Felleslab: Distillation Columns

Separation

September - October, 2021

Contents

1. Objectives

The purpose of this laboratory work is to understand and investigate the distillation principles, the parameters affecting the operation of distillation columns and how to find optimal operating conditions. The following three tasks have to be done in the lab:

1. determine time required to reach steady state condition;
2. determine the efficiency of a column as a function of the vapor velocity;
3. determine the flooding and weeping conditions of the column (**Not required this year)**

This is a list of documents that are expected to be delivered:

1. A photocopy of your lab journal (see description in section 6.6);
2. Work Plan three days before you are scheduled to run the experiment (see description in section 5.1);
3. Report (see description in section 7)

Note that the main purpose of the report is to show that you have learned and understood the objective behind the three tasks given in this experiment.

2. Required Knowledge/Supporting Literature

You should read/ know:

✓ Definition of flooding and weeping point
✓ Theory behind McCabe-Thiele's method
✓ Ability to apply McCabe-Thiele's method in an exercise
✓ Understanding of how an operating line changes with reflux ratio
✓ Ethanol-water density-composition data at 25°C from Perry's handbook (2008).
✓ You should read the HMS booklet thoroughly and sign it. There are copies in English available at the Department office. This must be done by every student before you are allowed to work in the lab.

Before the experiments, you MUST:

✓ Read and understand the risk assessment for the experiment. These are uploaded on the course website.
✓ Calculate the amount of ethanol (96 vol% purity) and water needed in order to prepare your start solution (see section 6)
✓ Submit the work plan (see section 5) to your supervisor at least 3 days before the experiment.

Required reading before doing the experiment:

1. Geankoplis (2003): This book is required for basic distillation theory, Chapters 11.1 - 11.5
2. King (1971): This book is required to read about the definitions of weeping and flooding points
3. Gmehling and Onken (1977): This book is required for getting the data for Vapor-Liquid Equilibrium (VLE)
4. Perry and Green (2008): This book is required to get the data for weight percentage of ethanol as a function of density

For those who are interested in learning more about these topics, a more complete list of literature is suggested:

1) Theoretical Background Material
   a) General distillation theory:
      i) Geankoplis (2003)
      ii) Lydersen (1983)
      iii) Terjesen and Lydersen (1958)
      iv) Perry and Green (2008)
      v) McCabe and Smith (1976)
   b) Definitions of weeping and flooding points:
      i) Lydersen (1983),
      ii) King (1971),
      iii) Traybal (1980).
2) Data and Constants
   a) Equilibrium data: Gmehling and Onken (1977)
   b) Weight percentage of ethanol as a function of density: Perry and Green (2008), Handbook of Chemistry and Physics.
3) Similar Experiments in the Literature
   a) McCabe and Smith (1976),
   b) Perry and Green (2008),
   c) Lydersen (1983).

3. Brief Theory

3.1. Definitions

Distillation is a process of physically separating a mixture into two or more products that have different boiling points, by partial vaporization of a liquid mixture and/or by partial condensation of a gas mixture thereby rendering liquid phase richer in less volatile (with higher boiling point) component and the vapor phase is richer in more volatile (with lower boiling point) component.

Weeping point: refers to a condition when the vapor velocity through the perforations of a tray is so low that the liquid can't be held out of the perforations and instead of overflowing over the downcomer, the liquid "weeps" through the perforations/holes of a sieve tray. The vapor at this point loses contact with the liquid. This definition does not apply to packed columns.

Flooding point: any countercurrent separation device has a capacity limit. This limit in distillation is the known as flooding and is a result of high vapor velocity. At high vapor velocities, the pressure exerted by the vapor from below balances the gravity head of the liquid, as a result liquid starts building up in the column. This condition is reflected by sudden increase in the pressure drop.

Column efficiency: the overall column efficiency in a distillation column is defined as the ratio of total number of ideal equilibrium stages to the number of actual trays in the column. An ideal distillation stage is one in which the outgoing liquid and vapor phase establish an equilibrium with each other. High column efficiency indicates a thorough contact between gas and liquid.

In case of packed column, for the estimation of number of actual trays we use the height equivalent to a theoretical plate (HETP). HETP is the height of packing that does the same separation as a theoretical plate.

