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Balancing and weighing

1. Switch on the balance and allow sufficient time for the device to achieve a thermal equilibrium.
2. Check for correct levelling.
3. Never weigh the material directly onto the balance. Use a suitable container or weighing boat.
4. Remove the container and then fill it with a substance to be weighed carefully.
5. To maintain the balance, keep it switched on during long activities, keep it clean and regularly check for **drifting** of the measurements.

Volumetric Glassware

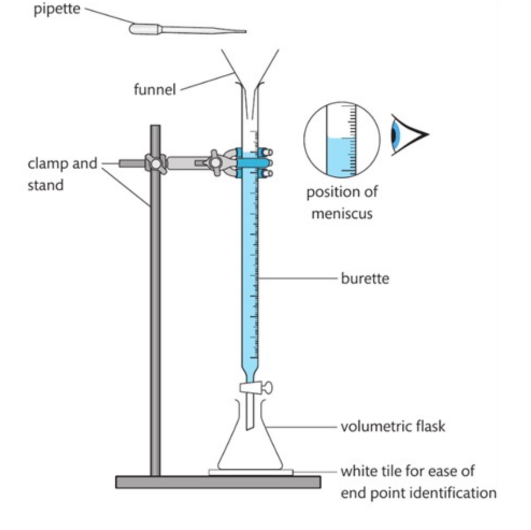
Name	Sizes	Uses	Limitations
Pipette (non-graduated)	5.0 ml to 50 ml	Taking volumes of solutions when accuracy of a single volume is needed, normally to add small volumes dropwise	Can be very fragile when handled and need some practice with pipette fillers to produce accurate volumes
Pipette (graduated)	Graduated between 1 ml and 1/10th ml	Taking volumes of solutions of specific random volumes	Practice is needed with pipette fillers to produce accurate volumes
Burette	25 ml and 50 ml	Measuring accurate volumes with a graduation of 0.1 ml allowing a meniscus to be observed	The tap can become stiff to use so silicon grease is needed for lubrication. The tip can become clogged or fill with air bubbles
Conical (Erlenmeyer) flask	50 ml to 500 ml	Mixing and swirling volumes of solutions with little risk of spillage	No real limitations in its use
Volumetric flask	10 ml to 2000 ml	Measuring, mixing and making up volumes to a given mark with a high degree of accuracy	Glass can expand as a result of chemical reactions producing heat
Beaker	Varying sizes, generally 10 ml to 1000 ml	Measuring non-accurate volumes of solutions, general usage, waste etc with a graduation to +/− 10%	No real limitations as fit for general purpose, but hot alkalis can damage the surface

Measuring Volume

$$\text{Volume } (\mathbf{V}) = \frac{\text{mass } (m)(\text{g})}{\text{density } (d)(\text{g/cm}^3)}$$

Titration

Quantitative analysis: Practical experiment producing numerical results.
Titration: the process of determining the concentration of the unknown solution using a solution of known concentration.



Titration Calculations

In this experiment, 20.0 cm^3 of 1.0 mol dm^{-3} NaOH neutralised 25.5 cm^3 of H_2SO_4 whose concentration was believed to be 0.4 mol dm^{-3}.

Step 1 Balance the chemical equation:

$$2NaOH(aq) + H_2SO_4(aq) \longrightarrow Na_2SO_4(aq) + 2H_2O(l)$$

2 moles of NaOH are needed to neutralise 1 mole of H_2SO_4. The **stoichiometry** is 2:1.

Step 2 Calculate the number of moles of NaOH:

$$20.0\ cm^3 = \frac{20}{1000} = 2 \times 10^{-2}\ dm^3 = \mathbf{0.02\ mol}$$

Step 3 Calculate how much H_2SO_4 has been neutralised (note 2:1 ratio)

$$\tfrac{1}{2} \text{ the amount of NaOH} = \tfrac{1}{2} \times 0.02 \text{ mol} = \mathbf{0.01\ mol}$$

