# CHEMISTRY - Paper 3

## Errors

$$absolute\ error\ =\ no.\ of\ readings\ \times \frac{smallest\ division}{2}$$

$$percentage\ uncertainty\ =\ \frac{absolute\ error}{reading} \times\ 100$$

Eg. maximum percentage error in a temperature change of 14.0°C = ((2 × 0.5)/14.0) × 100 = 7.14%

- Random error: usually result from the experimenter's inability to take consistent measurements e.g. in the disappearing cross experiment. It is often due to a problem which persists throughout the entire experiment e.g. random fluctuations in room temperature.
- Systematic error: usually caused by measuring incorrectly calibrated apparatus or incorrectly used apparatus e.g. thermometers that consistently read above the actual temperature, or reading volumes consistently from the wrong part of the meniscus.

Evaluate the effectiveness of control variables

- Comment on errors intrinsic in measuring devices, e.g. a thermometer consistently reading 1°C above actual temperature.
- Experiments where limitations of the method introduce errors: e.g. heat loss when trying to assess enthalpy change
- Systematic error = e.g. zero error in a balance. Thus the same balance is used for all weighings within an experiment.
- Random error = e.g. change in room temperature when investigating effect of changing concentration on the rate of a reaction.
- NOTE: A statement of 'human errors' is not acceptable; though there are occasionally errors arising in the observer's ability to observe, e.g. in the disappearing cross experiment, which would be a random error.)

## Accuracy

| Apparatus | Smallest division | Max Error |
|---|---|---|
| Burette | $0.05cm^3$ | $0.1cm^3$ |
| Pipette ($25cm^3$) | | $0.06cm^3$ |
| Volumetric Flask ($250cm^3$) | | $0.2cm^3$ |

- Give burette readings to 2 dp (last digit = 0 or 5)
- Give pipette readings to 1 dp

NOTE

- Record burette readings to the nearest 0.05cm³
- When using a thermometer calibrated at 1°C intervals, temperature readings should be recorded to the nearest 0.5°C.
- Measuring cylinder calibrated at 1.0cm³ should be read to the nearest 0.5cm³.

**Significant figures**

- Use the correct number of significant figures for calculated quantities (this should be the same as or one more than the smallest number of significant figures in the provided or experimentally determined data).
    - For example, if titre volume is measured to 4 significant figures, e.g. 23.45cm³, then the calculated molar concentrations from this should be given to four significant figures, e.g. 1.305mol dm−3 or 0.9876mol dm−3.
- However, if the concentration of one of the reactants is given to three significant figures, then the calculated concentration could be given to three or four significant figures.
    - For example, if the concentration of alkali in an acid–base titration is given as 0.100mol dm−3, then the concentration of the acid may be shown as 0.1305mol dm−3 or 0.131mol dm−3.

**Graphs**

- Plot appropriate variables on appropriate, clearly labelled x- and y-axes (the same convention for axis labels should be used as for table headings)
- Choose scales for graph axes that allow the graph to be read easily, such as 1, 2 or 5 units to a 20mm square; the data points should occupy at least half of the graph grid in both x- and y-directions
- Plot all points using a cross × or circled dot ʘ to an appropriate accuracy •
- Draw straight lines or smooth curves of best fit to show the trend of a graph.
- A line of best fit should show an even distribution of points on either side of the line along its entire length, and anomalous points should be identified.

## Quantitative analysis

**Titration experiments**

- acid–alkali titration (this could be weak or strong acid and weak or strong alkali).
- potassium manganate(VII) titration with hydrogen peroxide, iron(II) ions or ethanedioic acid or its salts.
- sodium thiosulfate and iodine titrations.

NOTE

- Burette has to be written to 2 DP.
- Carry out a rough titration first.
- 2 best titres must be within 0.1 cm3 of each other.

- If the first 2 titres are within 0.1 cm3 then no need for the 3rd titre.
- Repeat and find the average titre volume with a total spread of not more than 0.20 cm3.

Use of a burette

| Advantages | Disadvantage |
|---|---|
| Lower error | Takes longer to add the reagent |
| More accurately calibrated | |

**Rates experiments**
- Mix reagents and record the time for an observation to occur.
- Eg. time taken on mixing solutions of sodium thiosulfate and an acid for the print on a piece of paper to be obscured by the precipitate produced.