Examples:

A. Consider a **tray column** with the following characteristics:

   Number of theoretical stages (by McCabe Thiele diagram), N_t = 9

   Number of actual trays, N_a = 10

   Hence, the column efficiency is given by: $\frac{9-1}{10} = 80\%$

   Note that one stage is deducted from the numerator, as the reboiler is counted as one stage.

B. Consider a **packed column** with the following characteristics:

Number of theoretical stages (by McCabe Thiele diagram), N_t = 9

Height of packing = 1.3 m

Hence, the HETP is given by: $\frac{1.3}{9-1} = 0.1625$ m

Reflux: the portion of the vapor that is condensed and then returned to the distillation column. In this laboratory work most of time the distillation column will work with a total reflux, i.e., the entire vapor at the top of the column is condensed.

McCabe-Thiele Method: the simplest and perhaps most instructive method for analysis of binary distillation is the graphical approach presented by McCabe and Thiele in 1925. This method is based on the constant molar overflow assumption, which implies constant molar vapor flow rate and constant molar liquid flow rate leaving all stages in a section. Hence in each section of a distillation column, the operating line can be considered a straight line.

4. Equipment

The following figures highlight the main components of the equipment to be used. It is advised that you visit the lab and identify these parts when writing your working plan.
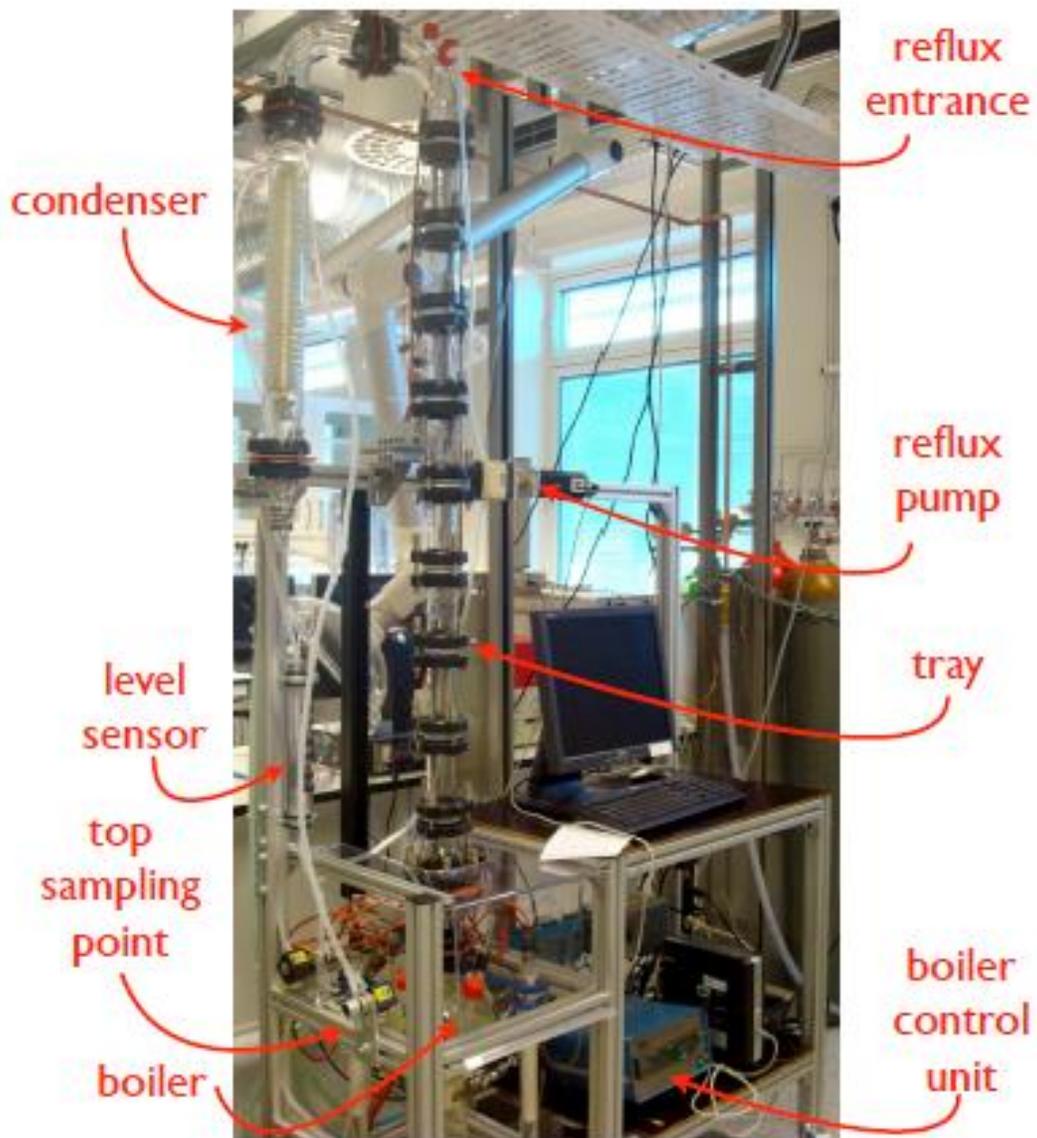

