Separation of Acetone-Water with Aspen Plus® V8.0

Liquid-Liquid Extraction with 3-Methylhexane as the Solvent

1. Lesson Objectives

- Learn how to build an extraction and solvent recovery flowsheet.
- Learn how to configure **EXTRACT** and **RadFrac** blocks.
- Learn how to examine results and compare direct distillation versus extraction

2. Prerequisites

- Aspen Plus V8.0

3. Background

Water has a high latent heat (heat of vaporization) compared to many other components. For the separation of a water-acetone mixture (50 wt-% each), it may be more energy efficient to use extraction instead of direct distillation. In this example, we compare direct distillation to extraction using 3-methylhexane as the solvent.

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

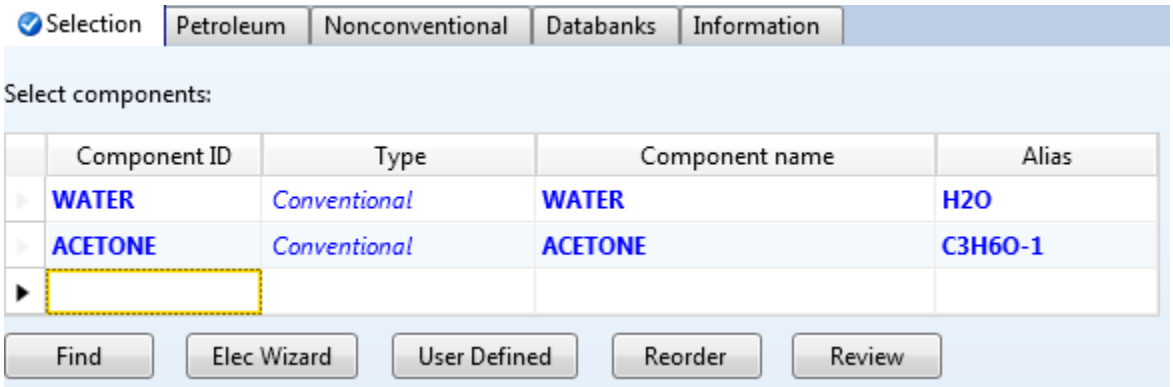
Determine whether distillation or extraction is more energy intensive and by how much for separating a 50 wt-% acetone 50 wt-% water stream.

Aspen Plus Solution

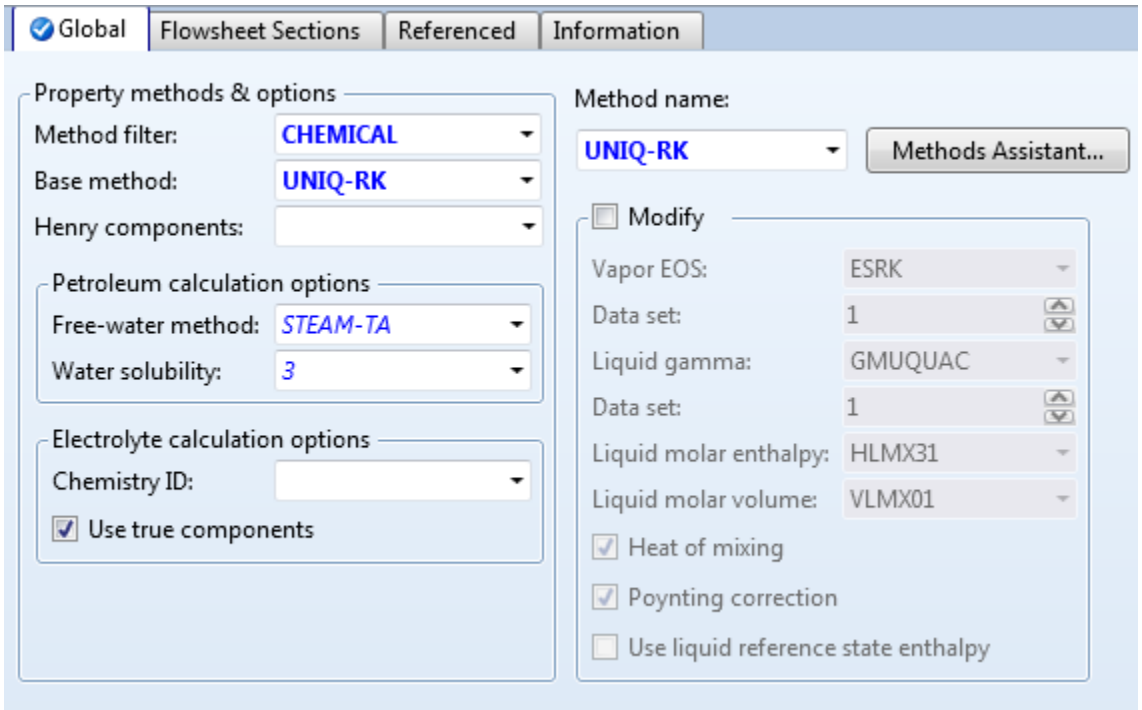
If you are unfamiliar with how to start Aspen Plus, select components, define methods, or construct a flowsheet, consult **Get Started Guide for New Users of Aspen Plus.pdf** for instructions.

4.01. Start a new simulation using the **Blank Simulation** template in Aspen Plus.

4.02. The **Components | Specification | Selection** sheet is displayed. Enter **WATER** and **ACETONE** in the **Component ID** column. Note that **Component name** and **Alias** are filled automatically.



4.03. Define methods. Press the **F4** key and the **Methods | Specifications | Global** sheet is displayed. Select **CHEMICAL** for **Method filter** and **UNIQ-RK** for **Base method**. This sheet should look like this.



4.04. Press the **F4** key. The **Methods | Parameters | Binary Interaction | UNIQ-1 | Input** sheet is displayed. Note that binary parameters are filled automatically.

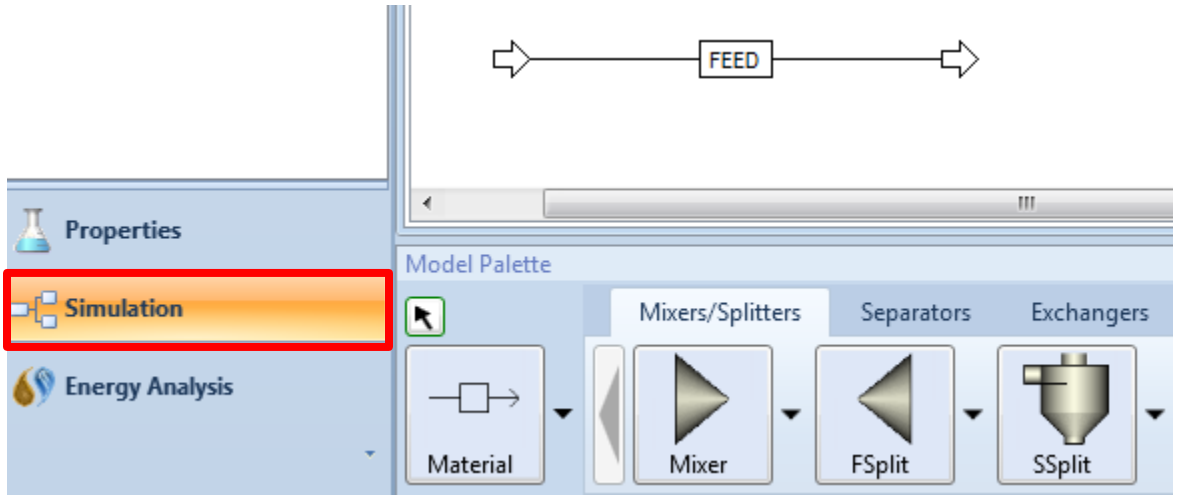
Input | Databanks | Information

Parameter: UNIQ Data set: 1

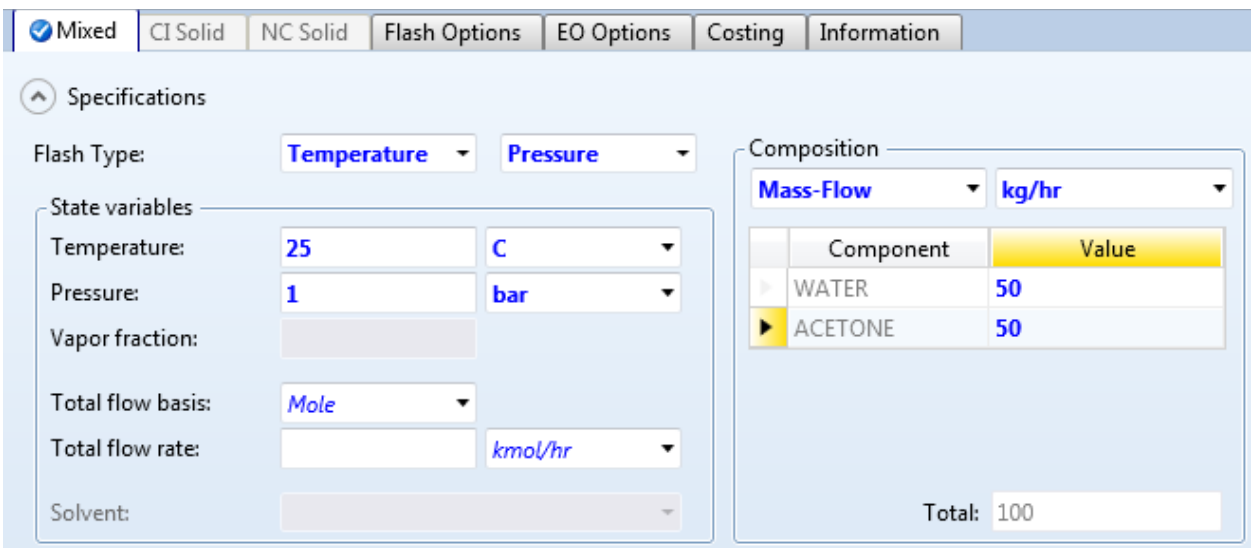
Temperature-dependent binary parameters

Component i	**WATER**
Component j	**ACETONE**
Temperature units	**C**
Source	**VLE-RK**
Property units	
AIJ	*-2.6066*
AJI	*3.0505*
BIJ	*924.15*
BJI	*-1380.05*
CIJ	*0*
CJI	*0*
DIJ	*0*
DJI	*0*
TLOWER	*20*
TUPPER	*230*
EIJ	*0*
EJI	*0*

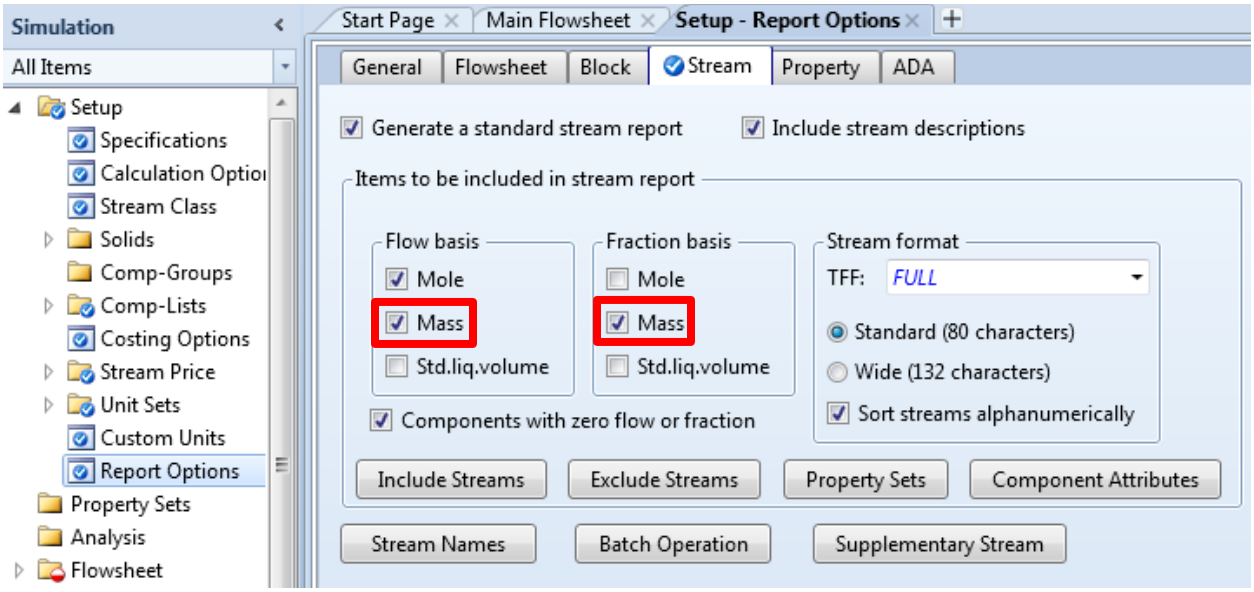
4.05. Move to the simulation environment by clicking the **Simulation** bar in the navigation pane. Draw one material stream called **FEED** on the flowsheet.



4.06. Double-click on stream **FEED** on the flowsheet or navigate to the **Streams | FEED | Input | Mixed** sheet. Enter **25** for **Temperature** and **1** for **Pressure.** In the **Composition** frame, select **Mass-Flow**. Enter **50** for both **WATER** and **ACETONE**. The **Streams | FEED | Input | Mixed** sheet should look like this.