**Gravimetric experiments**
- Heat a solid in a crucible on a pipe-clay triangle and record mass change
- Eg. the determination of the water of hydration of a hydrated salt by evaporation of the water and calculation of the change in mass.

**Thermometric experiments**
- Accurately use and take readings from thermometers.
- Eg. the determination of the enthalpy change of reaction by recording of temperature changes and subsequent calculation of enthalpy changes and use of Hess's law.

**Gas volume experiments**
- Set up apparatus for gas collection over water method.
- Eg.determination of the composition of a solid from the volume of carbon dioxide produced on reaction of a carbonate with an acid.

## Qualitative analysis

NOTE
- Record all observations.
- Record observations to the same level of detail, e.g. observations of qualitative variables such as colour should be recorded in simple language such as 'blue' or 'yellow'. Where fine discrimination is required, terms such as 'pale' or 'dark' should be used, and comparisons made such as 'darker brown than at three minutes' or 'paler green

- Write observations by looking at the observations mentioned in the salt analysis data sheet. Copy the observation which matches closely with your observation. There would be rare exceptions to this scenario.
- If there are series of colour changes observed, mention all of the colours.
- Use excess alkali where a precipitate is produced on addition of NaOH(aq) or $NH_3$(aq) to determine solubility.
- Tables for salt analysis should be constructed with separate columns for “1 cm3 of reagent X” and “reagent X in excess”.
- Identify a gas whose formation is shown by effervescence.
- When reagents are asked, write proper bench reagents.
  Eg. Instead of writing $H^+$ ions, write HCl (aq) or HNO3 (aq).
  Instead of writing $Cr_2O_7^{-2}$ ions, mention $K_2Cr_2O_7$ or $Na_2Cr_2O_7$.

**Cations**

| | **NaOH (aq)** | **$NH_3$ (aq)** |
|---|---|---|
| zinc, $Zn^{2+}$(aq) | white ppt. | white ppt. |
| | soluble in excess | soluble in excess |
| aluminium, $Al^{3+}$ (aq) | white ppt.<br><br>This precipitate formed by $Al^{3+}$ is very soluble and disappears very quickly. Use a very tiny quantity of NaOH at first (few drops). A small white ppt will form floating on top, which would dissolve very quickly. | white ppt. |
| | soluble in excess | insoluble in excess |
| magnesium, $Mg^{2+}$ (aq) | white ppt. | white ppt. |
| | insoluble in excess | insoluble in excess |
| manganese, $Mn^{2+}$ (aq) | off-white ppt. rapidly turning brown on contact with air<br><br>brown residues floating on top surface and on sides of the test tube, white ppt/light brown ppt at the bottom | off-white ppt. rapidly turning brown on contact with air<br><br>brown residues floating on top surface and on sides of the test tube, white ppt/light brown ppt at the bottom |
| | insoluble in excess | insoluble in excess |

| | | |
|---|---|---|
| ammonium, $NH_4^+$ (aq) | no ppt.<br>($NH_3$ produced on warming) | – |
| barium, $Ba^{2+}$ (aq) | faint white ppt / no ppt.<br><br>faint white ppt. is observed unless [Ba2+(aq)] is very low | no ppt. |
| calcium, $Ca^{2+}$(aq) | white ppt.<br><br>white ppt unless [Ca2+(aq)] is very low | no ppt. |
| | insoluble in excess | |
| chromium(III), $Cr^{3+}$(aq) | grey-green ppt. | grey-green ppt. |
| | ppt soluble in excess giving dark green solution | insoluble in excess |
| copper(II), $Cu^{2+}$(aq) | pale blue ppt. | pale blue ppt. |
| | insoluble in excess | soluble in excess giving dark blue solution<br><br>use very small amount of $Cu^{+2}$ (<1 cm3) or use a lot of aq. $NH_3$ (fill the entire test tube) and shake vigorously to dissolve the ppt. |
| iron(II), $Fe^{2+}$(aq) | green ppt. turning brown on contact with air | green ppt. turning brown on contact with air |
| | insoluble in excess | insoluble in excess |
| iron(III), $Fe^{3+}$(aq) | red-brown ppt. | red-brown ppt. |
| | insoluble in excess | insoluble in excess |