Sieved-tray column
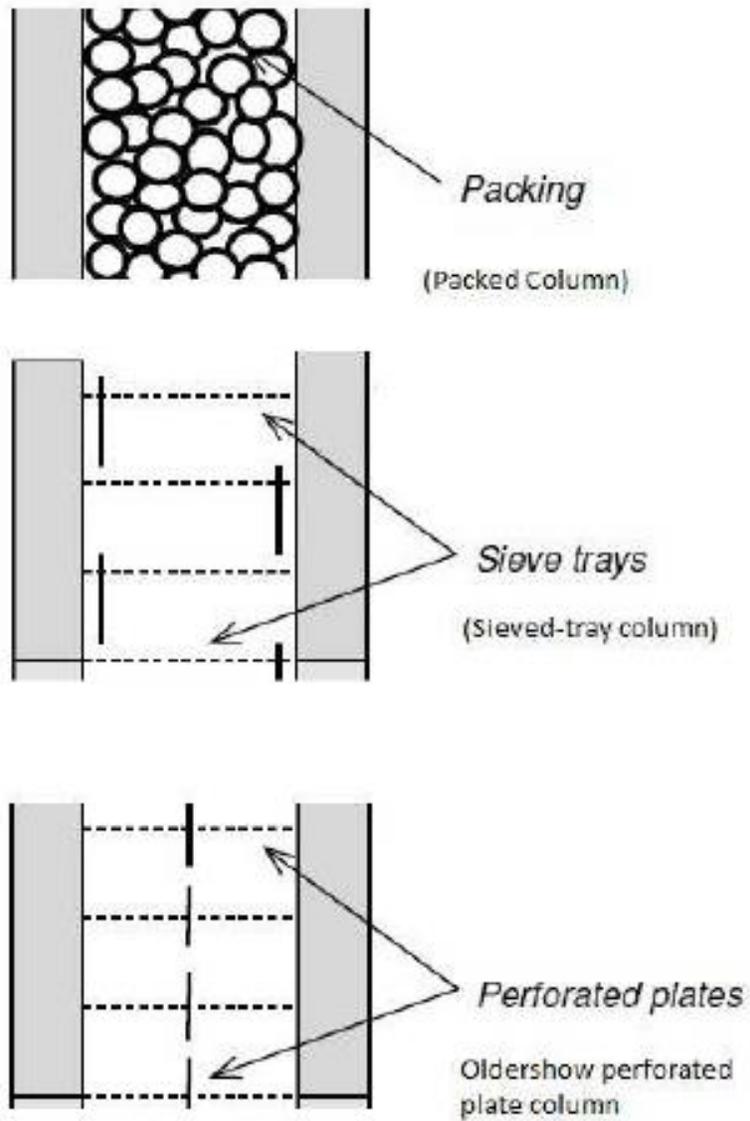
Figure 4.2: The different column tray types

5. 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 work plan, but do not include parts which are not of relevance to your exercise. Go to the lab and study the column. Locate every part marked on the apparatus description. Find the literature data you need.

5.1. Writing the Work Plan

The work plan should be delivered to your supervisor not later than 3 days before you make the experiment. A group will not be admitted to work on the experiment without having delivered the work plan.

The work plan should include information on:

- ✓ How to do the experiment;
- ✓ How to calculate results from the raw data (See Appendix A).

Your raw data will consist of composition of product (from analysis of your samples), pump flow rate at steady state, temperature of the column and time. You will have to calculate:

- ✓ Composition of top and bottom products
- ✓ Number of ideal trays needed
- ✓ Vapor velocity

A part of the work plan will be to do the Assignment exercise shown at the end of this document in the Appendix A.