Step 4 Change the volume of H_2SO_4 to dm^3 (1000 cm^3 = 1 dm^3):

$$25.5\ cm^3 = 25.5/1000 = \mathbf{2.55 \times 10^{-2}\ dm^3}$$

Step 5 The concentration of H_2SO_4 is:

$$\frac{0.01}{2.55} \times 10^{-2}\ mol/dm^{-3} = \mathbf{0.392}\ mol/dm^3$$

Steps in the investigation	Pay particular attention to...	Think about this...
1. Set up the apparatus as shown in Figure 2.3.	Make sure that the burette is absolutely vertical from all directions.	If the burette is at an angle, the measurement of acid or alkalis will be incorrect.
2. Ensure that the tap is open. Rinse the burette with distilled water and then with a small quantity of standard solution.	Fully rinse the burette with the standard solution to be sure that only this is recorded flowing from the burette afterwards.	
3. Close the tap and fill the burette.	Fill the burette above the graduation mark to have sufficient solution to release in order to set the first graduation level.	If you make a mistake, simply add extra standard solution and try again.
4. Release the titre solution slowly until the meniscus is on the first graduation level.	Make sure that the base of the curve sits on the graduation mark.	
5. If air bubbles are present, repeat steps 2 to 4, to ensure that no air bubbles interfere with the readings during titration.	Ensure that no air bubbles interfere with the readings during the titration.	An air bubble could put your eventual results out by a number of cm^3, having an important effect on results.
6. Transfer a known volume of unknown concentration solution to a conical flask.	Operate the pipette carefully and according to good practice.	
7. Add three drops of indicator (e.g. phenolphthalein) to the solution in the conical flask.	Be sure that the colour of the solution is clear enough to enhance the point at which the change in colour first appears.	
8. Slowly titrate – opening the tap and gently swirling the conical flask.	Observe the solution carefully to identify the first signs of colour change.	The swirling motion will ensure that the end point is a true neutralisation mark.
9. At the first indication of colour change, reduce the flow rate. Observe the **end point** as the complete colour change and record the level on the burette.	Your results table should show up to three separate volume readings for the same titration. Once you have two values the same (concordant), use this in your calculation. Other values can be discarded.	

Colourimetry

Steps in the investigation	Pay particular attention to...	Think about this...
1. Switch on the colorimeter and allow time for the equipment to stabilise.		
2. Choose the appropriate wavelength settings for the device. The 1.0 M $CuSO_4$ solution used in this experiment has a light blue colour.	You may need to access reference material for this, although the settings are usually displayed on the device.	A high concentration of the coloured solution absorbs more light (and transmits less) than a solution of lower concentration.
3. Make up serial dilutions from the 1.0 M $CuSO_4$ stock solution. (Put 10 ml of 1.0 M stock in a test tube and add 10 ml of distilled water. This is now 20 ml of 0.5 M solution. Repeat for further dilutions.)	You will need to ensure that your measurements are accurate so that your graph results will follow the expected pattern.	
4. Place a reference into the **cuvette**, press and release the reference button 'R'. The display should show 0.00 Absorbance (Abs) or 100%T.	Check that the display is correct. If not repeat step 4.	
5. Remove the reference cuvette and replace it with a sample solution cuvette. Wipe the cuvette on all sides with a lens tissue to clear fingerprints or spills from the side of the cuvette to avoid errors.	Wipe the cuvette on all sides with a lens tissue to clear away fingerprints or spills from the side of the cuvette.	If the light does not pass through a clean cuvette, the effect is to alter the concentration value of the sample.
6. Press and release the test (T) button. The result will be displayed in Absorbance or percentage Transmission units.		
7. Repeat the previous two steps with your remaining dilutions. If the process takes more than approximately 15 minutes, then you may need to 're-reference' with the reference solution to avoid some possible instrument error (drift).	If the process takes you more than 15 minutes to complete, you may need to re-reference the device with the reference solution.	Possible instrument error may occur due to drift.
8. Plot a graph of Absorbance (*y*-axis) against Concentration (*x*-axis).		

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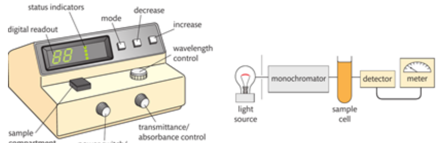
Colourimetry

Colorimetry is a technique which measures the intensity of **colour**. The level of colour in a solution can be used to provide a value of its concentration since the intensity of the colour from a chemical reaction is proportional to the concentration of the substance test-ed.

