Extractive Distillation for Heptane-Toluene Separation using Aspen Plus® V8.0

1. Lesson Objectives

- Essentials of extractive distillation
- How to compare design alternatives

2. Prerequisites

- Aspen Plus V8.0
- Introduction to distillation

3. Background

When the two components in a binary mixture have very close normal boiling points, their relative volatility is likely to be small if they do not form an azeotrope. For such cases, it may be more efficient to use extractive distillation with a solvent than normal distillation. In extractive distillation, a less volatile solvent is used to increase the relative volatilities of the original mixtures, allowing for easier separation. In this example, phenol is used as the solvent for the separation of n-heptane and toluene.

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

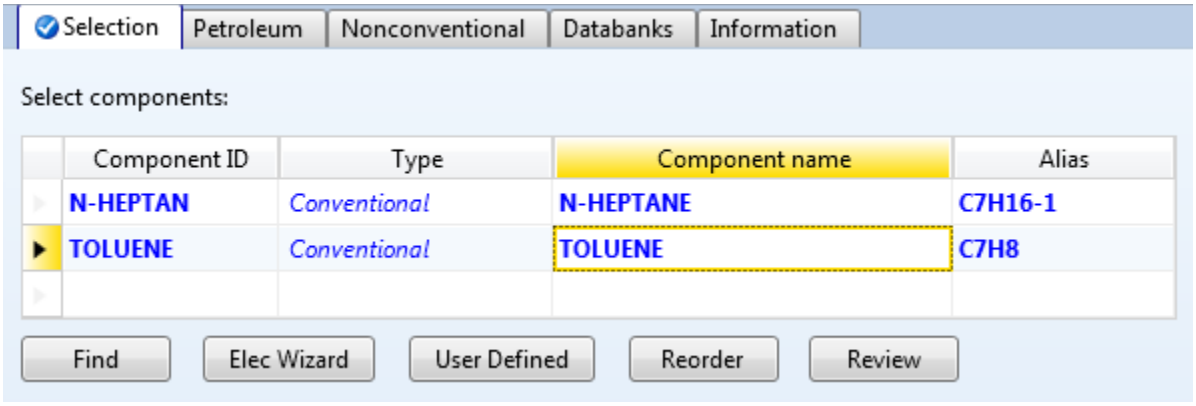
Determine whether conventional distillation or extractive distillation with phenol as a solvent is a more efficient method to separate n-heptane and toluene.

Aspen Plus Solution

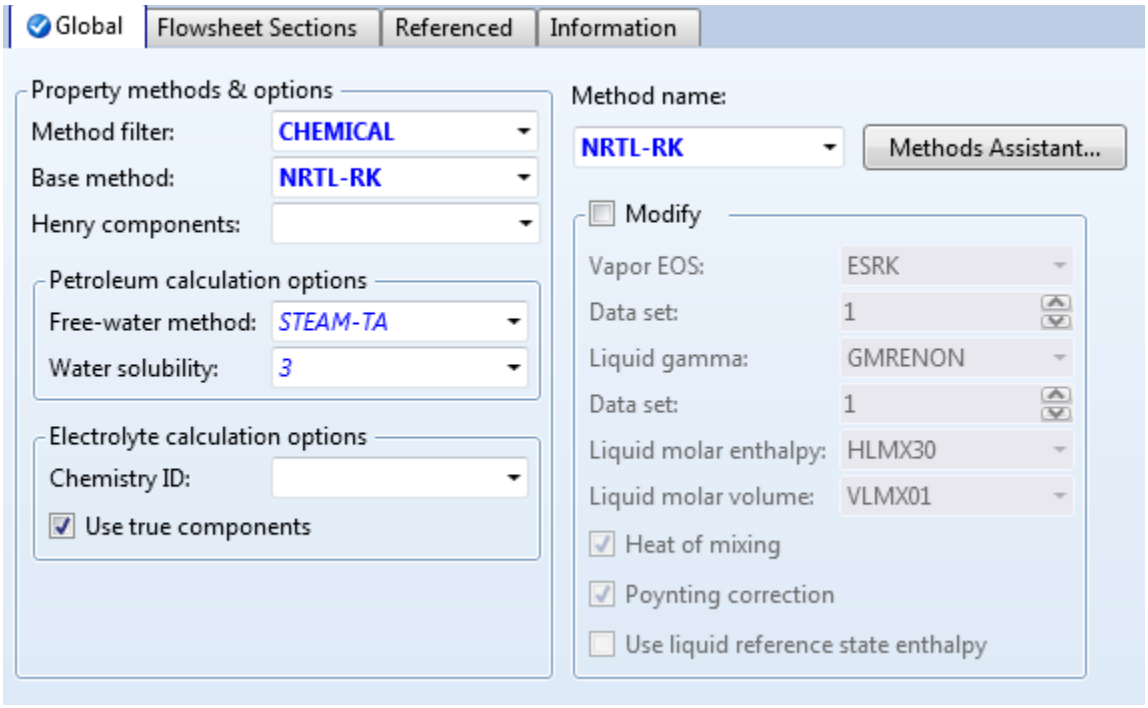
If you are unfamiliar with how to start Aspen Plus, select components, define methods, or construct a flowsheet, consult **Get Started Guide for New Users of Aspen Plus.pdf** for instructions.

4.01. We will build models to simulate the separation of n-heptane and toluene. One model has a single distillation column and the other uses the extractive distillation approach with two columns. First of all, we will build a base bkp file to be used as the starting point for both models. Start a new simulation using the **Blank Simulation** template in Aspen Plus.

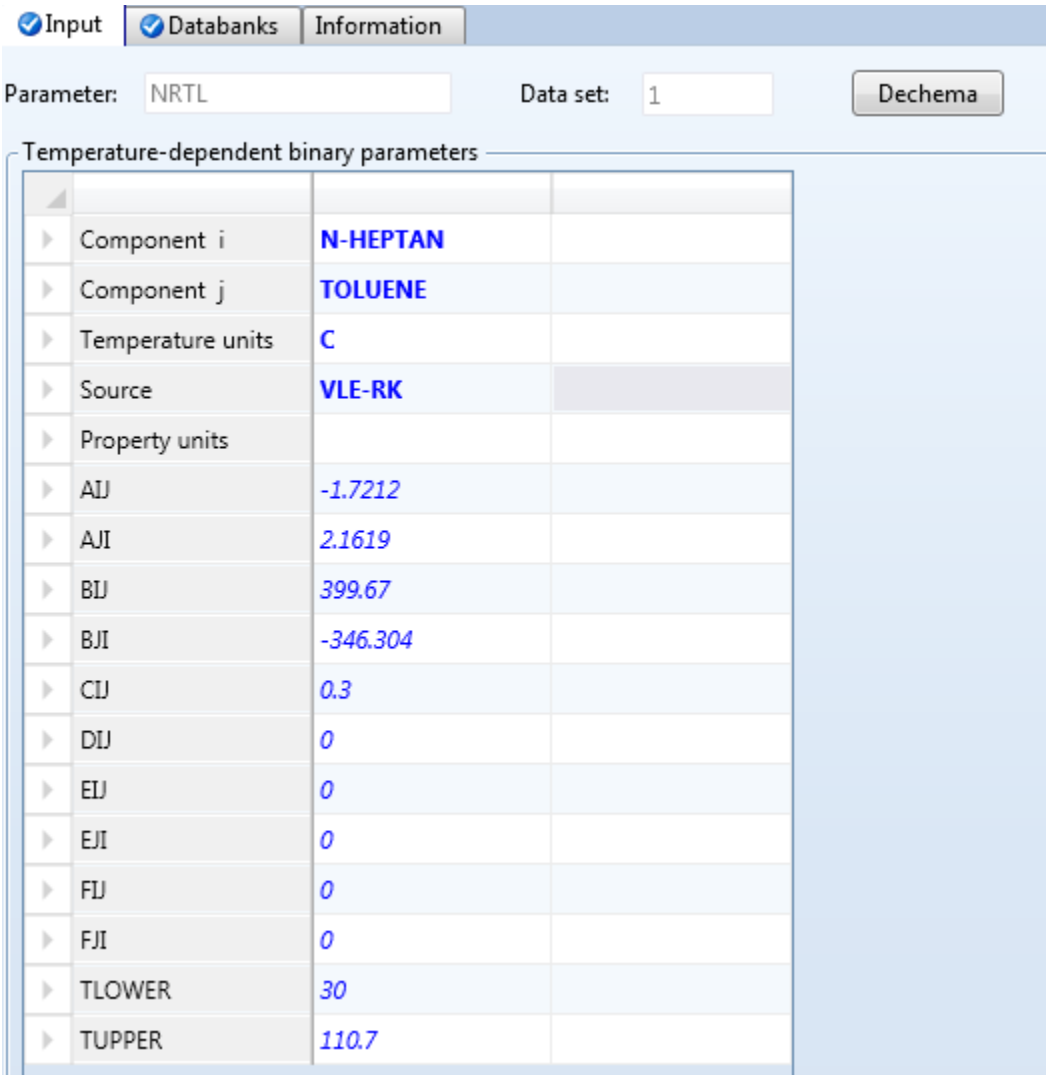
4.02. The **Components | Specification | Selection** sheet is displayed. Enter **N-HEPTAN** in **Component ID** column and **N-HEPTANE** in **Component name** column. Note that the Alias is filled automatically. Then, in the next row, enter **TOLUENE** in **Component ID** column. **Component name** and **Alias** are filled automatically for **TOLUENE**. The **Components | Specifications | Selection** sheet should look like this.



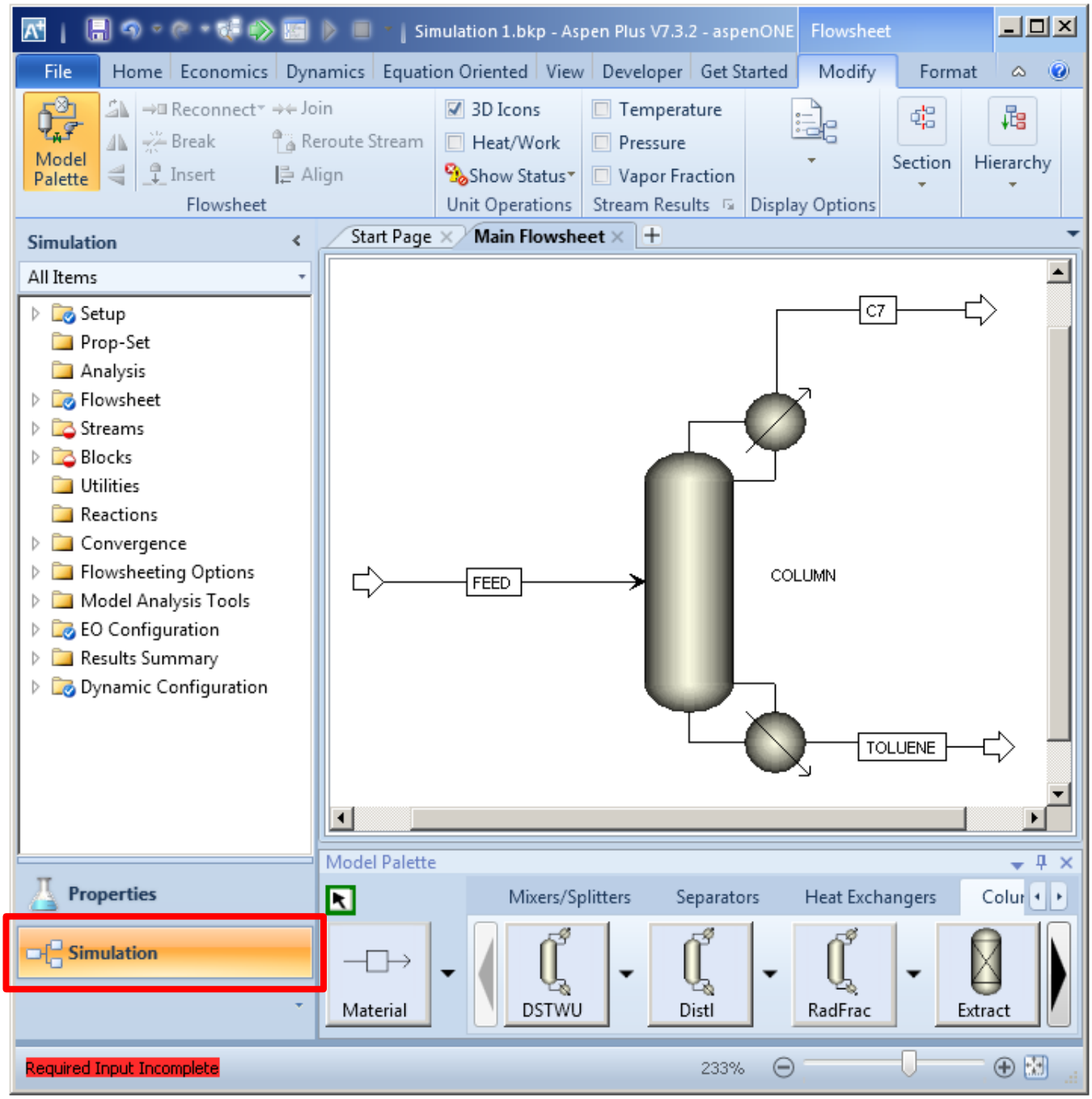
4.03. Define methods. Press the **F4** key and the **Methods | Specifications | Global** sheet is displayed. Select **CHEMICAL** for **Method filter** and **NRTL-RK** for **Base method**. Now, the sheet should look like this.