6. Experiment

You will run two different exercises:

EXERCISE 1: time required for reaching Steady State condition

EXERCISE 2: Efficiency vs. Vapor velocity

Generally, the experiment takes from 6 to 8 hours.

6.1. Column Startup
1.   Turn on the PC
2.   Turn on the cooling water
3.   Make sure that that the bottom and distillate tap lines are closed
4.   Charge the column with 5800 ml a mixture of water and ethanol as follow:
     - ✓ Rasching-ring column: $x_{ethanol}=0.1$
     - ✓ Sieve-tray column: $x_{ethanol}=0.12$
     - ✓ Oldershaw perforated plate: $x_{ethanol}=0.11$
5.   Set the column to total reflux
     - ✓ Using the control box, set T = 105°C
     - ✓ When the top temperature substantially changes, or the pressure difference over the column rises quickly, set the boiler's power duty as follows: Raschig-ring column 50%, Sieve-tray column 30%, Oldershaw perforated plate 50%.
6.   Wait until the vapor stream starts condensing: let this be "zero" time.

In case of **flooding** conditions, switch off the heater immediately.

In any **emergency** situation, turn off the heater but leave the cooling water on.

6.2. EXERCISE 1: Time Required for Reaching Steady State Condition
The purpose of this exercise is to calculate how long the column takes to reach steady state conditions:

1.   Set the reboiler power to a desired value (say 50% and 30% for sieve tray) and wait until the column starts to give condensate.
2.   Take samples from the top every 5 minutes within 1 hour, so in the end of this experiment you will have 12 top-samples.

3. After you take the sample number 12, take one sample from the bottom of the column, because you will need its composition for the next exercise. Note down the temperature and pump flow rate at this point to utilize it for next exercise.
4. Analyze all samples and find out how long the column takes to reach steady state: when the composition of the top-samples is constant from one sample to another, you can consider that you reached the steady-state.

6.3. EXERCISE 2: Efficiency vs. Vapor velocity

The goal now is to find how the column efficiency (or the theoretical number of trays from McCabe-Thiele) varies as a function of the vapor velocity. Run the column with total reflux for five different boiler powers, yielding different vapor velocity.

1) Set a desired power (say, 40%) in the reboiler.
2) Wait until the column reaches the steady-state. You can assume that the column reaches steady state conditions in the same time as in the exercise 1. For example, if in the exercise 1 you got constant composition of the top-samples in 40 minutes, you can consider that in exercise 2, the column reaches the steady-state also in around 40 minutes.
3) Take samples from the top and from the bottom, 20 ml each.
4) Write down the reflux rate at the time of sampling.
5) Set a new power in the reboiler. In total, you should get 5 different velocities using 5 different boiler power settings. (We will use 4 different power setting including the steady state experiment to reduce total experiment time).

Analyze the samples and calculate the efficiency of the column and the theoretical number of stages using McCabe-Thiele

To calculate the vapor velocity, use the following equation:

$$v = \frac{\dot{V}_{gas}}{A} \ [m/s]$$

where: $\dot{V}_{gas}$ is the volume rate of the gas, [m^3/s]. It can be calculated from the equation of state of the ideal gas; A is the cross-sectional area of the column, [m^2]. You can calculate it using diameter D of the column, given below:

- ✓ Oldershaw column: D = 0.07 m,
- ✓ Sieve-Tray column: D = 0.05 m,
- ✓ Packed column: D = 0.05 m.

Identify flooding and weeping points at the end. Record the considering the values of pressure drop and vapor velocity at flooding and weeping point. Write down the power of the boiler at the weeping and flooding points. The weeping point is not applied to the packed column.

**Due to corona situation, we will skip determination of flooding and weeping points.

6.4.Column Shutdown
1.   After taking the last needed sample, turn off the heater.
2.   Let the column with contents cool down.
3.   Empty the column and turn off the cooling water.
4.   Turn off the rest of the equipment.

6.5. Data Processing
When you have finished in the lab, you have to analyze the data. If you are using pycnometers to determine the sample densities, then Literature data will be needed to see how the density varies with the composition for the water-ethanol system. With that, you can derive your sample's composition. From the GC analysis, you will get the volumetric composition of your samples directly. A detailed description on how to proceed with the GC analysis is given in Appendix B.