Spectroscopy

This method of chemical analysis is used to determine the purity/ concentration of a chemical substance. Principle relies on absorption or scatter of **electromagnetic radiation.**



The Beer-Lambert Law

Provides a mathematical relationship between the absorbance of light and the concentration of a substance. The more light absorbed by a substance, the greater its concentration.

$$A = elc$$

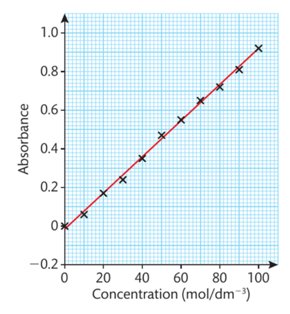
where:

A – absorbance

e – constant of proportionality or **molar absorptivity** (how well the substance absorbs light and can have the units cm^{-1} M^{-1})

l – the path length (usually the width of the cuvette)

c – concentration of solution ($mol\ dm^{-3}$).

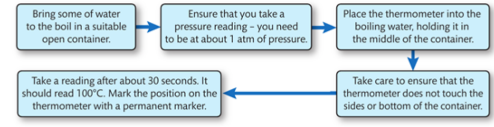


Thermometers

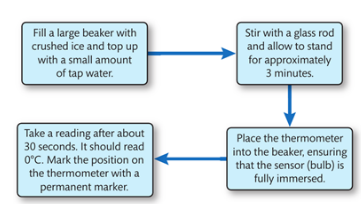
Thermometer type	Principle of operation	Main applications
Liquid-filled	**Alcohol in glass** – a specified amount of alcohol is coloured and placed in a pressurised glass tube. Alcohol expands with rise in temperature, although it is slow to respond. It only measures to +78°C. The alcohol is not harmful if the glass breaks.	Clinical usage in hospitals, industrial complexes, schools and colleges and for domestic use.
	Mercury in glass – a small amount of mercury is sealed in a glass tube. The metal responds quickly to temperature changes. There are two basic types: a straight tube and another which has a slight bend in the bottom to prevent the liquid from dropping quickly when removed from the mouth. Mercury is poisonous. It is difficult to read the display.	Clinical usage in hospitals. General industrial uses for liquid temperature measurement, schools and colleges, practical scientific investigation.
Electronic	**Thermistor** – a semiconductor component. The electrical resistance of the thermistor decreases in a circuit as the temperature increases. As a result, more current flows. The display may be digital.	Because they are sensitive to temperature changes and link to electrical circuit resistance, they are used in fire alarms and switching circuits for heater systems (range: −250 to 700 °C). Digital thermometers generally used in hospitals for patient care, schools and working environments in conjunction with 'thermometer skin strips'.
	Resistance – a conductive wire in a circuit which increases its resistance to current flow when its temperature rises. Suitable for high temperature changes. Easy to read display. Expensive and can be subject to 'drift'.	Used in industry because it can record temperatures of over 1000 °C.
	Thermocouple – when two wires of different metals are connected together, two junctions are formed which produce a potential difference with temperature. This is a very sensitive device. Short distance measurements can be taken. Difficult to calibrate.	Used in industry to record the temperature of furnaces, ovens, etc. because of its wide range of temperature recording (range: −250 to 1600 °C).
	Rotary – the coiled bimetallic strip principle is used so that when temperatures increase, the strip expands more and touches a calibrated pointer.	Simple construction allows general usage such as in greenhouses or fridge-freezers.
	Infra-red – this type detects various wavelengths of infra-red electromagnetic radiation and is extremely accurate. Can only measure surface temperatures. Affected by radio waves and other waves. Affected by ambient conditions.	Commonly used in hospitals to determine a patient's change in temperature by inserting the device into the ear.

