Chem Factsheet

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Essential Laboratory Procedures: PSA's - AS Part 1

Before using this Factsheet, use the internet to download a copy of your examination board's specification.

- Read the section on laboratory skills to ensure that you are familiar with what will be expected of you.
- Will you be allowed access to instructions prior to the practical session? (Some boards allow this whilst others keep instruction sheets and mark schemes confidential to assessors.)
  - If "yes", spend time reading them to understand what you will do and why you will do it.
  - If you understand *why* you do each step you are far less likely to make errors than if you blindly follow the instructions. This should also help you to work efficiently in the laboratory.

Prior Knowledge

To get the most from this Factsheet you should already be familiar with the following terms: qualitative, quantitative, evaluation and preparation.

Equipment - Advice

If you have little practical experience to date and/or English is not your first language, you could familiarise yourself with the names and types of commonly used laboratory equipment by using the internet to collect labelled images. Use the examples in this Factsheet starting with volumetric (graduated) flask, beaker and filter funnel.

To get the most from this Factsheet: Use the procedures to draw up flow charts with images of apparatus and very brief descriptions of what to do. Add explanations of what is happening at each stage and why each step is used. Summarise the tests for ions and their results in a table format.

After working through this Factsheet you should be able to:

- recall and understand simple procedures for preparing solutions, titrating solutions and testing for the common anions.
- present titration results clearly.
- do calculations based on titration results.
- be aware of what is expected from you during each practical session.

Chemistry is by its very nature a practical subject and, to ensure that all students of the subject are trained in the essential laboratory procedures, each of the Advanced level courses includes an assessment of practical skills at both AS level and again at A2 level, commonly contributing 20% of the total AS marks, 20% of the total A2 marks and hence 10% of the total GCE award. Normally the assessments fall naturally into the topics studied and so are continuous through the courses.

Some boards are very specific with regard to exactly what is assessed - setting common tasks with mark schemes, whilst others are less prescriptive, offering examples but also allowing centres / teachers to define and score their own tasks. AQA lists examples of tasks and their possible contexts in the specification.

Further differences exist when it comes to written tasks with some boards requiring formal writing up of practical reports and others setting written examination papers around specific practical tasks – PSAs, ISAs and EMPAs. In all cases you will have to set out your results in a clear and comprehensive manner.

You will be expected to complete tasks that fall into each of the categories inorganic, physical and organic chemistry. Also, within each of these categories, there will be qualitative and quantitative tasks, together with preparations. At times you will need to evaluate results and be aware of sources of errors.

Throughout you will be expected to be conscious of hazards and to carry out tasks safely without endangering yourself or others around you. You may be expected to prepare a risk assessment. To this end you should make yourself aware of "Hazcards" or similar sources of information in your laboratory.

During practical sessions wear safety glasses at all times. In your handling assume that all of the reagents and liquids are toxic, corrosive and flammable.

You may be expected to carry out calculation work.

General Practical Advice – Always:

1. use *clean* apparatus. There are occasions when the apparatus need not be dry. Before using wet vessels ask yourself "will the inclusion of water affect my results / experiment"?
2. label all vessels *before* putting solution/solid into them. (A colourless solution looks like all other colourless solutions. Confusing them could cost you marks and might even lead to dangerous procedures.)
3. always use a *pipette filler* to take up liquid into a pipette.

Key Points to doing well include:
1. being able to follow and interpret experimental instructions.
2. completing tasks in an appropriate time ie working efficiently.
3. manipulating apparatus skilfully.

Here follow examples of typical procedures at AS level.

Section 1 - Inorganic

(1A) Making A Volumetric Solution – A Standard Solution (A Quantitative Skill)

Almost any compound soluble in water could be chosen. You may be asked to calculate the mass of solid required to produce a solution with a concentration within a given range - see box 1. Commonly you will go on to use your prepared solution in a titration - procedure (1B).

<u>Task</u>: To Produce a Standard Solution from Solid X.

