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BATCHCOLUMN APPLICATION EXAMPLE

PURIFICATION OF THYMOL BY BATCH DISTILLATION

EXAMPLE PURPOSE
The main interest of this example is to describe two different heating systems for the boiler: an external jacket and a helical coil. Furthermore, a reactive calculator generated by Simulis Kinetics after the identification of the kinetic parameters for the thymol synthesis is also used. This way, the components, the thermodynamic model, and the chemical reactions can be easily loaded in the BatchColumn simulation.

ACCESS	☑ Free Internet	☐ Restricted to clients	☐ Restricted	☐ Confidential

CORRESPONDING BATCHCOLUMN FILE	*BATCHCOL_EX_EN - Thymol.pbpc*

TABLE OF CONTENTS

1. Introduction

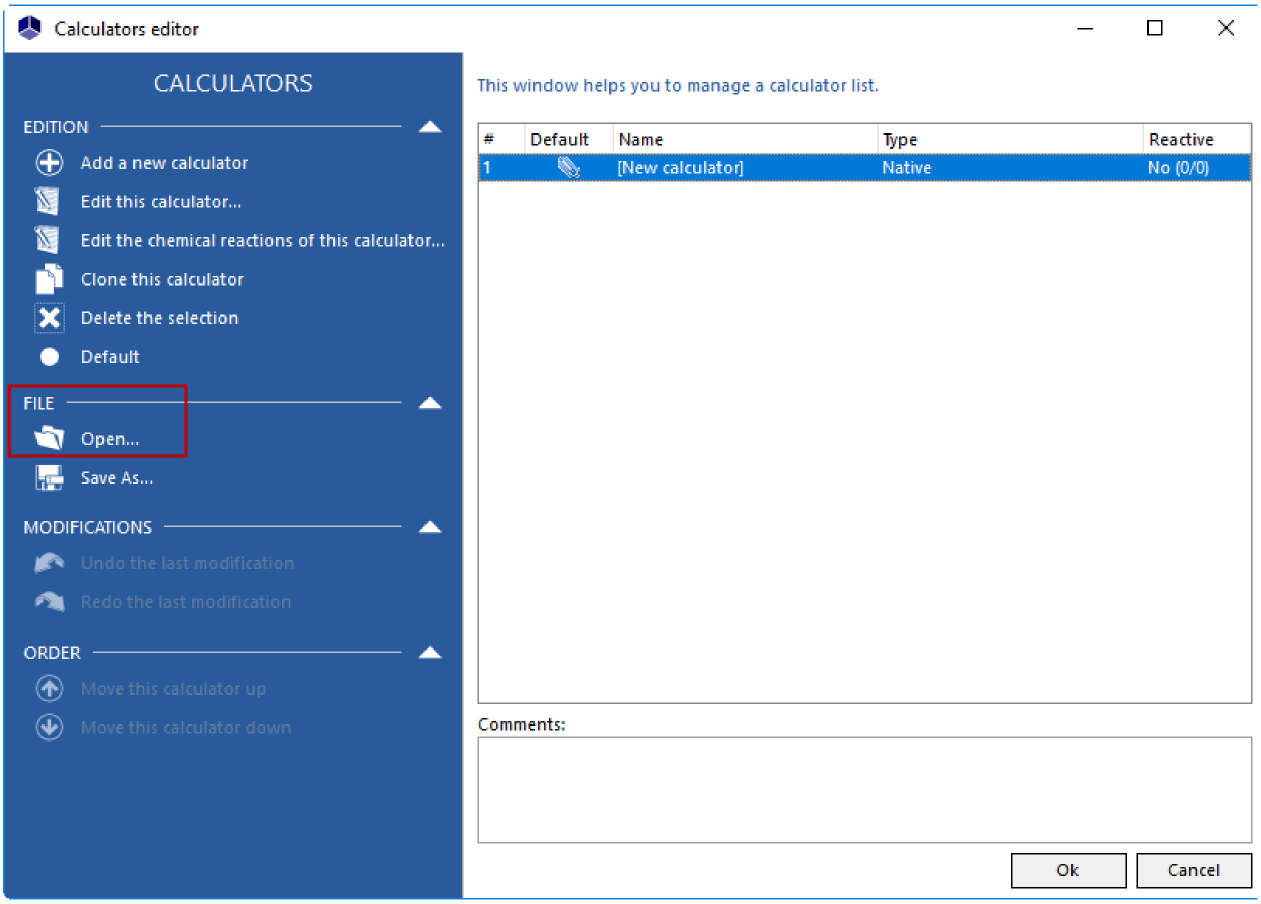
Thymol is a phenol contained in the thyme oil and in the volatile essential oils of other plants. It takes the form of colorless crystals with a specific aromatic smell. It is soluble in alcohols, in fat and oil, but slightly soluble in water. It is notably used for its antiseptic action, its antibacterial properties and its antifungal effect as well as to stabilize the pharmaceutical preparations.

