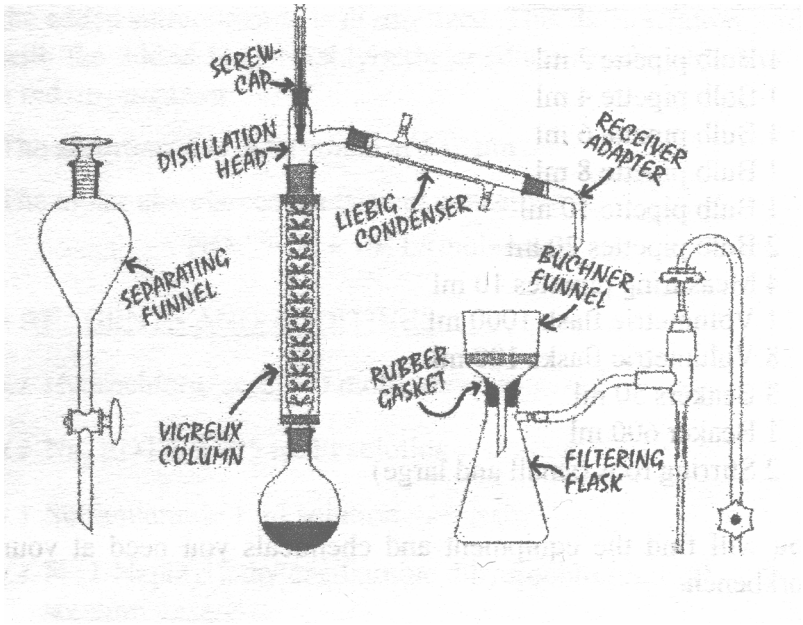
Synthesis of l-Butylacetate

Aim
The synthesis of l-butylacetate on the basis of l-butanol and acetic anhydride

Equipment
1 Round-bottom flask 250 ml
1 Measuring pipette 5 ml
1 Multiple adapter (two necks *Y-piece 3x*)
1 Dropping funnel 100 ml
1 Liebig condenser
1 Heating mantel 250 ml
1 Separating funnel 500 ml
1 Erlenmeyer flask100 ml
1 Distillation heads
1 Vigreux- column 300 mm
1 Screw-cap with cone
1 Distillation thermometer
1 Receiver adapter
1 Filtering flask (Erlenmeyer shape) 500 ml
1 Buchner funnel 90 mm
1 Beaker 600 ml
1 Funnel 70 ml
Stirring-rod / clips / robber tubing / pH paper / suberit-ring / pan / robber gasket
Refractometer



Chemicals
l-Butanol $C_4H_{10}O$
Concentrated sulphuric acid H_2SO_4
Acetic anhydride. $C_4H_3O_3$
Sodium chloride NaCl

Sodium hydrogen carbonate $NaHCO_3$
Anhydrous magnesium sulphate $MgSO_4$
Acetone C_3H_6O

Procedure

Do not start before making sure that all the glassware used in the synthesis reaction is dry.

Place 67 mL l-butanol into the round-bottom flask. Add 0.7mL concentrated sulphuric acid with a measuring pipette and mix immediately, otherwise carbonisation may occur. Concentrated sulphuric acid is a strong hygroscopic substance. Provide the flask with a multiple adapter, dropping funnel and Liebig condenser. Place 83 mL acetic anhydride into the dropping funnel.

Heat the mixture to boiling point and remove the heating mantel. Add the acetic anhydride drop by drop. The speed at which the drops are added should be such that the mixture remains at boiling point.

Should the temperature of the reaction mixture drop below boiling point, stop the additions and before doing anything else re-heat until it boils again. Resume the additions again after removing the heating mantel. After having added all the acetic anhydride, allow the reaction mixture to boil for another 5 minutes.

Cool the flask in ice-water and pour the cooled reaction mixture into 100 ml water to which 5g sodium chloride has been added. Separate the layers in a separation funnel whereby the organic layer remains in the separation funnel. Add 100 mL water, shake well and separate again.

Now add 100 mL solution of $NaHCO_3$ (5g per 100 mL) to the ester. This causes the formation of carbon dioxide, therefore be careful when shaking.

Should there still be an acidic reaction from the water layer, add extra $NaHCO_3$ solution. Stop the additions if there is no more acidic reaction from the water layer. Finally, wash with 30 mL water.

Dry the l-butylacetate on anhydrous magnesium sulphate. Bring the ester in an Erlenmeyer flask, add a spoonful of magnesium sulphate and mix around.

Repeat the addition until the added drying agent remains flaky. Leave it to dry for at least 30 min. Filter off the drying agent and distil the l-butylacetate. Use a Vigreux column and a heating mantel for this. Should the boiling point fluctuate, collect the distillation in fractions. A good separation is only obtained if distillation is slow (approx. one drop per second).

Do not boil until completely dry. Leave approx. 2 mL liquid in the flask.

Determine the refractive index and the mass of every fraction.
Make a 10% 1-butylacetate solution in acetone.
Calculate the yield percentage.

Waste-disposal

Collect water layers as waste in the storage tank
Dispose the filter with magnesium sulphate hydrate in the waste bin