Calibrating thermometers

Checking the calibration of a thermometer at boiling point

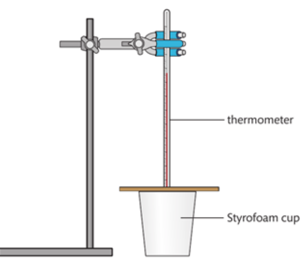


Checking the calibration of a thermometer



Cooling Curve

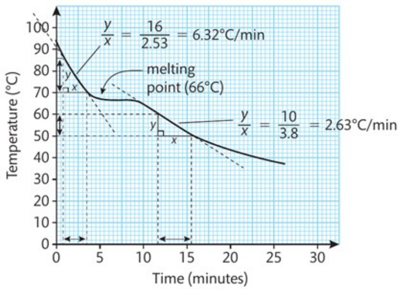
1. Set up the apparatus as shown in Figure 2.10.
2. Pour freshly boiled tap water into a 100 ml beaker to the mark.
3. Transfer this water to the calorimeter.
4. Record the temperature of the water and start the stop clock.
5. Record the temperature at 60-second intervals for approximately 10 minutes.
6. Plot the results on a graph of temperature (y-axis) against time (x-axis).
7. Draw a curve of best fit through your plots and draw a tangent to the curve at each point.
8. Calculate the 'rate of cooling' at each point from the gradients of the tangents.



Intermolecular forces are the sum of repulsive and attractive forces between molecules. This does not include atomic bonding of the substance, which is termed intramolecular forces.

The **strength** of the attractive forces and the kinetic energy of the atoms in a substance are the key factors which determine what physical state it will be in.

Greater attractive intermolecular forces means that **higher temperature** are needed to reduce the forces of attraction binding the molecules. Changing the state of a solid to liquid needs a lot of extra heat energy, changing the state of a liquid to a gas requires even more heat energy.



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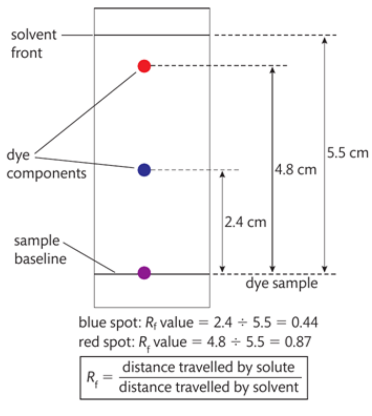
Chromatography

Chromatography: A method used to separate chemical mixtures for analysis.
Mobile phase: The liquid that transports the substance mixture through the absorbing material which travels along the stationary phase and carries the substance components with it.
Stationary phase: The solid material that absorbs the mixture flowing through it.
Chromatogram: The resulting paper or plate produced showing the substance separation.

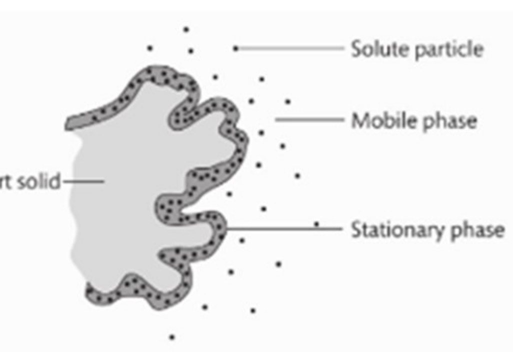
Paper Chromatography

In chromatography, substances are separated as they travel in a **mobile phase** which passes over a **stationary phase**. Different substances travel at different speeds, so some move further than others in the time specified.
In paper chromatography, the stationary phase is paper and the mobile phase may be either an aqueous liquid or a non-aqueous organic solvent.



Thin Layer Chromatography

This type of chromatography is used to analyse dyes in fibres, inks and taints. The **stationary** phase is a thin player of an unreactive substance (silica or aluminium oxide) supported on a flat, inert surface such glass, metal or plastic plate. A small amount of the mixture to be analysed is put near the bottom of the plate in the form of spots.
Each component of the mixture is adsorbed on the solid because of their differences in solubility and strength of their **adsorption**. When the solvent reaches the top of the plate, the plate is removed and dried.
For colourless samples, an ultra-violet (UV) lamp is used.



Preparative methods for samples

Solvent extraction

Process provides a method of separating two compounds from a chemical mixture based on the differences of their **solubilities.**
Immiscible: liquids that do not mix together.
When left to stand the two chemicals separate.