This example deals with the thymol purification and the recovery of m-cresol that has not reacted after a batch distillation. The operating mode consists of five steps: filling of the column, infinite reflux, m-cresol cut, inter-cup and thymol cut. The technological elements in relation with the heating devices of the boiler are taken into account (external jacket and helical coil).

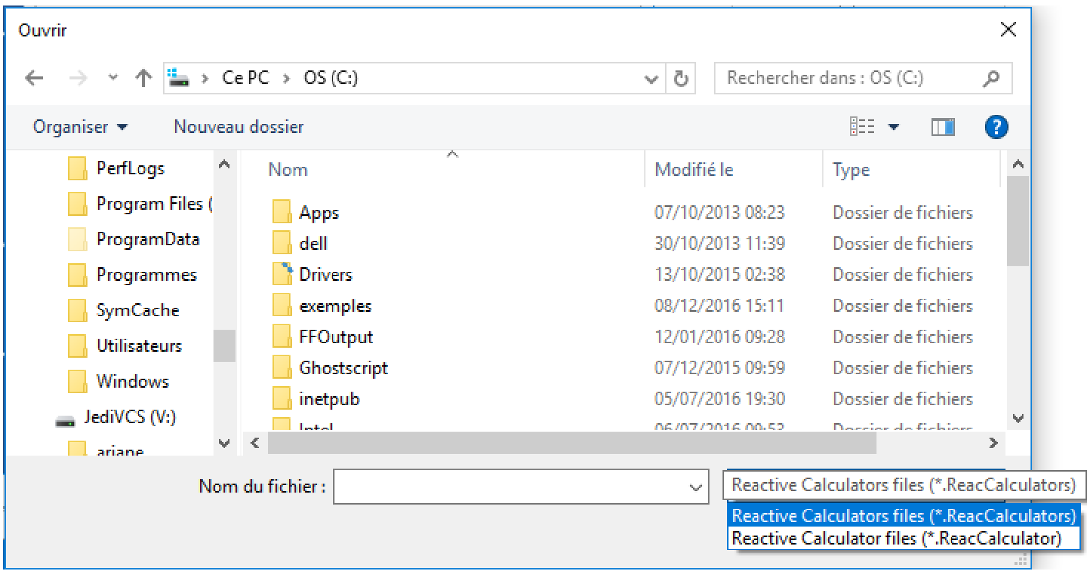
It is the last example of a series of three examples dealing with the synthesis and the purification of thymol. The first example “SIMKIN_EX_EN – Thymol” allows to identify the parameters of the chemical reactions. The second example “BATCHREA_EX_EN – Thymol” deals with the synthesis of thymol.

2. COMPONENTS, THERMODYNAMIC MODEL

The components, the thermodynamic model as well as the chemical reaction model (reaction scheme and kinetic parameters) are going to be loaded directly from the « .ReacCalcultor » file generated at the end of the « SIMKIN_EX_EN - Thymol » example. In the calculators editor, delete the default calculator, then click **File/Open**.