4.07. Modify report options. Navigate to the **Setup | Report Options | Stream** sheet. In the **Flow basis** frame, check the **Mass** check box. In the **Fraction basis** frame, select **Mass** as shown below.

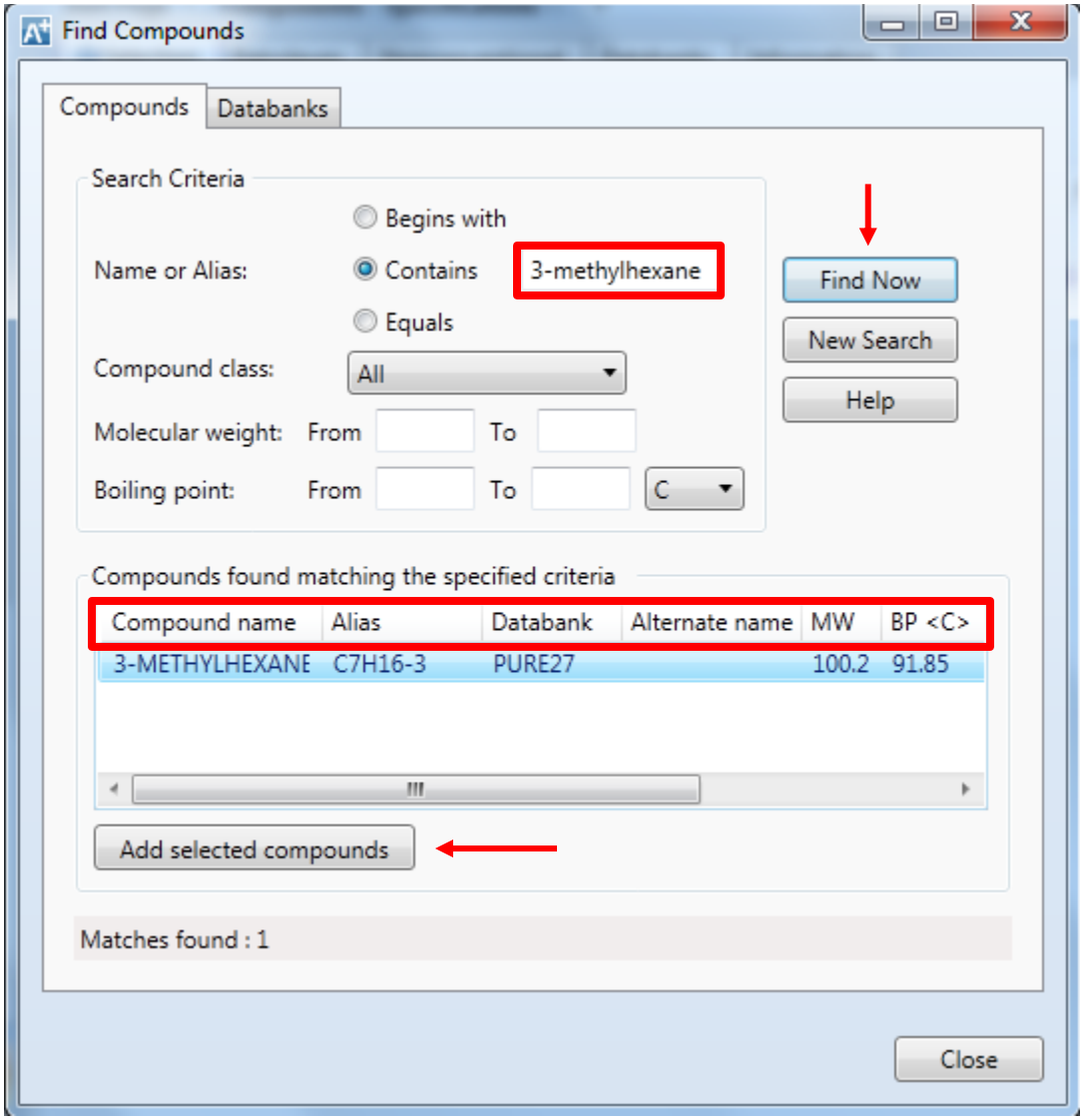


4.08. Save the simulation. On the ribbon, click **File** and click **Save As** to save the simulation as **Dist-009_Base.bkp**. Since we will compare the liquid-liquid extraction approach with direct distillation, we need to build one model for each approach. **Dist-009_Base.bkp** will be used as the starting point for building both models. Close the Aspen Plus Window.

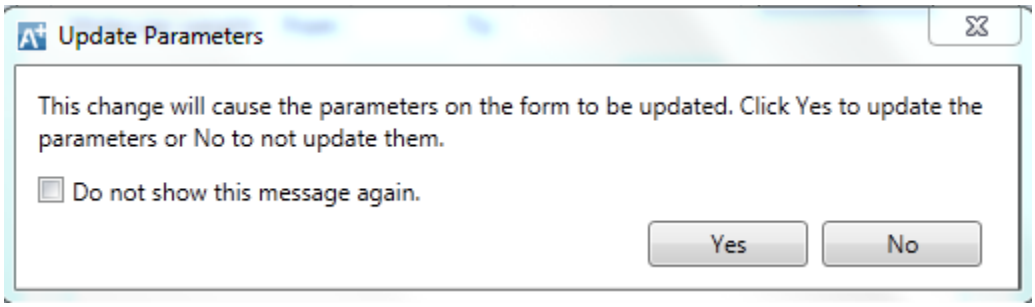
4.09. The next step is to build the model for the extraction approach. In Windows Explorer, make a copy of **Dist-009_Base.bkp** and rename it to **Dist-009_Acetone_Water_Separation_Extract.bkp**. Double-click the **Dist-009_Acetone_Water_Separation_Extract.bkp** to load it into Aspen Plus.

4.10. For the extraction approach, we will use 3-methylhexane as the solvent. Therefore, we need to add this component to our simulation. Click the **Properties** bar in the navigation pane (it is right above the **Simulation** bar) to move to the properties environment.

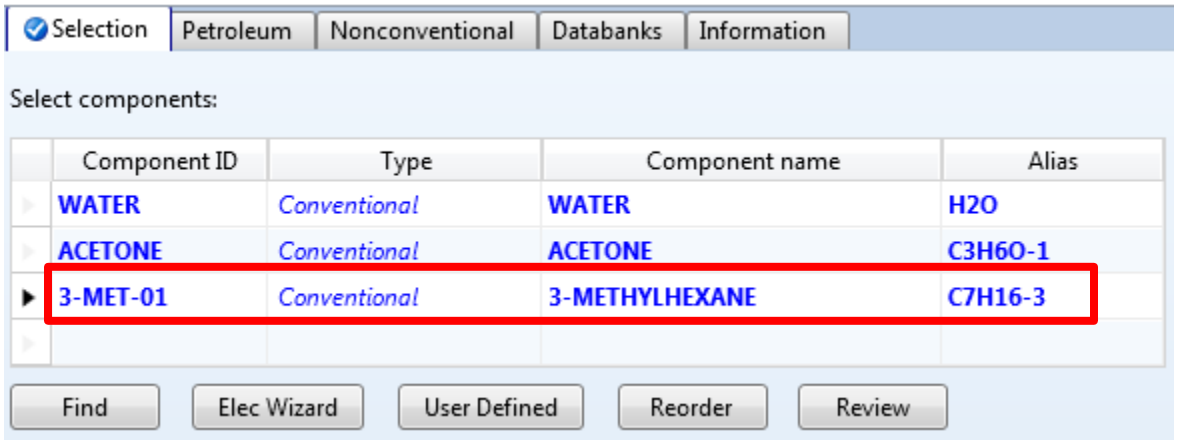
4.11. Go to the **Components | Specifications | Selection** sheet. Click the first empty cell in the third row, which is right under **ACETONE** in the **Component ID** column. With this click, none of the existing components is selected. Click the **Find** button. The **Find Compounds** window pops up. Enter **3-methylhexane** for **Contains**. Click the **Find Now** button and wait for searching to complete. Once completed, **3-METHYLHEXANE** should be listed. Click it in the list box to select it. Click the **Add selected compounds** button.



4.12. The **Update Parameters** dialog box pops up.



4.13. Click the **Yes** button and the **Update Parameters** dialog box disappears. Click the **Close** button in the **Find Compounds** window to close the **Find Compounds** window. Note that **3-METHYLHAXANE** has been added and appears on the **Components | Specifications | Selection** sheet as shown below.



Component ID	Type	Component name	Alias
WATER	Conventional	WATER	H2O
ACETONE	Conventional	ACETONE	C3H6O-1
3-MET-01	Conventional	3-METHYLHEXANE	C7H16-3

4.14. Press the **F4** key. The **Methods | Parameters | Binary Interaction | UNIQ-1 | Input** sheet is displayed. Note that binary parameters are updated automatically.

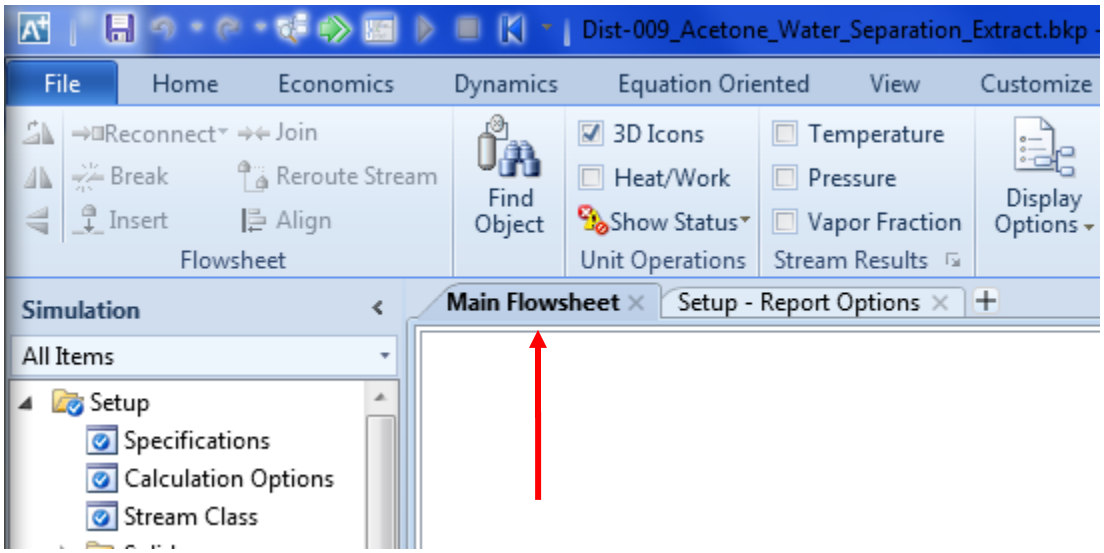
Input | Databanks | Information

Parameter: UNIQ Data set: 1 Dechema

Temperature-dependent binary parameters

Component i	WATER	WATER
Component j	ACETONE	3-MET-01
Temperature units	C	C
Source	VLE-RK	LLE-LIT
Property units		
AIJ	-2.6066	0
AJI	3.0505	0
BIJ	924.15	-535.55
BJI	-1380.05	-1401.7
CIJ	0	0
CJI	0	0
DIJ	0	0
DJI	0	0
TLOWER	20	20
TUPPER	230	40
EIJ	0	0
EJI	0	0

4.15. Move to the simulation environment by clicking the **Simulation** bar in the navigation pane. Click the **Main Flowsheet** tab as shown below.

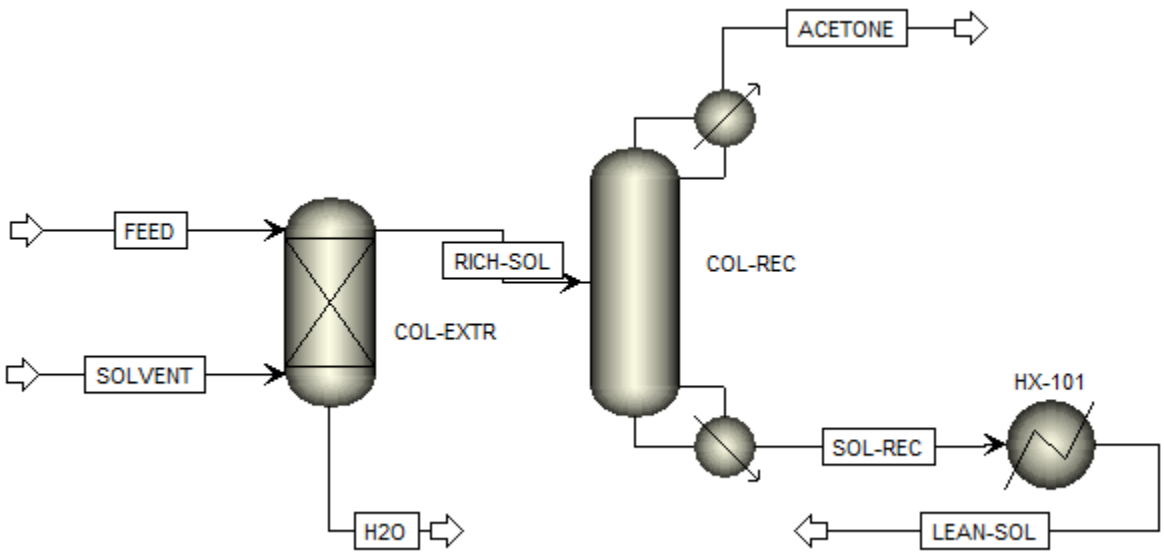


4.16. We need to expand the existing flowsheet (only one stream **FEED** now) to completion by adding the following blocks and streams.

