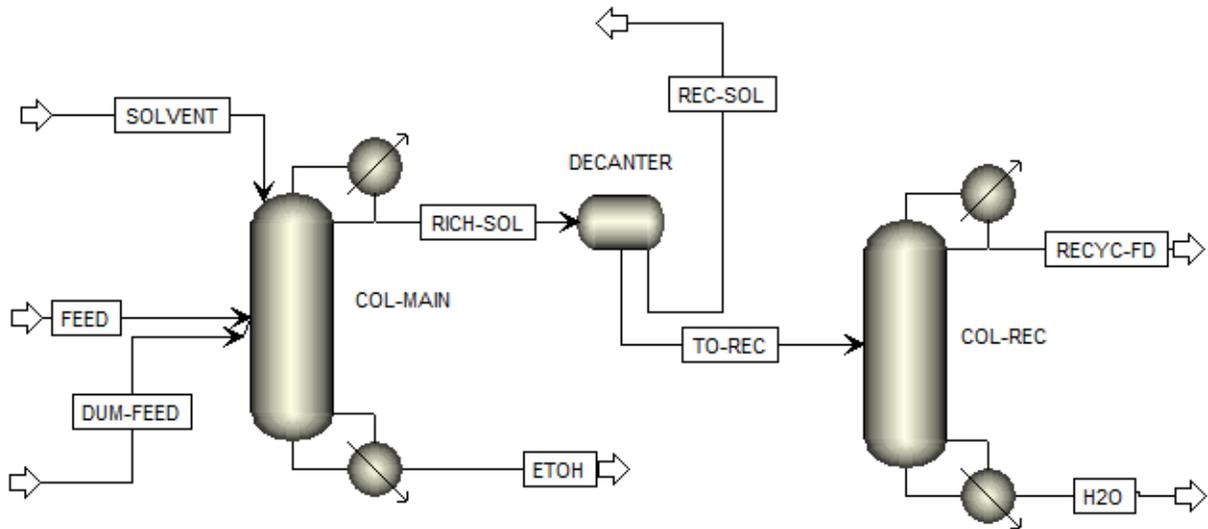
4.13. Create a flowsheet that consists of the following blocks and streams.

Name	Type
DUM-FEED	MATERIAL
ETOH	MATERIAL
FEED	MATERIAL
H2O	MATERIAL
REC-SOL	MATERIAL
RECYC-FD	MATERIAL
RICH-SOL	MATERIAL
SOLVENT	MATERIAL
TO-REC	MATERIAL

Name	Type
COL-MAIN	RadFrac
COL-REC	RadFrac
DECANTER	Decanter

4.14. These blocks and streams should be connected in the following way.



- 4.15. Enter the feed conditions. Go to the **Streams | FEED | Input | Mixed** sheet and specify **FEED** as shown below.

Mixed | CI Solid | NC Solid | Flash Options | EO Options | Costing | Information

Specifications

Flash Type: Vapor Fraction Pressure

State variables

Temperature: C

Pressure: 1 bar

Vapor fraction: 0.3

Total flow basis: Mole

Total flow rate: kmol/hr

Solvent:

Composition

Mole-Flow kmol/hr

Component	Value
ETHANOL	87
WATER	13
C6H12-1	

Total: 100

- 4.16. Go to the **Streams | SOLVENT | Input | Mixed** sheet and specify **SOLVENT** as shown below. This stream will eventually be connected with a stream recycled from **DECANTER**. The specifications here serve as an initial guess.

Mixed | CI Solid | NC Solid | Flash Options | EO Options | Costing | Information

Specifications

Flash Type: Vapor Fraction Pressure

State variables

Temperature: C

Pressure: 1 bar

Vapor fraction: 0

Total flow basis: Mole

Total flow rate: 100 kmol/hr

Solvent:

Composition

Mole-Frac

Component	Value
ETHANOL	
WATER	
C6H12-1	1

Total: 1

- 4.17. Go to the **Streams | DUM-FEED | Input | Mixed** sheet. This steam will later on be connected with a recycle stream from the top of **COL-REC**. The dummy feed stream is a place holder for the recycle stream from the top of the recycle column. For now, we enter a tiny flowrate with a rough guess for compositions as shown below.

Specifications

Flash Type: Vapor Fraction Pressure

State variables

Temperature: C

Pressure: 1 bar

Vapor fraction: 0

Total flow basis: Mole

Total flow rate: 1e-05 kmol/hr

Solvent:

Composition

Mole-Flow kmol/hr

Component	Value
ETHANOL	0.35
WATER	0.3
C6H12-1	0.35

Total: 1

- 4.18. Go to the **Blocks | COL-MAIN | Specifications | Setup | Configuration** sheet. Enter **62** for **Number of stages**. Select **Total** for **Condenser**, **Vapor-Liquid-Liquid** for **Valid phases** and **Strongly non-ideal** for **Convergence**. In the **Operating specifications** frame, enter **3.5** for **Reflux ratio**. Then, select **Bottoms rate** as the other specification and enter **50** for **Bottoms rate**. **Blocks | COL-MAIN | Specifications | Setup | Configuration** sheet should look like this.

Configuration

Setup options

Calculation type: Equilibrium

Number of stages: 62 Stage Wizard

Condenser: Total

Reboiler: Kettle

Valid phases: Vapor-Liquid-Liquid

Convergence: Strongly non-ideal liquid

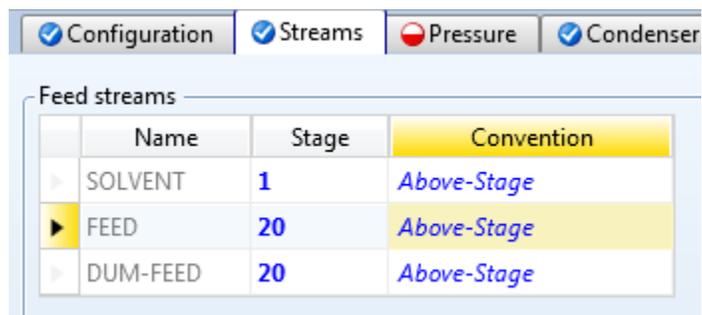
Operating specifications

Bottoms rate Mole 50 kmol/hr

Reflux ratio Mole 3.5

Free water reflux ratio: 0 Feed Basis

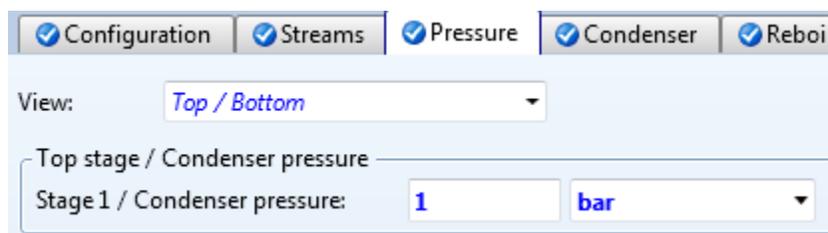
- 4.19. Go to the **Blocks | COL-MAIN | Specifications | Setup | Streams** sheet. In the **Feed streams** frame, enter **20** in the **Stage** column for **FEED** and **DUM-FEED**. Enter **1** for **SOLVENT** as shown below.



The screenshot shows a software interface with four tabs: Configuration, Streams, Pressure, and Condenser. The 'Streams' tab is active. Below the tabs is a 'Feed streams' section containing a table with three columns: Name, Stage, and Convention.

	Name	Stage	Convention
▶	SOLVENT	1	Above-Stage
▶	FEED	20	Above-Stage
▶	DUM-FEED	20	Above-Stage

- 4.20. Go to the **Blocks | COL- MAIN | Specifications | Setup | Pressure** sheet. Enter **1** for **Stage 1 / Condenser pressure**.



The screenshot shows a software interface with five tabs: Configuration, Streams, Pressure, Condenser, and Reboiler. The 'Pressure' tab is active. Below the tabs is a 'View:' dropdown menu set to 'Top / Bottom'. Underneath is a section for 'Top stage / Condenser pressure' with a text input field containing '1' and a unit dropdown menu set to 'bar'.