Then you must draw McCabe-Thiele diagrams to find the number of ideal trays. You may use a computer program to draw the McCabe-Thiele diagrams. If you program it yourself (for example, in Matlab) you should also include the program code as an appendix in your report. However, at least one diagram should be drawn by hand. Use mm-scale paper in A3 format.

Remember to draw the equilibrium line as accurately as possible.

6.6. Lab Journal
While conducting experiments you will have to maintain a lab journal. In this journal you will write all your "raw" measurements, that is, before any calculation. This will help you to spot errors, and provides a data backup. It is wise to make notes on anything worth noting, as "Flooding conditions reached", "Error in valve operation, run must be repeated", "Unknown failure, all ethanol seems gone", "Bad measurement, possible cause is … ".

It is recommended to prepare the journal before starting the lab, with tables ready to be filled in and memos. This will help you remembering what measurements to take and when. You would not be the first students that get halfway through the day only to realize they did not write some important data, and must start from scratch. Make sure to account for all the data you have to collect.

7. Report

7.1. What Your Report Should Contain

For general guidelines in writing the report, refer to the lab course's home page. Below are given some tips for the distillation experiment, as follows:

• Introduction part, where you should explain what the goals of the experiment.

• Theoretical part, where you should describe the main information about the distillation process in a concise way.

• Explain in the theoretical part how the operation line should look in the McCabe-Thiele diagram at total reflux.

• Describe the procedure clearly, including all relevant details.

• Include the description how you made exercises 1 and 2, with calculations and plots:

  ✓ for exercise 1 – a plot of top-samples composition vs. time (12 points);
  ✓ for each power duty – a plot of total column efficiency (HETP) vs. vapor velocity;
  ✓ McCabe-Thiele diagrams of mole fraction in liquid phase vs. mole fraction in gas phase, separate for each power duty (total: 5 plots)
  ✓ One McCabe-Thiele diagram should be made by hand, on the mm-scale paper in A3 format.

• Write the conclusion and obtained results of both experiments, including the flooding and weeping points, discuss the time required to reach the steady-state condition, describe the tendency of the change of the vapor velocity versus heater's power and column efficiency.

• In a separate "Discussion" part give a description on the possible error sources in your experiment. In this section calculations are not required. If your results do not relate to the theory, try to come up with a reasonable explanation (also you can discuss these things with your supervisor).

• A photo copy of the lab journal shall be handed in as an appendix to the report. This will make evaluation of your work routines easier and will also make it simpler to figure out the reason when some results seem strange.

• You'll send a first version of the report to your instructor, who will correct it and give it back to you with comments to help you improve your report. The final version of the report will then be evaluated.

Note: For typesetting guidelines, see Beccari (1997).

7.2. Common Mistakes (Read carefully and take them into account)

• Work plan not delivered in time (at least 3 days before the experiment itself). Group will not be admitted to work on experiment without delivered work plan.

• Incomplete listing of results in abstract or conversely too long-winded abstract.

• Too much theory, covering parts that are not necessary.

• Incomplete description of the methods: they must be clear and thorough enough that another group might run the same experiments you did only by reading that.

• Results presented with the wrong parameters (e.g. plots of efficiency vs. heater's power instead of vapor velocity).

• Not mentioning units of calculated or measured values. Write both in the Work Plan and in your Report.

• Wrong units used in plots and calculations (wrong ideal gas constant's unit or wrong temperature unit, and so on).

• Not mentioning the name of axes in the plots, including the units of plotted values. You should also include the mole fraction of top and bottom composition on the McCabe-Thiele diagrams.

• Aberration in the results is possible, try to explain them in your report based on your experimental procedure.

• Incomplete appendices: remember to attach all relevant material, especially the lab journal with raw data and any computer scripts/programs you have written.

• Please avoid making this list longer for next year's students!!

Bibliography

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McCabe, W. L. and J. C. Smith (1976). Unit Operations of Chemical Engineering (3ed.). McGraw-Hill, USA.

Perry, R. H. and D. W. Green (2008). Perry's Chemical Engineers' Handbook (8 ed.). McGraw-Hill, USA.

Terjesen and Lydersen (1958). Kjemiteknikk II. Trondheim.