Name	Type
COL-EXTR	Extract
COL-REC	RadFrac
HX-101	Heater

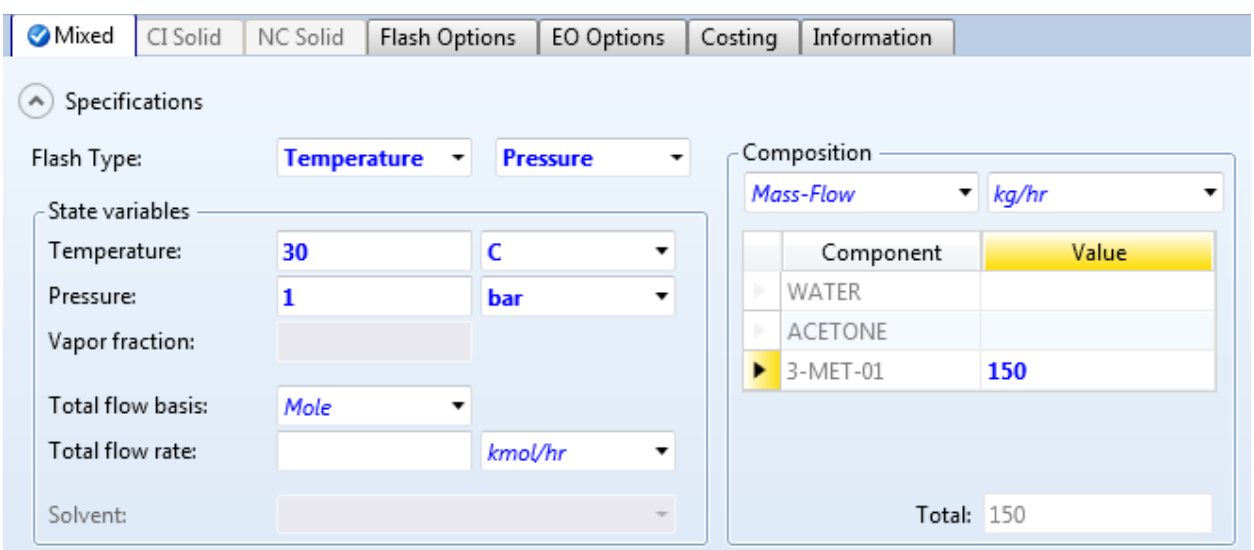
Name	Type
ACETONE	MATERIAL
FEED	MATERIAL
H2O	MATERIAL
LEAN-SOL	MATERIAL
RICH-SOL	MATERIAL
SOL-REC	MATERIAL
SOLVENT	MATERIAL

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

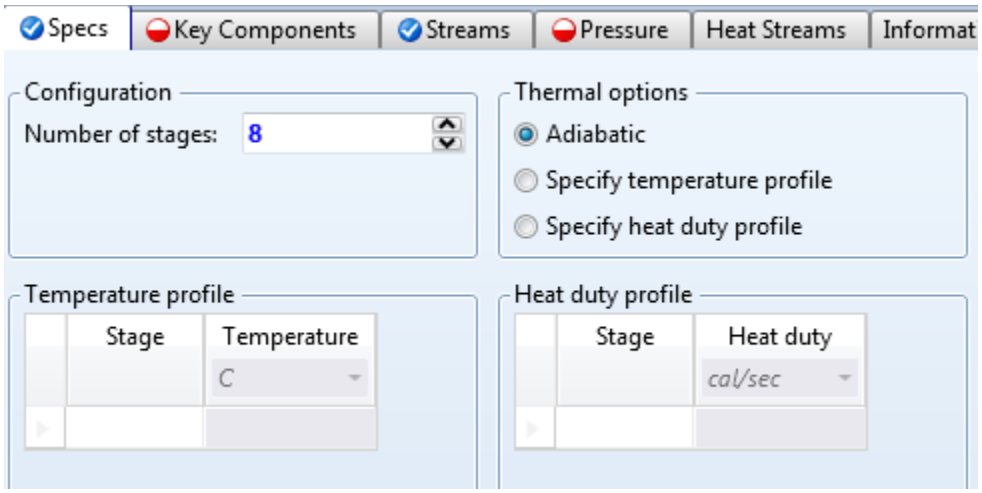


4.18. Stream **SOLVENT** will be connected with stream **LEAN-SOL** later. Heater **HX-101** is needed because the purities of product streams **H2O** and **ACETONE** heavily depend on the temperature of the solvent stream entering **COL-EXTR**.

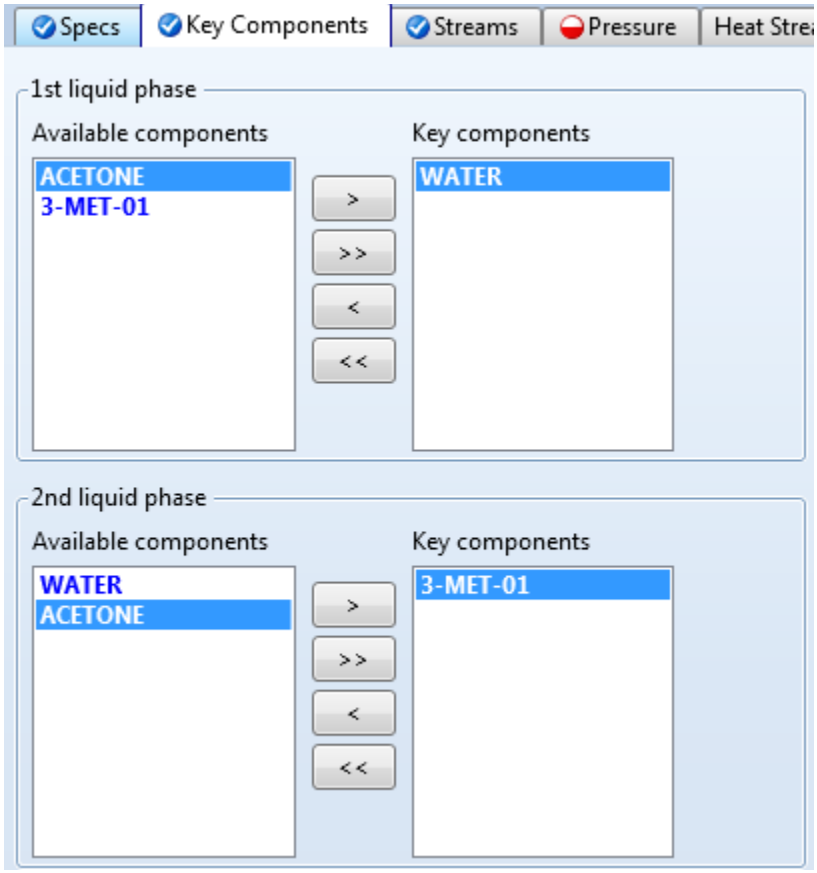
4.19. Go to the **Streams | SOLVENT | Input | Mixed** sheet. Enter **30°C** for **Temperature** and **1 bar** for **Pressure**. In the **Composition** frame, select **Mass-Flow**. Enter **150 kg/hr** for **3-MET-1**. We use 30 °C because (1) it is low enough for the extraction of acetone from the water phase to the solvent phase; and (2) cooling in **HX-101** can be achieved using water. We use 150 kg/hr because this flow rate is large enough to extract acetone from water. The **Streams | SOLVENT | Input | Mixed** sheet should look like this.



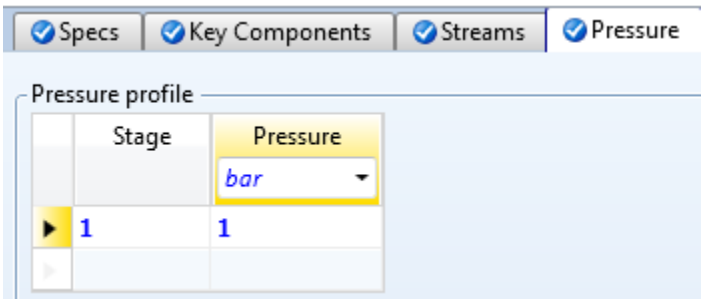
4.20. Specify **COL-EXTR**. Go to the **Blocks | COL-EXTR | Setup | Specs** sheet. Enter **8** for the **Number of stages** as shown below.



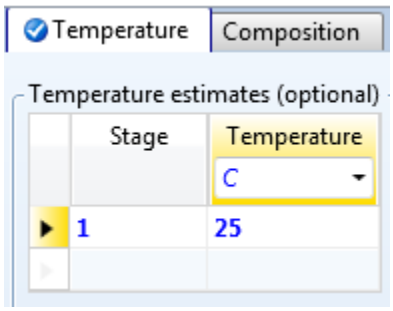
4.21. Go to the **Blocks | COL-EXTR | Setup | Key Components** sheet. In the **1st liquid phase** frame, move **WATER** to the **Key components** list. In the **2nd liquid phase** frame, move **3-MET-1** to the **Key components** list. The sheet looks like this.



4.22. Go to the **Blocks | COL-EXTR | Setup | Pressure** sheet. Enter **1** under **Stage** and **1** under **Pressure** as shown below.

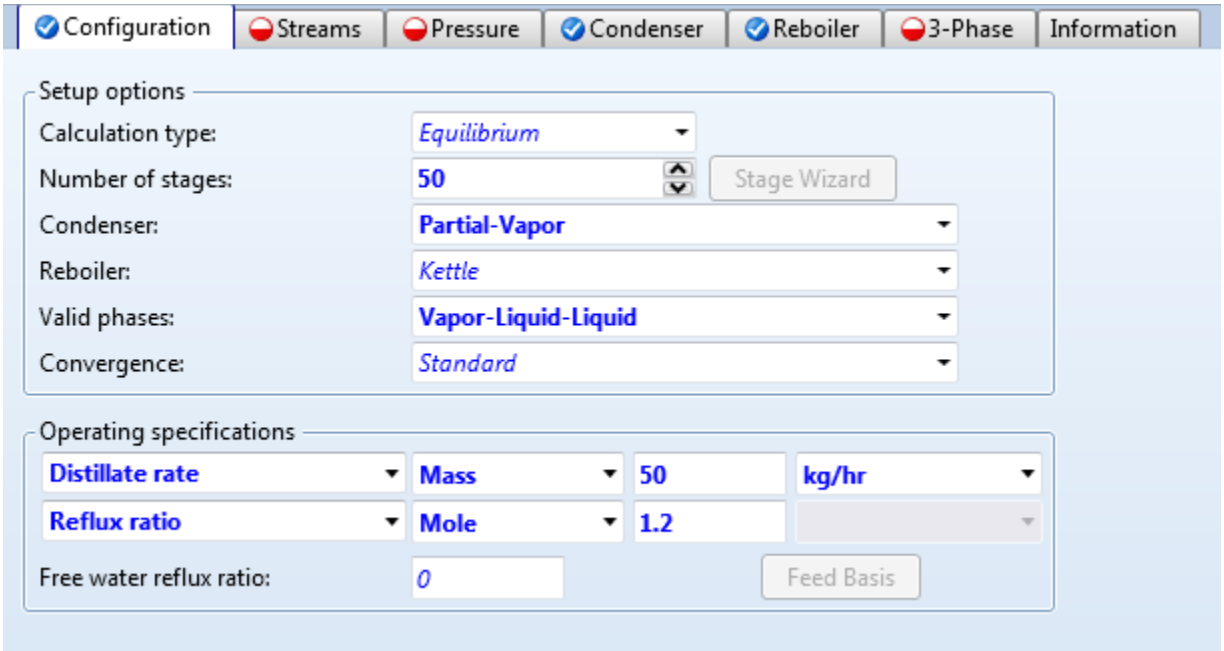


4.23. Go to the **Blocks | COL-EXTR | Estimates | Temperature** sheet. Enter **1** under **Stage** and **25** under **Temperature** as shown below.

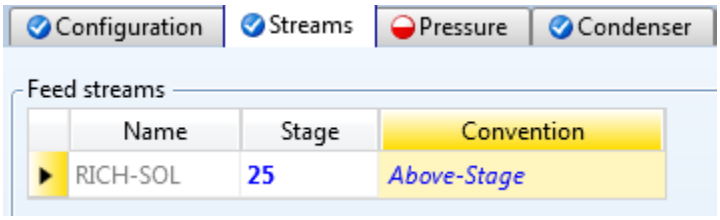


4.24. Go to the **Blocks | COL-EXTR | Convergence | Outside Loop** sheet. Enter **200** for **Maximum iterations** and **1e-07** for **Error tolerance**.