- 4.21. Go to the **Blocks | COL-MAIN | Specifications | Setup | 3-Phase** sheet. Enter **1** for **Starting stage** and **62** for **Ending stage**. Specify **WATER** as **Key components** in the **Key components to identify 2nd liquid phase** frame. This tells Aspen Plus that we expect the second liquid phase to be predominantly water, and since we do not know exactly which stages of the column will contain two liquid phases, we will ask Aspen Plus to check every stage.

Pressure  Condenser Thermosiphon Config.  Reboiler  3-Phase In

Stages to be tested for two liquid phases

	Starting stage	Ending stage
▶	1	62
▶		

Key components to identify 2nd liquid phase

Available components: ETHANOL, C6H12-1

Key components: WATER

- 4.22. Go to the **Blocks | COL-MAIN | Convergence | Convergence | Basic** sheet. Increase **Maximum iterations** from **25** to **200**.
- 4.23. Go to the **Blocks | COL-REC | Specifications | Setup | Configuration** sheet. Enter **100** for **Number of stages**. Select **Total** for **Condenser**, **Vapor-Liquid-Liquid** for **Valid phases** and **Strongly non-ideal** for **Convergence**. In the **Operating specifications** frame, enter **5** for **Reflux ratio**. Then, select **Bottoms rate** as the other specification and enter **8** for **Bottoms rate**. Note that the value for Bottoms rate is an initial guess and it will be varied to meet the product purity specification. **Blocks | COL-REC | Specifications | Setup | Configuration** sheet should look like this.

Configuration sheet showing Setup options and Operating specifications:

**Setup options**

- Calculation type: *Equilibrium*
- Number of stages: **100**
- Condenser: **Total**
- Reboiler: *Kettle*
- Valid phases: **Vapor-Liquid-Liquid**
- Convergence: **Strongly non-ideal liquid**

**Operating specifications**

- Bottoms rate: **Mole**, **8**, **kmol/hr**
- Reflux ratio: **Mole**, **5**
- Free water reflux ratio: **0**

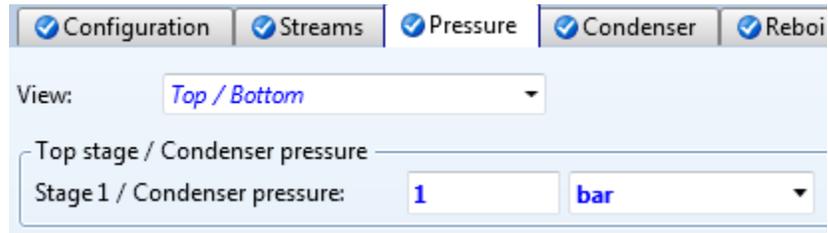
Buttons: Stage Wizard, Feed Basis

- 4.24. Go to the **Blocks | COL-REC | Specifications | Setup | Streams** sheet. In the **Feed streams** frame, enter **30** for **Stage** as shown below.

Streams sheet showing Feed streams:

Name	Stage	Convention
TO-REC	<b>30</b>	<i>Above-Stage</i>

- 4.25. Go to the **Blocks | COL- REC | Specifications | Setup | Pressure** sheet. Enter **1** for **Stage 1 / Condenser pressure**.



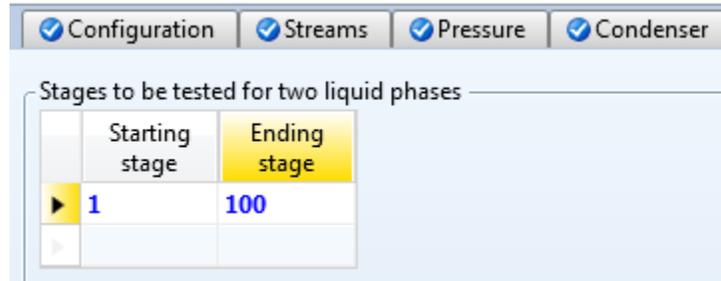
Configuration Streams Pressure Condenser Reboiler

View: *Top / Bottom*

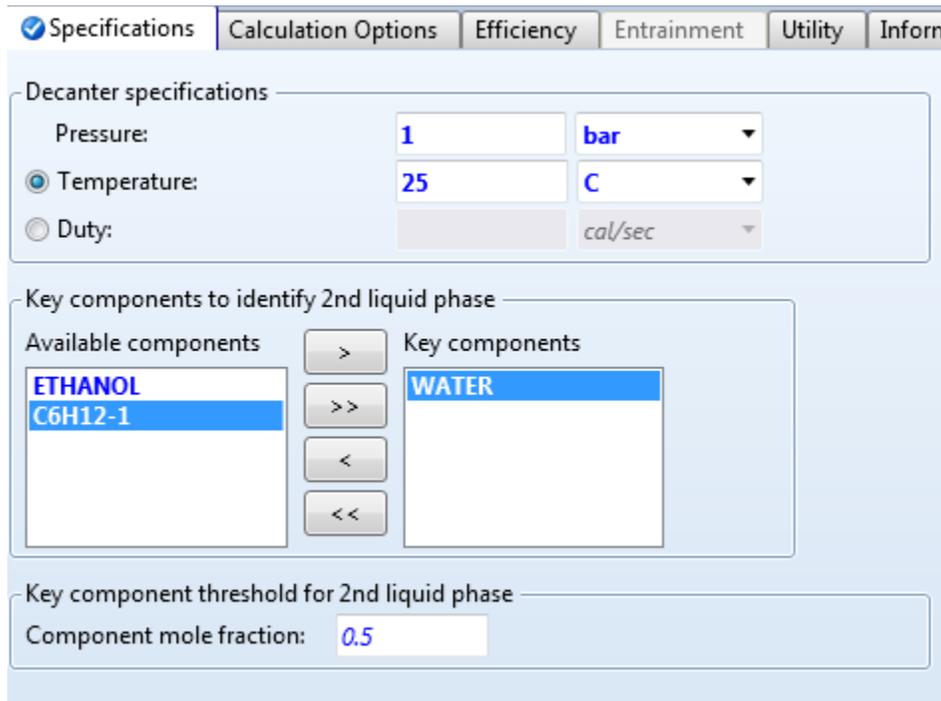
Top stage / Condenser pressure

Stage 1 / Condenser pressure: **1** **bar**

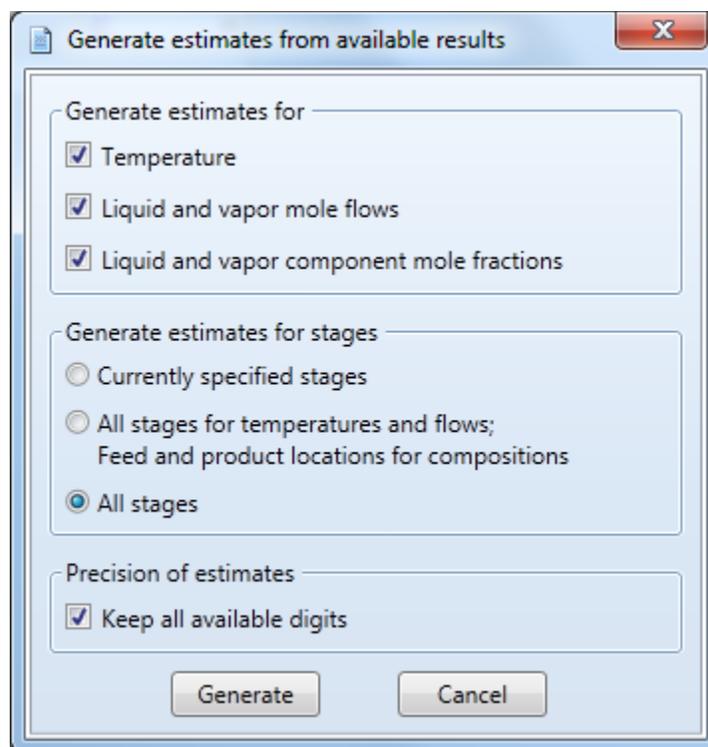
- 4.26. Go to the **Blocks | COL-REC | Specifications | Setup | 3-Phase** sheet. Enter **1** for **Starting stage** and **100** for **Ending stage**. Specify **WATER** as **Key components** in the **Key components to identify 2nd liquid phase** frame. This tells Aspen Plus that we expect the second liquid phase to be predominantly water, and since we do not know exactly which stages of the column will contain two liquid phases, we will ask Aspen Plus to check every stage.