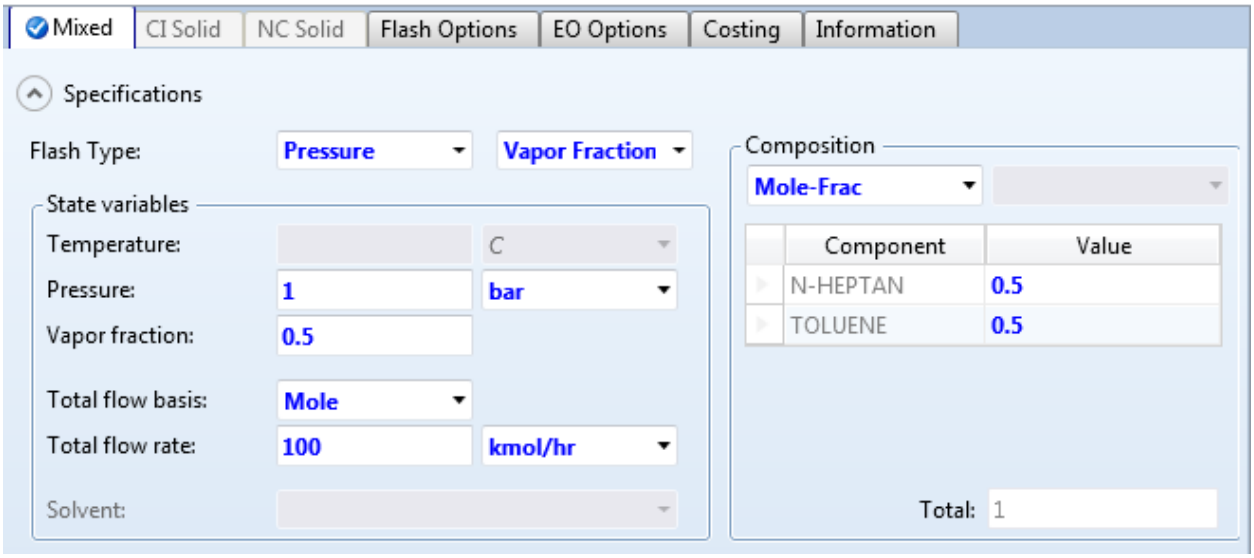
4.04. Press the **F4** key. The **Methods | Parameters | Binary Interaction | NRTL-1 | Input** sheet is displayed. Note that binary parameters are filled automatically.



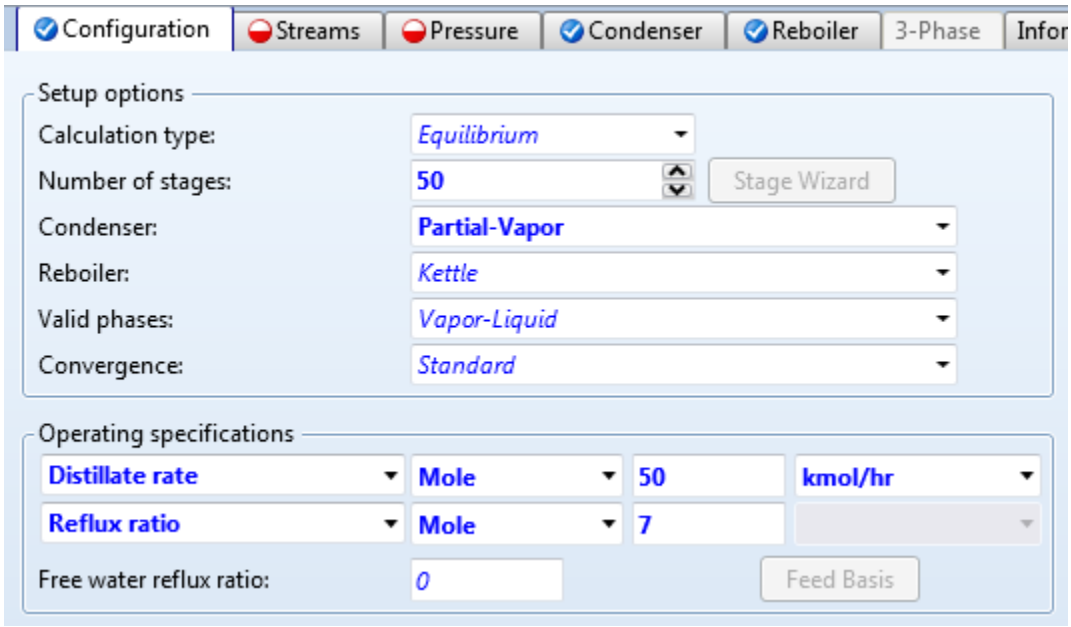
4.05. Move to the simulation environment by clicking the **Simulation** bar in the navigation pane. Then, draw a flowsheet to match the one shown below by placing a **RadFrac** block on the flowsheet and creating feed and effluent streams.



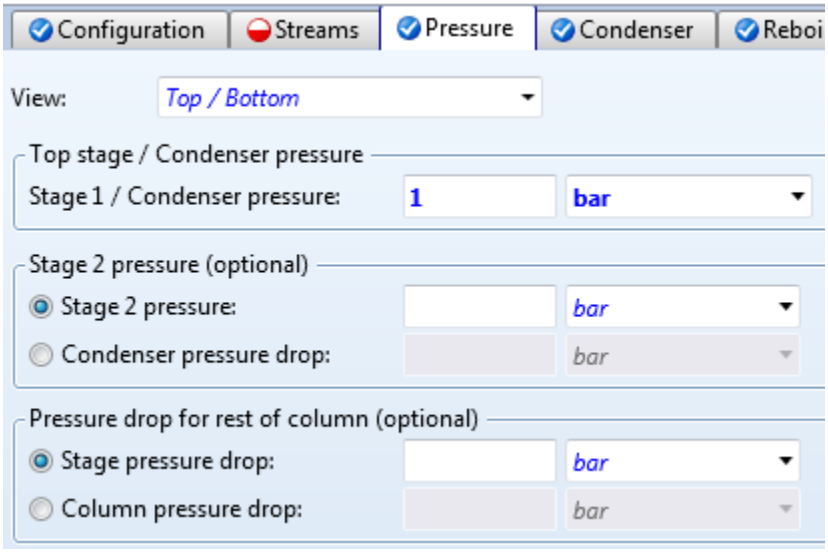
4.06. Press the **F4** key and the **Streams | FEED | Input | Mixed** sheet. Is displayed. Select **Vapor Fraction** and **Pressure** for **Flash Type**. Enter **1** for **Pressure**, **0.5** for **Vapor fraction** and **100** for **Total flow rate**. In the **Composition** frame, select **Mole-Frac** and enter **0.5** for both components. Now the **Streams | FEED | Input | Mixed** sheet should look like this.



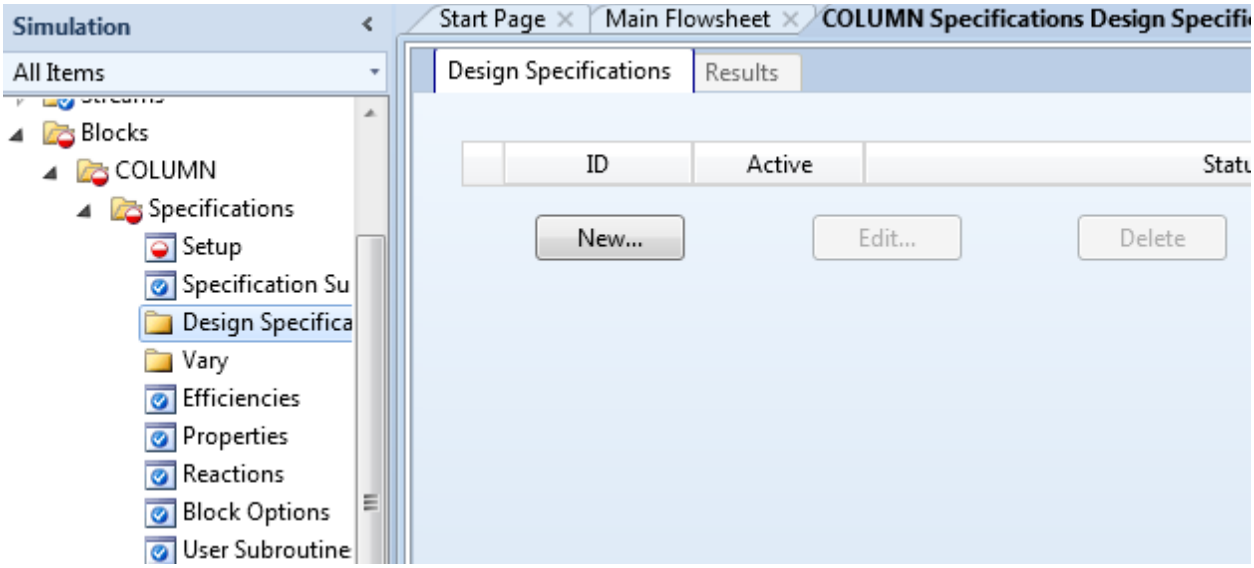
4.07. Press the **F4** key and the **Blocks | COLUMN | Specifications | Setup | Configuration** sheet is displayed. Enter **50** for **Number of stages**. Select **Partial-Vapor** for **Condenser**. In the **Operating specifications** frame, enter **50** for **Distillate rate** and **7** for **Reflux ratio**. The **Blocks | COLUMN | Specifications | Setup | Configuration** sheet should look like this.



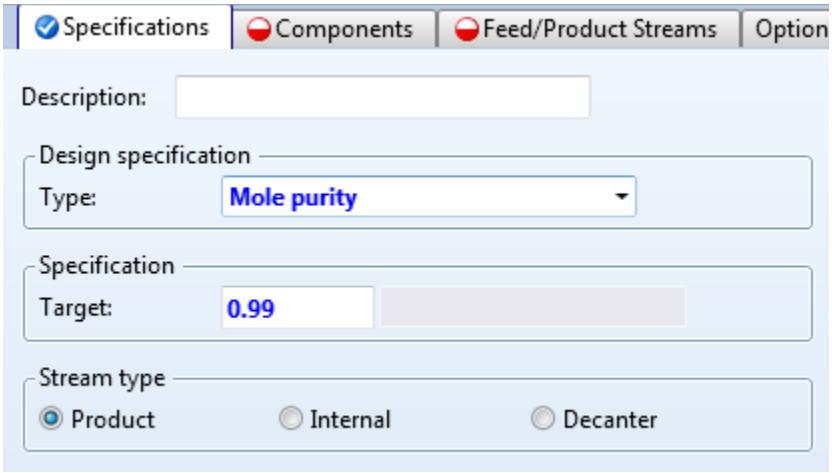
4.08. Go to the **Blocks | COLUMN | Specifications | Setup | Pressure** sheet. Enter **1** for **Stage 1 / Condenser pressure**.