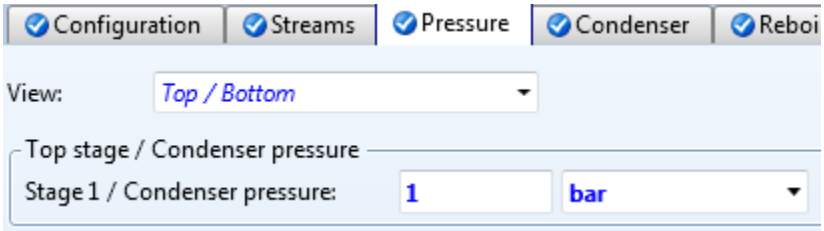
4.25. Specify **COL-REC**. Go to the **Blocks | COL-REC | Specifications | Setup | Configuration** sheet. Enter **50** for **Number of stages**. Select **Partial-Vapor** for **Condenser** and **Vapor-Liquid-Liquid** for **Valid phases**. In the **Operating specifications** frame, select **Mass** basis for **Distillate rate**. Enter **50** for **Distillate rate** and **1.2** for **Reflux ratio**. The **Blocks | COL-REC | Specifications | Setup | Configuration** sheet should look like the screenshot below.



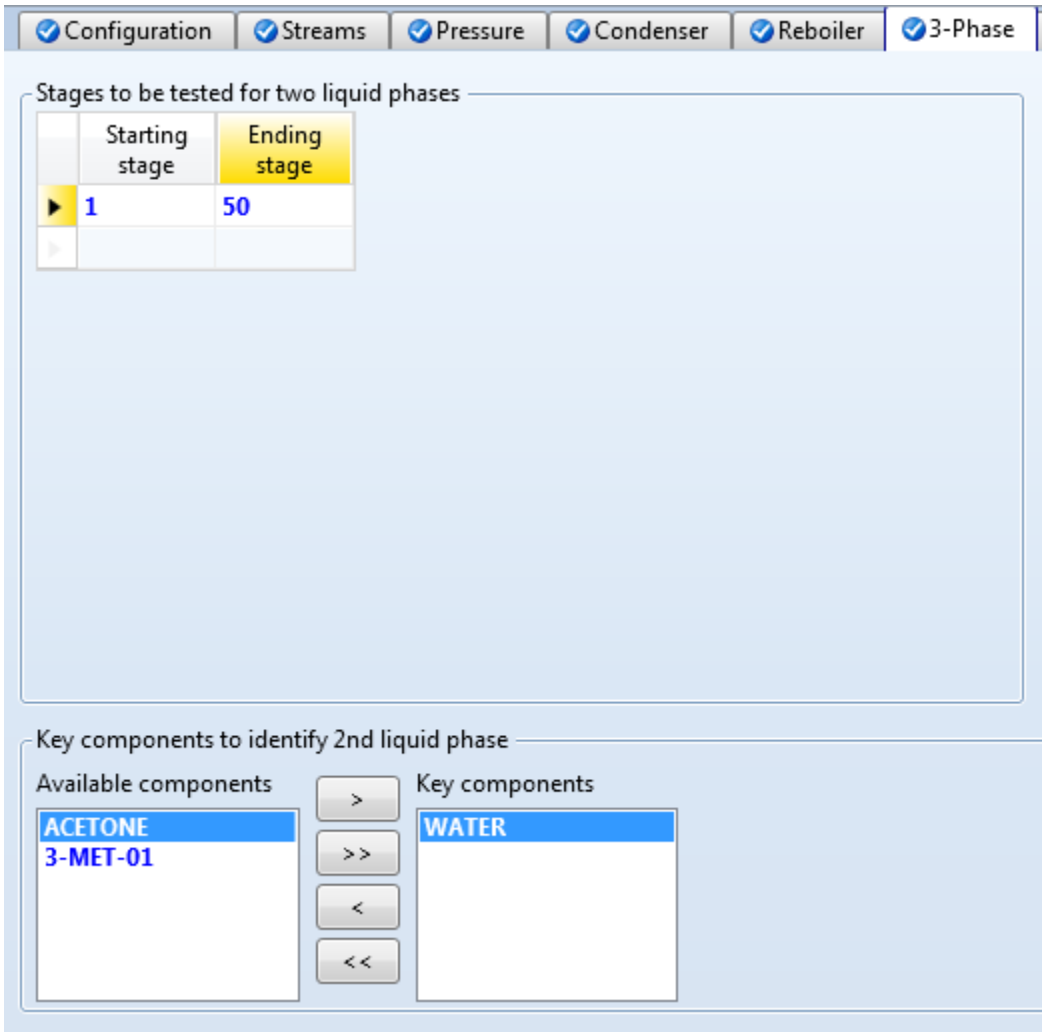
4.26. Go to the **Blocks | COL-REC | Specifications | Setup | Streams** sheet. In the **Feed streams** frame, enter **25** in the **Stage** column as shown below.



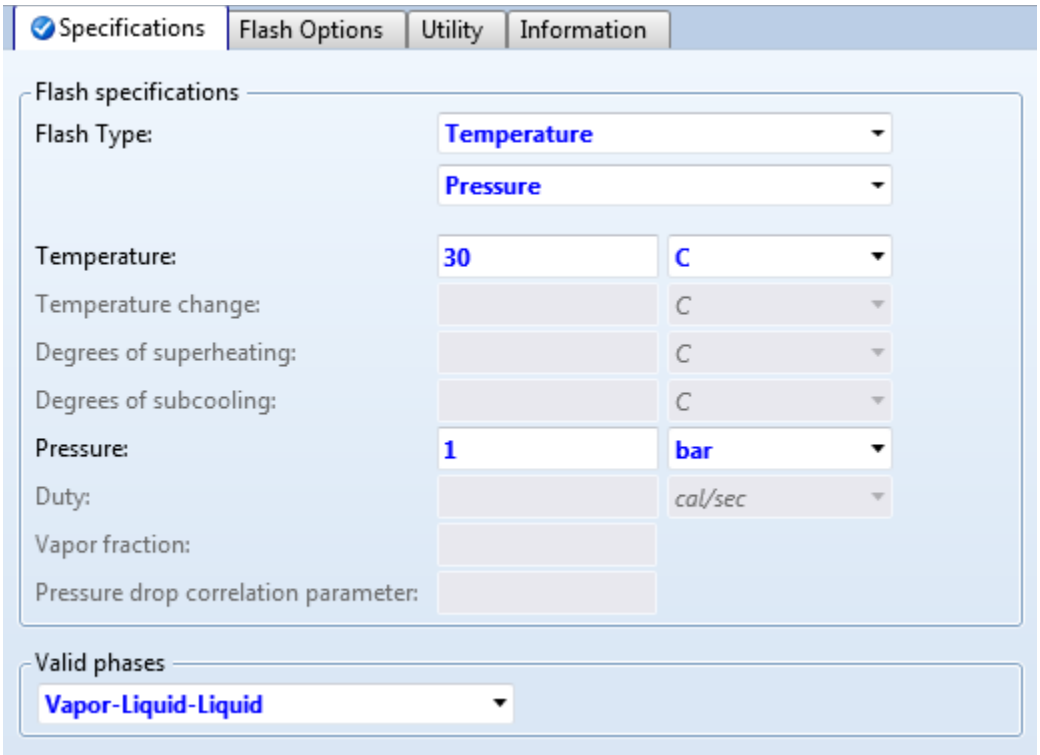
4.27. Go to the **Blocks | COL-REC | Specifications | Setup | Pressure** sheet. Enter **1** for **Stage 1 / Condenser pressure**.



4.28. Go to the **Blocks | COL-REC | Specifications | Setup | 3-Phase** sheet. Enter **1** for **Starting stage** and **50** for **Ending stage**. In the **Key components to identify 2nd liquid phase** frame, move **WATER** to the **Key components** list as shown below.



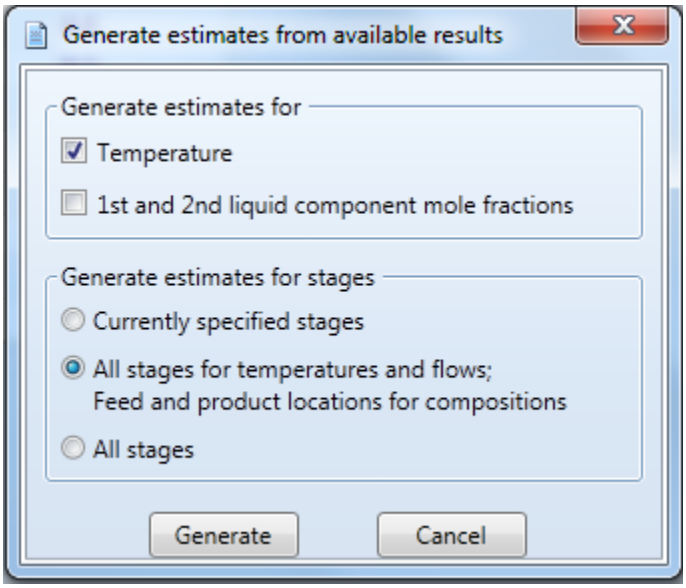
4.29. Specify **HX-101**. Go to the **Blocks | HX-101 | Input | Specifications** sheet. Enter **30** for **Temperature** and **1** for **Pressure**. Select **Vapor-Liquid-Liquid** for **Valid phases** as shown below.



4.30. Press the **F5** key to run the simulation. The simulation completes without any error or warning.

4.31. Generate temperature and composition estimates for the extractor. Now that the simulation has converged, we can generate estimates based on previous results which will help the simulation converge when we reinitialize and close the recycle loop.

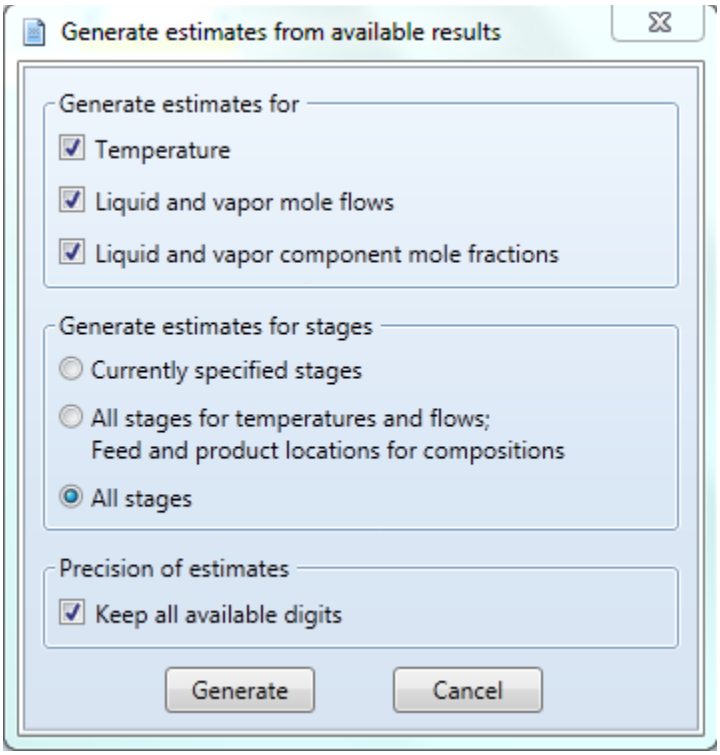
4.32. Go to the **Blocks | COL-EXTR | Estimates | Temperature** sheet. Click the **Generate Estimates…** button. The **Generate estimates from available results** window pops up. To generate the most estimates, make the selections shown below. Click the **Generate** button.



4.33. Once all estimates are generated, click the button in the **Home | Run** group of the ribbon to reinitialize simulation. Run the simulation again to ensure that it still converges.

4.34. We also want to make the **COL-REC** column more numerically robust. Go to the **Blocks | COL-REC | Specifications | Setup | Configuration** sheet. Select **Custom** for **Convergence**.

4.35. Go to the **Blocks | COL-REC | Convergence | Estimates | Temperature** sheet. Click the **Generate Estimates…** button. The **Generate estimates from available results** window pops up. Select the options that will generate the most estimates as shown below.

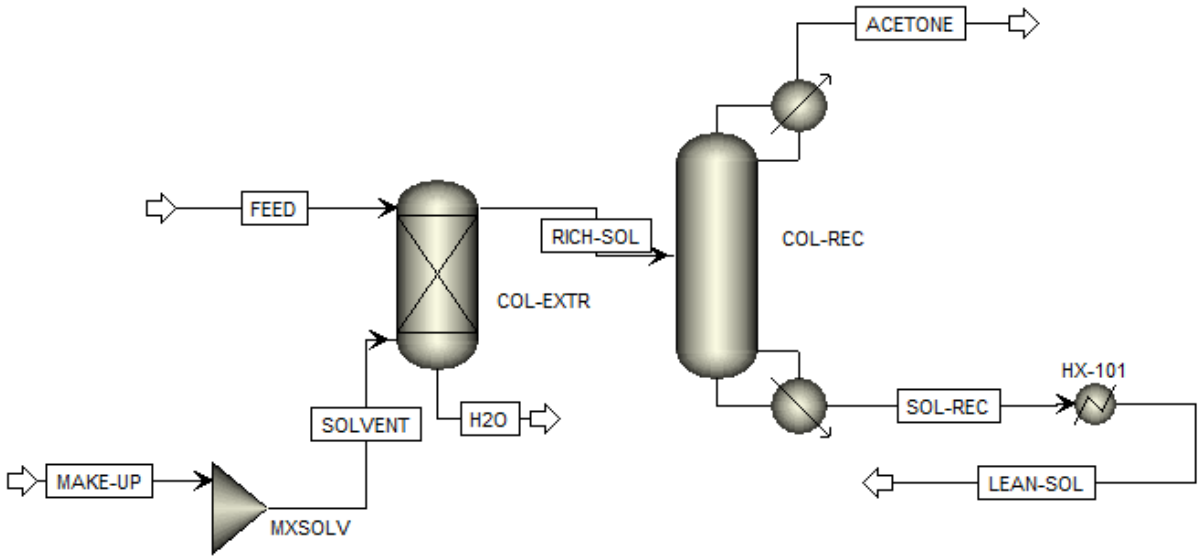


4.36. After all estimates are generated, go to the **Blocks | COL-REC | Convergence | Convergence | Basic** sheet. Select **Newton** for **Algorithm**. Increase **Maximum iterations** to **200** and Enter **1e-07** for **Error tolerance**.