| **$Pb^{2+}$** | **$Al^{3+}$** |
|---|---|
| Forms white ppt with NaOH(aq) and $NH_3$(aq) | Forms white ppt with NaOH(aq) and $NH_3$(aq) |

| | |
|---|---|
| Lead halides are insoluble: $PbCl_2$, $PbI_2$, $PbBr_2$ | Aluminium halides are soluble |
| $PbSO_4$, $PbCr_2O_7$ are insoluble | These aluminium salts are soluble |
| Use reagents containing $Cl^-$ / $I^-$ / $Br^-$ / $SO_4^{2-}$ / $Cr_2O_7^{2-}$ / $CrO_4^{2-}$ AND $Na^+$ / $K^+$ / $H^+$ ions to test for $Pb^{2+}$;<br>eg. HCl, KI, $K_2Cr_2O_7$<br>precipitate forms. | No precipitate forms with these reagents. |

| **$NH_4^+$** | **$Ba^{2+}$** |
|---|---|
| Forms no ppt with NaOH(aq) and $NH_3$(aq) | Forms no ppt with NaOH(aq) and $NH_3$(aq) |
| - Warm $NH_4^+$ with NaOH.<br>- $NH_3$ is produced.<br>- Test for $NH_3$ using damp red litmus. | - Add $H_2SO_4$ to $Ba^{2+}$<br>- White ppt forms |

| **$Cr^{3+}$** | **$Fe^{2+}$** |
|---|---|
| Forms grey-green ppt with NaOH(aq) and $NH_3$(aq). | Forms green ppt with NaOH(aq) and $NH_3$(aq). |
| No browning occurs. | Ppt. turns brown on contact with air. |
| Ppt dissolves in excess NaOH(aq) to give a dark green solution. | Ppt does not dissolve in excess NaOH(aq) or NH3(aq) |

| **$Ca^{2+}$** | **$Ba^{2+}$** |
|---|---|
| Forms white ppt with NaOH(aq) unless [Ca2+(aq)] is very low and no ppt with $NH_3$(aq). | Forms faint white ppt with NaOH(aq) unless [Ba2+(aq)] is very low and no ppt with $NH_3$(aq). |
| Forms white ppt with $H_2SO_4$ | Forms slight white ppt with $H_2SO_4$ |
| To distinguish between $Ca^{2+}$ and $Ba^{2+}$, flame test has to be done, which is not required at this level. Mark schemes usually allow both as the correct answer. | |

**Anions**

| | |
|---|---|
| carbonate, $CO_3^{2-}$ | $CO_2$ liberated by dilute acids |
| | - add dil. $HNO_3$<br>- effervescence is produced.<br>- gas gives a white ppt. with limewater<br>NOTE: dil. HNO3 is preferred for $CO_3^{2-}$ test, because this sample can then be used to test for further ions like $SO_4^{2-}$ and halides, which would not be possible with H2SO4 or HCl. |
| chloride, $Cl^-$ (aq) | gives white ppt. with Ag+ (aq) (soluble in $NH_3$(aq)) |
| | - add aq. $AgNO_3$<br>- white ppt<br>- add dil. aq. $NH_3$ to the ppt<br>- ppt dissolves |
| bromide, $Br^-$ (aq) | gives cream/off-white ppt. with Ag+ (aq) (partially soluble in $NH_3$(aq)) |
| | - add aq. $AgNO_3$<br>- cream ppt<br>- add dil. aq. $NH_3$ to the ppt<br>- ppt does not dissolve<br>- add conc. aq. $NH_3$ to the ppt<br>- ppt dissolves |
| iodide, $I^-$ (aq) | gives pale yellow ppt. with Ag+ (aq) (insoluble in $NH_3$(aq)) |
| | - add aq. $AgNO_3$<br>- pale yellow ppt<br>- add dil. aq. $NH_3$ to the ppt<br>- ppt does not dissolve<br>- add conc. aq. $NH_3$ to the ppt<br>- ppt does not dissolve |
| nitrate, $NO_3^-$ (aq) | $NH_3$ liberated on heating with $OH^-$(aq) and Al foil |
| | - use boiling tube<br>- add same amount of NaOH(aq)<br>- add Al foil and heat<br>- effervescence produced<br>- gas turns damp red litmus paper blue |
| nitrite, $NO_2^-$ (aq) | $NH_3$ liberated on heating with $OH^-$(aq) and Al foil; decolourises acidified aqueous $KMnO_4$ |
| | - use boiling tube<br>- add same amount of NaOH(aq) |