Procedure:

1) Zero / tare the balance and place the clean dry weighing bottle or weighing boat on the top pan digital balance. Record its mass[a]. Into the weighing bottle/boat, using a spatula, transfer the ***approximate***[b] mass required. (The assessor will be looking to see that no solid is spilled. If you do spill any you must clean it up immediately.)
2) Weigh ***accurately*** the weighing vessel and contents, recording the ***precise*** mass.
3) Empty the contents of the weighing vessel into a beaker and reweigh the weighing vessel. (Some solid may have adhered to the container and not been transferred.) Determine the mass transferred and use this value for future calculations.
4) To the beaker add about 100 cm^3 distilled (or de-ionised) water. Stir with a glass rod until **<u>all</u>** of the solid has dissolved[c].
5) Transfer the contents of the beaker to the volumetric/graduated flask[d]. Rinse the beaker with distilled water (from a wash bottle) and carefully transfer the washings to the volumetric flask. Finally rinse the funnel and the stirring rod into the flask.
6) Carefully add distilled water to the flask to make up the volume to the mark exactly[e].
7) With the stopper securely held in place, invert the flask several times[f]. Add more water to the mark if shrinkage has occurred. Then remix.

Comments And Guidance On This Procedure

see points a – f above:

a Use all of the figures displayed on the balance. Do not round off. The assessor will be looking to see that you are reading the balance correctly and recording the values correctly.

b ***Approximate:*** If the instruction states for example "approximately 5 g" or "about 5 g", you do not have to waste time trying to get an exact mass of 5.000g. A mass close to this, say between 4.9 and 5.1g will be fine. However, the mass must be ***accurately and precisely*** weighed and recorded eg 4.984g might be recorded.

c If the solid does not dissolve easily, add a little more water. You must ensure that no solid remains at this stage and also must not "splash" when you stir as this would lose some of your carefully weighed material.

d Use a funnel to transfer liquids. Again, you must avoid losing any material through leaving it behind or splashing. Remember to rinse the funnel, transferring its washings too.

e To make up the volume to the mark exactly the final few drops of water can be added from a teat pipette. This will ensure that you do not go above the mark.

f You must mix very thoroughly and hence secure homogeneity of the solution. Otherwise any one sample taken from the flask will be different in its concentration to any other. It is usual for the teacher/supervisor to inspect the flask to see that the bottom of the liquid's meniscus is level with the mark on the flask.

Question 1

(a) In the above procedure (1A), is it important to use only a **<u>dry</u>** vessel to weigh out the solid? Explain.

(b) Suggest a source of error and explain its effect on any subsequent use of the "standard" solution.

Answers

(a) Yes. If water was present some of the moisture might get transferred along with the solid. Your calculations would suggest that you have transferred more solid than is true. (However, if you only had wet apparatus: Weighing the empty **<u>wet</u>** vessel and then again with added solid would not affect results provided that (a) the difference in mass was used to calculate the mass of solid used and, (b) the vessel was then rinsed and all of the washings transferred to the beaker.)

(b) A likely source of error would be the loss of solid (spilling) after weighing or liquid (splashing) when transferring it to the volumetric flask.

As there are less particles in the solution than there should be, the concentration of this "standard" solution will be **<u>lower</u>** than that based on the recorded mass of solid used.

Thus if it is used to find the concentration of a second solution via titration, the value for the concentration of the second solution will appear **<u>higher</u>** than it actually is.

Box 1 : Calculating Masses

Calculate the mass of anhydrous sodium hydrogen carbonate required to produce a 250 cm^3 solution of concentration 0.100 mol dm^{-3}.

<u>Method 1</u>

(1) $NaHCO_3$: $Mr = 23.0 + 1.00 + 12.0 + (16.0 \times 3) = 84.0$

(2) 0.100 mol is $0.100 \times 84.0 = 8.40$g. (If this was in 1dm^3 it would be a 0.100 mol dm^{-3} solution. However, we only want to make 250 cm^3 of the solution. ie 250/1000 or ¼ of this volume.)