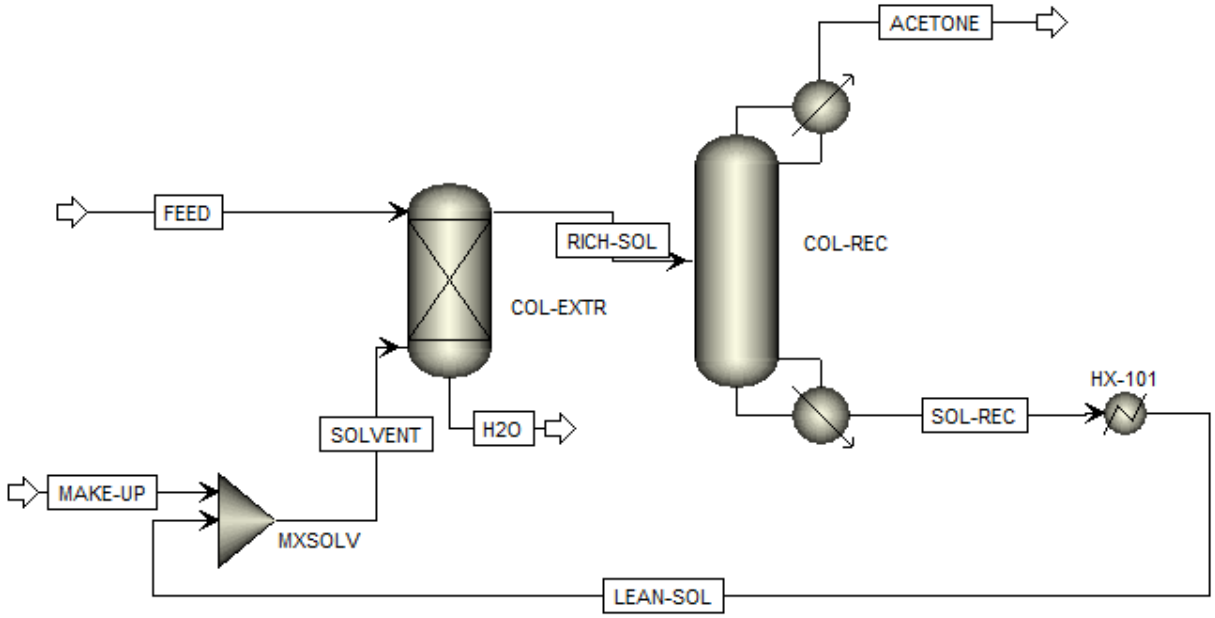
4.37. Go to the **Blocks | COL-REC | Convergence | Advanced** sheet and select **Dogleg strategy** for **Stable-Meth**.

4.38. Click the button in the **Home | Run** group of the ribbon to reinitialize simulation. Run the simulation again to ensure that it still converges.

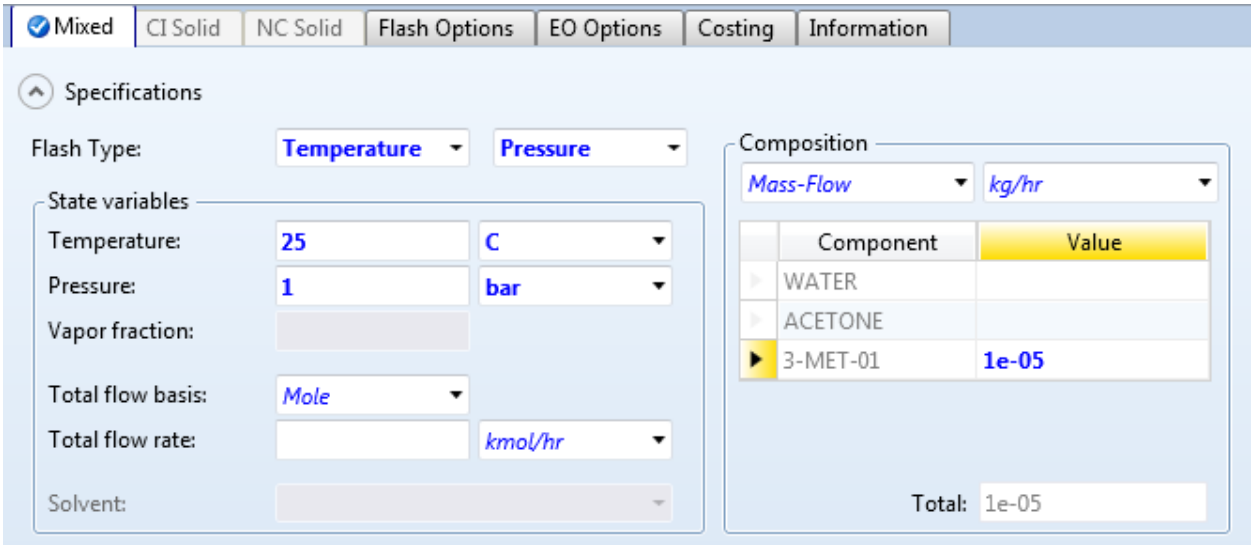
4.39. We need to connect the recycle streams. In the ribbon, click the **View** tab. Click the **Flowsheet** button in the **Show** group. The **Main Flowsheet** is displayed. Add a **Mixer** block, **MXSOLV**, before stream **SOLVENT** and add a solvent make-up stream, **MAKE-UP**. The flowsheet should look like the screenshot below.



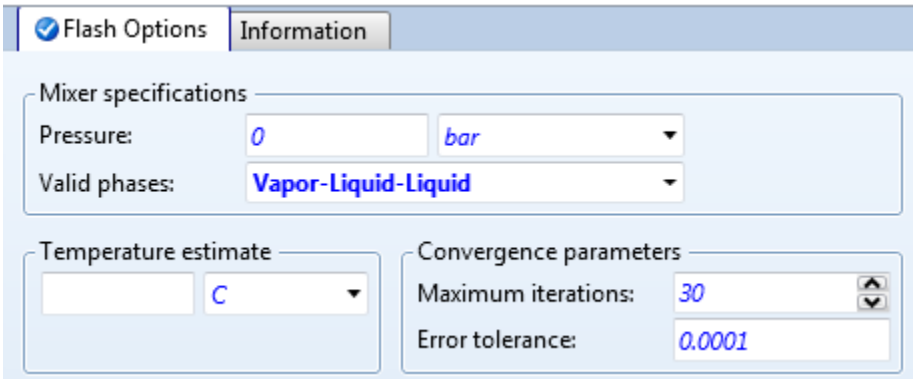
4.40. Right click stream **LEAN-SOL** and select **Reconnect Destination** on the context menu. Move the mouse cursor over the inlet port icon of **MXSOLV** and click the inlet port icon. The flowsheet should look like the screenshot below.



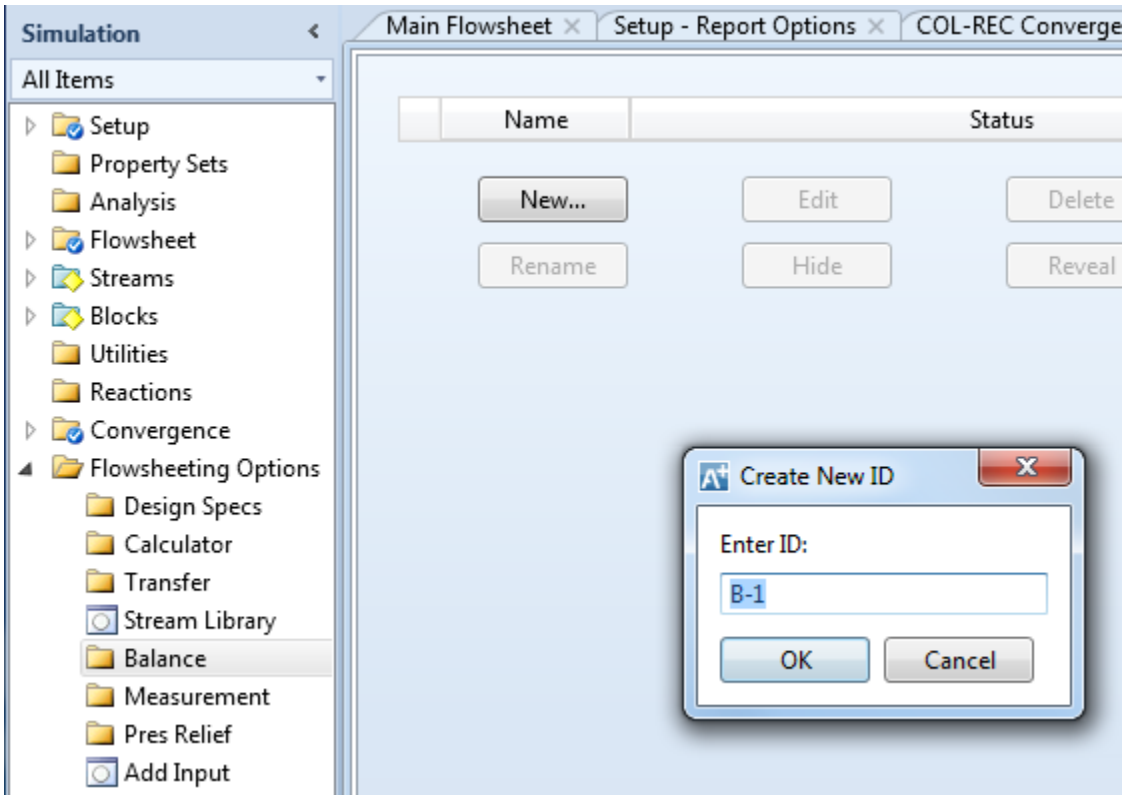
4.41. Specify stream **MAKE-UP**. Go to the **Streams | MAKE-UP | Input | Mixed** sheet. Enter **25** for **Temperature** and **1** for **Pressure**. In the **Composition** frame, select **Mass-Flow**. Enter **0.00001** for **3-MET-1**. For now, we just enter a tiny flow for the solvent make-up stream as an estimate. Later on, we will use a **Balance** block to calculate its flowrate. The **Streams | MAKE-UP | Input | Mixed** sheet should look like this.



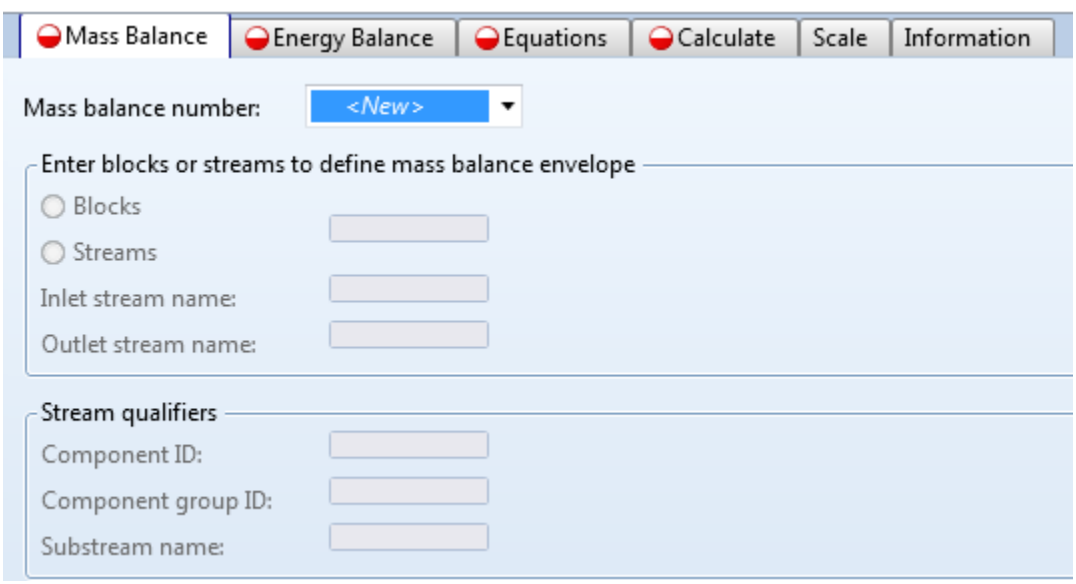
4.42. Go to the **Blocks | MXSOLV | Input | Flash Options** sheet. Select **Vapor-Liquid-Liquid** for **Valid phases** as shown below.