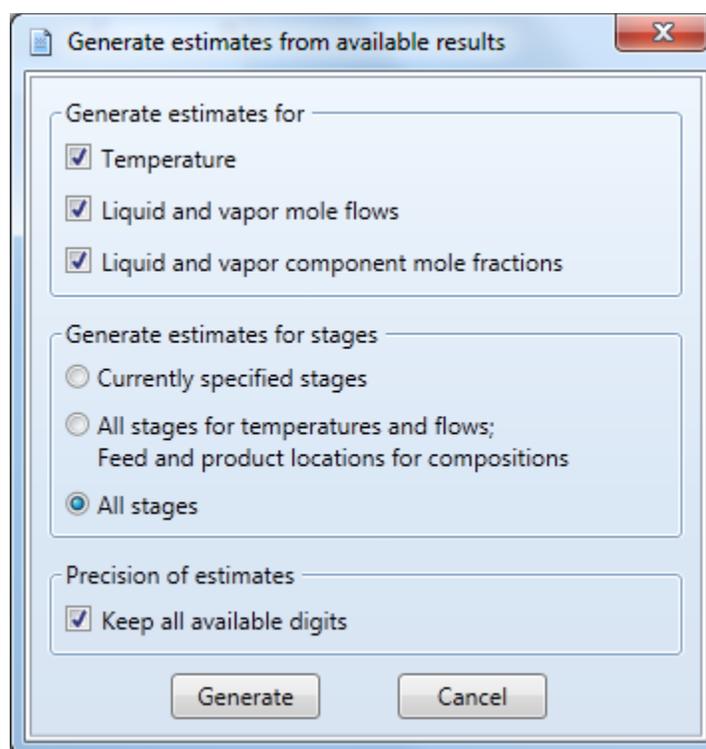
- 4.27. Go to the **Blocks | COL-REC | Convergence | Convergence | Basic** sheet. Increase **Maximum iterations** from **25** to **200**.
- 4.28. Go to the **Blocks | DECANTER | Input | Specifications** sheet. Enter **1** for **Pressure** and **25** for **Temperature**. In **Key components to identify 2nd liquid phase** frame, move **WATER** to Key components list. The **Blocks | DECANTER | Input | Specifications** sheet look like this:



- 4.29. Press the **F5** key to run the simulation and the simulation should complete without any error or warning.
- 4.30. Make the two columns numerically more robust. Go to the **Blocks | COL-MAIN | Specifications | Setup | Configuration** sheet. Change **Convergence** to **Custom**.
- 4.31. Go to the **Blocks | COL-MAIN | Convergence | Estimates | Temperature** sheet. Click the **Generate Estimates...** button. In the popup dialog box, select options to generate the most estimates as shown below. Then click **Generate** button and wait for estimate generation to complete.



- 4.32. Go to the **Blocks | COL-MAIN | Convergence | Convergence | Basic** sheet. Select **Newton** for **Algorithm**.
- 4.33. Go to the **Blocks | COL-MAIN | Convergence | Convergence | Advanced** sheet and select **Dogleg strategy** for **Stable-Meth**.
- 4.34. Now, we do the same for **COL-REC**. Go to the **Blocks | COL- REC | Specifications | Setup | Configuration** sheet. Change **Convergence** to **Custom**.
- 4.35. Go to the **Blocks | COL- REC | Convergence | Estimates | Temperature** sheet. Click the **Generate Estimates...** button. In the popup dialog box, select options to generate the most estimates as shown below. Then click **Generate** button and wait for estimate generation to complete.



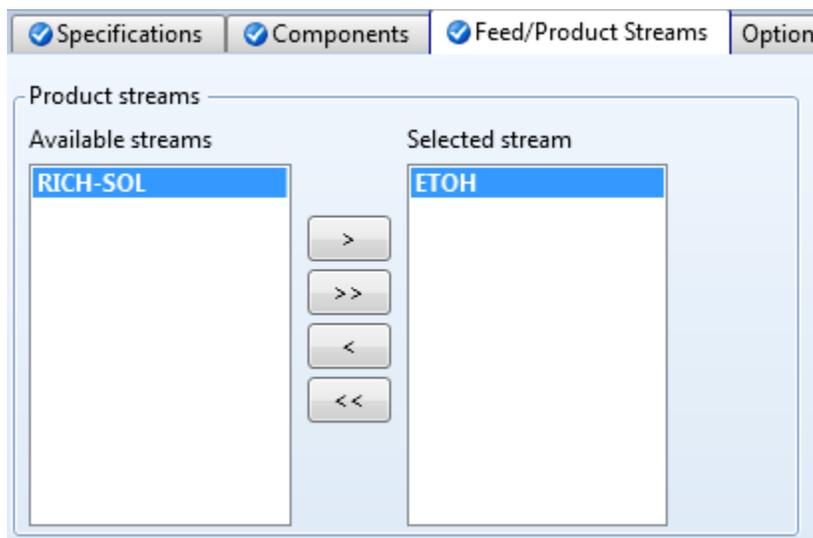
- 4.36. Go to the **Blocks | COL- REC | Convergence | Convergence | Basic** sheet. Select **Newton** for **Algorithm**.
- 4.37. Go to the **Blocks | COL- REC | Convergence | Convergence | Advanced** sheet and select **Dogleg** strategy for **Stable-Meth**.
- 4.38. Now, we use **Design-Specs / Vary** to achieve the desired product purity. Go to the **Blocks | COL-MAIN | Specifications | Design Specifications** form in the navigation pane. The object manager for **Design Specifications** is displayed. Click the **New...** button to create a new **Design Specification** called **1**.
- 4.39. Go to the **Blocks | COL-MAIN | Specifications | Design Specifications | 1 | Specifications** sheet. Select **Mole purity** for **Type** and enter **0.9995** for **Target**.

The screenshot shows the 'Specifications' tab of a software interface. The 'Description' field is empty. Under 'Design specification', the 'Type' dropdown is set to 'Mole purity'. Under 'Specification', the 'Target' field contains the value '.9995'. Under 'Stream type', the 'Product' radio button is selected, while 'Internal' and 'Decanter' are unselected.

- 4.40. Go to the **Blocks | COL-MAIN | Specifications | Design Specifications | 1 | Components** sheet. In the **Components** frame, move **ETHANOL** to the **Selected components** list as shown below.

The screenshot shows the 'Components' tab of the software interface. It features two lists: 'Available components' and 'Selected components'. The 'Available components' list contains 'WATER' and 'C6H12-1'. The 'Selected components' list contains 'ETHANOL'. Between the lists are four directional buttons: '>', '>>', '<', and '<<'. The 'Selected components' list is highlighted in blue.

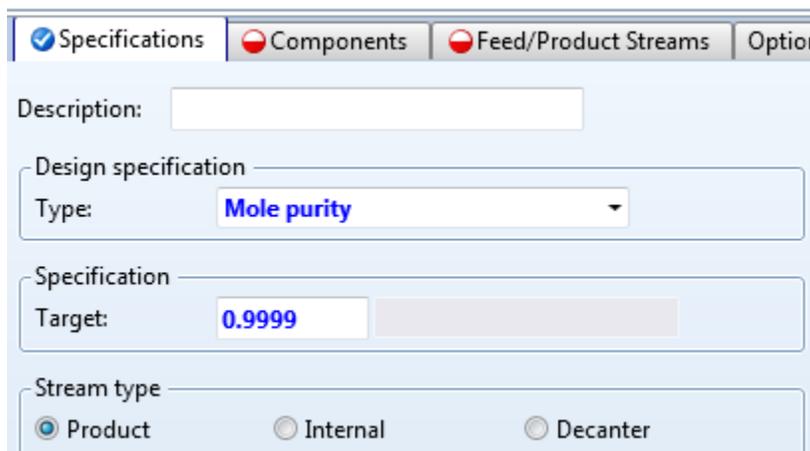
- 4.41. Go to the **Blocks | COL-MAIN | Specifications | Design Specifications | 1 | Feed/Product Streams** sheet. In the **Product streams** frame, move **ETOH** to the **Selected stream** list as shown below.