(3) For 250 cm^3 solution of concentration 0.100 mol dm^{-3}
$(250/1000) \times 8.40\text{g} = \underline{2.10\text{g (3sig.figs.)}}$

<u>Method 2</u>

(1) $NaHCO_3 - Mr = 23.0 + 1.00 + 12.0 + (16.0 \times 3) = 84.0$
i.e. 1 mole is 84.0g

(2) $$\text{Concentration} = \frac{\text{number mol} \times 1000}{\text{volume}(cm^3)}$$

$$0.100\ \text{mol dm}^{-3} = \frac{\text{number mol} \times 1000}{250\ (cm^3)}$$

$$\frac{0.100 \times 250}{1000} = \text{number mol} = 0.025\ \text{mol}$$

(3) 0.025 mol needed (in the 250 cm^3).
$0.025\ \text{mol} = 0.025 \times 84.0 = \underline{2.10\text{g (3sig.figs.)}}$

<u>Tip</u>: When practising calculations use a copy of the data sheet that you will be issued with for the examinations and use the values as given therein. If you do not have a copy you can download one from your exam board's web site.

(1B) Carrying Out A Simple Titration - eg Acid – Base

For an acid-base titration you might be provided with a solution of an acid of known concentration and asked to titrate this against an alkali of unknown concentration or vice versa. Most usually it is the acid that is delivered from the burette, but occasionally it is the alkali. The volume you are required to titrate may also vary. Before you begin the practical task construct a table ready for your results with space for 5 titrations. It is usual to do at least three but not more than five titrations. (See Box 2.)

Task:

You are provided with a solution of a monoprotic acid*. Find its concentration by titration with a solution of 0.100 mol dm^{-3} sodium hydroxide.

*A **mono**protic acid has only one proton available per molecule eg **H**Cl, **H**NO$_3$. An example of a **di**protic acid is sulfuric acid, **H**$_2$SO$_4$.

Procedure:

1) Into a clean, dry beaker^A pour approximately 100 cm^3 of the acid solution. Rinse the burette^B with a small volume of this. Fill the burette with the acid. Fill the burette with the acid and record the initial burette reading.
2) Into a second clean, dry beaker^C pour approximately 100 cm^3 of the alkali solution. Rinse the pipette^D with a small volume of this.
3) Into a 250 cm^3 conical flask^E pipette exactly 25.00 cm^3 of the alkali.
4) Add two or three drops of indicator^F to the solution in the conical flask.
5) Titrate^G the contents of the conical flask by adding acid solution to it from the burette. Record your results in a table.
6) Repeat the titration^H until you obtain **two** results, which are **concordant^I.**
7) Calculate and record the mean titre. Use this to calculate^J the concentration of the alkali.

Comments And Guidance On This Procedure

see points A – J above:

A. Label the beaker "acid".

B. Ensure that the tap of the burette is closed. Use a funnel to pour about 10 cm^3 of the acid into the burette. Over a sink, "roll" the burette, tilting slowly so that the liquid washes the entire glass surface. Continue rolling and tilting until the acid pours out of the top of the burette and down the sink.

Fill the burette beyond the zero mark. Hold the burette over the sink and open the tap until the bottom of the meniscus is on the burette scale, allowing the excess acid to flow down the sink. This also rinses the tap region and fills the tap with acid solution. Remove the funnel from the burette so that no solution "drips" into the burette during the titration.

C. Label the beaker "alkali".

D. To rinse the pipette: Using the pipette filler take up about 5 – 10 cm^3 of the alkali. In the same manner as with the burette earlier, roll the pipette and draw the solution up past the bulb of the pipette to ensure that the entire surface is touched and so washed with the alkali. Do not allow the liquid to enter the pipette filler. Expel the liquid from the pipette into the sink.

E. Label the conical flask "sodium hydroxide".

F. Add sufficient indicator so as to give the solution a distinct colour but no more. The indicator "reacts" with the acid so if too much indicator is added it will "use up" too much of the acid. Commonly phenolphthalein is used for a strong acid – strong base titration. It is pink in alkali and colourless in acid. Place a white tile (or piece of paper) under the conical flask so as to see the colour change easily.

Note the colour of the indicator in the alkali.

G. Record all results in the table. The first titration should be done very quickly to establish approximately the titre required. Add from the burette, about 1 cm^3 at a time, the acid with swirling. Stop when the indicator changes colour. This is the approximate end-point of the titration and must be less than 1 cm^3 from the exact end point – because the indicator did not change after the previous addition from the burette.

H. Repeat but **<u>add quickly</u>** from the burette until you have delivered 1 cm^3 less than at the end of the first titration. (In this example this would be 24.0 cm^3.) Now add dropwise from the burette with swirling and washing down with distilled water until the indicator changes colour.

