1 INTRODUCTION

The purpose of this exercise is to determine the efficiency of the column as a function of vapour velocity and reflux ratio. The flooding and weeping conditions of the column is also to be determined

2 RELEVANT LITERATURE

2.1 THEORETICAL BACKGROUND MATERIAL

- General distillation theory can be found in Lydersen (1985),Terjesen and Lydersen, Perry (1984), McCabe and Smith (1976) and King (1971)

- Definitions of weeping- and flooding point is given in Lydersen(1985), King(1971) and Traybal(1980)

2.2 DATA AND CONSTANTS

- Equilibrium data can be found in Gmelin and Onken(1977)

- Weight% ethanol as a function of density can be found in Perry(1984)

2.3 SIMILAR EXPERIMENTS IN THE LITERATURE

- According to McCabe and Smith(1984), Perry(1984) and Peavy & Baker(1937), the average plate efficiency increases with increasing vapour velocity up to an maximum. Around maximum, the efficiency is almost independent of the vapour velocity. At even higher vapour velocities, the efficiency drops sharply with vapour velocities. Lydersen(1985) reports that the maximum average plate efficiency is achieved with a vapour velocity equal to 0.4 to 0.85 times the flooding velocity.

- According to Lydersen(1985), the average plate efficiency decreases with increased reflux ratio for a given separation. No prediction of the efficiency is given in the literature if the separation changes with changed reflux ratio.

3 THEORY

3.1 DEFINITIONS

Weeping point: The point where the vapour velocity is too low to penetrate the liquid layers in the column. As a result of this, the liquid starts to leak through the holes in the plates.

Flooding point: With countercurrent flow, there are upper limits to the vapour and liquid amounts that can be brought in contact with each other. At high vapour velocities, the vapour will carry liquid droplet upwards in the column. This will lead to liquid accumulation in the upper part of the column.
King(1971) claims that the flooding point is reached when the liquid level in a downcomer exceeds the distance between the plates. A small increase in the pressure drop can in this situation no longer be compensated with an increase in the liquid level in the downcomers. Accumulation in the upper parts of the column will lead to a sudden increase in pressure drop over the column

Total plate efficiency: The number of ideal plates divided by the number of practical trays in the column

Trountons rule: For chemically similar substances, the molar heat of vaporization is approximately equal for ideal systems.

Ideal stage: The liquid stream leaving the stage is in equilibrium with the vapour leaving the same stage.

3.2 THEORETICAL BACKGROUND

By distilling a two-component system one wishes to separate the two components and achieve as pure products as possible. This happens in a distillation column, where a part of the cold vapour is sent back through the column as liquid at its boiling point.
By bringing this liquid stream in contact with the vapour, the concentration of the lightest component in the vapour will increase, at the same time as the concentration of the heaviest component decreases. The liquid stream fed back into column is called reflux.

A certain amount of the condensate will be collected as product.

The simplest and perhaps most instructive method for analysis of binary distillation is the graphic approach presented by McCabe & Thiele in 1925. This method uses the fact that the composition at each point is completely determined by the mole fraction of one of the two components. The method is described in McCabe & Smith(1976)

The problem with the McCabe-Thiele method is that it is based on simplified enthalpy balances, making it possible to use the approximation of constant molar flows. When this approximation is not valid, the operating lines will not be straight. Using mass- and enthalpy balances in addition to equilibrium data and enthalpy/concentration data, operation lines can be constructed based on Ponchon-Savarits method. This method is described in King(1971).

By simulated continuous distillation with varying reflux ratio, the mole fraction of the lightest component in the liquid in the top part of the column will decrease as the reflux ratio decreases. Each new reflux ratio will alter the gradient of the operating line.

