Practical procedures:

Making a standard solution:

Stage 1: transfers known mass of solid

a) Weigh the sample bottle containing the solid on a (2 dp) balance

b) Transfer to beaker* and reweigh sample bottle

c) Record the difference in mass

Stage 2: Dissolves in water

a) Add distilled / deionised water

b) Stir (with a glass rod) or swirl

c) Until all solid has dissolved

Stage 3: Transfer, washing and agitation

a) Transfer to volumetric / graduated flask. Allow if a clear description/diagram given eg long necked flask with 250 cm^3 mark

b) With washings

c) Make up to 250 cm^3 / mark with water

d) Shakes/inverts/mixes

Recrystallisation:

Dissolve the product in the **minimum** volume of Hot water / solvent

Allow the solution to cool and allow crystals to form.

Filter off the pure product under reduced pressure / using a Buchner funnel and side arm flask

The crystals were compressed in the funnel

A little cold water was poured through the crystals

Melting point determination

Measure the melting point using melting point apparatus

Sharp melting point / melting point matches data source value

Experiment to determine the solubility:

Take a known volume of the saturated solution

Evaporate the filtrate to dryness

Weigh the residue

Measuring temperature drop (eg in an enthalpy calculation):

Place thermometer in an insulated cup.

Start a clock

Record the temperature every subsequent minute for 3 minutes

Add the ***salt / solution*** at minute 4

Record the temperature every subsequent minute from minute 5

Plot a graph of temperature vs time

Extrapolate back to time of mixing = 4 and determine the temperature

Measuring temperature rise (eg in an enthalpy calculation):

Start a clock when ***salt*** is added to water

Start a clock when ***salt*** is added to water

Record the temperature every subsequent minute for about 5 minutes

Plot a graph of temperature vs time

Extrapolate back to time of mixing = 0 and determine the temperature

Preparing a pure sample of AgBr from a mixture of NaCl and NaBr

Stage 1*: formation of precipitates*

- *Add silver nitrate*
- *to form precipitates of AgCl and AgBr*
- $AgNO_3 + NaCl \rightarrow AgCl + NaNO_3$
- $AgNO_3 + NaBr \rightarrow AgBr + NaNO_3$

Stage 2*: selective dissolving of AgCl*

- *Add excess of dilute ammonia to the mixture of precipitates*
- *the silver chloride precipitate dissolves*
- $AgCl + 2NH_3 \rightarrow Ag(NH_3)_2^+ + Cl^-$

Stage 3*: separation and purification of AgBr*

- *Filter off the remaining silver bromide precipitate*
- *Wash to remove soluble compounds*
- *Dry to remove water*

Producing a pH curve

- Measure pH of 25cm^3 of the acid
- Add alkali in 1cm^3 portions
- Stir mixture
- Measure pH (after each addition)
- Repeat until 24cm^3
- Add 0.5cm^3 near endpoint 24 – 26cm^3
- Add alkali in 1cm^3 portions until in excess

Thin layer chromatography:

Set up:

- Wearing plastic gloves to hold a TLC plate, draw a pencil line 1.5 cm from the bottom of the plate.
- Use a capillary tube to apply a very small drop of the solution of amino acids to the mid-point of the pencil line.
- Allow the spot to dry completely.
- Add the developing solvent to a depth of not more than 1 cm.
- Place your TLC plate in the developing tank.
- Allow the developing solvent to rise up the plate to the top.
- Remove the plate and quickly mark the position of the solvent front with a pencil.
- Allow the plate to dry in a fume cupboard.

Developing:

- Spray with developing agent
- Measure distances from initial pencil line to the spots (x)
- Measure distance from initial pencil line to solvent front line (y)
- R_f value = x / y
- Amino acids have different polarities
- Therefore, have different retention on the stationary phase or different solubility in the developing solvent

How would you determine a suitable solvent for recrystalisation:

Place a small amount of pure crystals in a test tube.

Add small volume (0.5–1.0 cm3) of possible solvent.

Shake/stir.

If pure crystals dissolve then solvent unsuitable.

If the pure crystals are insoluble in cold solvent then heat in a water bath.

If pure crystals do not dissolve = unsuitable.

If pure crystals dissolve partially add more solvent.

If/when completely dissolved place in ice water bath.

If crystals form = suitable.

Effectiveness determined by measuring melting point.

Purity indicated by melting point being sharp and close to data book melting point

Colorimetry to measure rates:

Setting up

Calibrate the colorimeter with known concentrations

Plot a graph of absorbance against concentration

Carrying out

Fill a cuvette with the solution at regular time intervals during the reaction

Read the absorbance from the colorimeter

Read from the graph to find the concentration of the solution

Plot a graph of concentration against time

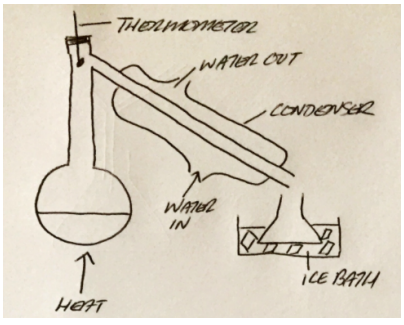
If it is a straight line = 0 order

Constant half lives = 1st order

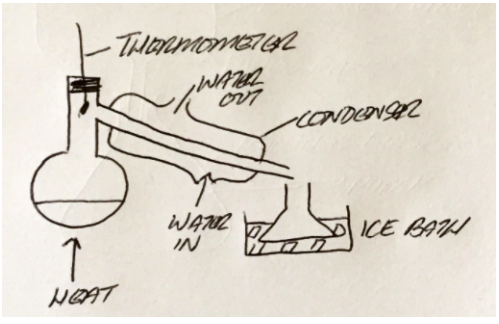
Increasing half lives = 2nd order

Practical techniques:

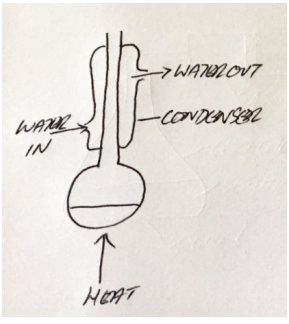
Fractional distillation



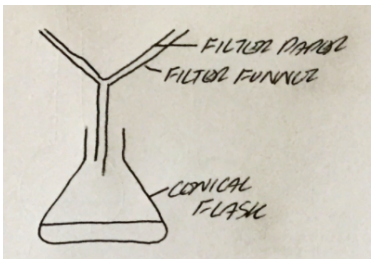
Distillation



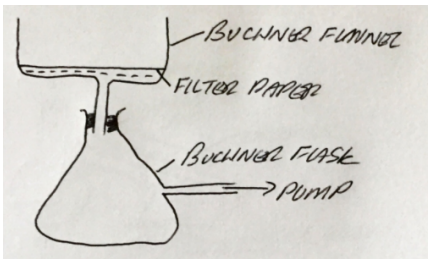
Reflux



Filtration



Filtration under reduced pressure



Gas collection