

Methyltetrahydrofuran

How to Recover and Dry MeTHF Batchwise

MeTHF Recovery

The solubility behavior of MeTHF/water mixtures and the favorable MeTHF-water azeotrope make it possible to recover dry MeTHF with conventional distillation equipment.

The solubility of water in MeTHF does not increase very much as the temperature is increased from 0 °C to 70 °C.

Table 1.
Solubility of Water in MeTHF

Temperature °C	Wt% Water
0.0.....	4.0
9.5.....	4.1
19.3.....	4.1
29.5.....	4.2
39.6.....	4.3
50.1.....	4.4
60.7.....	4.6
70.6.....	5.0

However, the solubility of MeTHF in water dramatically drops as the temperature is increased.

Table 2.
Solubility of MeTHF in Water

Temperature °C	Wt% Water
0.0.....	21.0
9.5.....	17.8
19.3.....	14.4
29.5.....	11.4
39.6.....	9.2
50.1.....	7.8
60.7.....	6.6
70.6.....	6.0

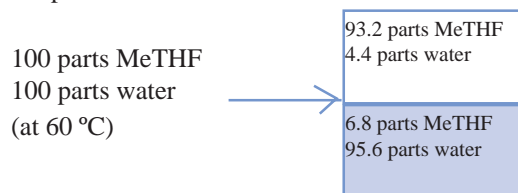
Because of this drop in solubility of MeTHF in water as the temperature is increased, mixtures of water and MeTHF should always be separated at temperatures above 60 °C to minimize the loss of MeTHF to the water phase.

The MeTHF-water azeotrope contains 10.6 % water. Wet

MeTHF can be dried at atmospheric pressure in a batch or continuous distillation process using this azeotrope. Also, products dissolved in MeTHF can be dried using this azeotrope.

Example of Batch MeTHF Recovery

As an example of how dry MeTHF can be recovered, consider a process that has a stream that contains 100 parts MeTHF and 100 parts water. Separation of this mixture in a decanter that is held at 60 °C gives a MeTHF layer that has 4.4 parts water and a water layer that has 6.8 parts MeTHF.



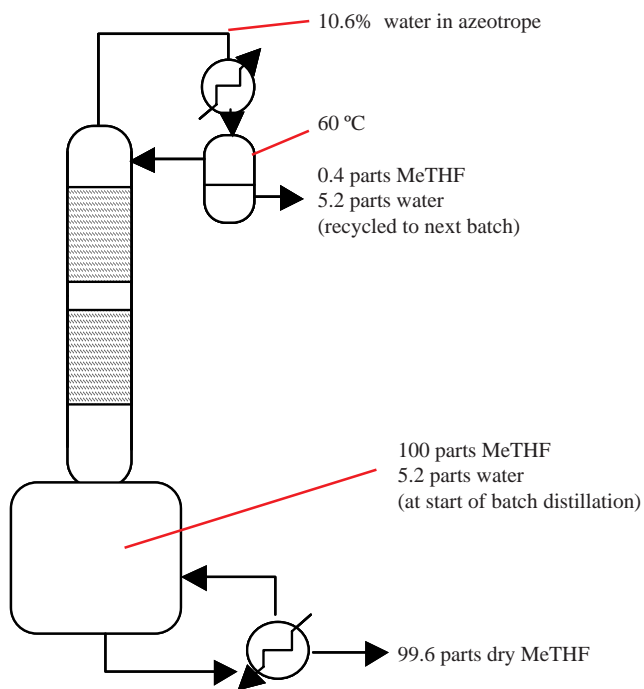
The MeTHF in the water phase and the MeTHF phase can both be recovered as dry MeTHF in batch distillations. The MeTHF in the water phase can be recovered by distilling the azeotrope (10.6% water) and adding the collected overheads to the MeTHF phase. A distillation set-up consisting of a reboiler, a fractionation tower and an overhead condenser with a liquid-liquid decanter can be used to recover dry MeTHF. The overhead condenser and decanter are operated at 60 °C to minimize the loss of MeTHF in the decanted water phase. The condensed MeTHF in the decanter is added back to the column as reflux. The water phase from the decanter is recycled to the next batch distillation to recover the MeTHF dissolved in it. With this process, essentially all of the MeTHF can be recovered as dry MeTHF.

The MeTHF azeotropic distillation is similar to drying with toluene except that the effective MeTHF azeotrope is 6% water since the refluxed MeTHF contains about 4.6% water.

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How to Recover and Dry MeTHF

The figure below shows a schematic of the distillation equipment needed to recover dry MeTHF. The overhead decanter should be sized so that at the end of the batch distillation when no more water phase can be removed from the overhead decanter, the liquid and vapor hold-up in the column can be distilled without reflux. The MeTHF in the decanter can be used in the next batch distillation. After a batch distillation, the reboiler product typically has about 300 ppm water.

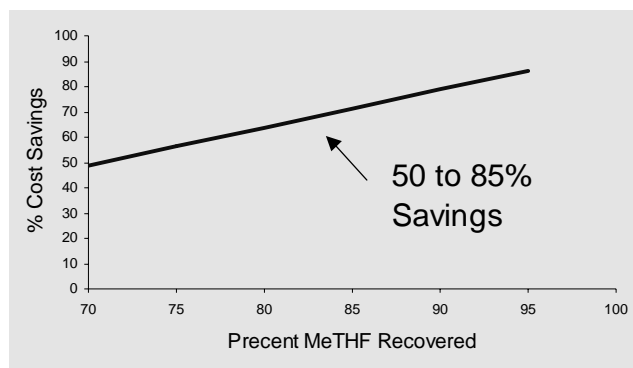


Switch to MeTHF and Save Money

Although the initial cost of MeTHF may be higher than THF, significant cost savings can be realized from a switch to MeTHF if the THF is not currently being recovered. The total cost of THF is equal to the cost of buying and disposing of the product. The adjacent figure shows the % cost savings that can be realized by installing and operating a MeTHF recycle process. Penn will help you design a customized recovery system that will reduce your THF

costs by as much as 50 to 85%. In addition to the cost savings, recovering MeTHF is a more environmentally friendly practice than disposing of THF.

% Cost Savings versus % MeTHF Recovered



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