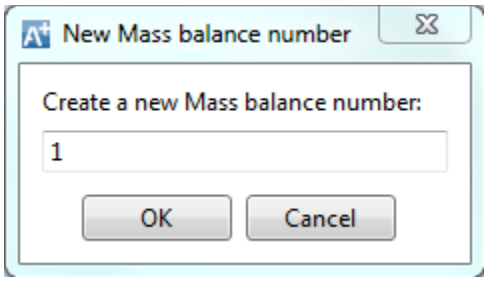
4.43. In the navigation pane, click the **Flowsheeting Options | Balance** node. The object manager for **Balance** is displayed. Click the **New…** button and click the **OK** button to create a **Balance** block called **B-1**.



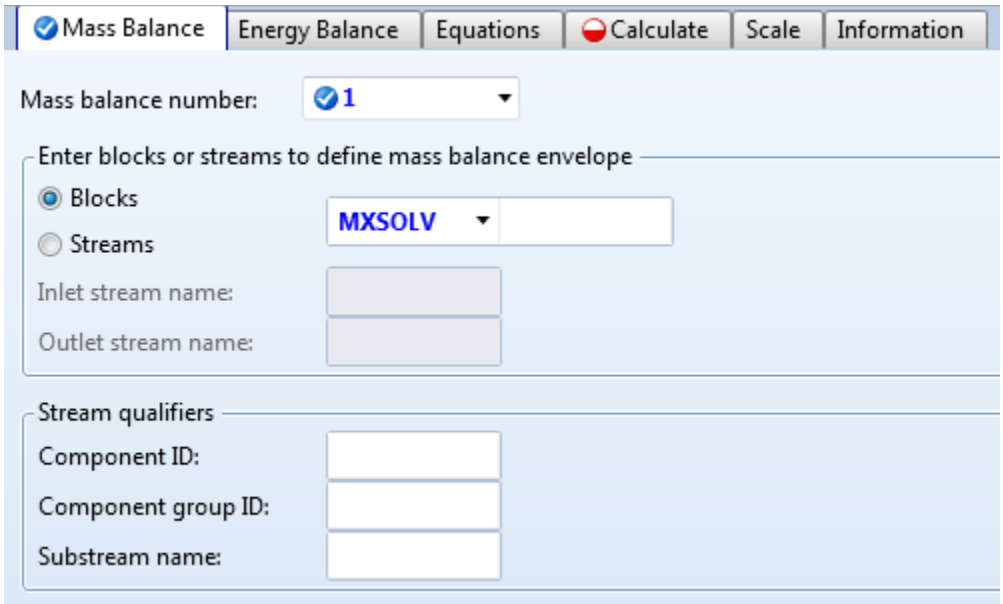
4.44. Go to the **Flowsheeting Options | Balance | B-1 | Setup | Mass Balance** sheet. Click the **Mass balance number** dropdown box and click **<New>**.



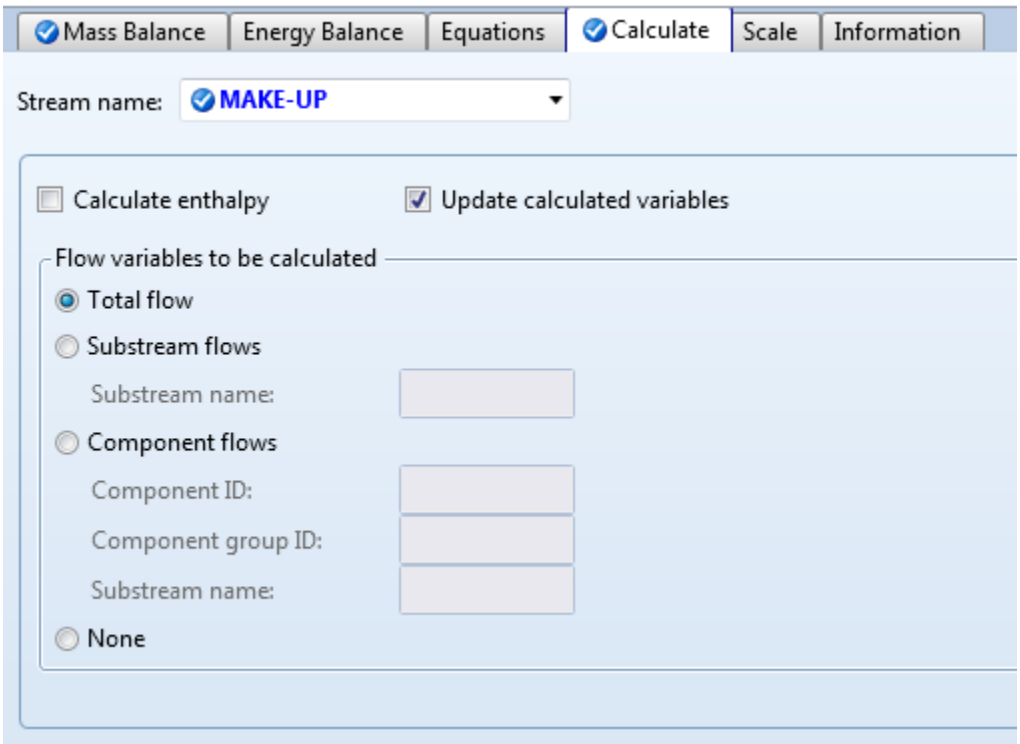
4.45. The **New Mass balance number** window pops up. Click the **OK** button.



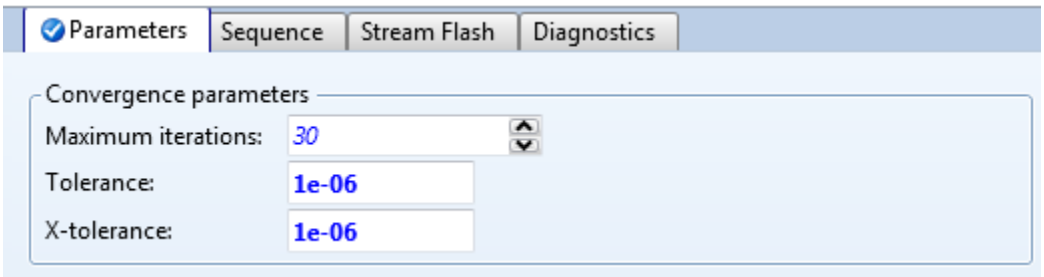
4.46. In the **Enter blocks or streams to define mass balance envelope** frame, select **MXSOLV** for **Blocks**.



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



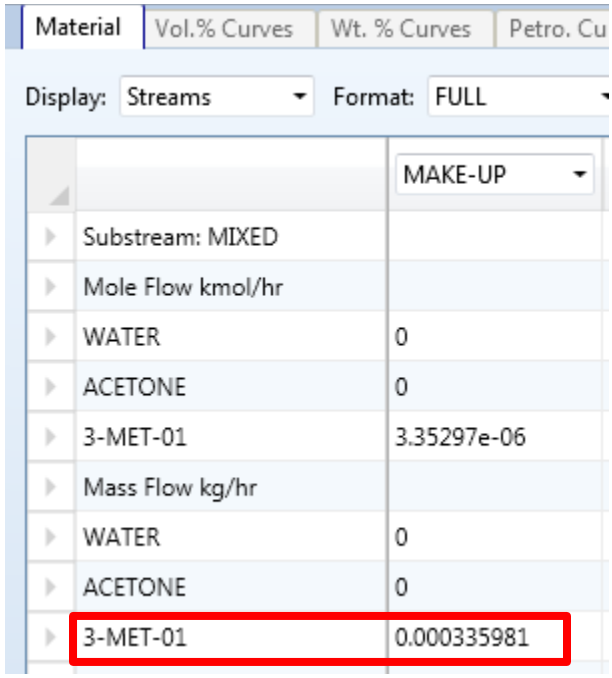
4.48. Go to the **Flowsheeting Options | Balance | B-1 | Advanced | Parameters** sheet. Change both **Tolerance** and **X-tolerance** to **1e-6** as shown below.



4.49. Click the button in the **Home | Run** group of the ribbon to reinitialize simulation.

4.50. Press the **F5** key to run the simulation. Note that the simulator reports 2 errors for the first 2 executions of **B-1**. There is no error associated with subsequent executions of **B-1**. Therefore, these 2 error messages can be safely ignored.

4.51. Go to the **Streams | MAKE-UP | Results | Material** sheet. Note that the flowrate for **3-MET-01** is **0.000335981 kg/hr** instead of the originally entered 0.00001 kg/hr.



4.52. Go to the **Results Summary | Streams | Material** sheet to review results. The mass purity of acetone in stream **ACETONE** is **0.995093**. The purity of water in stream **H2O** is **0.995088**.

Material | Heat | Load | Work | Vol.% Curves | Wt. % Curves | Petro. Curves | Poly. Curves

Display: All streams Format: FULL Stream Table Copy All

	ACETONE	FEED	H2O
Substream: MIXED			
Mole Flow kmol/hr			
WATER	0.0136152	2.77542	2.76181
ACETONE	0.856657	0.860881	0.00422391
3-MET-01	4.67827e-07	0	2.93484e-06
Mass Flow kg/hr			
WATER	0.245283	50	49.7547
ACETONE	49.7547	50	0.245325
3-MET-01	4.68781e-05	0	0.000294083
Mass Frac			
WATER	0.00490566	0.5	0.995088
ACETONE	0.995093	0.5	0.00490647
3-MET-01	9.37563e-07	0	5.88161e-06

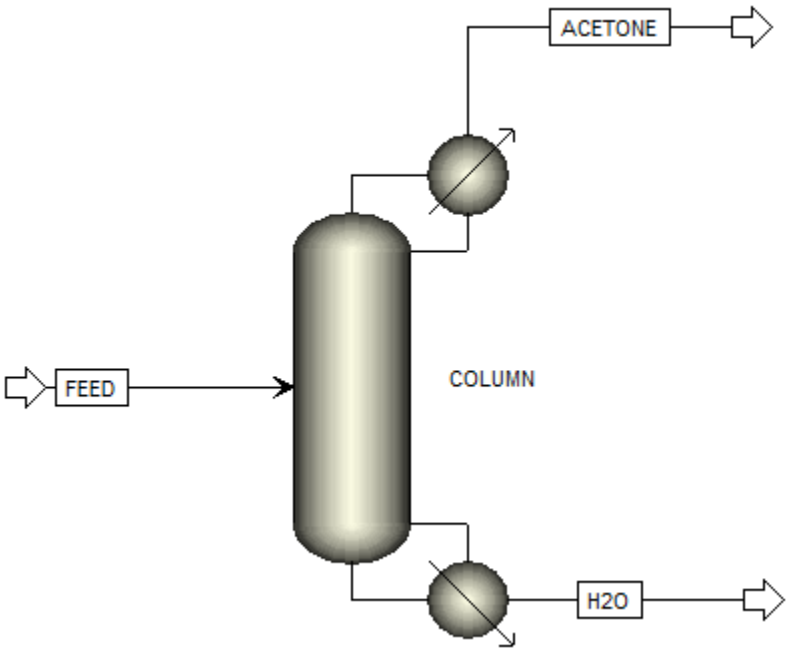
4.53. Go to the **Blocks | HX-101 | Results | Summary** sheet. The **Heat Duty** is **-1454.43 cal/sec**.

4.54. Got to the **Blocks | COL-REC | Results | Summary** sheet. The **Heat duty** for the **Condenser** is **-2063.45 cal/sec**. The **Heat duty** for the **Reboiler** is **6003.03 cal/sec**.

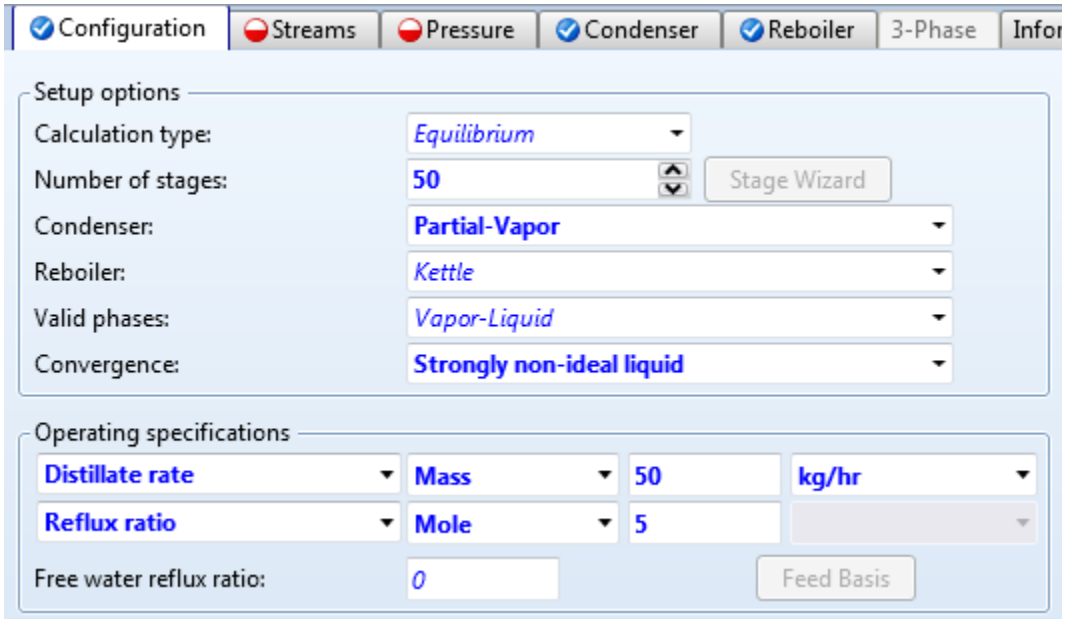
4.55. On the ribbon, click **File** and click **Save As** to save the simulation. Close the Aspen Plus Window.