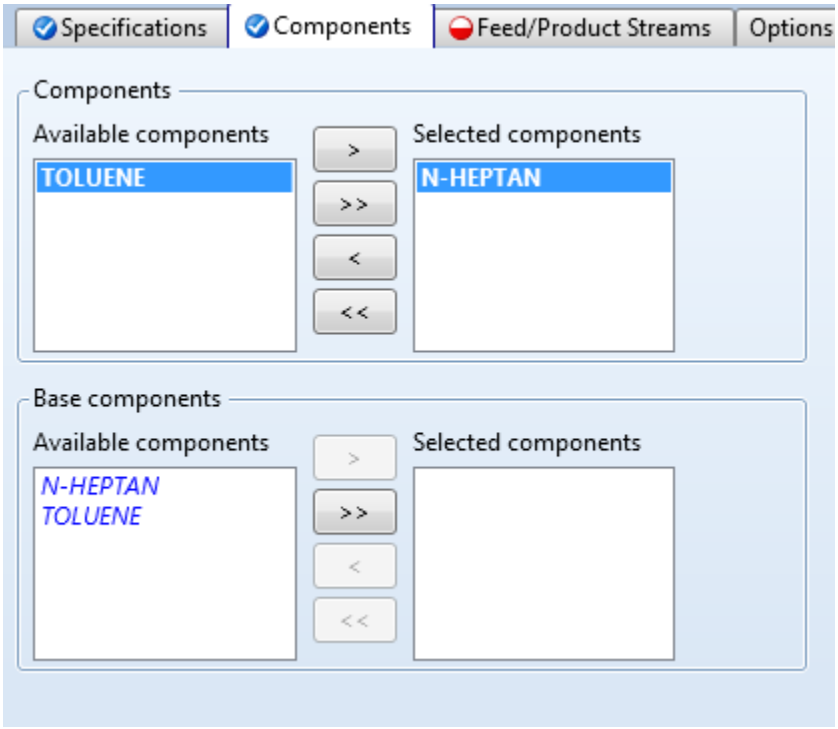
4.09. In the navigation pane, select **Blocks | COLUMN | Specifications | Design Specifications**. The object manager for **Design Specifications** is displayed. Click the **New...** button to create a new **Design Specs** called **1**.



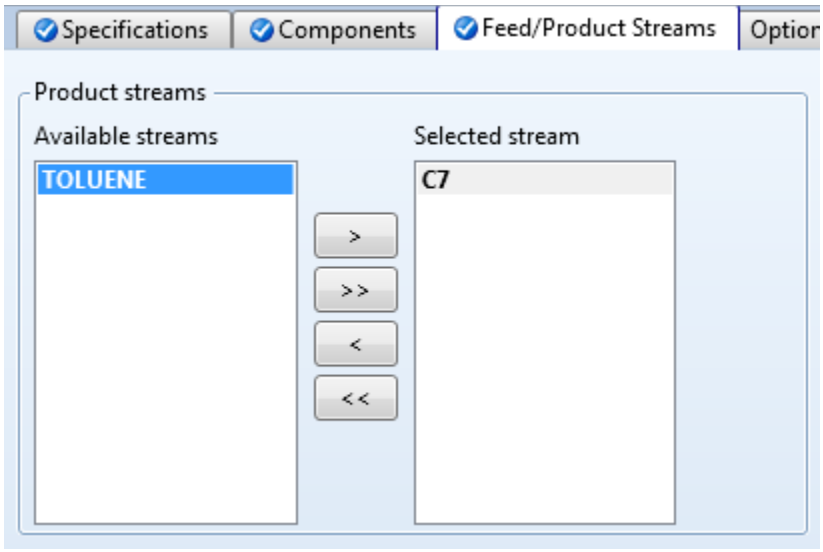
4.10. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 1 | Specifications** sheet. Select **Mole purity** for **Type** and enter **0.99** for **Target** as shown below.



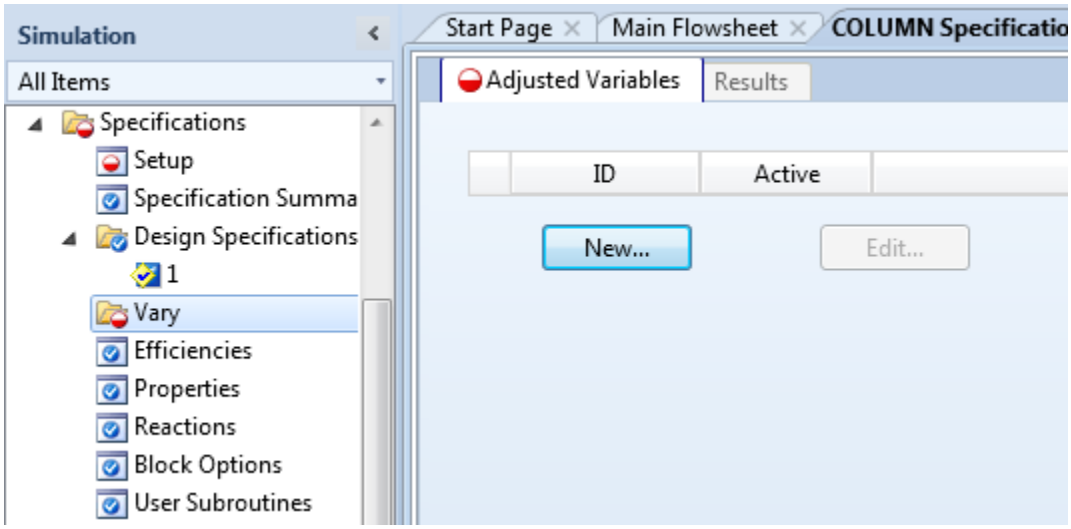
4.11. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 1 | Components** sheet. In the **Components** frame, move **N-HEPTAN** to the **Selected components** list as shown below.



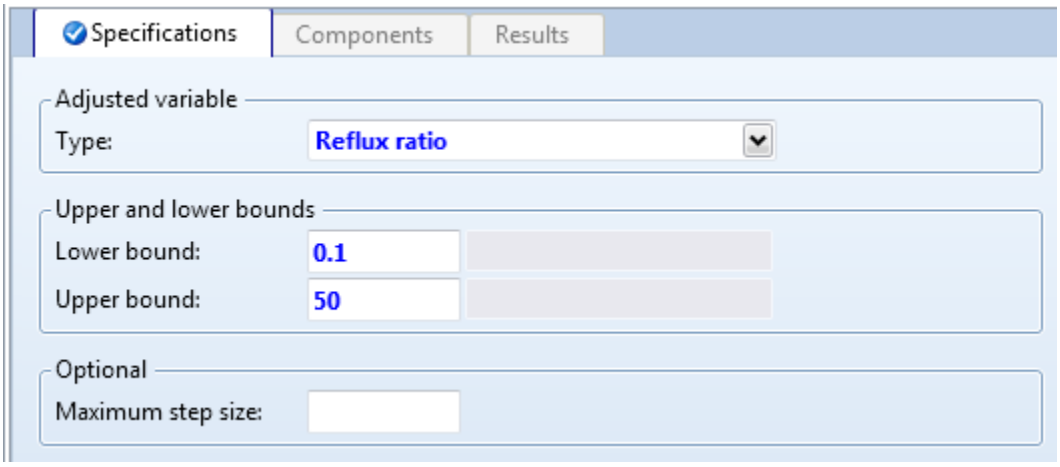
4.12. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 1 | Feed/Product Streams** sheet. In the **Product streams** frame, move **C7** to the **Selected stream** list as shown below.



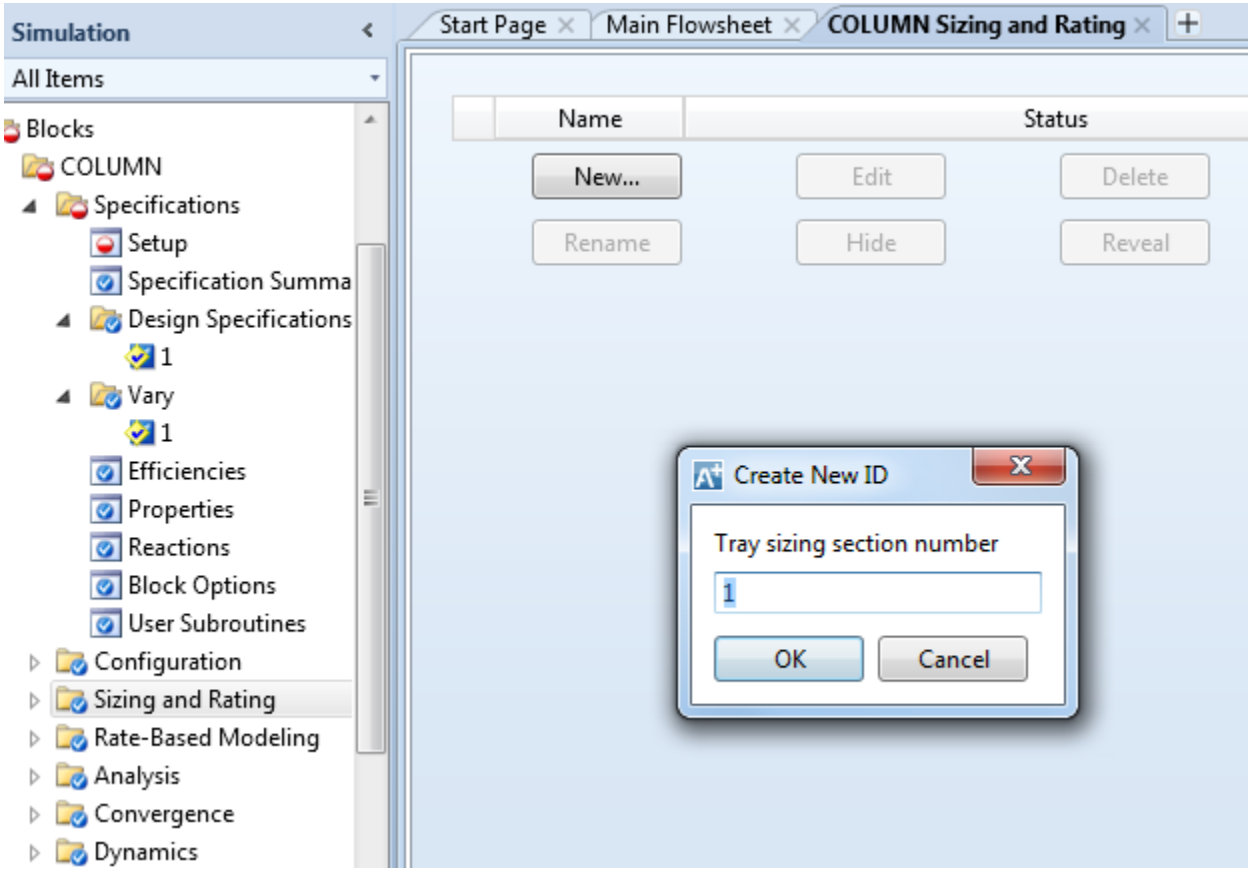
4.13. In the navigation pane, select **Blocks | COLUMN | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New...** button to create a new **Vary** called **1**.



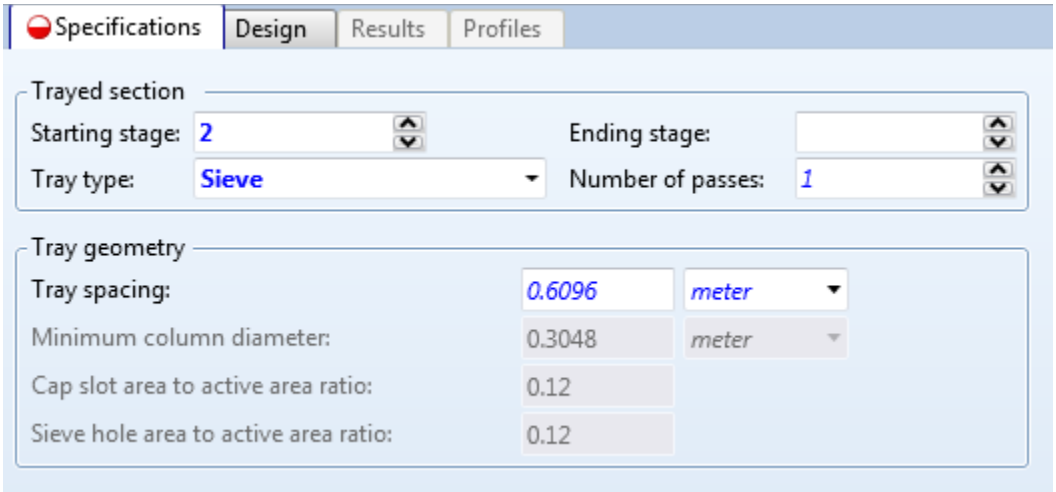
4.14. Go to the **Blocks | COLUMN | Vary | 1 | Specifications** sheet. Select **Reflux ratio** for **Type**. Enter **0.1** for **Lower bound** and **50** for **Upper bound**. Now, the sheet should look like this.