In the **Open** window, select a Reactive calculator file (.ReacCalculator) as file type.



Note: the remaining propylene at the end of the synthesis is eliminated by degassing and then is not transferred to the column boiler. The "propylene" component can then be deleted from the components list. Due to the absence of propylene, the chemical reactions will not occur. Therefore they can be deleted or deactivated.

3. SIMULATION

3.1. Process description

3.1.1. Column

The column is made up with 56 theoretical stages, including the boiler and the condenser.

Three collection tanks will be used during the distillation step.

The initial load is detailed below. It corresponds to the content of the reactor at the end of the synthesis without the propylene (see the "BATCHREA_EX_EN – Thymol" example).

✓	Temperature	25°C	
✓	Total mass	1 825 kg	
✓	Composition	m-cresol	27.9541% wt.
		Thymol	24.3378% wt.
		3M2P	18.7774% wt.
		3M5P	2.3200% wt.
		3M4P	26.6107% wt.

The liquid hold-ups are supposed to be constant all along the distillation:

- ✓ Condenser 5 l
- ✓ Column 4 l for each theoretical stage

The pressure profile is also supposed to be constant all along the distillation process

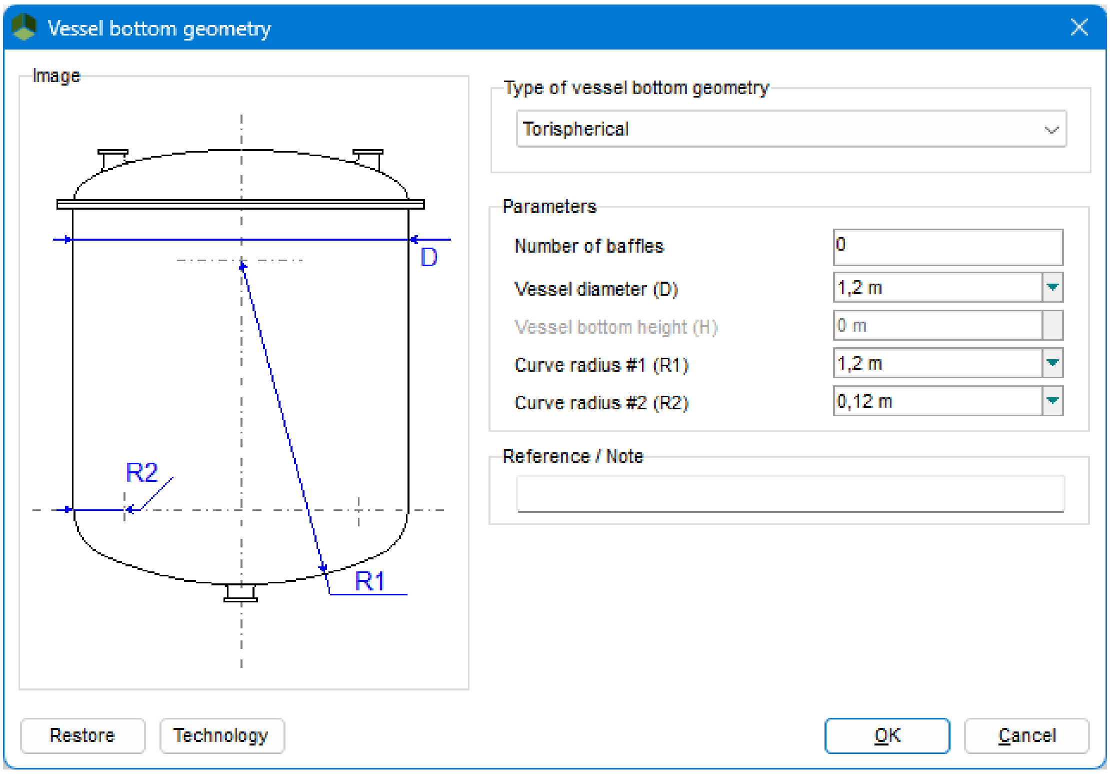
- ✓ Condenser 40 mmHg
- ✓ Pressure drop 10 mmHg