4.56. We will build a model based on direct distillation. In Windows Explorer, make a copy of **Dist-009_Base.bkp** and rename it to **Dist-009_Acetone_Water_Separation_RadFrac.bkp**. Double-click the **Dist-009_Acetone_Water_Separation_RadFrac.bkp** to load it into Aspen Plus.

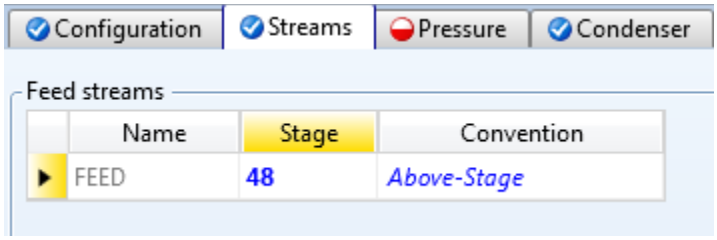
4.57. In the ribbon, click **View** tab. Click the **Flowsheet** button in the **Show** group. The **Main Flowsheet** is displayed. Create the flowsheet shown below by adding a **RadFrac** block and effluent streams.



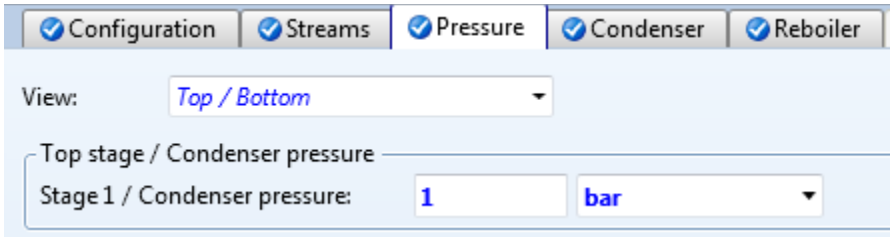
4.58. Define column operating conditions. Double-click the **COLUMN** block on the main flowsheet or navigate to the **Blocks | COLUMN | Specifications | Setup | Configuration** sheet. Enter **50** for **Number of stages**. Select **Partial-Vapor** for **Condenser** and **Strongly non-ideal liquid** for **Convergence**. In the **Operating specifications** frame, select **Mass** as the basis for **Distillate rate**. Enter **50** for **Distillate rate** and **5** for **Reflux ratio**. Values entered here for **Distillate rate** and **Reflux ratio** are initial guess and will be varied later on for target product purity. The **Blocks | COLUMN | Specifications | Setup | Configuration** sheet should look like the screenshot below.



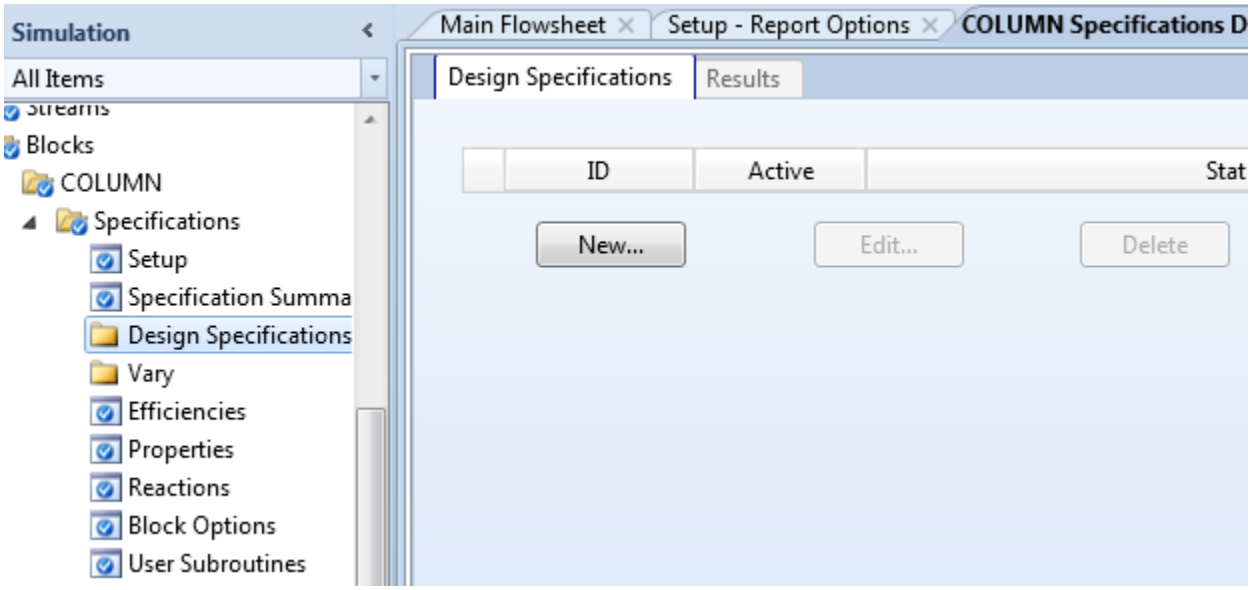
4.59. Go to the **Blocks | COLUMN | Specifications | Setup | Streams** sheet and enter **48** for **FEED** as shown below. We use 48 because it should lead to lowest reboiler duty.



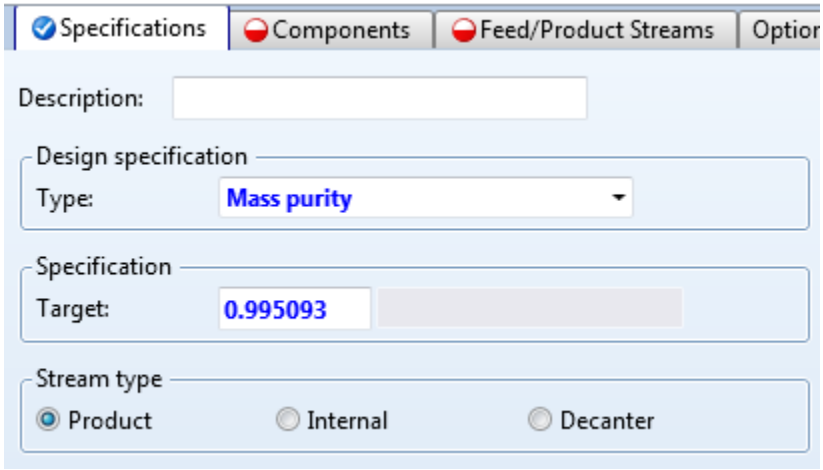
4.60. Go to the **Blocks | COLUMN | Specifications | Setup | Pressure** sheet and enter **1** for **Stage 1 / Condenser pressure** as shown below.



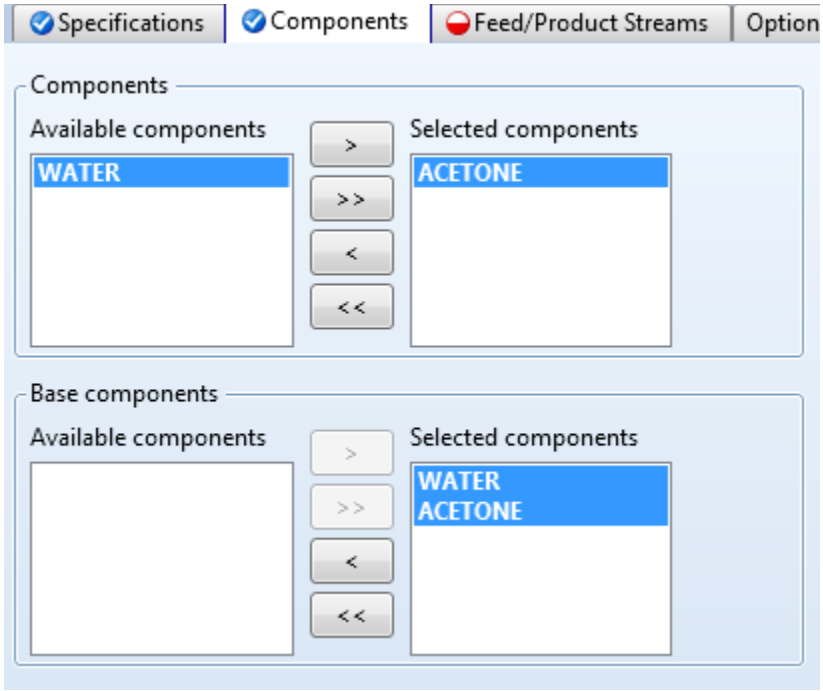
4.61. We will define two pairs of Design Specs / Vary to specify purity for product streams to match purity from the extraction approach. In the navigation pane, select **Blocks | COLUMN | Specifications | Design Specifications**. The object manager for **Design Specifications** is displayed. Click the **New…** button to create a new **Design Specs** called **1**.



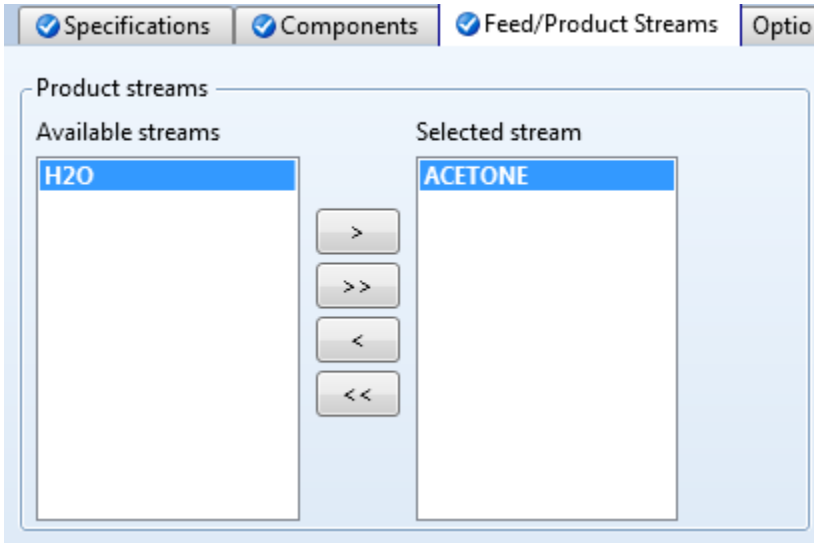
4.62. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 1 | Specifications** sheet. Select **Mass purity** for **Type** and enter **0.995093** for **Target** as shown below.



4.63. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 1 | Components** sheet. In the **Components** frame, move **ACETONE** to the **Selected components** list. In the **Base components** frame, move all components to the **Selected components** list. This sheet should look like the screenshot below.

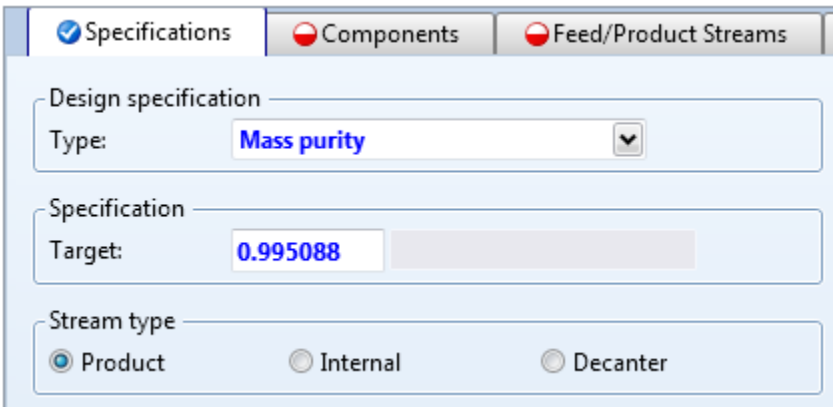


4.64. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 1 | Feed/Product Streams** sheet. In the **Product streams** frame, move **ACETONE** to the **Selected stream** list as shown below.

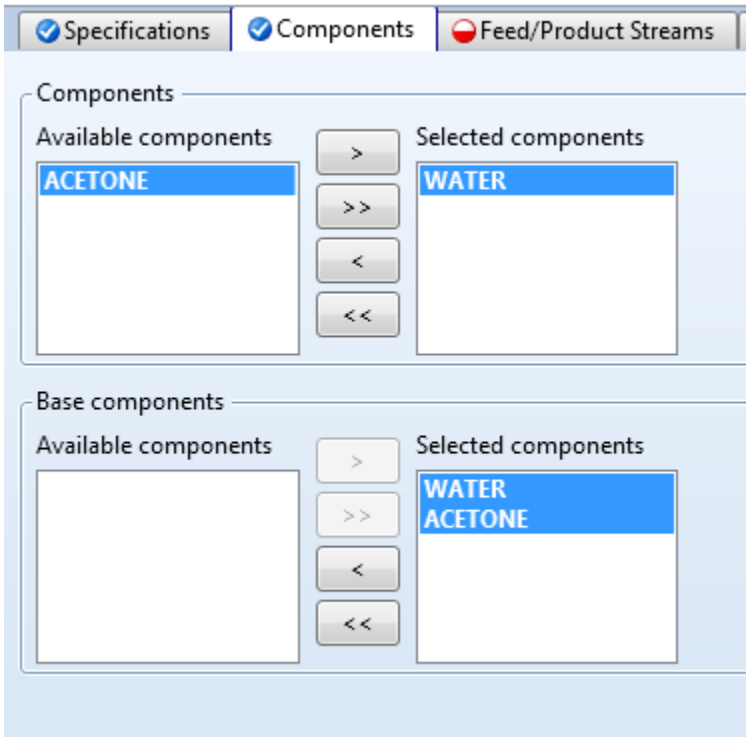


4.65. We create the second Design Specs to specify purity for stream **H2O**. In the navigation pane, select **Blocks | COLUMN | Specifications | Design Specifications**. The object manager for **Design Specs** is displayed. Click the **New…** button to create a new **Design Specs** called **2**.