- 4.42. In the navigation pane, select **Blocks | COL-MAIN | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New...** button to create a new **Vary** called **1**.
- 4.43. Go to the **Blocks | COL-MAIN | Specifications | Vary | 1 | Specifications** sheet. Select **Bottoms rate** for **Type**. Enter **1** for **Lower bound** and **120** for **Upper bound**.

The screenshot shows the 'Specifications' tab for a 'Vary' object. The 'Adjusted variable' section has a 'Type' dropdown menu set to 'Bottoms rate'. The 'Upper and lower bounds' section has two rows: 'Lower bound' with a value of '1' and 'Upper bound' with a value of '120', both with units of 'kmol/hr'. The 'Optional' section has a 'Maximum step size' input field.

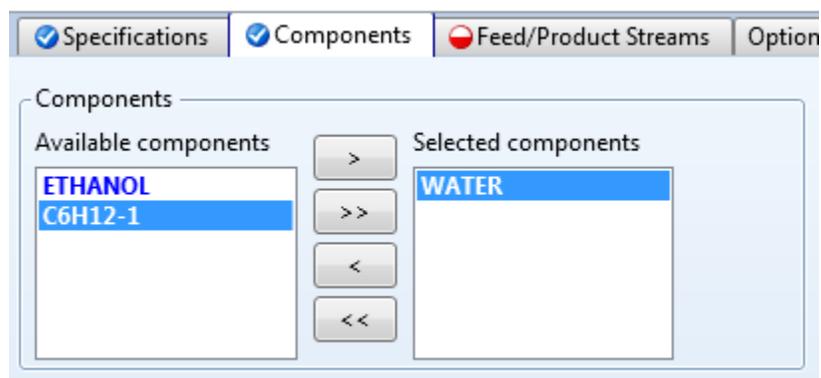
- 4.44. Now, we create Design Specs for **COL-REC**. In the navigation pane, select **Blocks | COL-REC Specifications | Design Specifications**. The object manager for **Design Specs** is displayed. Click the **New...** button to create a new **Design Specs** called **1**.
- 4.45. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 1 | Specifications** sheet. Select **Mole purity** for **Type** and enter **0.9999** for **Target**.



The screenshot shows a software interface for configuring Design Specifications. At the top, there are four tabs: 'Specifications' (selected with a blue checkmark), 'Components' (with a red circle), 'Feed/Product Streams' (with a red circle), and 'Options' (with a red circle). Below the tabs, there are several input fields and controls:

- Description:** An empty text input field.
- Design specification:** A section containing a 'Type' dropdown menu set to 'Mole purity'.
- Specification:** A section containing a 'Target' input field set to '0.9999'.
- Stream type:** A section with three radio buttons: 'Product' (selected), 'Internal', and 'Decanter'.

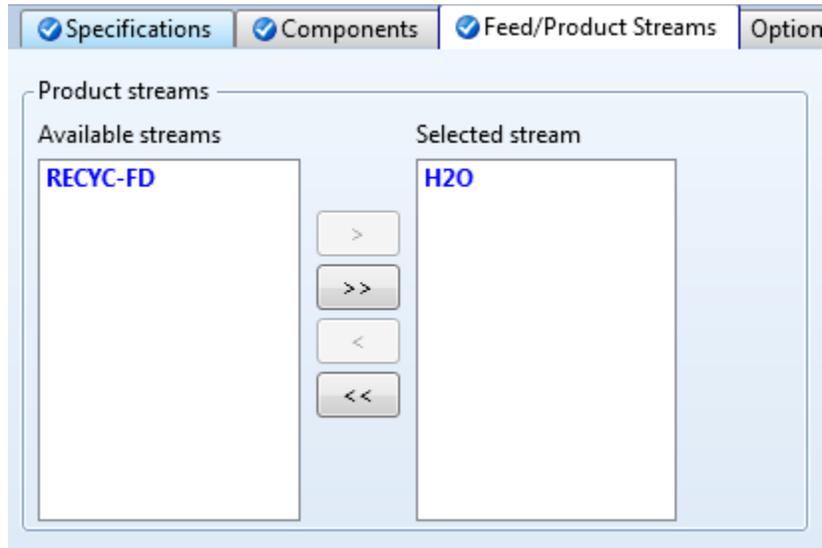
- 4.46. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 1 | Components** sheet. In the **Components** frame, move **WATER** to the **Selected components** as shown below.



The screenshot shows a software interface for managing components. At the top, there are four tabs: 'Specifications' (with a blue checkmark), 'Components' (selected with a blue checkmark), 'Feed/Product Streams' (with a red circle), and 'Options' (with a red circle). Below the tabs, there is a 'Components' section with two lists and four control buttons:

- Available components:** A list containing 'ETHANOL' and 'C6H12-1'.
- Selected components:** A list containing 'WATER'.
- Control buttons:** Four buttons between the lists: '>', '>>', '<', and '<<'.

- 4.47. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 1 | Feed/Product Streams** sheet. In the **Product streams** frame, move **H2O** to the **Selected stream** list as shown below.



- 4.48. In the navigation pane, select **Blocks | COL-REC | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New...** button to create a new **Vary** called **1**.
- 4.49. Go to the **Blocks | COL-REC | Specifications | Vary | 1 | Specifications** sheet. Select **Bottoms rate** for **Type**. Enter **1** for **Lower bound** and **25** for **Upper bound**.

The screenshot shows a configuration window with a tabbed menu at the top containing 'Specifications', 'Components', and 'Results'. The 'Specifications' tab is active. The window is divided into three sections: 'Adjusted variable', 'Upper and lower bounds', and 'Optional'. In the 'Adjusted variable' section, the 'Type' dropdown menu is set to 'Bottoms rate'. In the 'Upper and lower bounds' section, the 'Lower bound' is set to '1' and the 'Upper bound' is set to '25', both with units of 'kmol/hr'. The 'Optional' section contains a 'Maximum step size' input field which is currently empty.

- 4.50. Press the **F5** key to run the simulation and the simulation should complete without any error or warning.
- 4.51. Before the recycle loops can be closed, the open-loop simulation should approximate the closed-loop solution. Go to the **Streams | RECYC-FD | Results | Material** sheet. Copy the mole-based composition of the **RECYC-FD** stream.

Material	Vol.% Curves	Wt. % Curves	Petro. Curves
Display: Streams    Format: FULL			
			RECYC-FD
▶ Mole Flow kmol/hr			
▶ ETHANOL		43.4097	
▶ WATER		6.10179	
▶ C6H12-1		7.20126	
▶ Mole Frac			
▶ ETHANOL		0.765431	
▶ WATER		0.107591	
▶ C6H12-1		0.126978	
▶ Mass Frac			
▶ ETHANOL		0.736364	
▶ WATER		0.0404757	

- 4.52. Paste the mole fractions into the **Composition** field on **Streams | DUM-FEED | Input | Mixed** sheet. Increase the **Total flow rate** to **5 kmol/hr**.

Mixed   
  CI Solid   
  NC Solid   
  Flash Options   
  EO Options   
  Costing   
  Information

Specifications

Flash Type: Pressure    Vapor Fraction

State variables

Temperature:  C

Pressure:  bar

Vapor fraction:

Total flow basis: Mole

Total flow rate:  kmol/hr

Solvent:

Composition

Mole-Frac

Component	Value
▶ ETHANOL	0.765431
▶ WATER	0.107591
▶ C6H12-1	0.126978

Total:

- 4.53. Run the simulation without reinitializing.
- 4.54. Because the RadFrac blocks are using Newton's method to converge, large changes in the feed conditions can create errors in the simulation. Change the **Total flow rate** of **DUM-FEED** to each of the values in the table below and run the simulation after each change of the flow rate.

7	kmol/hr
9	kmol/hr
15	kmol/hr
25	kmol/hr
35	kmol/hr
57	kmol/hr

- 4.55. Because the flow rate of **DUM-FEED** has been increased, the flow rate of **SOLVENT** must be increased so that the column operates within the same distillation region. Change the **Total flow rate** to **150 kmol/hr** and run the simulation.

Mixed
  CI Solid
  NC Solid
  Flash Options
  EO Options
  Costing
  Information

Specifications

Flash Type:

State variables

Temperature:

Pressure:

Vapor fraction:

Total flow basis:

Total flow rate:

Solvent:

Composition

Component	Value
ETHANOL	
WATER	
C6H12-1	1

Total:

- 4.56. Copy the composition of **RECYC-FD** and paste it into **DUM-FEED** again. Increase the **Total flow rate** to **76 kmol/hr**.

