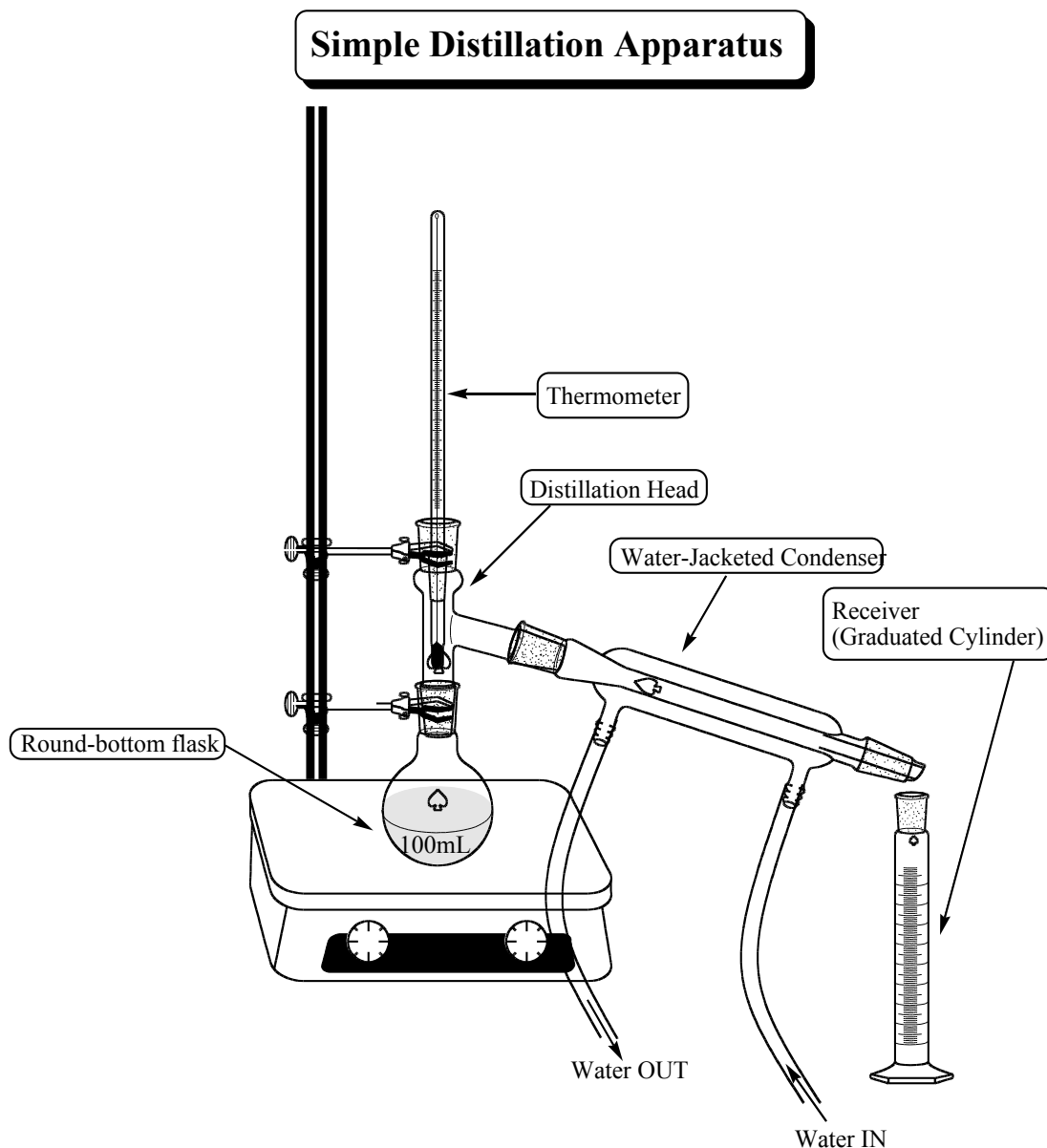


Organic Chemistry 211 Laboratory Simple Distillation



In this experiment simple distillation will be performed in order to separate ethyl acetate from butyl acetate. Assemble the apparatus as shown in the diagram. Add 20 mL of the mixture which contains unknown amounts of ethyl acetate and butyl acetate and a boiling stone (one per person) to the distillation flask. Begin the heating with the heating mantle (instead of hot plate indicated in the diagram). Since butyl acetate is relatively a high-boiling (low vapor pressure) substance, it may be appropriate to initiate the heating on the maximum voltage setting (turn all the way on). Watch the distillation flask to see that it does not boil so vigorously that the liquid runs over the top. Distillation should be dropwise. Record the temperature on the thermometer for every 0.5 mL of the distillate received. Later, make a graph (in your laboratory notebook) of temperature

versus the volume of the distillate. Continue distillation for two or three more data points on your graph after a temperature plateau is reached close to the boiling point of butyl acetate (for graphing purpose). **Important:** Do not allow the round-bottom distilling flask to go dry.

For later GC analysis: before and during the distillation, you will collect 4 fractions according to the chart below and store each of them in a capped vial for the next experiment (Experiment 6: GC). You need not collect an exact volume. Record the exact temperature range and volume for each fraction you collected. Parafilm tightly each vial for storage.

| | Original mixture* | 1 st fraction | 2 th fraction | 3 th fraction |
|------------------------|-------------------|--------------------------|--------------------------|--------------------------|
| Temperature range (°C) | Room temp. | 76–80 | 88–92 | >110 |
| Volume (drops or mL) | ~2 mL | ~1 mL | ~1 mL | ~1 mL |

* It should be collected from your original unknown solution that you obtained from your instructor and should not be counted toward the 20 mL of the beginning solution for distillation.

Safety:

- The whole system needs to be open for gas to escape. NEVER HEAT A CLOSED SYSTEM.
- Do not turn on the heat until you get the instructor's initial.

For the report:

- You should write one combined lab report for both Simple Distillation and Fractional Distillation experiments.
- Two graphs are required: (do graphs horizontally on paper)
(1) One graph of Temp (y) vs. Volume (x) (for both simple and fractional distillations on the same graph; i.e. two sets of data plotted on the same graph);
(2) One graph of Time (y) vs. Volume (x) (for both simple and fractional distillations on the same graph). Remember to label axes.
- There are more requirements related to Fractional Distillation. See next lab manual.