4 APPARATUS

4.1 COLUMN

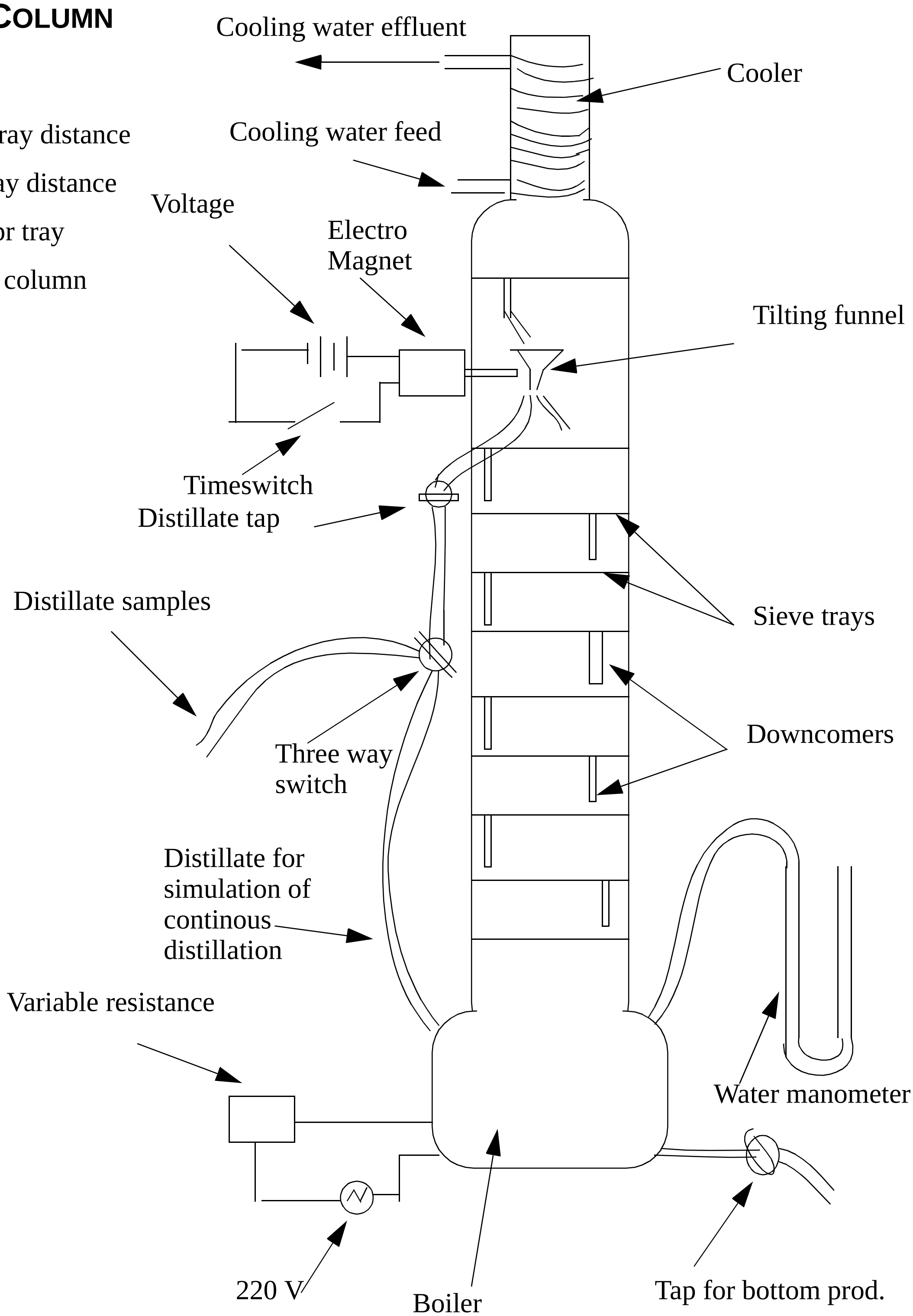
10 trays

150 mm tray distance

75 mm tray distance

55 holes pr tray

1700 mm column



4.2 TIMING PULS GENERATORS

The reflux to the column is set by a timing generator witch controls the tilting funnel and thus the condensate flow. The pulse and pause time can be varied between 2.5 and 100%, witch corresponds to a pause and puls times from 0.5 to 15 s. 50% reflux corresponds to pause time = pulse time, e.g pulse = 20%, pause = 20%. Increasing the ratio between pause and pulse increases the reflux. 30% pause and 10% pulse gives 75% reflux. The total cycle time (pause + pulse) should not exeed 20 s.

5 EXPERIMENTAL

5.1 OPERATION AND SAFETY

Operating the column is fairly simple. The two most important points is to turn on the cooling water first, and to fill the boiler with enough liquid to cover the heater **at all times**. For this, 4 litres of feed should be sufficient. The heater is about 20 cm high.

Before filling the column, make sure that the bottom and distillate tap lines are **closed.**

After filling the column, some liquid might have accumulated in the distillate tap. Remove this and feed it back into the top of the column.

When the feed is cold (first run), it takes some time to heat up the mixture and the column. To speed up this process, increase the boiler effect (about 2A) and use some reflux. When liquid starts to appear in the distillate product tube, switch to full reflux and set the desired input effect to the boiler.

Use a feed mixture with 0.1 mole fraction ethanol.

Compositions are measured using pyknometers. These pyknometers needs to be calibrated first, using distilled water as reference. Use 3 pyknometers pr. sample.

In an emergency situation, turn off the heating, but leave the cooling water on.

5.2 EXERCISE 1: EFFICIENCY VS. VAPOUR VELOCITY

Run the column with total reflux. Do 6 experiments at different boiler effects, giving different vapour velocities. Vapour velocities, pressure drop, top and bottom composition must be recorded. In what order should you do the measurements?

Compute average plate efficiency based on comparing the real number of plates and the theoretical number computed using M-T.

Flooding and weeping point can be found by considering the pressure drop.

5.3 EXERCISE 2: EFFICIENCY VS. REFLUX RATIO

Fix the boiler effect at the optimal point found from the previous experiment. Determine the effect of reflux ratio on efficiency by simulating continuous distillation. To do this, feed the distillate back into the boiler.

5.4 PLANNING THE EXPERIMENT

After reading this description, study the literature and make sure that you understand the process and the theoretical background. Make sure that you understand the McCabe-Thiele method. A brief overview of distillation theory and McCabe-Thiele must be included in the experiment plan.

Go to the lab and study the column. Locate every part marked on the apparatus description.

Find the litterature data you need. Generate a McCabe-Thile diagram and make enough copies.

Write a detailed plan for the experiment. This plan should include not only how to do the experiment, but also how to calculate the results needed.

Before you can start the experiments, the plan has to be approved by the supervisor. You will also have to prove that you are familiar with the apparatus and know how to operate it. You must also read and sign the HMS booklet.

5.5 VERSIONS

Students taking version 2 or 3 of the course will perform both exercises. Students taking version 1 can choose between exercise 1 and 2.

6 REPORT

More info later.

7 LITTERATURE

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