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A. Assignment

To make sure you understand how to calculate your results, you should make an example calculation, and deliver it in your work plan.

You have the following measurements:

• Weight of empty pycnometer 1 - 11.98170 g.

• Weight of empty pycnometer 2 - 11.77385 g.

• Weight of pycnometer 1 with water - 16.95955 g.

• Weight of pycnometer 2 with water - 16.75745 g.

• Weight of pycnometer 1 with distillate - 16.0903 g.

• Weight of pycnometer 2 with bottom product - 16.6962 g.

From the GC analysis, you have the following measurements:

Distillate composition: $x_{ethanol}$ = 0.744

Bottom composition: $x_{ethanol}$ = 0.033

i. Show that both methods lead to approximately the same results.

ii. Calculate the vapor velocity in m/s, knowing that the column was operated under total reflux, and the reflux rate was 53 ml/min. All measurements were done at 25 °C and 1 bar.

iii. Estimate of the number of ideal stages (McCabe-Thiele) and attach the plot of this diagram to your work plan.

Beside these calculations, it is necessary to include some theory about Gas Chromatography (we use a FID detector). Explain how we can calculate the ethanol concentration with GC (the quantitative analysis by using an internal standard).

B. Composition Measurements

The composition measurements are performed with **Gas Chromatography (GC)**. The following sections include the information needed for the laboratory exercise. More information about how GC works can be obtained from pertinent literature.

Sampling

The needed equipment is shown in Figure 7.1. To determine the ethanol content, the ethanol sample must be mixed in a fixed ratio with a standard component (in this case, isopropanol). Pour isopropanol into a small glass container (you will probably not need more than 5 ml).
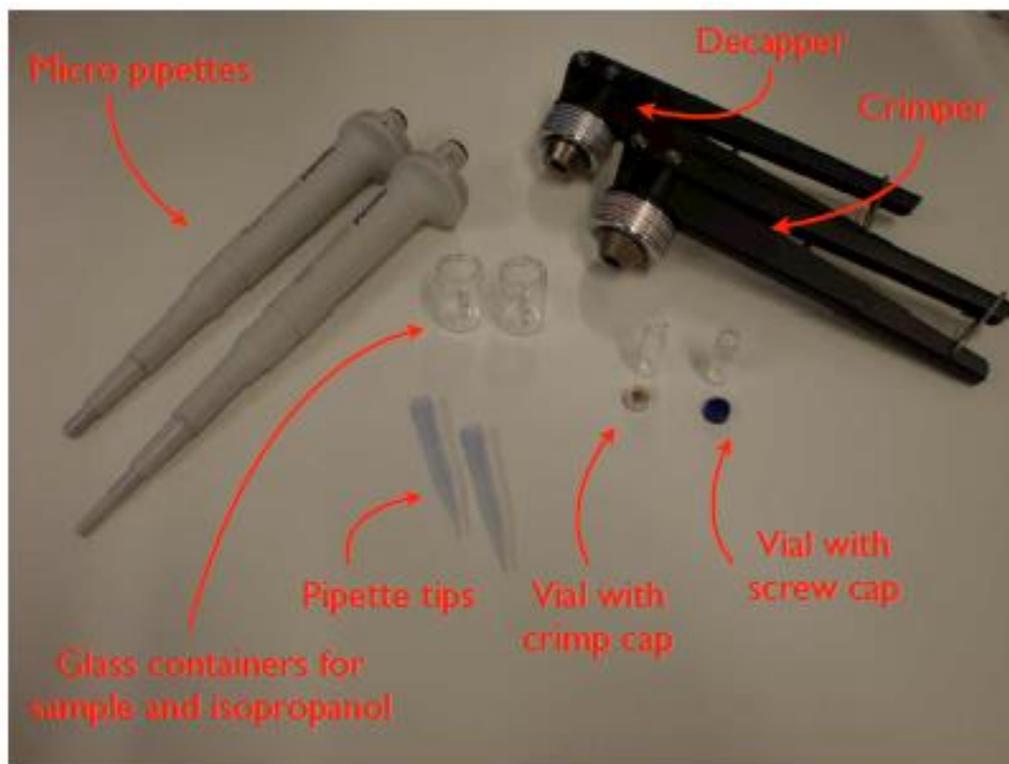
Figure B.1: Equipment for sample preparation

To prepare a sample for the GC, mix the following volumes into a glass vial by using the micropipettes:

   • 500 μL of ethanol sample

   • 250 μL of isopropanol

When pipetting, use the reversed pipetting technique (see the subsequent section). Seal the vial with a cap. If the vial has a screw top use screw caps. If the vial has a crimp top, use the aluminum cap and crimp it on using the crimper. Remember to clearly mark the vial for later identification. Make sure to mix the

vial very well with the provided table-top shaker. Do this the same way for all samples. Here lies big potential for errors, since the two fluids to not readily mix.