3.1.2. Condenser

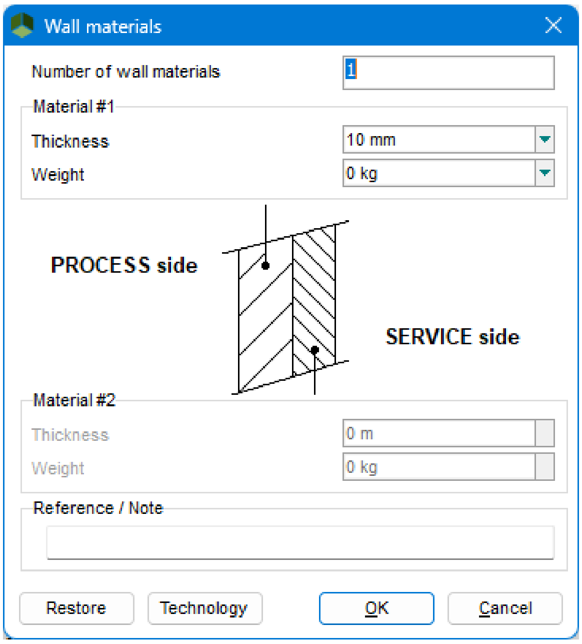
The condenser is supposed to be ideal total all along the distillation process.

3.1.3. Boiler

The column's boiler characteristics are detailed below.



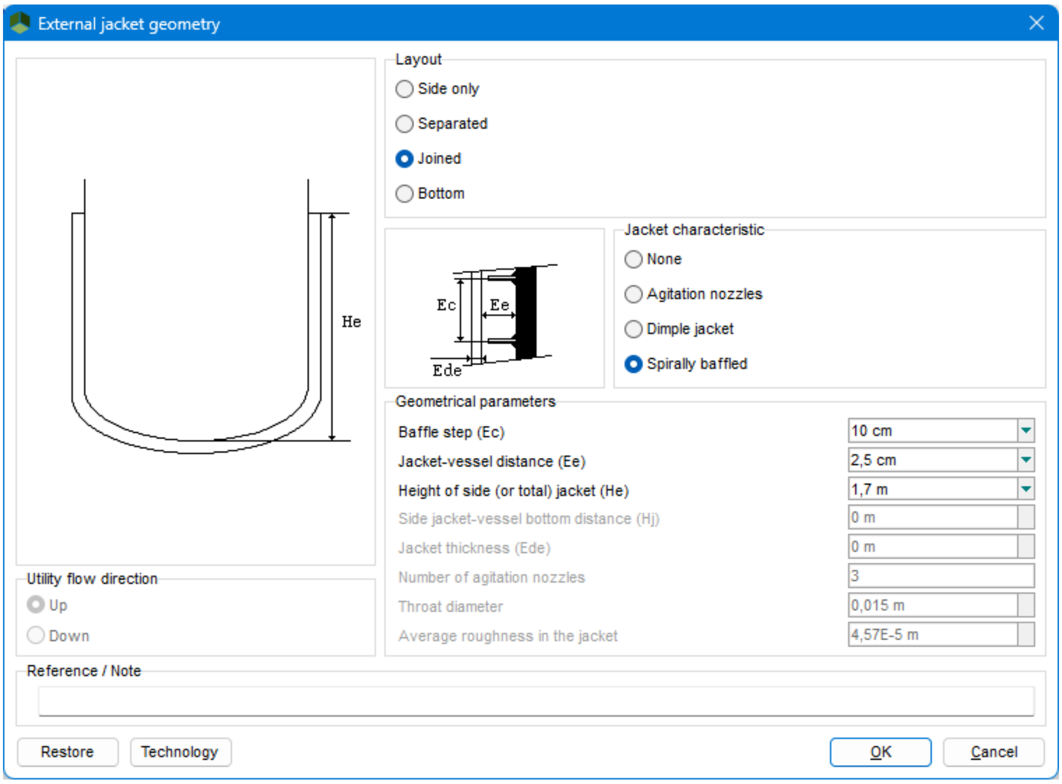
With a wall material defined as follows:



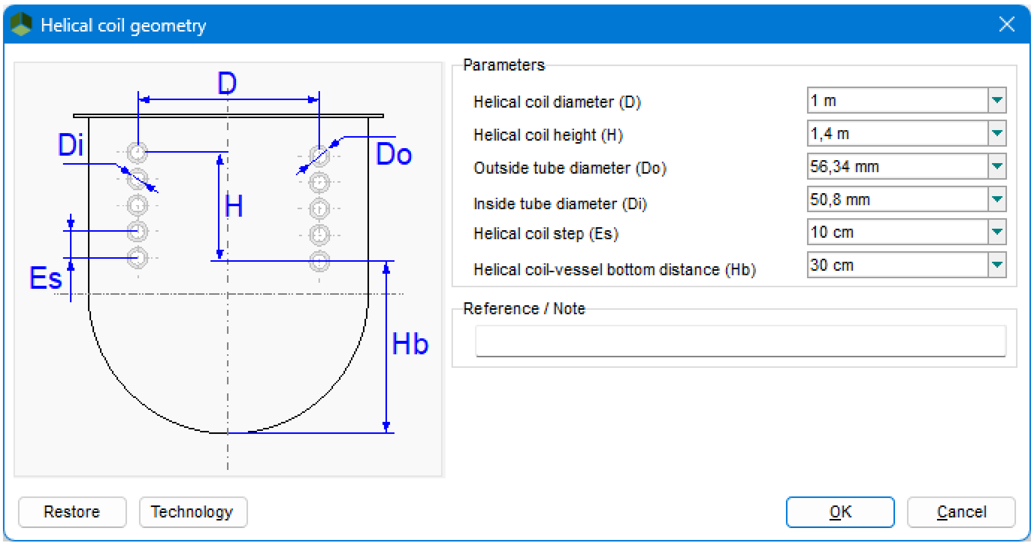
The thermal conductivity of the wall is considered to be equal to 19 $W.m^{-1}.K^{-1}$. The thermal conductivity is specified for each step.

3.1.4. Boiler heating device

The column's boiler is equipped with a wall heat exchanger (external jacket) with the following characteristics:



And with an immersed heat exchanger (helical coil) detailed below:



The thermal fluid used is the same for both systems and is described in the table below:

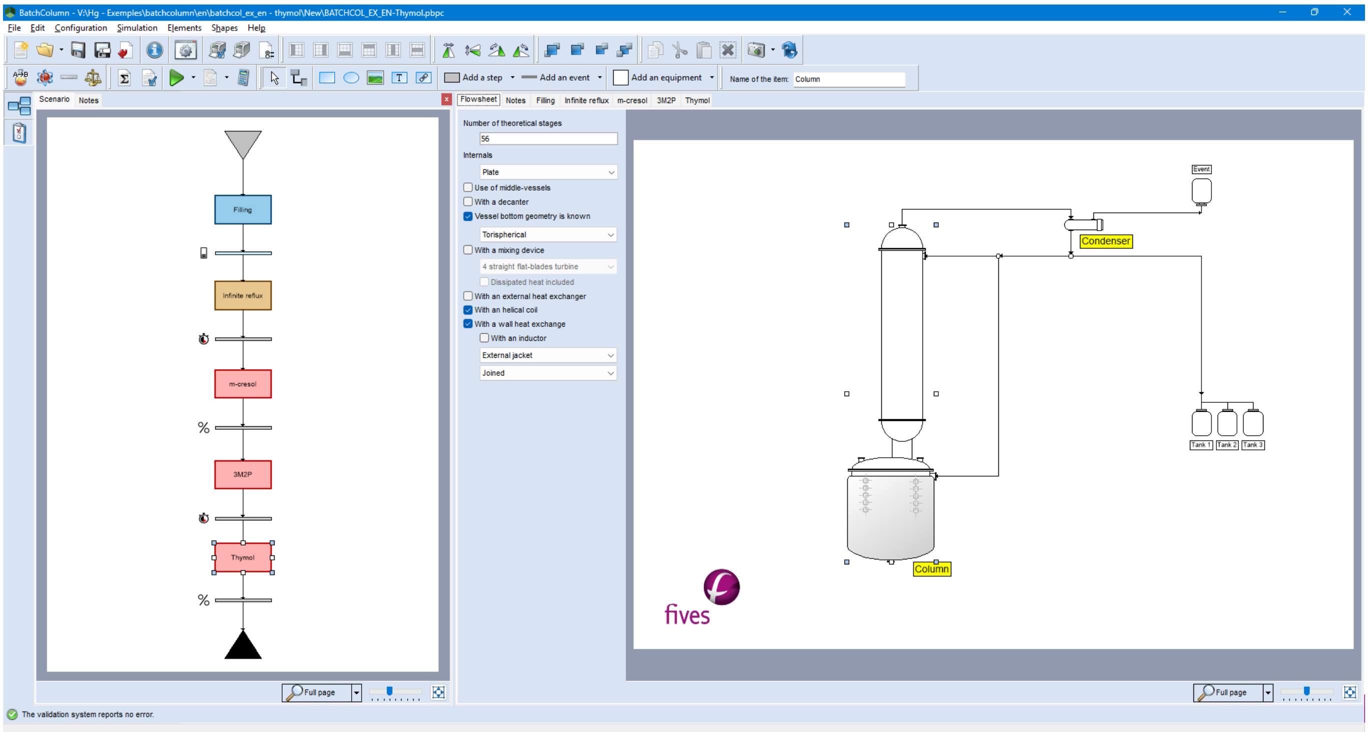
Heat transfer fluid (the same for all the steps)		
Exchanger	Wall heat exchanger	Helical coil
Fluid	Gilotherm DO RP (150 300)	
Flowrate	400 kg/h	1 350 kg/h
Inlet temperature	300°C	

3.1.5. Operating mode

The operating mode is made up with five steps. During the first step, the column is filled. A second step at infinite reflux allows to classify the products in the column. The third step allows to recover the maximum quantity of m-cresol to recycle it. The next step, an intercut, allows to eliminate mainly the 3M2P. The last step is the thymol production. The operating parameters of these steps appear in the table below.