Mixed | CI Solid | NC Solid | Flash Options | EO Options | Costing | Information

Specifications

Flash Type: Pressure Vapor Fraction

State variables

Temperature: C

Pressure: 1 bar

Vapor fraction: 0

Total flow basis: Mole

Total flow rate: 57 kmol/hr

Solvent:

Composition

Mole-Frac

Component	Value
ETHANOL	0.758251
WATER	0.106379
C6H12-1	0.13537

Total: 1

- 4.57. Increase the **Total flow rate** of **SOLVENT** to **170 kmol/hr** as well. Run the simulation.

Mixed | CI Solid | NC Solid | Flash Options | EO Options | Costing | Information

Specifications

Flash Type: Pressure Vapor Fraction

State variables

Temperature: C

Pressure: 1 bar

Vapor fraction: 0

Total flow basis: Mole

Total flow rate: 170 kmol/hr

Solvent:

Composition

Mole-Frac

Component	Value
ETHANOL	
WATER	
C6H12-1	1

Total: 1

- 4.58. Change the **Total flow rate** of **DUM-FEED** and **SOLVENT** to match the rows in the table below. Each time after changing both flow rates, run the simulation.

DUM-FEED Total flow rate		SOLVENT Total flow rate	
96	kmol/hr	190	kmol/hr
116	kmol/hr	210	kmol/hr
136	kmol/hr	230	kmol/hr
156	kmol/hr	250	kmol/hr
176	kmol/hr	270	kmol/hr
196	kmol/hr	290	kmol/hr

- 4.59. At this point, the dummy feed stream has composition and flow rate very similar to the top of the second column. Go to the **Results Summary | Streams | Materials** sheet. You should be able to see the following if you change the second stream to be **RECYC-FD**.

Material	Heat	Load	Work	Vol.% Curves	Wt. % Curves	Petro.
Display:	Streams		Format:	FULL		Stream Table
				DUM-FEED		RECYC-FD
▶ Substream: MIXED						
▶ Mole Flow kmol/hr						
▶ ETHANOL				148.617		145.098
▶ WATER				20.8502		20.249
▶ C6H12-1				26.5326		29.1036
▶ Mole Frac						
▶ ETHANOL				0.758251		0.746195
▶ WATER				0.106379		0.104134
▶ C6H12-1				0.13537		0.149671

- 4.60. Re-generate all estimates for **COL-MAIN** and **COL-REC**. Re-initialize and re-run the simulation. The simulation should converge.
- 4.61. Go to the **Streams | REC-SOL | Results | Material** sheet. Copy the composition of the **REC-SOL** stream from the stream results.

Material	Vol.% Curves	Wt. % Curves	Pet
Display: Streams Format: FULL			
	REC-SOL		
▶ Substream: MIXED			
▶ Mole Flow kmol/hr			
▶ ETHANOL		33.3822	
▶ WATER		0.857474	
▶ C6H12-1		287.4	
▶ Mole Frac			
▶ ETHANOL		0.103787	
▶ WATER		0.00266594	
▶ C6H12-1		0.893547	

- 4.62. Paste the compositions into the **SOLVENT | Input | Mixed** sheet. Increase the **Total flow rate** to **360 kmol/hr**.

Mixed	CI Solid	NC Solid	Flash Options	EO Options	Costing	Information
Specifications						
Flash Type:		Pressure	Vapor Fraction			
State variables						
Temperature:		C				
Pressure:	1	bar				
Vapor fraction:	0					
Total flow basis:	Mole					
Total flow rate:	360	kmol/hr				
Solvent:						
Composition						
Mole-Frac						
	Component	Value				
▶	ETHANOL	0.103787				
▶	WATER	0.00266594				
▶	C6H12-1	0.893547				
Total:		1				

- 4.63. Run the simulation. There should be no errors. Re-generate all estimates again for both **COL-MAIN** and **COL-REC** according to instructions in step 4.31.
- 4.64. Go to the **Blocks | COL-MAIN | Convergence | Convergence** form. Change the **Initialization method** to **Azeotropic** and select **Medium** for **Damping level**.

Basic convergence

Algorithm: **Newton**

Maximum iterations: **200**

Error tolerance:

Methods

Initialization method: **Azeotropic**

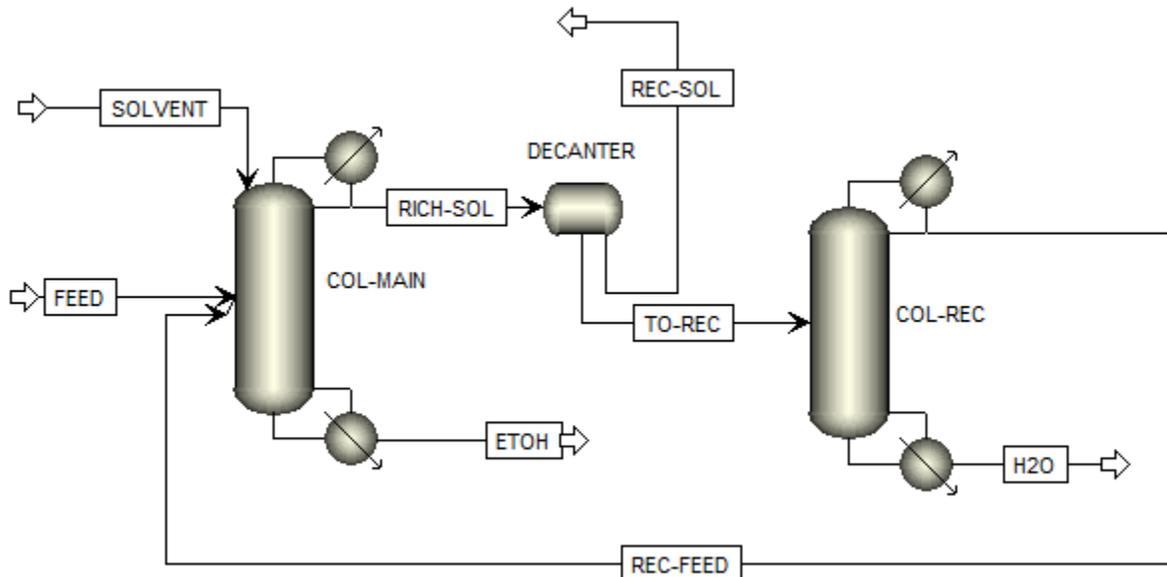
Damping level: **Medium**

Liquid-liquid phase splitting method: *Gibbs*

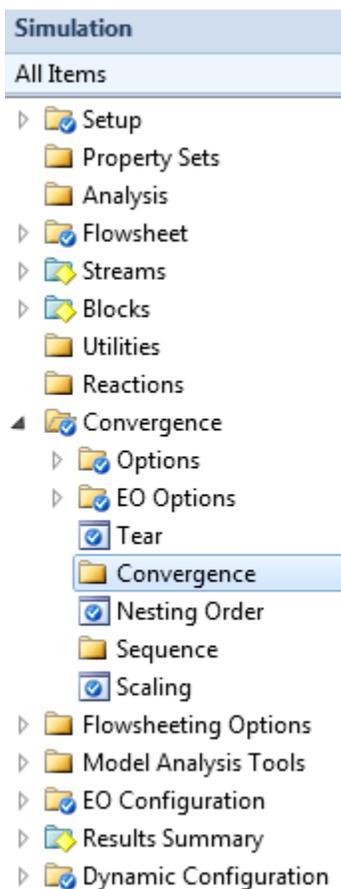
Solids handling: *Overall*

Salt precipitation handling: *Include*

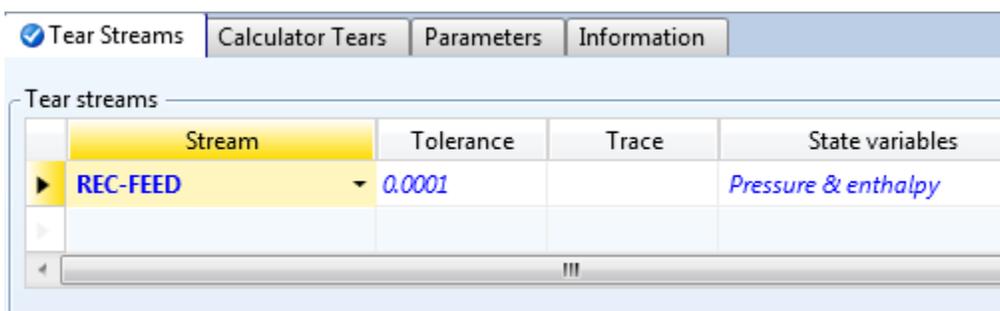
- 4.65. Make the same change for **COL-REC**. This increases the robustness of the convergence in these blocks.
- 4.66. Select both **DUM-FEED** and **RECYC-FD**. Right-click one of them and select **Join Streams**. Rename the stream **REC-FEED**. The connectivity is shown below.