Filtration

The process involves passing a mixture of solids and liquids through filter paper placed in a funnel. The solids are insoluble. The liquid flows through the paper. You should pass the liquid through multiple time to complete the process.
Choosing the filter paper that has the correct **porosity** (A measure of the volume of tiny holes) is crucial in the filtering process of the medium and the length of time needed for it to filter

Evaporation

Process involves separating the soluble chemical compound by the means of removing excess water from a chemical solution by **heating** the solution. E.g. NaCl and other salts from water. The heating process must be carried out carefully and slowly to prevent over-evaporation of the solution. Repeated evaporation cycles increase the concentration of the sample to be separated.

Locating agents

Samples, such as amino acids, are colourless and need some means to make them visible for analysis. Locating agents are **chemical substances** which are added to the samples by a spray. The results are the colourless compounds are now able to be seen.

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Plant pigment extraction

Steps in the investigation	Pay particular attention to...
1. Place a few leaves from the same plant into a mortar.	
2. Add a small amount of grit or sand (to break the cell walls) and approximately six drops of propanone.	A small amount of grit or sand is needed help tear and expose the inner leaf.
3. Grind the mixture for a few minutes.	
4. Put a pencil line 3 cm from the bottom on the TLC plate.	
5. Use a micro-capillary tube to put a small spot in the centre of the pencil line.	Practise putting the spots onto paper before doing it 'for real'. This will help you to develop a good technique.
6. Add the solvent to the beaker.	You may need to add a small quantity at first.
7. Place the TLC plate into the beaker and ensure that the level of solvent is below the spot.	Add more solvent if the initial level is too low.
8. Allow the solvent to rise until it stops. Mark this point with a pencil line.	You will need to observe the solvent movement over a suitable time period.
9. Calculate the R_f of each pigment.	

Amino Acid Identification

Steps in the investigation	Pay particular attention to...
1. Put a pencil line across the chromatography paper 3 cm from the bottom.	
2. Place a small pencil dot every 2.5 cm apart for each of the amino acids under test and label them.	Be sure to label the pencil dots before testing.
3. Pipette 2 ml of an amino acid onto one of the dots. Repeat for the other two, using a different pipette for each.	
4. Repeat this another four times.	Each dot should applied at least four times to develop a suitable concentration of all amino acids.
5. Allow to dry and roll the paper into a cylinder. Be careful not to contaminate the paper with your fingers.	
6. Add the solvent to the beaker.	Be careful when adding the solvent to avoid splashing the paper in random areas.
7. Place the cylinder into the beaker and ensure that the level of solvent is below the line.	
8. Allow the solvent to rise until it stops. Mark this point with a pencil line.	
9. The technician will now spray ninhydrin onto the paper and heat-develop in an oven.	Ninhydrin is flammable, harmful if swallowed, an irritant on skin and respiratory system, and can cause dizziness if inhaled.
10. Calculate the R_f of each amino acid.	

Problems in technique

- **Correct spotting of samples** – if your samples are not spotted above the solvent level, they will not travel with the solvent but are more likely to be washed into the solvent before the mobile phase occurs.
- **Uneven movement of the solvent in TLC** – using water as the solvent is not advised because of its surface tension which produces a curved solvent front and possible errors in the R_f calculation. Use a large amount of solvent so that the mobile phase can advance to its full limit. Ensure that the plate is positioned flat against the chamber floor and is not tilted. Use a ruler or even spirit level to check this.
- **Over-concentration of spot** – if this occurs during your preparation, streaking will happen rather than separations. Make sure that your sample is accurately prepared in accordance with the procedures outlined in this section.
- **Excessive spot sizes** – generally, spot sizes must not be larger than 1 mm to 2 mm in diameter. If the spot size is too large, overlapping can occur with spots of similar R_f values. As a result, the differences in R_f cannot be easily calculated.

Molecule size and mobility

The molecules of substances are different in size and weight. This physical property will allow separation of chemical substances in a process similar to a mechanical sieve. Smaller molecules can permeate though the gel porous, larger molecules will travel further in the mobile phase.
Retention factor (R_f): distance moved by the solute/distance moved by the solvent on chromatography paper/plate.

$$R_f = \frac{\text{distance travelled by solute}}{\text{distance travelled by solvent}}$$