| | |
|---|---|
| | - add Al foil and heat<br>- effervescence produced<br>- gas turns damp red litmus paper blue<br><br>- add acidified aqueous $KMnO_4$<br>- turns from purple to colourless |
| sulfate, $SO_4^{2-}(aq)$ | gives white ppt. with $Ba^{2+}(aq)$ (insoluble in excess dilute strong acids); gives white ppt. with high $[Ca^{2+}(aq)]$ |
| | - add $BaCl_2(aq)$ or $Ba(NO_3)_2(aq)$<br>- white ppt<br>- add excess dilute HCl / $HNO_3$ (fill the test tube)<br>- white ppt remains insoluble<br>NOTE: $Ba(NO_3)_2(aq)$ is preferred, because the same sample can then be used to test for halide ions. |
| sulfite, $SO_3^{2-}(aq)$ | gives white ppt. with $Ba^{2+}(aq)$ (soluble in excess dilute strong acids); decolourises acidified aqueous $KMnO_4$ |
| | - add $BaCl_2(aq)$ or $Ba(NO_3)_2(aq)$<br>- white ppt<br>- add excess dilute HCl / $HNO_3$ (fill the test tube)<br>- white ppt dissolves<br><br>- add acidified aqueous $KMnO_4$<br>- turns from purple to colourless |
| thiosulfate, $S_2O_3^{2-}(aq)$ | gives off-white/pale yellow ppt. slowly with $H^+$ |
| | - add HCl / $HNO_3$ (one drop at a time)<br>- off-white / pale yellow ppt (slowly turns cloudy) |

**Gases**

<u>When to look for gases</u>

To check if a gas is being liberated, put the thumb on top of the test tube and check if pressure builds up in the tube.

| | |
|---|---|
| ammonia, $NH_3$ | If NaOH is added to the salt and then heated with aluminium foil, $NH_3$ is liberated if $NO_3^-$ / $NO_2^-$ is present. |
| carbon dioxide, $CO_2$ | If <u>acid + salt</u> produces effervescence, carbonate ion is present, so the gas evolved is carbon dioxide.<br>- effervescence produced<br>- gas gives a white ppt. with limewater |
| hydrogen, $H_2$ | If <u>acid + metal</u> produces bubbles, the gas evolved is |

| | |
|---|---|
| | hydrogen gas.<br>- effervescence produced<br>- gas ‘pops’ with a lighted splint |
| oxygen, $O_2$ | |
| (sulphur dioxide, $SO_2$) | If dilute acid is added to a salt, $SO_2$ is liberated if $SO_3^{2-}$ is present. |
| (nitrogen dioxide, $NO_2$) | If dilute acid is added to a salt, $NO_2$ is liberated if $NO_3^-$ is present. |

Test for gases

| | |
|---|---|
| ammonia, $NH_3$ | turns damp red litmus paper blue<br>- DAMP a red litmus paper with distilled water.<br>- Hold it near the mouth of the test tube; it shouldn't touch the tube.<br>- Red litmus should turn blue. |
| carbon dioxide, $CO_2$ | gives a white ppt. with limewater |
| hydrogen, $H_2$ | ‘pops’ with a lighted splint<br>- Put your thumb on top of the test tube and allow pressure to build up.<br>- Place a lighted matchstick at the mouth of the tube. |
| oxygen, $O_2$ | relights a glowing splint |
| (sulphur dioxide, $SO_2$) | - dip a paper in $K_2Cr_2O_7$ and hold it near mouth of tube: $K_2Cr_2O_7$ colour change from orange to green.<br>- hold damp blue litmus paper near mouth of tube: blue litmus turns red.<br>- pipe the gas into solution of $KMnO_4$: it turns from purple to colourless.<br>- smells of rotten eggs or burnt matches. |
| (nitrogen dioxide, $NO_2$) | - put the thumb on top of the test tube.<br>- test tube turns pale brown and colour disappears when thumb is removed; pale brown plume released.<br>- it is visible when the reactants are thrown in a white sink - brown vapours can be noticed in the sink.<br>- also turns damp blue litmus paper red. |

NOTE: before testing for any gas, press down the thumb at the mouth of the test tube for some time to build up pressure, and then test.