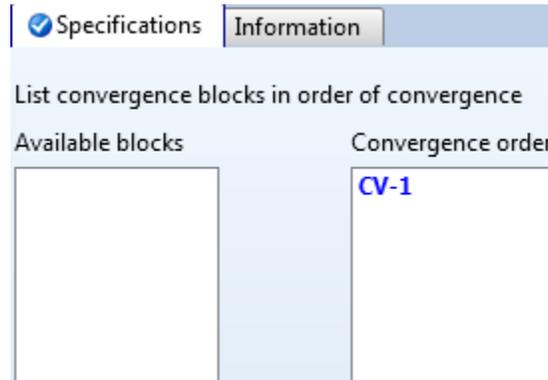
4.67. In the navigation pane, click **Convergence | Convergence**.



4.68. Click the **New** button. Let Aspen Plus name it **CV-1** and select **Wegstein** as the **Type**. Select **REC-FEED** as the **Stream**.



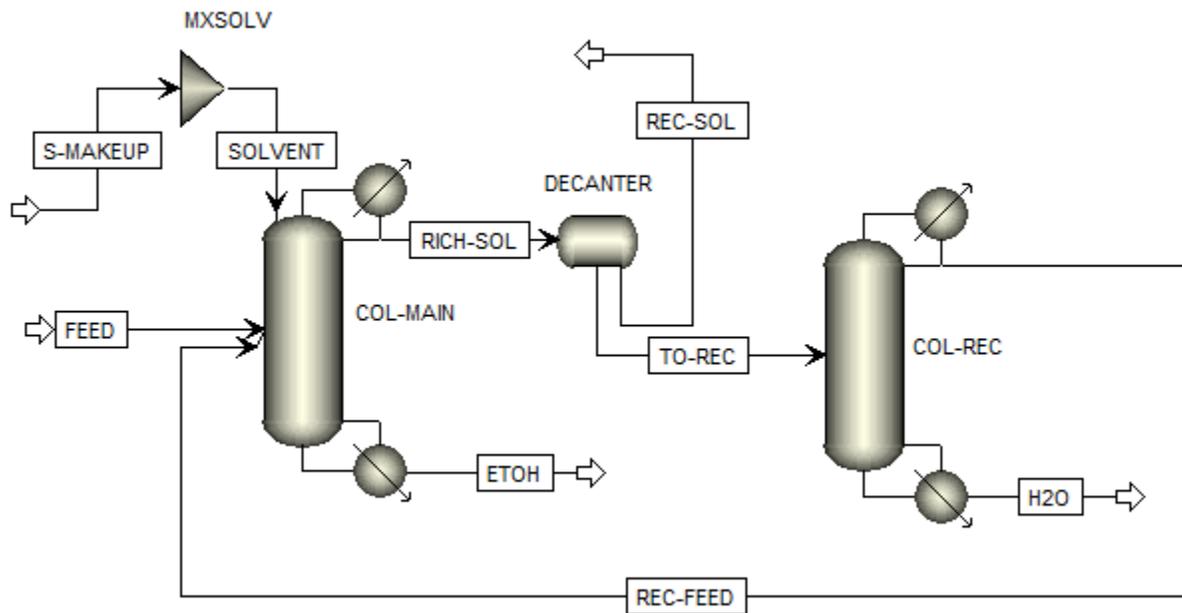
- 4.69. In the navigation pane, go to the **Convergence | Nesting Order | Specifications** sheet. Move **CV-1** to the **Convergence order** list as shown below.



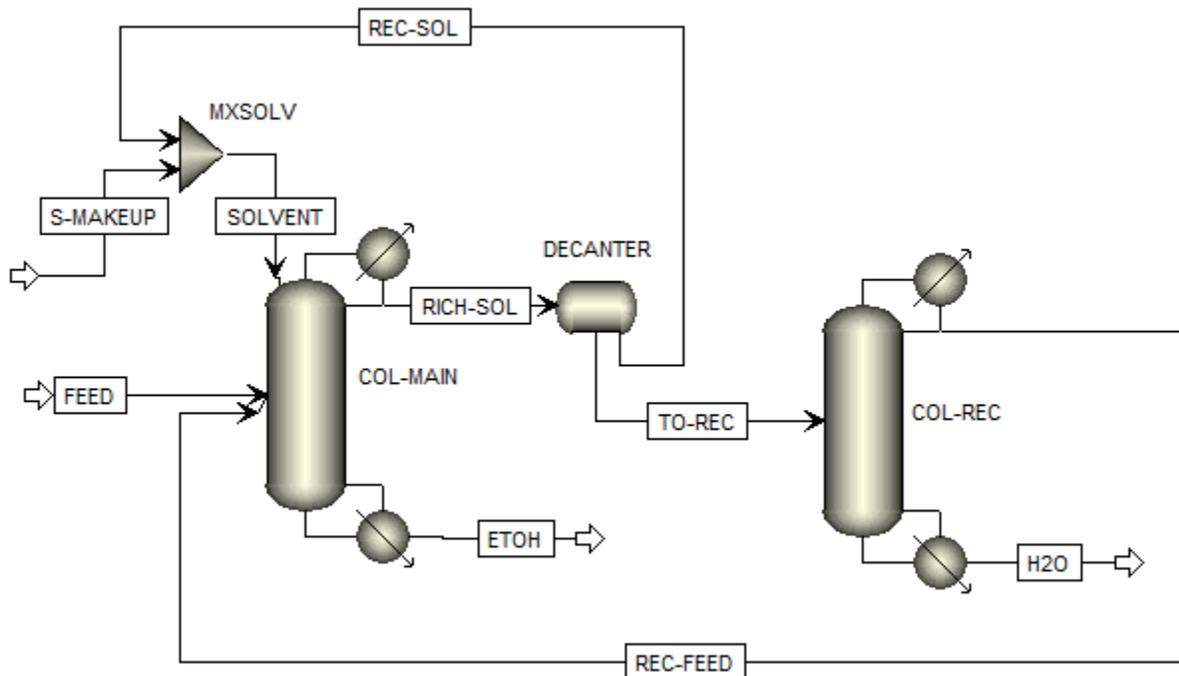
- 4.70. Run the simulation. There should be no errors.
- 4.71. Now, let us check solvent loss to **ETOH** and **H2O**. Go to the **Results Summary | Streams | Material** sheet. Select **ETOH** and **H2O**. We lose **0.0432032 kmol/hr** of **cyclohexane** to stream **ETOH**. Therefore, we need to create a solvent makeup stream.

Material	Heat	Load	Work	Vol.% Curves	Wt. % Curves	Petro. Curves	Poly. Curves
Display: Streams		Format: FULL		Stream Table		Copy All	
	ETOH	FEED	H2O				
▶ Substream: MIXED							
▶ Mole Flow kmol/hr							
▶ ETHANOL	86.3633	87	0.00130065				
▶ WATER	7.0312e-17	13	13.0052				
▶ C6H12-1	0.0432032	0	1.712e-19				
▶ Mole Frac							
▶ ETHANOL	0.9995	0.87	0.0001				

- 4.72. Add a **Mixer** block, **MXSOLV**, before stream **SOLVENT** and add a solvent make-up stream, **S-MAKEUP**. The flowsheet should look like the screenshot below.



- 4.73. Close the second recycle loop. In the **Main Flowsheet** window, right click stream **REC-SOL** and select **Reconnect Destination** on the context menu. Move the mouse cursor over the inlet port icon of **MXSOLV** and click the inlet port icon. The flowsheet should look like the screenshot below.