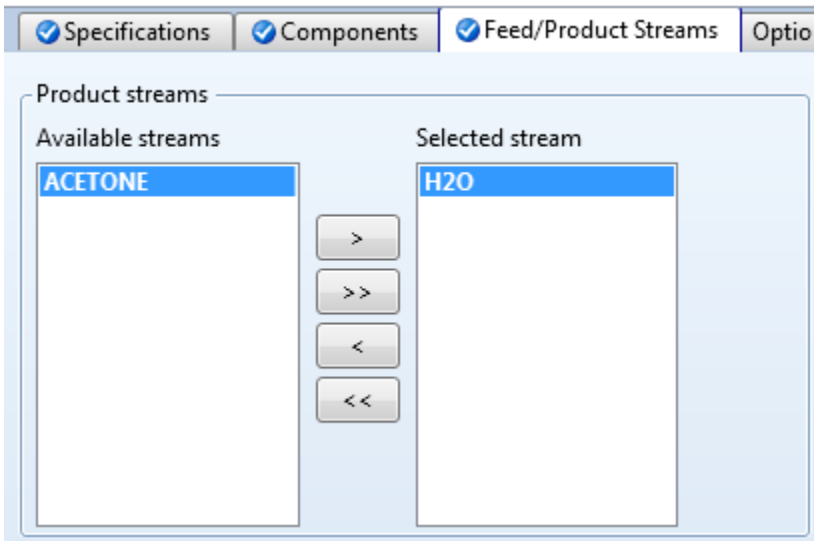
4.66. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 2 | Specifications** sheet. Select **Mass purity** for **Type** and enter **0.995088** for **Target** as shown below.



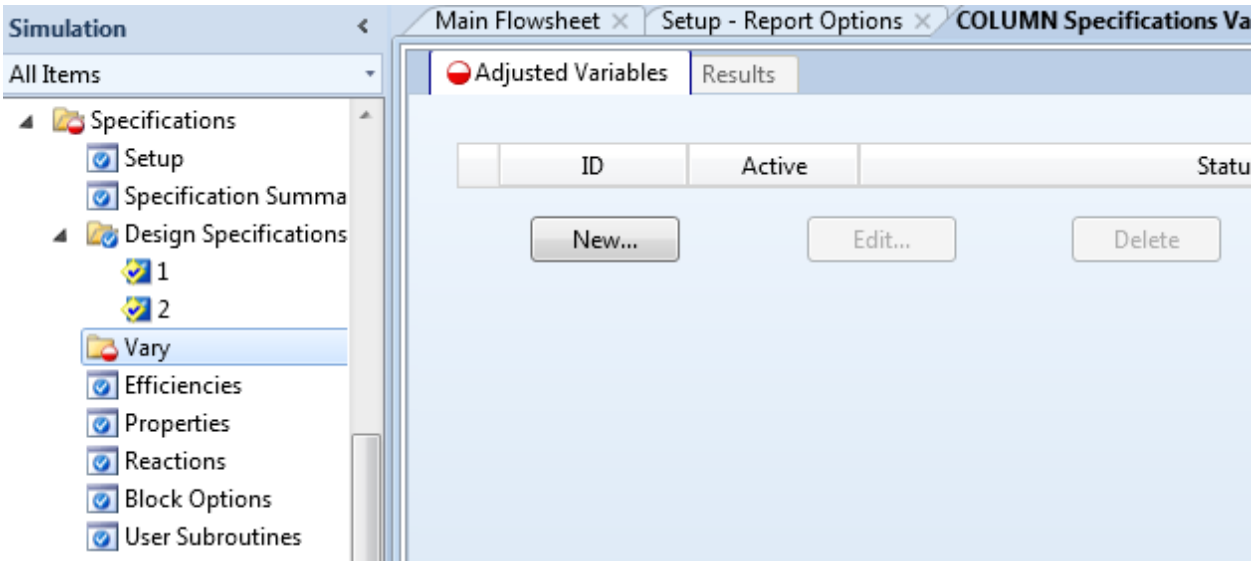
4.67. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 2 | Components** sheet. In the **Components** frame, move **WATER** to the **Selected components** list. In the **Base components** frame, move all components to the **Selected components** list. This sheet should look like the screenshot below.



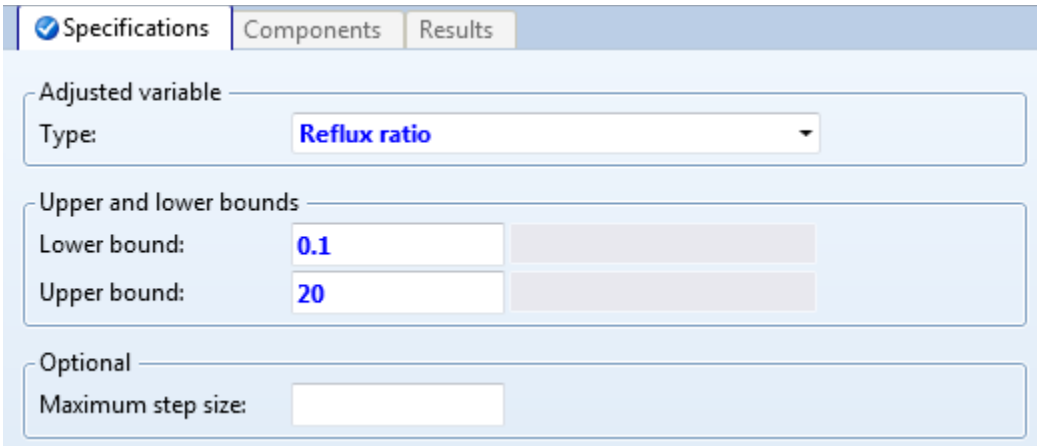
4.68. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 2 | Feed/Product Streams** sheet. In the **Product streams** frame, move **H2O** to the **Selected stream** list as shown below.



4.69. We create corresponding **Varys**. In the navigation pane, select the **Blocks | COLUMN | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New...** button to create a new **Vary** called **1**.

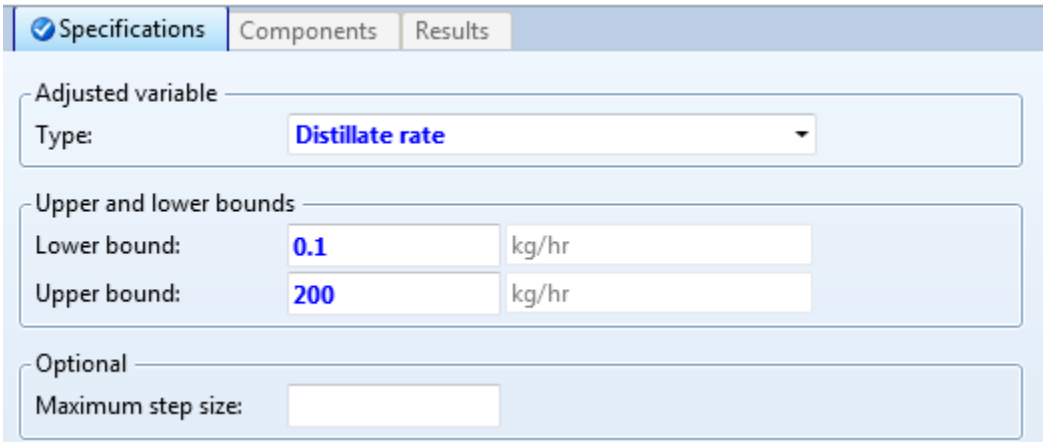


4.70. Go to the **Blocks | COLUMN | Specifications | Vary | 1 | Specifications** sheet. Select **Reflux ratio** for **Type**. Enter **0.1** for **Lower bound** and **20** for **Upper bound**. This sheet should look like this.



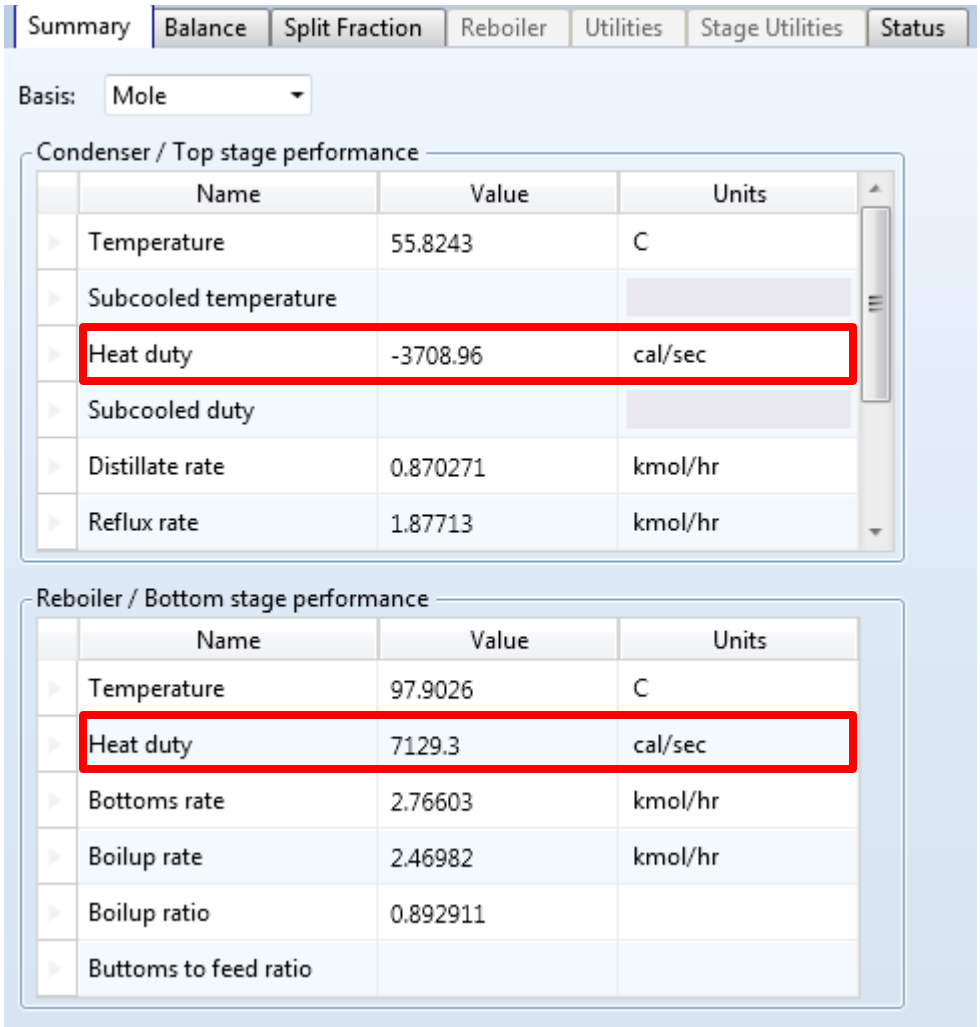
4.71. In the navigation pane, select **Blocks | COLUMN | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New…** button to create a new **Vary** called **2**.

4.72. Go to the **Blocks | COLUMN | Specifications | Vary | 2 | Specifications** sheet. Select **Distillate rate** for **Type**. Enter **0.1** for **Lower bound** and **200** for **Upper bound** as shown below.



4.73. Go to the **Blocks | COLUMN | Convergence | Convergence | Basic** sheet. Increase **Maximum iterations** from **25** to **200**. Enter **1e-7** for **Error tolerance**.

4.74. Press the **F5** key to run the simulation and the simulation converges without any error or warning. Go to the **Blocks | COLUMN | Results | Summary** sheet to view results. Note that the **Heat duty** of **Condenser** is **-3708.96 cal/sec** and the **Heat duty** of **Reboiler** is **7129.3 cal/sec**.



4.75. The table below shows the heat duties for both approaches. We can clearly see the potential energy saving with the extraction approach.

	Case 1 (Extractor + Distillation)	Case 2 (Single Distillation Column)
Total Heating Duty (cal/sec)	6003.03	7129.3
Total Cooling Duty (cal/sec)	3517.88	3708.96

5. Conclusions

Based on the simulation results, achieving the same degree of separation using extraction requires about 16% less heating capacity than direct distillation. It also requires less cooling capacity. The reason is that the heat of vaporization for 3-methylhexane is significantly less than that of water. Of course, the extraction approach does require more hardware.

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