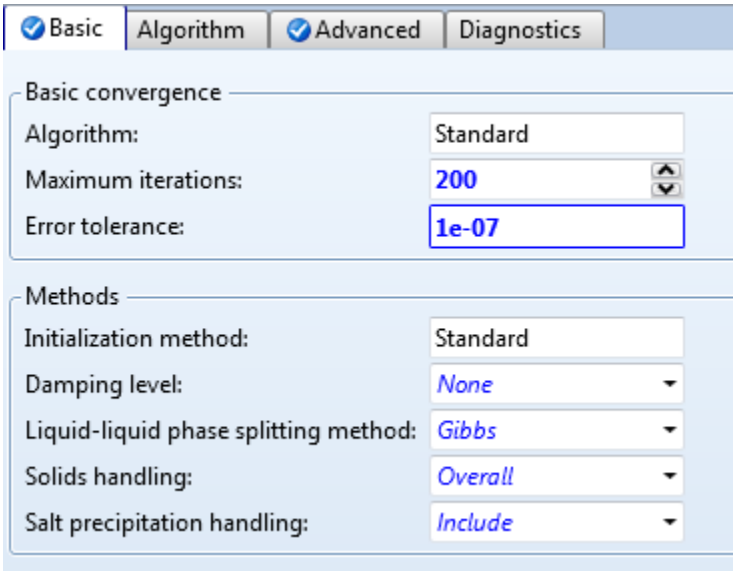
4.15. In the navigation pane, select **Blocks | COLUMN | Sizing and Rating**. The object manager for **Sizing and Rating** is displayed. Click the **New...** button to create a new **Tray Sizing** section called **1**.



4.16. Go to the **Blocks | COLUMN | Sizing and Rating | Tray Sizing | 1 | Specifications** sheet. Enter **2** for **Starting stage**. Select **Sieve** for **Tray type**. The sheet should look like this.



4.17. Go to the **Blocks | COLUMN | Convergence | Convergence | Basic** sheet. Enter **200** for **Maximum iterations** and **1e-7** for **Error tolerance** as shown below.

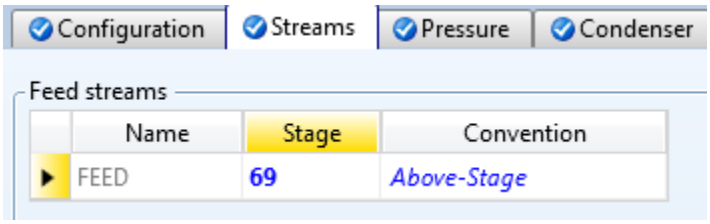


4.18. Now, save the simulation. On the ribbon, click **File** and then click **Save As** to save the simulation as **Dist-012_Base.bkp**. This is the starting point for building both models. Now, close the Aspen Plus Window.

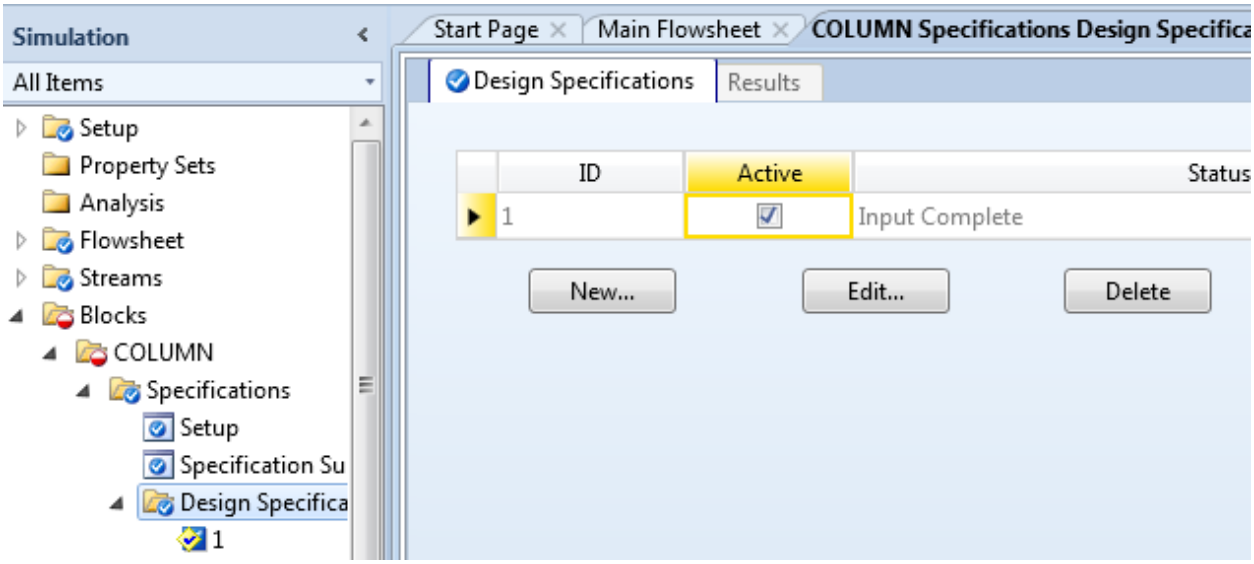
4.19. The next step is to build the model that contains one distillation column. In Windows Explorer, make a copy of **Dist-012_Base.bkp** and rename the new file to **Dist-012_One_Column_Distillation.bkp**. Double-click the **Dist-012_One_Column_Distillation.bkp** to load it into Aspen Plus.

4.20. Go to the **Blocks | COLUMN | Specification | Setup | Configuration** sheet. Change the **Number of stages** to **80**.

4.21. Go to the **Blocks | COLUMN | Setup | Streams** sheet. In the **Feed streams** frame, enter **69** in the **Stage** column as shown below. We use 69 because it leads to minimum reboiler heat duty for the same degree of separation.



4.22. Add the second Design Spec to specify the purity of the bottom product. 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 Spec** called **2**.



4.23. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 2 | Specifications** sheet. Select **Mole purity** for **Type** and enter **0.99** for **Target**.

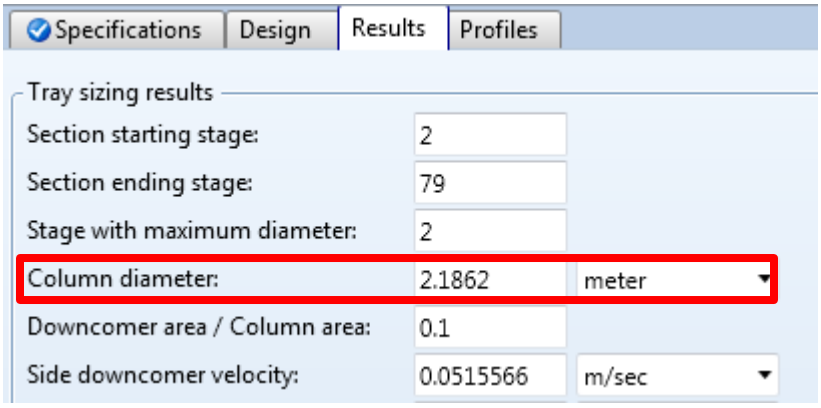
4.24. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 2 | Components** sheet. In the **Components** frame, move **TOLUENE** to the **Selected components** list.

4.25. Go to the **Blocks | COLUMN | Specifications | Design Specifications | 2 | Feed/Product Streams** sheet. In the **Product streams** frame, move **TOLUENE** to the **Selected stream** list.

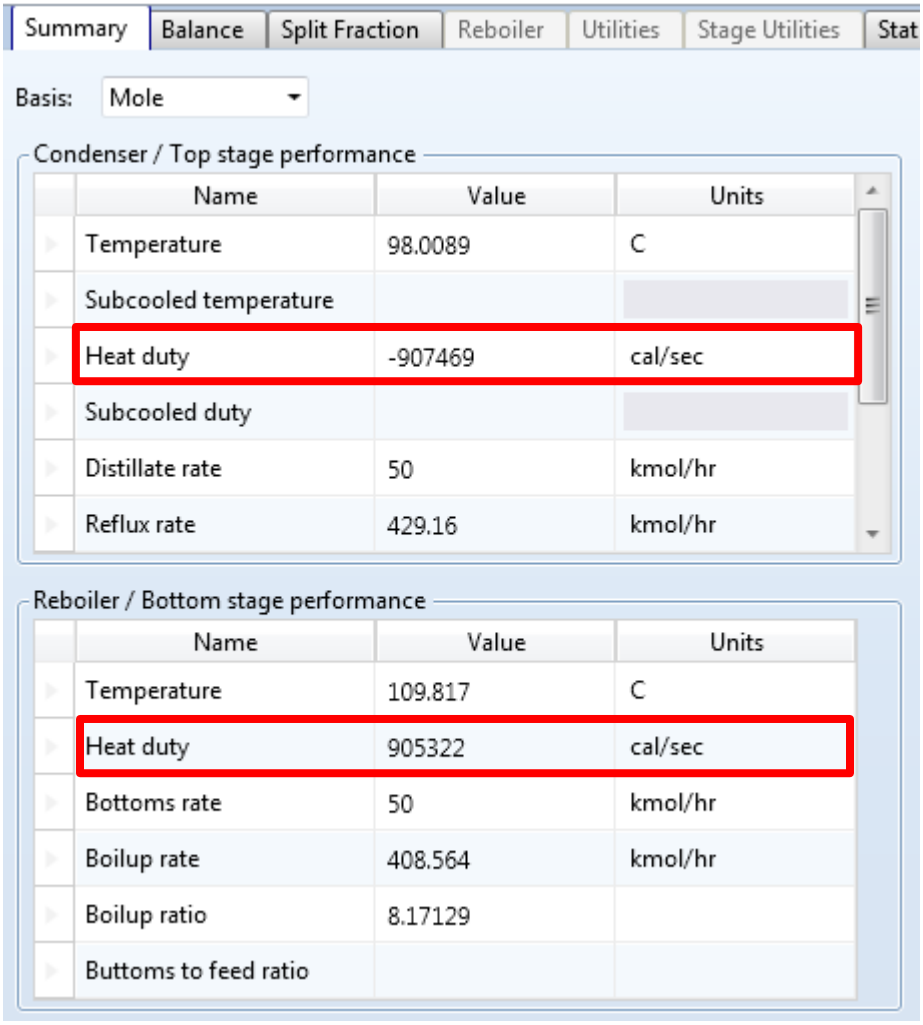
4.26. Go to the **Blocks | COLUMN | Sizing and Rating | Tray Sizing | 1 | Specifications** sheet. Enter **79** for the **Ending stage**.

4.27. Press the **F5** key to run the simulation. There should be no warnings or errors.

4.28. Go to the **Blocks | COLUMN | Tray Sizing | 1 | Results** sheet. Note that the calculated **Column diameter** is **2.1862 meter**.



4.29. Go to the **Blocks | COLUMN | Results | Summary** sheet and review **Condenser duty** and **Reboiler duty**.

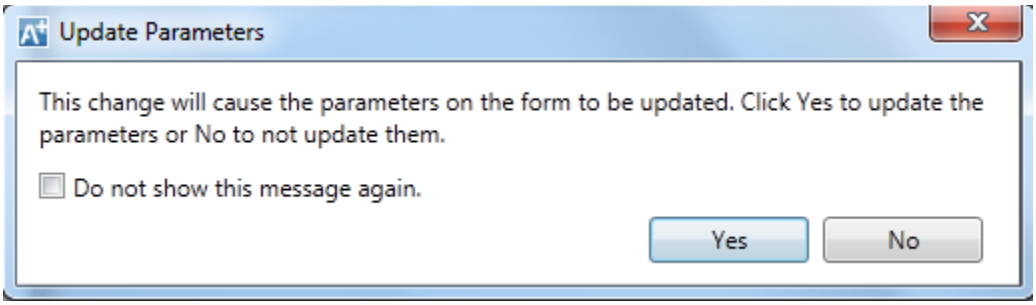


4.30. Now, press **Ctrl+S** to save the simulation. Close the Aspen Plus Window. This concludes our first case study.

4.31. Start the second case study. It uses extractive distillation with phenol as the solvent. In Windows Explorer, make a copy of **Dist-012_Base.bkp** and rename the new file to **Dist-012_Extractive_Distillation.bkp**. Double-click on file **Dist-012_Extractive_Distillation.bkp** to load it into Aspen Plus.

4.32. For extractive distillation, we will use phenol 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.

4.33. Go to the **Components | Specifications | Selection** sheet. In the third row, enter **PHENOL** in the **Component ID** column. The following dialog box will pop up. Click **Yes** to close the dialog box.