I. Continue repeating until concordant results are obtained. **Concordant** results are within 0.1 cm^3 of each other. You must decide whether or not to "top up the burette". If you have used less than 25.0 cm^3 you know that you have enough in the burette to do another titration. When you top it up you do not have to fill it to the zero mark as long as (a) there is enough in it for a titration – here, say 25.0 cm^3 – and (b) you record its actual reading in the table.

All values from a burette should be recorded to 2 decimal places with the final number being 5 or 0.

Show all working.

The assessor will be looking to see that you are reading and recording the values on the burette correctly.

J. Calculation – See box 3.

Box 2 A Typical Results Table:

Indicator: phenolphthalein
Indicator colour in alkali: pink
Indicator colour in acid: colourless

Titration Number:	1(rough)	2	3	4	5
Final Burette Reading/ cm^3	30.20	24.60	49.10		
Initial Burette Reading/ cm^3	5.00	00.00	24.60		
*Titre/ cm^3	25.20	24.60	24.50		

*Titre - the volume delivered from the burette.

Question 2

(a) In the above procedure (1B), is it important to use only a **<u>dry</u>** vessel
   (i) For the acid solution (step (1)?
   (ii) For the alkali solution (step (2)?
   (iii) For the conical flask (step (3)?

Answers

(a) (i) Yes. If water is present it will dilute the acid solution changing its concentration.

(ii) Yes. If water is present it will dilute the alkali solution changing its concentration.

(iii) No. A measured volume so a "fixed" number of particles will be transferred by pipette to this flask. Only these particles will react with the acid added from the burette. Any water in the conical flask will make no difference to the titration.

Box 3 Titration Calculation

Find the concentration of the monoprotic acid, HX.

(1) Calculate the number of moles of sodium hydroxide in the 25.0 cm^3 of 0.100 mol dm^{-3} solution used. As the acid reacts with the alkali 1:1, this is the same as the number of moles of the acid in the mean titre.

(2) Hence, knowing how many moles of acid are in the mean titre, calculate how many are in 1000 cm^3 ie in 1 dm^3 - its concentration.
Concordant results: 24.50 cm^3, 24.60 cm^3 of the monoprotic acid
Mean titre = $(24.60 + 24.50)/2 = 24.55$ cm^3 of the monoprotic acid
This reacted with 25.0 cm^3 of 0.100 mol dm^{-3} sodium hydroxide.
Number of moles sodium hydroxide = $(0.100 \times 25.0)/1000 = 0.00250$
Number of moles of the acid in 24.55 cm^3 is 0.00250.
In 1000 cm^3 (1 dm^3): $(0.00250/24.55) \times 1000 = 0.10183$ mol
Concentration of the monoprotic acid is <u>0.102 mol dm^{-3}</u> (3 sig figs)

(1C) Part 1 - Summary of Common Inorganic Tests - Aqueous Solutions

Anion:	Carbonate CO_3^{2-}
Test:	To the solution under test add an equal volume of dilute hydrochloric acid and bubble any gas through a small volume of limewater (calcium hydroxide solution).
Observation:	A milky white precipitate.
Notes:	All carbonates produce carbon dioxide on reaction with an acid. Carbon dioxide reacts with calcium hydroxide to produce insoluble calcium carbonate as a milky precipitate. $2H^+(aq) + CO_3^{2-}(aq) \rightarrow H_2O(l) + CO_2(g)$ $Ca(OH)_2(aq) + CO_2(g) \rightarrow H_2O(l) + CaCO_3(s)$
Anion:	Sulfate SO_4^{2-}
Test:	To the solution under test add an equal volume of dilute hydrochloric acid then an equal volume of barium chloride solution.
Observation:	A dense white precipitate.
Notes:	Barium ions react with sulfate ions to produce insoluble barium sulfate as a white precipitate. $Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$ The hydrochloric acid is added first so as to decompose any carbonate ions present since barium carbonate is also insoluble and white.
Anions:	Halides: chloride Cl^-, bromide Br^-, iodide I^- in aqueous solution
Test 1:	To the solution under test add an equal volume of dilute nitric acid followed by 1 - 2 cm^3 of silver nitrate solution.
Observations and Conclusions	A white precipitate – chloride ions present. A cream precipitate – bromide ions present. A yellow precipitate – iodide ions present.
Notes:	Halide ions (X^-) react with silver ions to produce insoluble silver halides. You should learn their colours. $Ag^+(aq) + X^-(aq) \rightarrow AgX(s)$ The nitric acid is added first so as to decompose any carbonate ions present as silver carbonate is also insoluble. The resulting precipitates from test (1) should each be divided into two test tubes. They can then be used for confirmation tests (2) and (3) below.
Test 2:	To the precipitate formed in test 1 add an excess of <u>**dilute**</u> aqueous ammonia.
Observations and Conclusions:	The white precipitate (chloride) dissolves completely. The cream precipitate (bromide) dissolves only sparingly. The yellow precipitate (iodide) does not dissolve.
Test 3:	**In a fume cupboard** to the precipitate formed in test 1, add an excess of <u>**concentrated**</u> ammonia solution
Observation:	The white precipitate (chloride) dissolves completely. The cream precipitate (bromide) dissolves completely. The yellow precipitate (iodide) does not dissolve.
Notes:	The solubility of the silver halides in ammonia solution decreases in the order: AgF → AgCl → AgBr → AgI