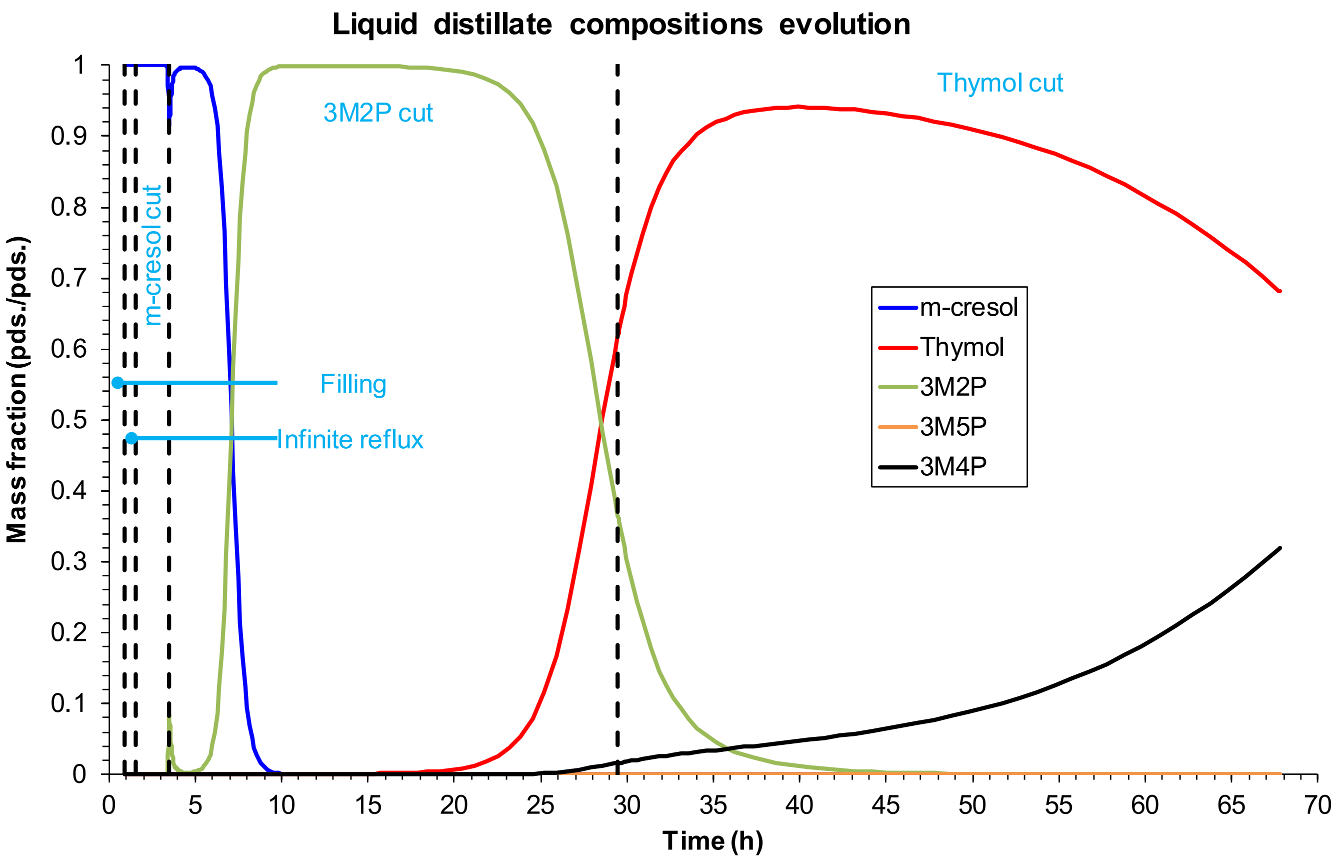
Parameter	Step 1 Filling	Step 2 Infinite reflux	Step 3 m-cresol cut	Step 4 3M2P elimination	Step 5 Thymol cut
Type	Filling	Infinite reflux	Distillation		
Operation	Variable heat duty				
Reflux ratio	-		2,5	50	
Collection tank	-		Tank #1	Tank #2	Tank #3
Stop event	Column filled	Step duration = 40 min	m-cresol content in the tank #1 < 99,9% wt.	Duration of the step = 26 h	Thymol content in the tank #3 < 87% wt.

The scenario is presented on the left of the following screenshot and the flowsheet on the right part.



3.2. Results

During the filling step, there is no liquid distillate. During the third step, the liquid distillate consists mainly of m-cresol. The m-cresol cut is stopped as soon as the content in 3M2P begins to be significant. The 3M2P cut contains 3M2P, the remaining m-cresol and the thymol that begins to be at the top of the column. The thymol cut is stopped when the collection tank meets the specification requirement.



The table below shows the content of the collection tanks, of the boiler and of the liquid holdups (condenser and plates) after the operation is completed. The 3M5P and 3M4P mainly stay in the boiler, and the liquid holdups in the column.

	Tank 1	Tank 2	Tank 3	Boiler	Liquid holdups
Mass (kg)	405	408	401	376	184
Composition (wt. %)					
m-cresol	99,90	13,51	0	0	0
Thymol	8 ppm wt.	5,89	87,00	5,69	27,01
3M2P	0,10	80,47	3,43	0,1 ppm wt.	1 ppm wt.
3M5P	0	Traces	Traces	10,89	0,75
3M4P	0,4 ppm wt.	0,13	9,57	83,42	72,24