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

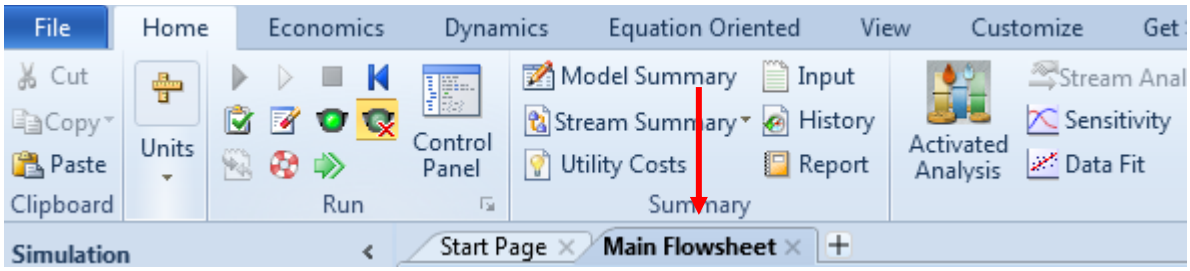
Input | Databanks | Information

Parameter: NRTL Data set: 1 Dechema

Temperature-dependent binary parameters

Component i	N-HEPTAN	N-HEPTAN	TOLUENE
Component j	TOLUENE	PHENOL	PHENOL
Temperature units	C	C	C
Source	VLE-RK	VLE-RK	VLE-RK
Property units			
AIJ	-1.7212	*-8.0029*	*-14.2162*
AJI	2.1619	*-4.9666*	*18.3762*
BIJ	399.67	*3646.19*	*5510.81*
BJI	-346.304	*2396.81*	*-6210.48*
CIJ	0.3	*0.47*	*0.3*
DIJ	0	*0*	*0*
EIJ	0	*0*	*0*
EJI	0	*0*	*0*
FIJ	0	*0*	*0*
FJI	0	*0*	*0*
TLOWER	30	*85.1*	*110.5*
TUPPER	110.7	*142*	*172.7*

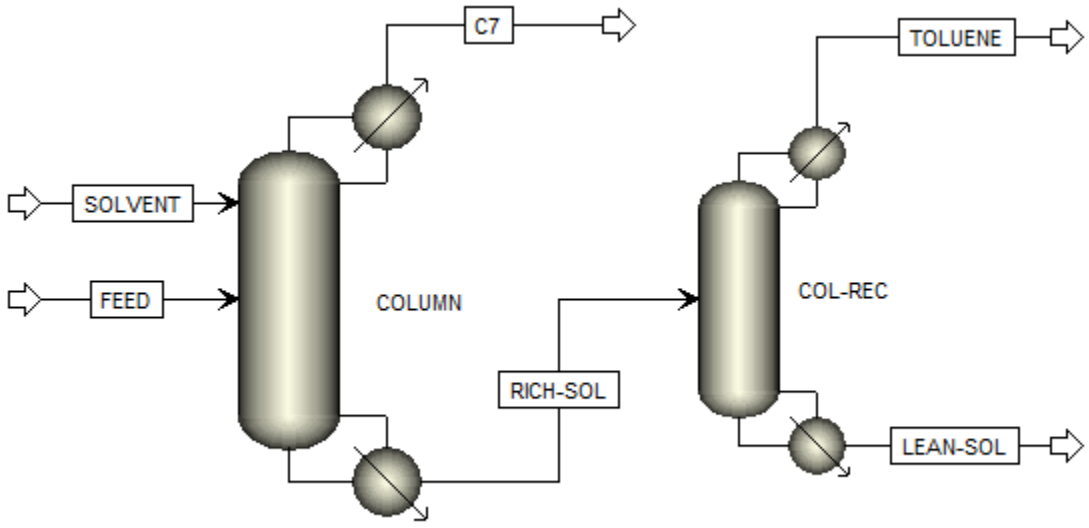
4.35. Move to the simulation environment by clicking the **Simulation** bar in the navigation pane. Then, click the **Main Flowsheet** tab as shown below.



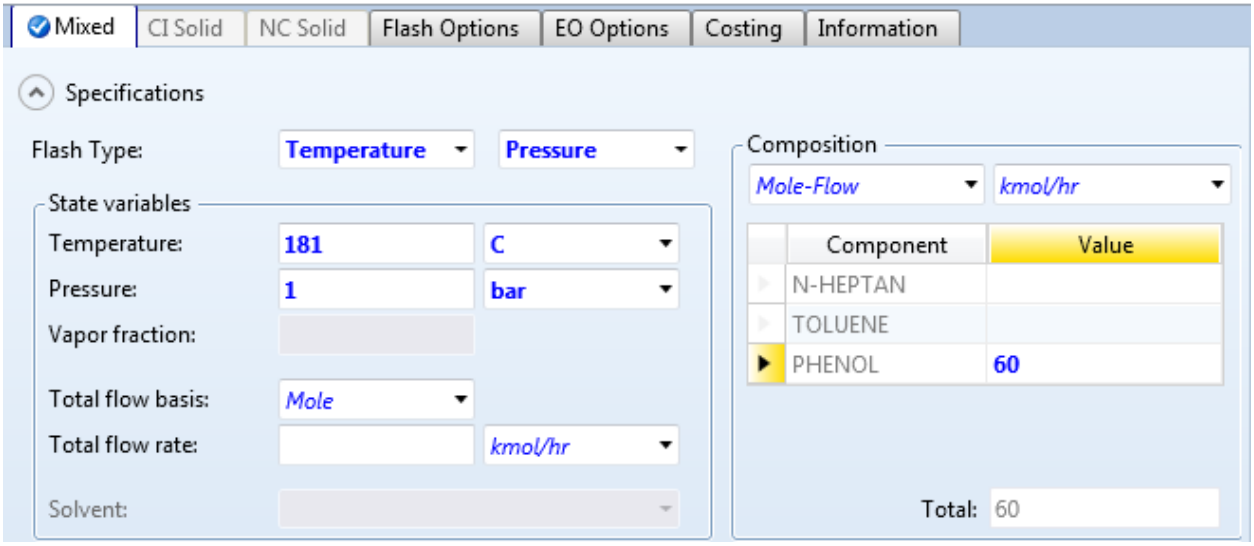
4.36. In the **Main Flowsheet**, rename stream **TOLUENE** to **RICH-SOL**. Then, modify the flowsheet to include two **RadFrac** blocks and the following streams.

Name	Type
C7	MATERIAL
FEED	MATERIAL
LEAN-SOL	MATERIAL
RICH-SOL	MATERIAL
SOLVENT	MATERIAL
TOLUENE	MATERIAL

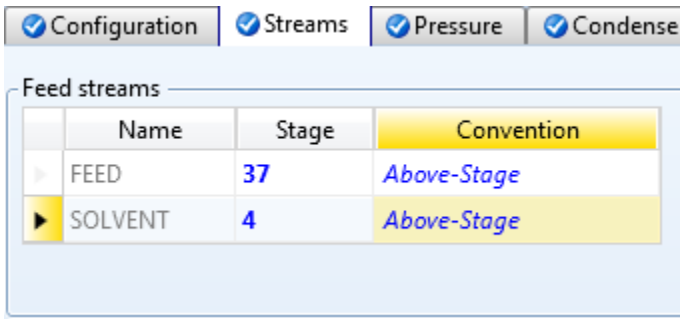
4.37. These blocks and streams should be connected in the following way.



4.38. Specify stream **SOLVENT**. Go to the **Streams | SOLVENT | Input | Mixed** sheet. Enter **181** for **Temperature** and **1** for **Pressure**. In the **Composition** frame, enter **60** for **PHENOL**. We use 181 °C because it is roughly the boiling point temperature of phenol at 1 bar. We use 60 kmol/hr because it leads to low energy consumption for the process. The **Streams | SOLVENT | Input | Mixed** sheet should look like the screenshot below.

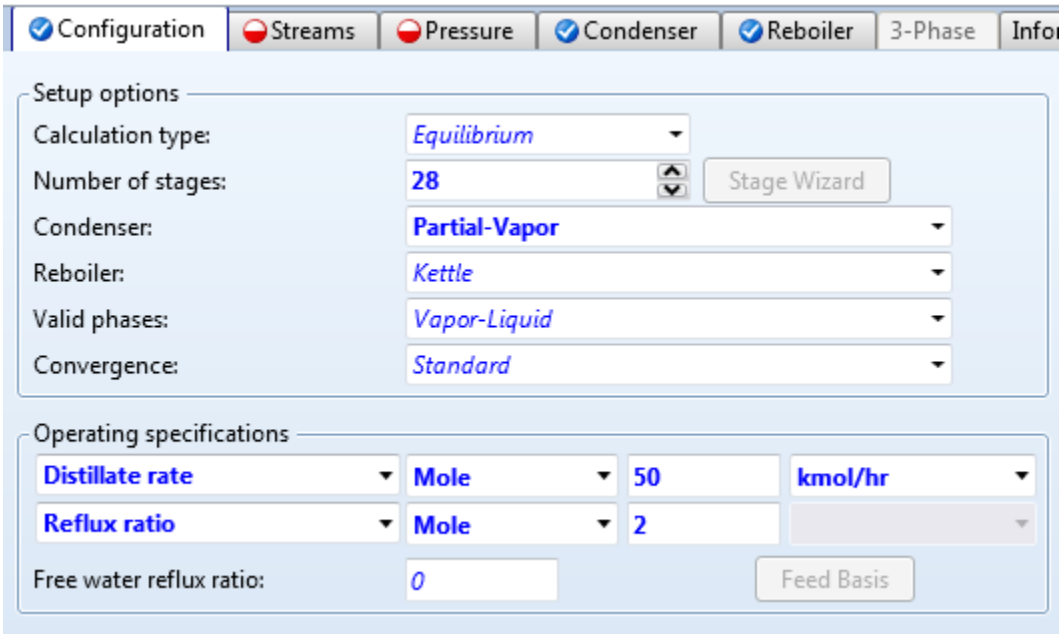


4.39. Go to the **Blocks | COLUMN | Specifications | Setup | Streams** sheet. In the **Stage** column in the **Feed streams** frame, enter **37** in for **FEED** and **4** for **SOLVENT**. We use 37 because it will lead to low energy consumption for this process. The **Blocks | COLUMN | Specifications | Setup | Streams** sheet should look like the screenshot below.

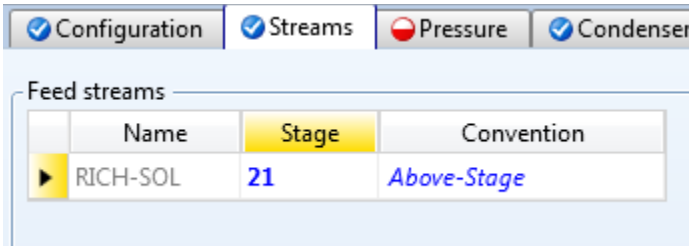


4.40. Go to the **Blocks | COLUMN | Sizing and Rating | Tray Sizing | 1 | Specifications** sheet. Enter **49** for **Ending stage**.

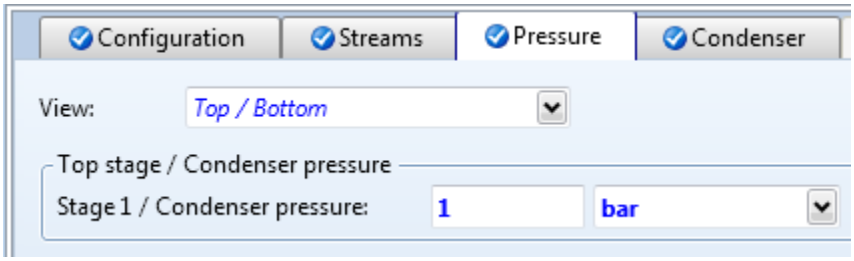
4.41. Specify **COL-REC**. Go to the **Blocks | COL-REC | Specifications | Setup | Configuration** sheet. Enter **28** for **Number of stages**. Select **Partial-Vapor** for **Condenser**. In the **Operating specifications** frame, enter **50** for **Distillate rate** and **2** for **Reflux ratio**. The **Blocks | COL-REC | Setup | Configuration** sheet should look like the screenshot below.



4.42. Go to the **Blocks | COL-REC | Specifications | Setup | Streams** sheet. In the **Feed streams** frame, enter **21** in the **Stage** column as shown below. We use 21 because it will lead to low energy consumption.

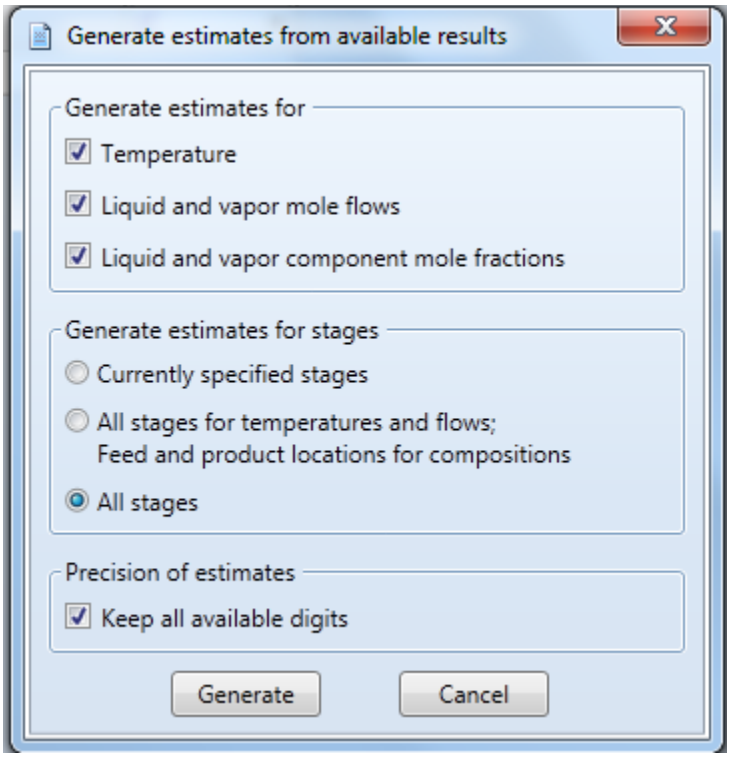


4.43. Go to the **Blocks | COL-REC | Setup | Specifications | Pressure** sheet. Enter **1** for **Stage 1 / Condenser pressure**.