- 4.74. Go to the **Streams | S-MAKEUP | Input | Mixed** sheet. Enter **25** for **Temperature**, **1** for **Pressure** and **0.00001** for **Total flow rate**. In the **Composition** frame, select **Mole-Frac** and enter **1** for **C6H12-1**. For now, we just enter a tiny total flow rate for the solvent make-up stream as an estimate. Later on, we will use a **Balance** block to calculate its flowrate. Now the **Streams | S-MAKEUP | Input | Mixed** sheet should look like this.

Mixed | CISolid | NC Solid | Flash Options | EO Options | Costing | Information

Specifications

Flash Type: **Temperature** | **Pressure**

State variables

Temperature: **25** | **C**

Pressure: **1** | **bar**

Vapor fraction: [ ]

Total flow basis: **Mole**

Total flow rate: **1e-05** | **kmol/hr**

Solvent: [ ]

Composition

**Mole-Frac**

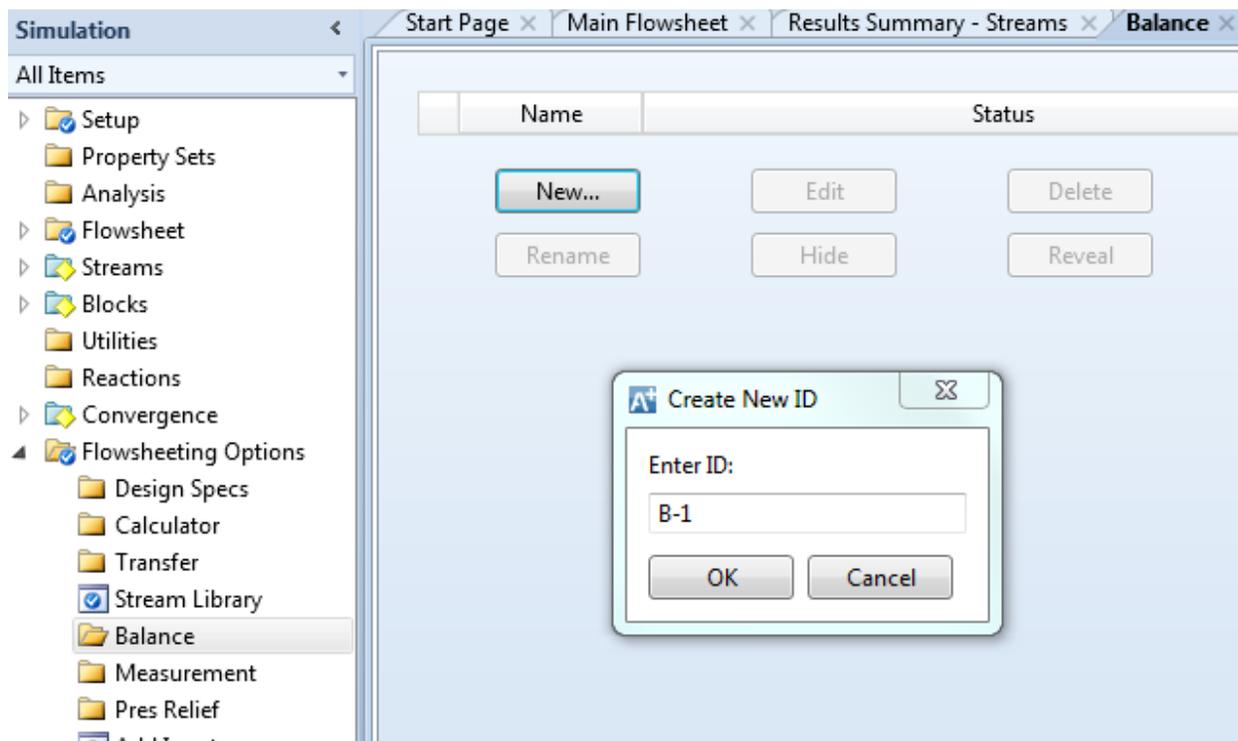
Component	Value
ETHANOL	
WATER	
C6H12-1	<b>1</b>

Total: 1

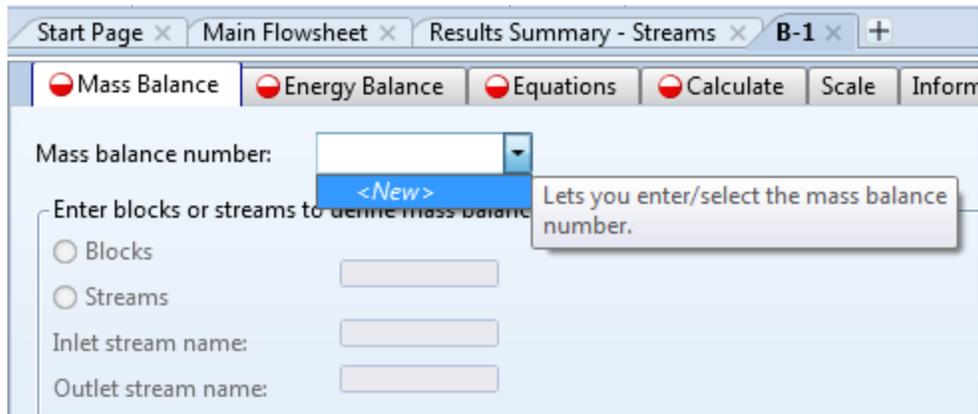
- 4.75. Go to the **Blocks | MXSOLV | Input | Flash Options** sheet. Note that the value for **Pressure** is **0**, indicating no pressure drop.

The screenshot shows the 'Flash Options' dialog box with the 'Information' tab selected. The 'Mixer specifications' section includes a 'Pressure' field set to '0' with a unit dropdown set to 'bar', and a 'Valid phases' dropdown set to 'Vapor-Liquid'. The 'Temperature estimate' section has a unit dropdown set to 'C'. The 'Convergence parameters' section includes a 'Maximum iterations' spinner set to '30' and an 'Error tolerance' field set to '0.0001'.

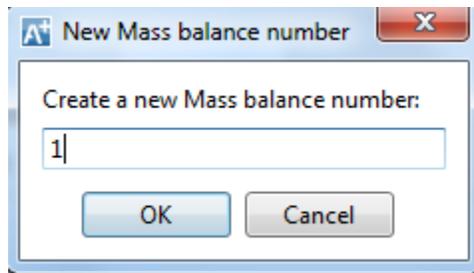
- 4.76. In the navigation pane, click the **Flowsheeting Options | Balance** node. The object manager for **Balance** is displayed. Click the **New...** button and click the **OK** button to create a **Balance** block called **B-1**.



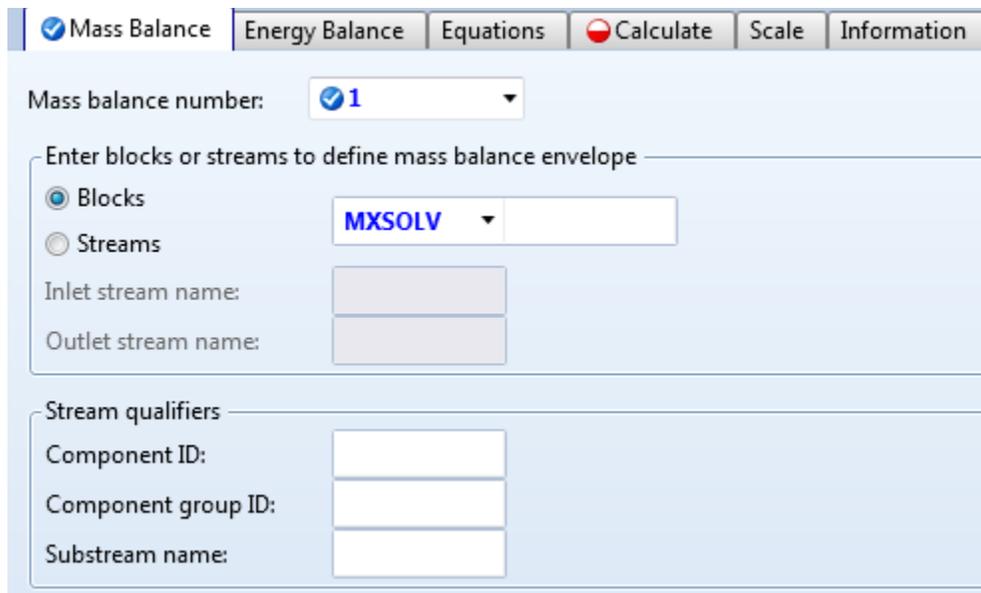
- 4.77. Go to the **Flowsheeting Options | Balance | B-1 | Setup | Mass Balance** sheet. Click the **Mass balance number** dropdown box and click **<New>**.