(1C) Part 2 - Summary of Common Inorganic Tests - Solid Salts

Anions:	Halides: chloride Cl^-, bromide Br^-, iodide I^- in a solid sample
Test 1:	Place a spatula measure of the solid in a **dry** test tube. **In a fume cupboard** add a few drops of concentrated sulfuric acid. Place **moist** blue litmus (or Universal) indicator paper at the mouth of the test tube so that any gas evolved comes into contact with it.
Observations and Conclusions:	All chlorides, bromides and iodides produce the corresponding hydrogen halide (HX where X = any halogen) gas which turns moist blue litmus paper pink and moist Universal indicator paper red because of its acidic nature.
Notes:	$2NaX (s) + H_2SO_4 (l) \rightarrow HX (g) + Na_2SO_4 (aq)$
Test 2:	Repeat test 1 replacing the moist litmus/Universal indicator paper with a small piece of filter paper that has been soaked in acidified potassium dichromate(VI) solution.
Observations and Conclusions:	The orange solution on the filter paper becomes green. Bromide ions **or** iodide ions (X below) are present.
Notes:	Orange dichromate(VI) ions are reduced to green chromium(III) ions by sulfur dioxide gas. The gas is produced when either bromide or iodide ions (X^-) reduce sulfate(VI) ions. Factsheets 78 and 104 are helpful in revising the construction of redox equations. Chloride ions do not reduce sulfate(VI) $2X^- (aq) + 4H^+(aq) + SO_4^{2-}(aq) \rightarrow SO_2 (g) + X_2(g) + H_2O (l)$ $3SO_2(g) + Cr_2O_7^{2-}(aq) + 2H^+(aq) \rightarrow 3SO_4^{2-}(aq) + 2Cr^{3+}(aq) + H_2O(l)$
Test 3:	Repeat test (1) replacing the moist litmus/Universal indicator paper with a small piece of filter paper that has been soaked in either lead(II) nitrate or lead(II) ethanoate solution.
Observations and Conclusions:	The white filter paper turns black. Iodide ions are present.
Notes:	Hydrogen sulfide is produced when iodide ions reduce sulfuric acid. Chloride and bromide ions are not able to do this. Hydrogen sulfide reacts with lead(II) ions to produce black insoluble lead(II) sulfide. $8X^- (aq) + 10H^+(aq) + SO_4^{2-}(aq) \rightarrow H_2S (g) + 4X_2(g) + 4H_2O (l)$ $Pb^{2+}(aq) + H_2S(g) \rightarrow PbS(s) + 2H^+(aq)$

Further Notes On Solid Halides With Concentrated Sulfuric Acid

As well as the results of the tests above there are other very important observations that you should note. When the halide reacts with the concentrated sulfuric acid:

- the hydrogen halide gas (HX) is seen as **misty fumes in moist air**.
- the bromide produces bromine gas which is seen as **brown fumes.**
- the iodide produces solid iodine which is seen as **black solid.** (If the mixture gets very hot, this can sublime and be seen as **purple fumes.**)

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