4.44. Press the **F5** key to run the simulation and it should run without any error or warning.

4.45. Now, we need to make these two **RadFrac** blocks numerically more robust. Go to the **Blocks | COLUMN | Convergence | Estimates | Temperature** sheet. Click the **Generate Estimates…** button. In the popup dialog box, select the options shown below to generate the most estimates. Then click the **Generate** button and wait for the estimate generation to complete.

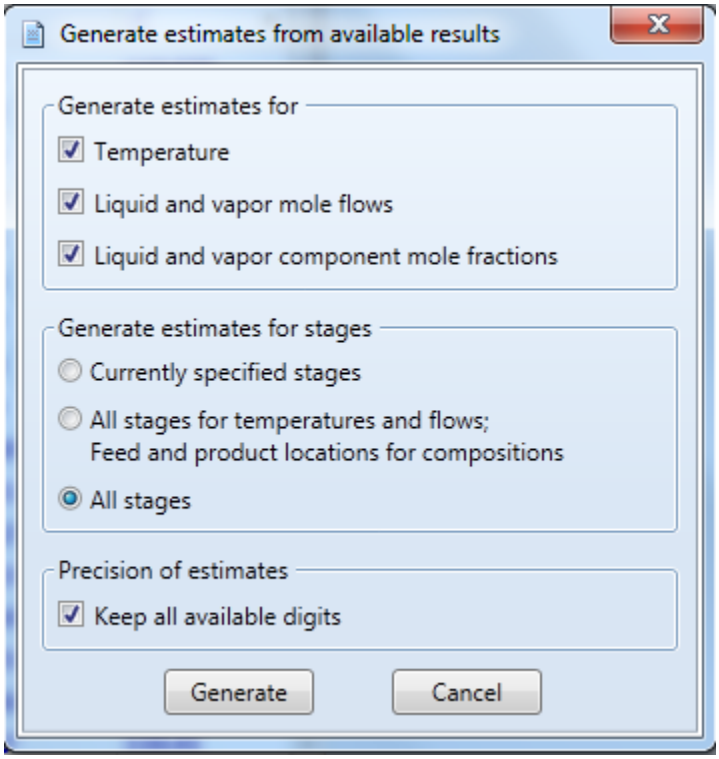


4.46. Go to the **Blocks | COLUMN | Specifications | Setup | Configuration** sheet. Select **Custom** for **Convergence**.

4.47. Go to the **Blocks | COLUMN | Convergence | Basic** sheet. Select **Newton** for **Algorithm**.

4.48. Go to the **Blocks | COLUMN | Convergence | Convergence | Advanced** sheet and select **Dogleg strategy** for **Stable-Meth**.

4.49. Go to the **Blocks | COL-REC | Convergence | Estimates | Temperature** sheet. Click the **Generate Estimates…** button. In the popup dialog box, select the options to generate the most estimates as shown below. Then click **Generate** button and wait for estimate generation to complete.



4.50. Go to the **Blocks | COL-REC | Specifications | Setup | Configuration** sheet. Select **Custom** for **Convergence**.

4.51. Go to the **Blocks | COL-REC | Convergence | Convergence | Basic** sheet. Select **Newton** for **Algorithm**. Enter **200** for **Maximum iterations** and **1e-7** for **Error tolerance**.

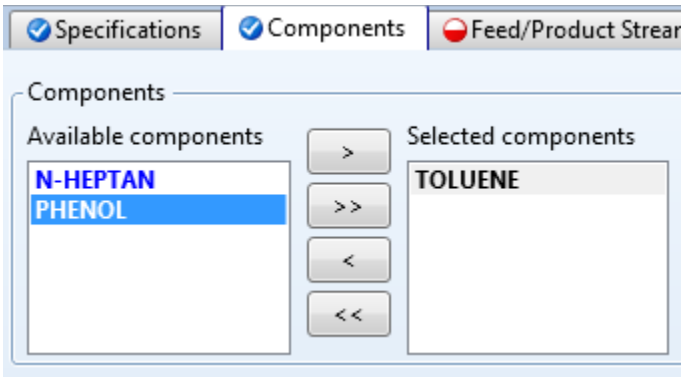
4.52. Go to the **Blocks | COL-REC | Convergence | Convergence | Advanced** sheet and select **Dogleg strategy** for **Stable-Meth**.

4.53. Now, click the button in the **Home | Run** group of the ribbon to reinitialize the simulation. Then, run the simulation again to ensure it still converges.

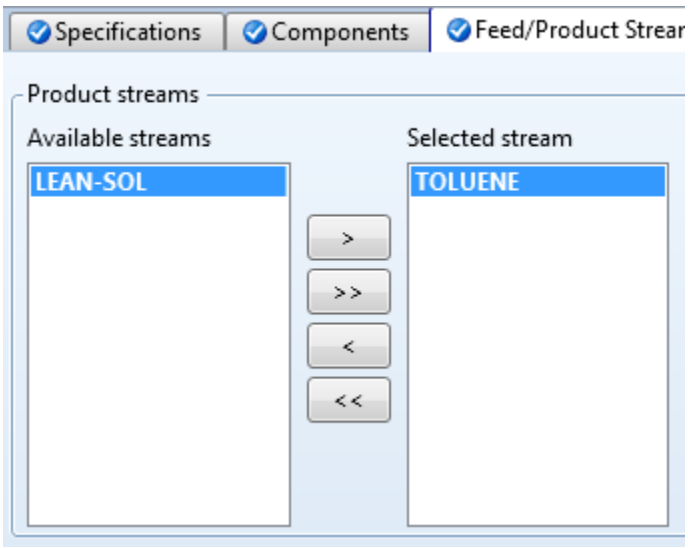
4.54. Now, we will define two pairs of **Design Specs** / **Vary** for **COL-REC** to specify the purity of the two outlet streams of the column. In the navigation pane, select **Blocks | COL-REC | Specifications | Design Specifications**. The object manager for **Design Specifications** is displayed. Click the **New…** button to create a new **Design Specification** called **1**.

4.55. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 1 | Specifications** sheet. Select **Mole purity** for **Type** and enter **0.9** for **Target**. We will later change this target to 0.99.

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



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



4.58. Now, we create the second Design Specs to specify the purity of stream **LEAN-SOL**. In the navigation pane, select **Blocks | COL-REC | Specifications | Design Specifications**. The object manager for **Design Specifications** is displayed. Click the **New…** button to create a new **Design Specification** called **2**.

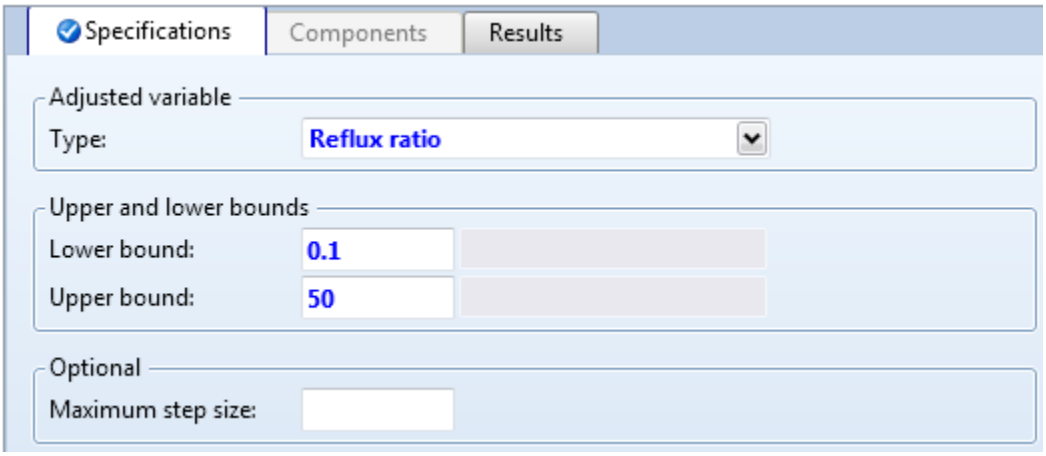
4.59. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 2 | Specifications** sheet. Select **Mole purity** for **Type** and enter **0.99999** for **Target**.

4.60. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 2 | Components** sheet. In the **Components** frame, move **PHENOL** to the **Selected components** list.

4.61. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 2 | Feed/Product Streams** sheet. In the **Product streams** frame, move **LEAN-SOL** to the **Selected stream** list.

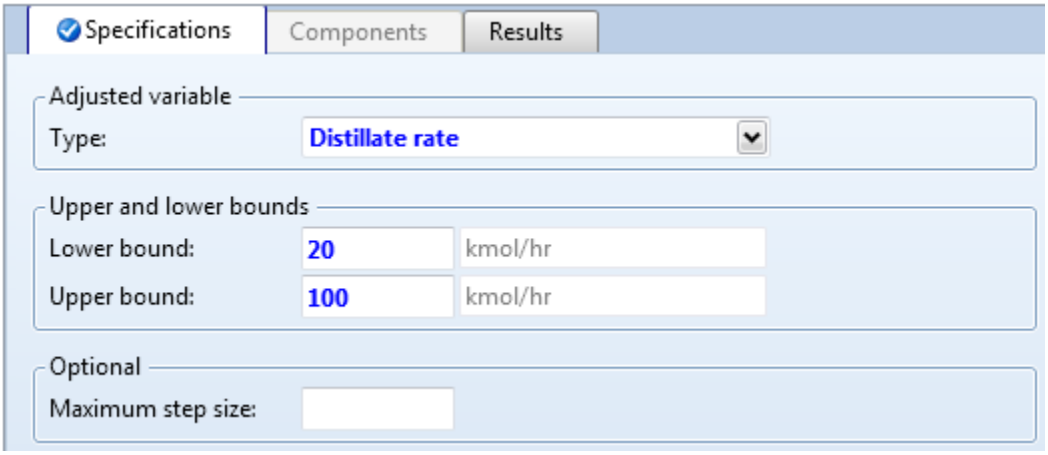
4.62. Now, we create corresponding **Varys**. In the navigation pane, select **Blocks | COL-REC | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New…** button to create a new **Vary** called **1**.

4.63. Go to the **Blocks | COL-REC | Specifications | Vary | 1 | Specifications** sheet. Select **Reflux ratio** for **Type**. Enter **0.1** for **Lower bound** and **50** for **Upper bound**. Now, the sheet should look like this.



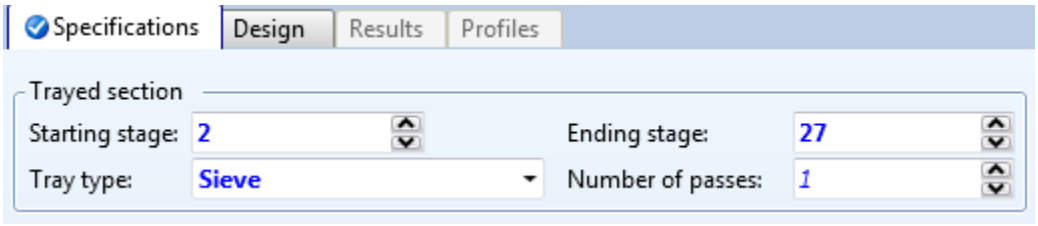
4.64. In the navigation pane, select **Blocks | COL-REC | Specifications | Vary**. The object manager for **Vary** is displayed. Click the **New…** button to create a new **Vary** called **2**.

4.65. Go to the **Blocks | COL-REC | Specifications | Vary | 2 | Specifications** sheet. Select **Distillate rate** for **Type**. Enter **20** for **Lower bound** and **100** for **Upper bound** as shown below.



4.66. In the navigation pane, select **Blocks | COL-REC | Sizing and Rating | Tray Sizing**. The object manager for **Tray Sizing** is displayed. Click the **New…** button to create a new **Tray Sizing** section called **1**.