Using Micropipettes

The micropipettes use air displacement to dispense the liquid. The pipettes have an adjustable dispensing volume, the quantity is marked on the top button (plunger). The plunger has two steps (stops) when pushed down:

First stop: used for aspirating and dispensing liquid into and out of the tip.

Second stop: used to blow-out the remaining liquid from the tip.

Reversed pipetting technique

Below is a description of a different kind of micropipette **NOT** used in this exercise, described here to illustrate a second technique with slightly different micropipettes, useful for viscous samples, the so called

The pipetting will be done in 5 steps:

1. Wetting

2. Aspiration

3. Dispensing

4. Blow-out

5. Tip-release

Wetting and Aspiration First submerge the tip about 3 mm into the liquid, push the plunger down to the second stop, release the plunger slowly, push it down to the second stop again, and slowly release. The first aspiration and displacement step is done to wet the tip and increase the accuracy.

Dispensing, Blow-out, and Tip Release Hold the tip at an angle against the glass wall, and dispense the liquid into the glass vial by pushing the plunger down to the first stop. Hold the plunger at the first stop, and lift the tip out of the vial. Blow out the remaining liquid into a glass container marked for waste by pushing the plunger down to the second stop. Release the tip into the trash by pushing the plunger down to the third stop.

Sample analysis in GC

A. Turn on the H2 supply for GC and check gas flow to GC.

B. Preparing to run the samples
1. Place the prepared samples into the auto sampler tray.
2. Take note of the position of the respective samples.
3. Start the **Control Panel** by double clicking icon in the desktop.
4. In **Control Panel** select **Launch Ethanol** project. This will open **Acquisition** window.
5. Select **Method** from toolbar of **Acquisition** window.
6. Open **Ethanol_Quadrex_50_iso.amx** method and download (send) it to the GC.

C. Running a batch of samples as sequence
1. In **Acquisition** window, select **Sequence** in the toolbar**.** A table appears where you can enter the sample information. If you cannot see the table, make sure that **Table** is selected under **Injections**.
2. You can add more samples by clicking in table symbol.
3. Type or select following sample information.
   a. **Vial**: This is the samples' position on the auto sampler tray. It is important that you enter the correct position. Else, you will analyze the wrong sample. If the vial position is 1 enter 101, position 2 as 102, position 12 as 112 and so on.
   b. **Sample ID***:* Select **Sample**
   c. **Run Type, Level, Volume, Injection source,** and **Sample amount***: Skip these boxes*
   d. **Acq. Method***:* Select and browse **Ethanol_Quadrex_50_iso.amx**
   e. **Proc. Method**: Select and browse **Ethanol_QuadrexCalibrated3.pmx**
   f. **Sample name**, **Data file***,* and **Label***:* Type same unique id to all three fields
4. For each sequence provide unique file name to save the result. YourGroupID_Date_DistiilationRigID (B01-19-8-ST4)
5. For *Result Path* select and browse folder named after corresponding lab assistant.
6. **Run** the sequence. The GC will analyze the samples in ascending order. Each sample takes roughly 7 minutes. You can see the progress of the acquisition if you select **Run Queue**. You can let the sample analysis run overnight and copy the result next day.

D. Data analysis
1. Once all sample acquisition and analysis are complete (probably next day), select **Run Queue** and right click on your sequence. Choose **Review Selected Run In Data Analysis** to open **Data Analysis** window.
2. Click **Reprocess All** in the **Data Analysis** window toolbar.
3. You can record the concentration and area of the ethanol signal and isopropanol signal area under **Injection Results** area. You have to select individual sample in the **Data Processing** area to see the results for individual samples.

E. Turn off the H2 Gas supply after the GC use.

Vial Disposal

Do not throw the vials away, the assistant will deal with this.