4.78. The **New Mass balance number** window pops up. Click the **OK** button.



4.79. In the **Enter blocks or streams to define mass balance envelope** frame, select **MXSOLV** for **Blocks**.



- 4.80. Go to the **Flowsheeting Options | Balance | B-1 | Setup | Calculate** sheet. Select **S-MAKEUP** for **Stream name**. This **B-1** balance block will calculate the flowrate of stream **S-MAKEUP**.

Mass Balance Energy Balance Equations Calculate Scale Information

Stream name: S-MAKEUP

Calculate enthalpy  Update calculated variables

Flow variables to be calculated

Total flow

Substream flows

Substream name:

Component flows

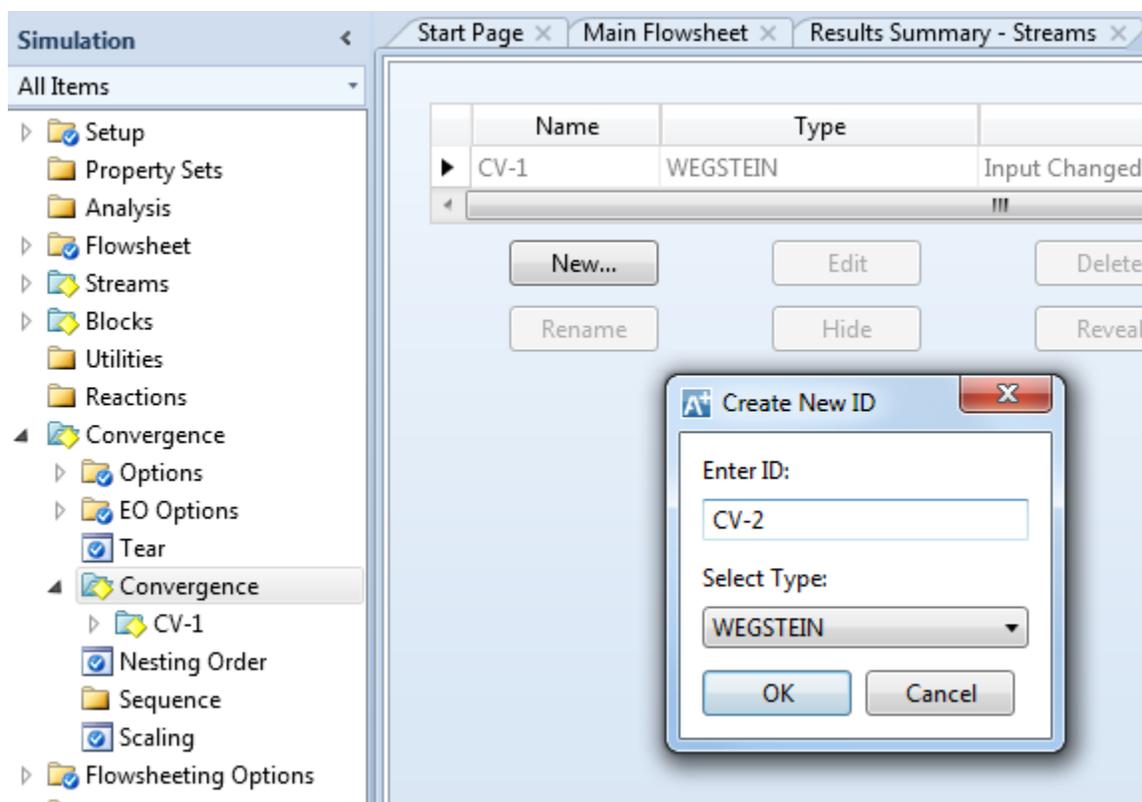
Component ID:

Component group ID:

Substream name:

None

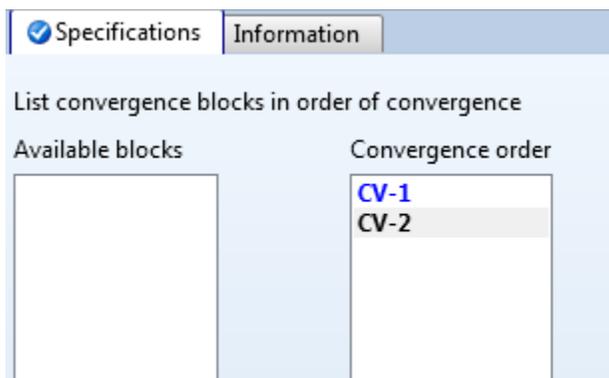
- 4.81. Create a convergence block for the second recycle stream. In the navigation pane, click **Convergence | Convergence**. The **Convergence** object manager is displayed. Click the **New...** button and the **Create New ID** window pops up. Select **WEGSTEIN** for **Select Type** and click the **OK** button.



- 4.82. On the **Convergence | Convergence | CV-2 | Input | Tear Streams** sheet, select **SOLVENT** in the first row in the **Stream** column as shown below.

Tear Streams				
Calculator Tears				
Parameters				
Information				
Tear streams				
	Stream	Tolerance	Trace	State variables
▶	<b>SOLVENT</b>	0.0001		Pressure & enthalpy
▶				

- 4.83. Go to the **Convergence | Nesting Order | Specifications** sheet. Move **CV-2** from the **Available blocks** list to the **Convergence order** list as shown below.



- 4.84. Click the **K** button in the **Home | Run** group of the ribbon to reinitialize simulation. Then, press the **F5** key to run the simulation again. Note that the simulator reports an error when the **B-1** block is executed for the first time and there is no error or warning for subsequent executions of the **B-1** block. Therefore, this error can be safely ignored.
- 4.85. Go to the **Streams | S-MAKEUP | Results | Material** sheet. Note that the flowrate for **C6H12-1** is **0.459136 kmol/hr** instead of the originally entered 0.00001 kmol/hr.

Material		Vol. % Curves	Wt. % Curves	Petro. Curves	Poly. Curves
Display: Streams		Format: FULL		Stream Table	
		S-MAKEUP			
▶ Substream: MIXED					
▶ Mole Flow kmol/hr					
▶ ETHANOL		0			
▶ WATER		0			
▶ C6H12-1		0.459136			

- 4.86. Go to the **Results Summary | Streams | Material** sheet. We can find information related to the feed stream and two product streams there.

	ETOH	FEED	H2O
▶ Substream: MIXED			
▶ Mole Flow kmol/hr			
▶ ETHANOL	86.9981	87	0.00130012
▶ WATER	7.401e-17	13	12.9999
▶ C6H12-1	0.0435208	0	1.5744e-20
▶ Mole Frac			
▶ ETHANOL	0.9995	0.87	0.0001
▶ WATER	8.5028e-19	0.13	0.9999
▶ C6H12-1	0.0005	0	1.2109e-21
▶ Mass Frac			
▶ ETHANOL	0.999087	0.944793	0.000255682
▶ WATER	3.3236e-19	0.0552068	0.999744
▶ C6H12-1	0.000913048	0	5.6562e-21
▶ Total Flow kmol/hr	87.0417	100	13.0012
▶ Total Flow kg/hr	4011.58	4242.21	234.257

## 5. Conclusions

In this example, cyclohexane is used as the entrainer to separate water and ethanol to produce anhydrous ethanol. By using the proper amount of solvent, we obtain pure ethanol from the bottom of the first column. The stream from the top of the first column is separated into two streams using a decanter: One stream contains mainly the solvent and is recycled back to the first column as solvent; the other stream is well within another distillation region so that we can use the second column to obtain pure water from the bottom of the second column. The top stream of the second column is recycled back to the first column as feed.

Please note that this process has two solutions and this example only presents one of them. Dist-016 provides details on how to obtain both solutions.

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