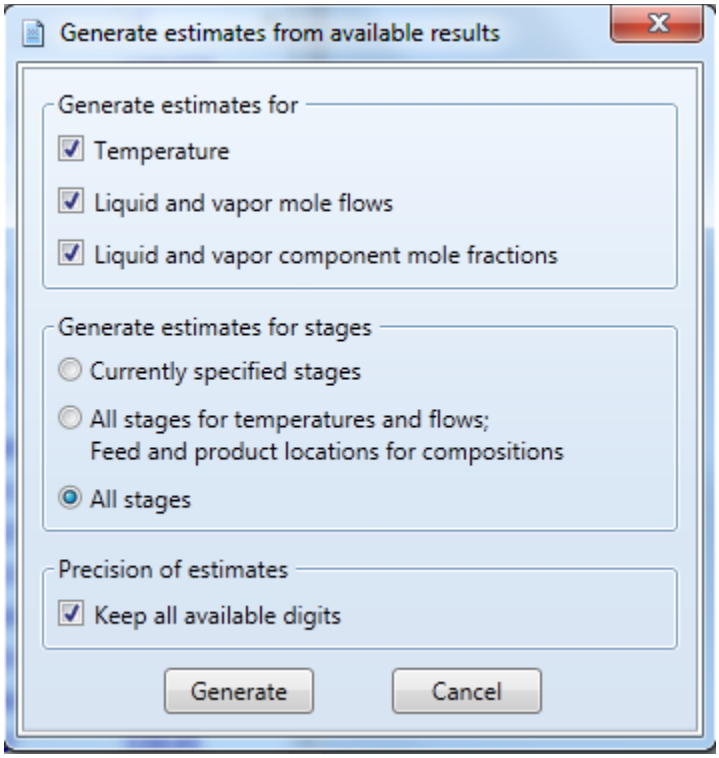
4.67. Go to the **Blocks | COL-REC | Sizing and Rating | Tray Sizing | 1 | Specifications** sheet. Enter **2** for **Starting stage** and **27** for **Ending stage**. Select **Sieve** for **Tray type** as shown below.



4.68. Press the **F5** key to run the simulation and it should complete without error or warning.

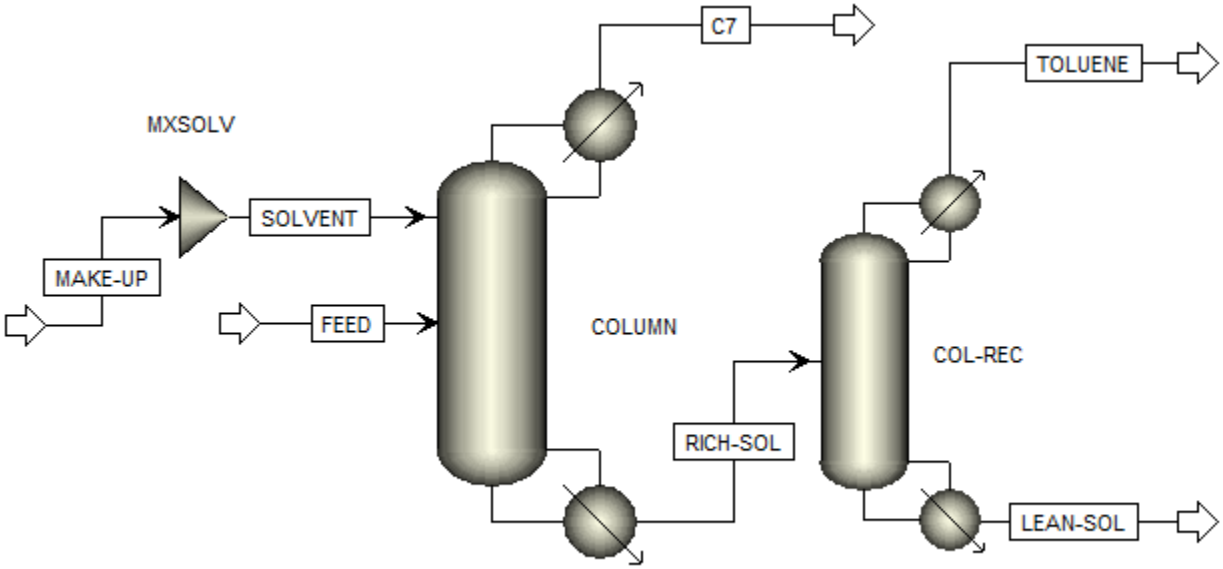
4.69. Go to the **Blocks | COL-REC | Specifications | Design Specifications | 1 | Specifications** sheet. Change **Target** from **0.9** to **0.99**. Press the **F5** key to run the simulation again.

4.70. 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 the **Generate** button and wait for the estimate generation to complete.

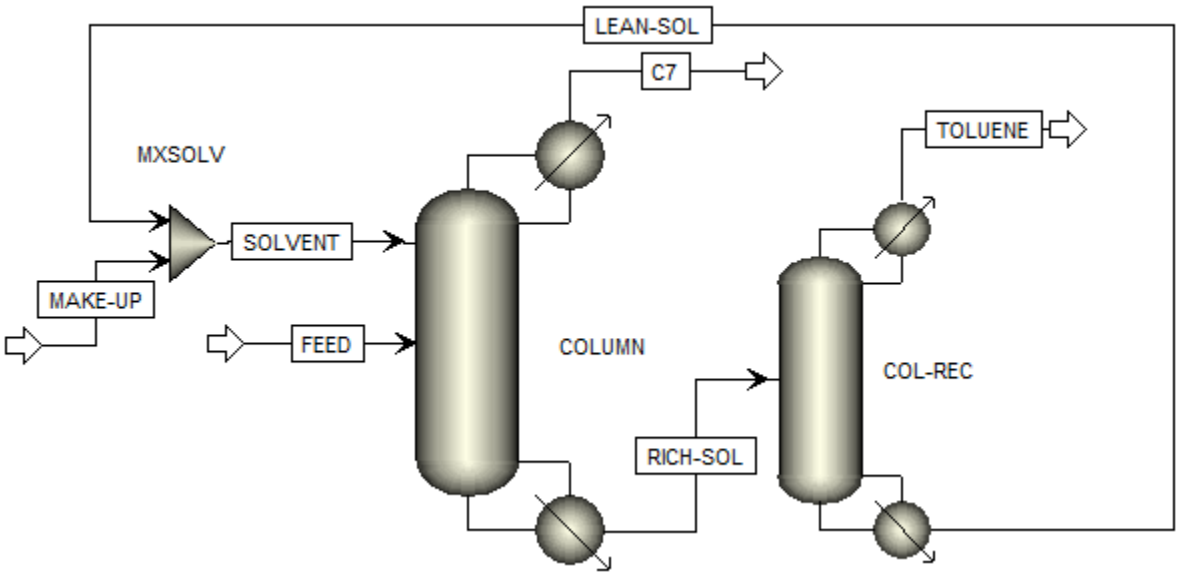


4.71. Now, click the button in the **Home | Run** group of the ribbon to reinitialize simulation. Then, run the simulation again to ensure that it still converges.

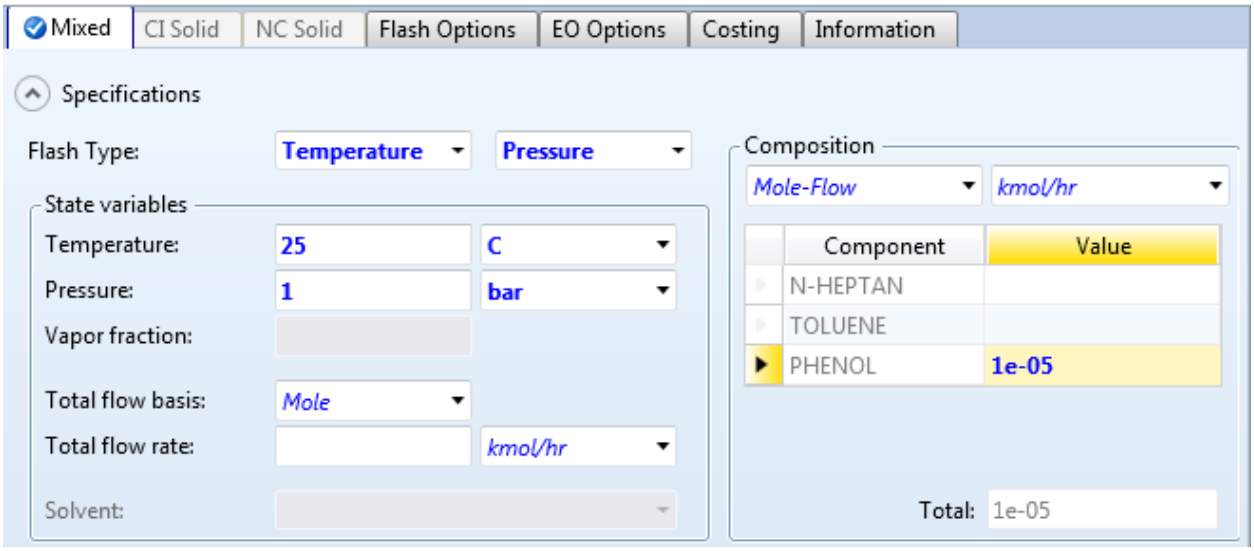
4.72. Add a solvent make-up stream. Add a **Mixer** block, **MXSOLV**, before stream **SOLVENT** and add a solvent make-up stream, **MAKE-UP**. The flowsheet should look like the screenshot below.



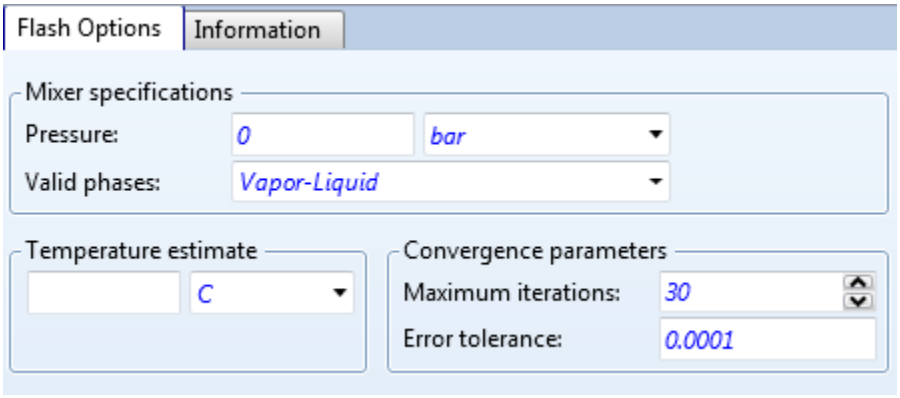
4.73. Close recycle loop. In the **Main Flowsheet** window, right click stream **LEAN-SOL** and select **Reconnect Destination** on the context menu. Move the mouse cursor over the inlet port icon of **MXSOLV** and click the inlet port icon. The flowsheet should look like the screenshot below.



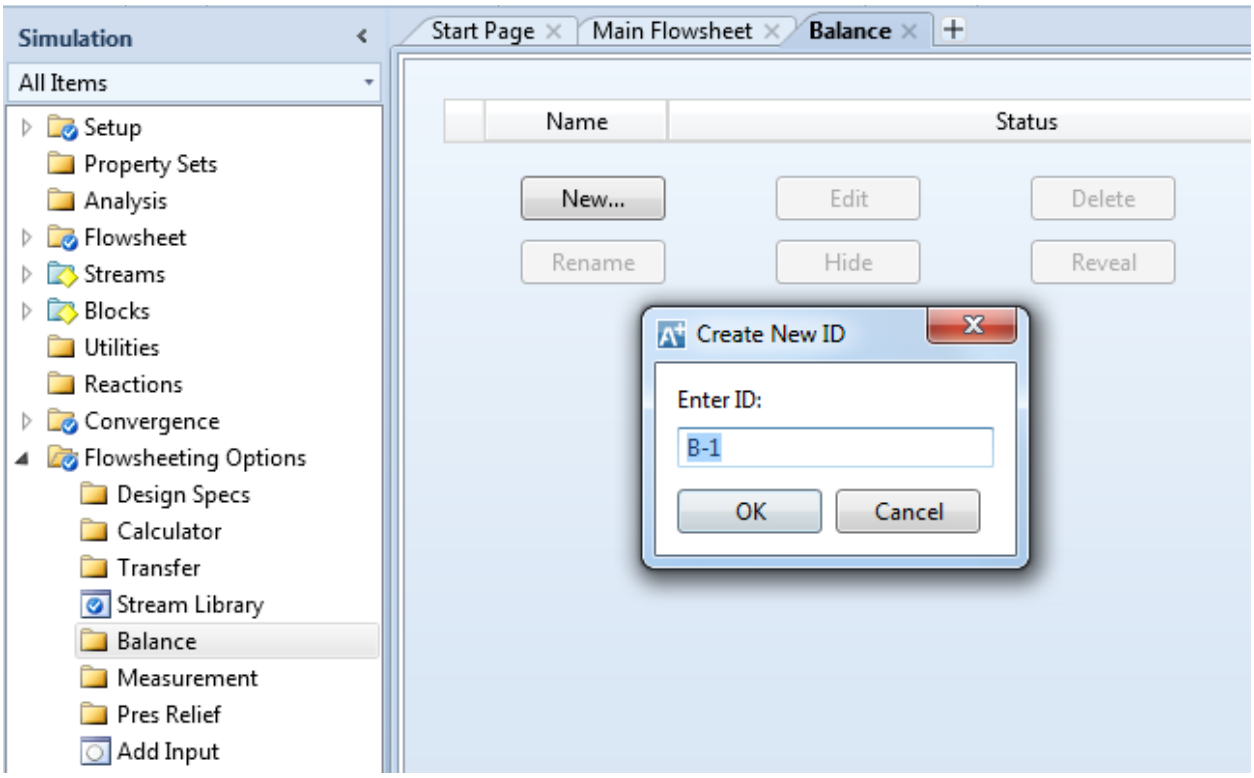
4.74. Specify stream **MAKE-UP**. Go to the **Streams | MAKE-UP | Input | Mixed** sheet. Enter **25** for **Temperature** and **1** for **Pressure**. In the **Composition** frame, enter **0.00001** for **PHENOL**. For now, we just enter a tiny flow for the solvent make-up stream as an estimate. Later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



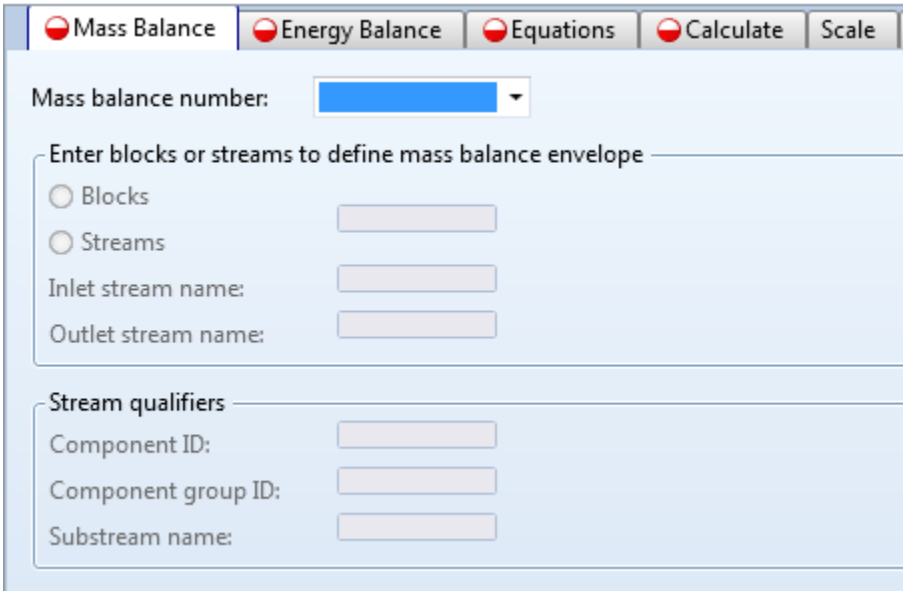
4.75. Go to the **Blocks | MXSOLV | Input | Flash Options** sheet. Note that the value for **Pressure** is **0**, indicating no pressure drop.



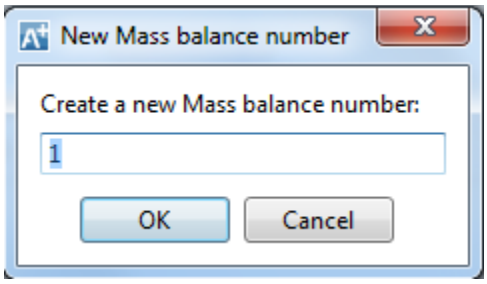
4.76. In the navigation pane, click the **Flowsheeting Options | Balance** node. The object manager for **Balance** is displayed. Click the **New...** button and click the **OK** button to create a **Balance** block called **B-1**.



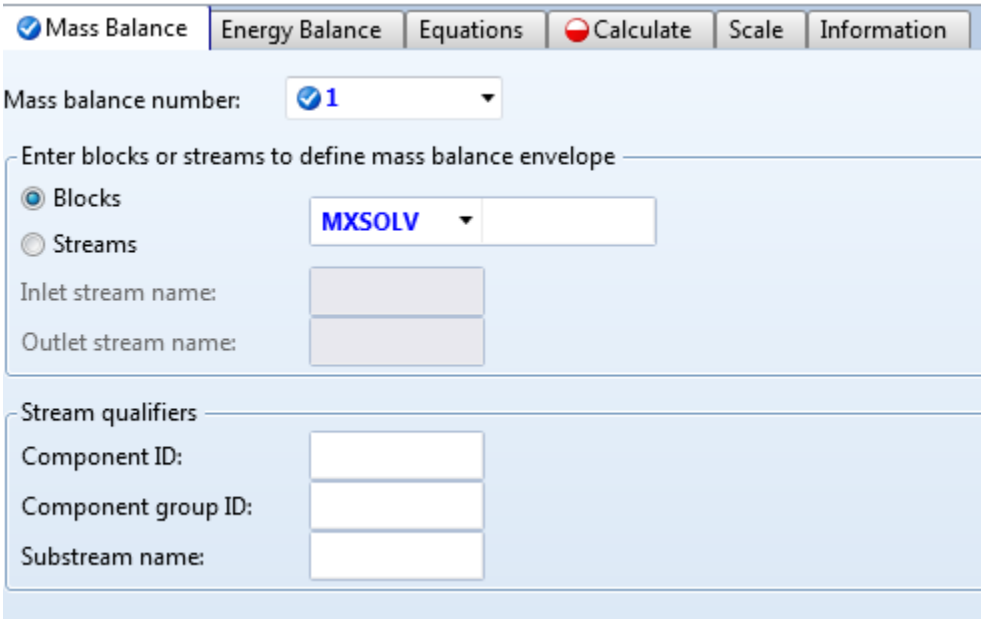
4.77. Go to the **Flowsheeting Options | Balance | B-1 | Setup | Mass Balance** sheet. Click the **Mass balance number** dropdown box and click **<New>**.



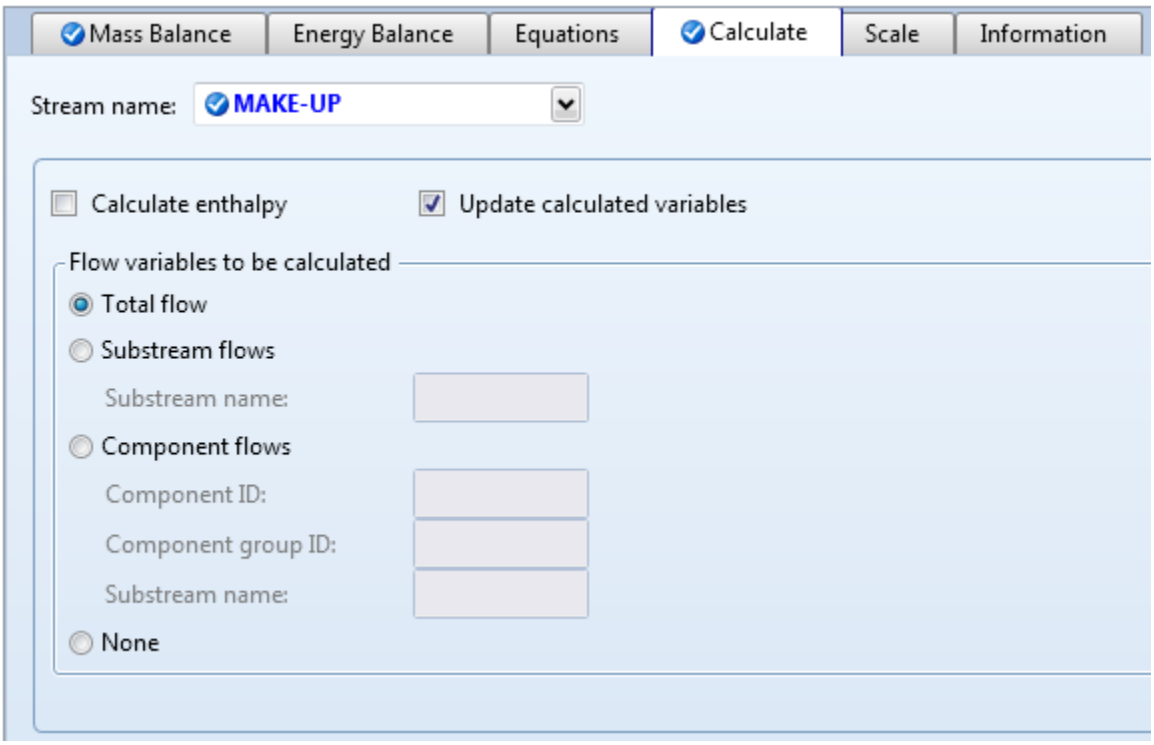
4.78. The **New Mass balance number** window pops up. Click the **OK** button.



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

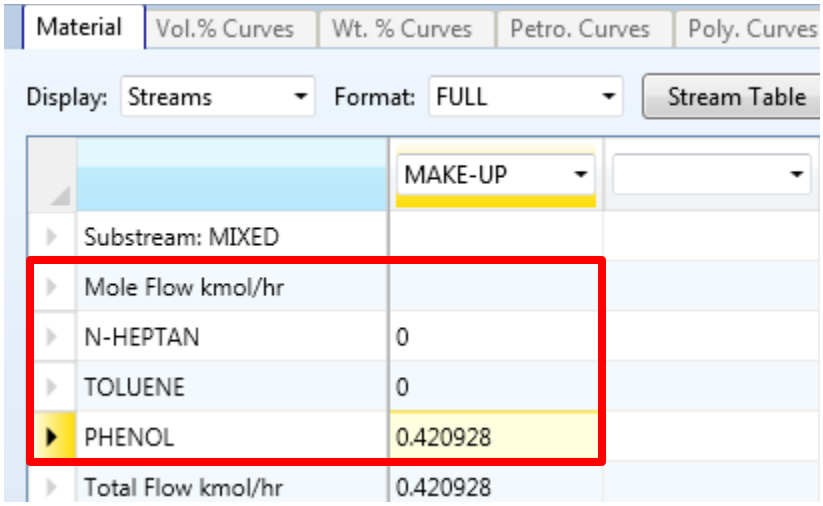


4.80. Go to the **Flowsheeting Options | Balance | B-1 | Setup | Calculate** sheet. Select **MAKE-UP** for **Stream name**. This **B-1** balance block will calculate the flowrate of stream **MAKE-UP**.



4.81. Click the button in the **Home | Run** group of the ribbon to reinitialize simulation. Then, press the **F5** key to run the simulation again and the simulation completes without any error or warning.

4.82. Go to the **Streams | MAKE-UP | Results | Material** sheet. Note that the flowrate for **PHENOL** is **0.420928 kmol/hr** instead of the originally entered 0.00001 kmol/hr.



4.83. Go to the **Blocks | COLUMN | Results | Summary** sheet to view the results. The **Heat duty** of the **Condenser** is **-551854 cal/sec.** The **Heat duty** of the **Reboiler** is **584371 cal/sec**.

4.84. Go to the **Blocks | COLUMN | Tray Sizing | 1 | Results** sheet. The calculated **Column diameter** is **1.73609 meter**.

4.85. Go to the **Blocks | COL-REC | Results | Summary** sheet to view results. The **Heat duty** of the **Condenser** is **-84495 cal/sec.** The **Heat duty** of the **Reboiler** is **162587 cal/sec**.

4.86. Go to the **Blocks | COL-REC | Tray Sizing | 1 | Results** sheet. The calculated **Column diameter** is **0.833042 meter**.

4.87. The table below compares the required duties and column diameters for the extractive distillation approach against those for the direct distillation approach with one column.

	Extractive Distillation	Single Column Distillation
Total Heating Duty (cal/sec)	746958	905322
Total Cooling Duty (cal/sec)	636349	907469
Hardware	50 stages; D=1.73609 meter 28 stages; D=0.833042	80 stage; D=2.1862 meter

5. Conclusions

For the separation of n-heptane and toluene, extractive distillation has the following advantages over single-column distillation:

- 17.5% less heating duty
- 30% less cooling duty
- Less hardware: 2 fewer stages and much smaller column diameter

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