GCE

Rewarding Learning

Revised GCE

Chemistry

AS Chemistry practical support document

For first teaching from September 2016

AS Chemistry practical support document

Please note that it is the responsibility of the centre to determine risk assessments, and quantities of chemicals used, for all experiments. Observations should be recorded where appropriate and is not indicated specifically in this document. This is not a practical manual but a guide to suitable practical methods.

Determine the formula of a hydrated compound by weighing and heating a hydrated salt to constant mass;

- weigh an evaporating basin
- add some hydrated solid and weigh the evaporating basin and solid
- heat to constant mass - heat for a few minutes, cool then weigh and repeat this procedure until the mass does not change

Use the deflection of a stream of liquid from a burette to indicate polarity or lack of polarity within a molecule;

- place the liquid in a burette and place a beaker underneath the jet
- open the tap and allow a small stream of liquid to run from the burette
- hold a charged polythene rod close to the stream of liquid and note any deflection
- to compare the polarity of two liquids use two burettes simultaneously with the stream of liquid at same rate and the distance between the charged rod and liquid constant

Carry out tests of electrical conductivity on solids, liquids and aqueous solutions of ionic and covalent substances;

- set up a circuit using a dc power pack and bulb
- add graphite electrodes into the circuit and dip them into a beaker of liquid/aqueous solution, ensuring they do not touch
- switch on the power pack and record if the bulb lights
- to test electrical conductivity of a solid, place the solid between crocodile clips

Determine the solubility of chlorine and iodine in aqueous and non-aqueous solvents.

- add 1 cm^3 of each of the aqueous halogen solutions (chlorine water, iodine solution) into separate test-tubes
- add equal volumes of a saturated hydrocarbon solvent e.g. hexane to each tube, stopper the tube and, shake the mixture by inverting the test-tube a few times.
- allow the two layers to settle; observe and record the colour

Quantitative determination of chlorine and iodine in aqueous solution is not required at AS. An estimate of the relative solubility of iodine could be made by comparing the colour of an iodine solution with the colour of solutions of known concentration.

Produce a reactivity order of the halogens using the displacement reactions of the halogens with other halide ions in solution;

- add 1 cm^3 of each of the aqueous halogen solutions (chlorine water, bromine water, iodine solution) into separate test-tubes
- add 1 cm^3 of potassium chloride solution to each and record any colour change
- repeat the experiment with potassium bromide solution and then with potassium iodide solution
- a saturated hydrocarbon e.g. hexane could be added to distinguish between bromine and iodine or starch solution could be added to detect iodine

Carry out the reactions of the halides with concentrated sulfuric and phosphoric acids and perform chemical tests for the products (excluding hydrogen sulfide);

- place 1 spatula of solid potassium chloride in test tube
- add a few drops of concentrated sulfuric acid and record observations
- test the gas evolved
- repeat the experiment using solid potassium bromide
- repeat the experiment using solid potassium iodide
- repeat the experiment using concentrated phosphoric acid instead of concentrated sulfuric acid

Carry out an acid-base titration to determine the concentration of acid/base, the degree of hydration in a hydrated metal carbonate and the percentage of ethanoic acid in vinegar

Steps to ensure accuracy

- rinse the apparatus with the appropriate solution
- add the solution from the burette dropwise just before the endpoint
- swirl the flask
- read the burette at the bottom of the meniscus

Carrying out the titration

- rinse the pipette with the solution to be transferred to the conical flask
- using a pipette and pipette filler place (25.0 cm^3) of the solution in the conical flask
- rinse the burette with the solution to be placed in it
- fill the burette with this solution and record the reading on the burette to one or two decimal places; the second decimal place should be a zero or a five
- Add 3-5 drops of a suitable indicator to the conical flask
- Add the solution from the burette, with swirling, until the indicator just changes colour
- Record the reading to one or two decimal places reading to the bottom of the meniscus (the second decimal place should be 0 or 5)
- Repeat the titration to achieve 2 concordant results. (within 0.2(0) cm^3 of each other)

Prepare solutions of known concentration

- calculate the required mass of the solid/liquid required to make the solution (or the required volume of liquid)
- weigh the solid/liquid in a beaker on a top pan balance (or measure the volume of the liquid using a graduated pipette/burette/measuring cylinder, as appropriate)
- dissolve the solid in a small volume (50–100 cm^3) of deionised water
- transfer the solution to the appropriately sized volumetric flask
- rinse the beaker and glass rod with deionised water and add washings to the volumetric flask
- make up to the mark by adding deionised water until the bottom of the meniscus is on the mark
- stopper the flask and invert to mix

Use chemical tests listed in 'Qualitative tests' to identify unknown substances

Flame tests

- dip the nichrome wire into concentrated hydrochloric acid
- dip the wire into the test solid
- hold the wire in a blue Bunsen burner flame
- note the colour of the flame

The test for ammonium ions
- warm the solid with a solution of sodium hydroxide
- test any gas produced with a glass rod dipped in concentrated hydrochloric acid solution or the stopper of a concentrated hydrochloric acid bottle

The test for sulfate ions
- if supplied with a solid, first make a solution of the salt e.g. a spatula measure in a test tube half-filled with deionised water
- add (1 cm^3) of barium chloride solution or barium nitrate solution

The test for halide ions
- if supplied with a solid, first make a solution of the halide salt in dilute nitric acid
- add (1 cm^3) silver nitrate solution
- add (1 cm^3) dilute ammonia solution
- add excess (5 cm^3) dilute ammonia solution
- if the precipitate remains repeat the process using concentrated ammonia solution

The test for carbonate ions
- to the solid add some dilute acid. If there is effervescence, bubble any gas produced into limewater

Test for unsaturation
- add a few cm^3 of bromine water and shake

Preparation of a halogenoalkane using the techniques of refluxing, separating with a funnel, removing acidity, drying and distillation;

Method for the preparation
- place (10 g) of sodium bromide in a pear shaped/round bottomed flask
- add (7.5 cm^3) of butan-1-ol and (10 cm^3) of water
- lower into a beaker of cold water
- add (10 cm^3) of concentrated sulfuric acid slowly and with swirling.
- add some anti-bumping granules
- gently reflux the mixture (for 30–45 minutes).
- distil off the impure bromobutane
- remove unreacted butan-1-ol by shaking with concentrated hydrochloric acid in a separating funnel
- remove the acid impurities from the impure product as described on page 4
- dry the impure product as described on page 4
- purify the product using simple distillation as described on page 4

Refluxing - a labelled diagram should include:
- the condenser in the upright position
- a flask
- a heat source
- the water flowing correctly through the condenser
- use of anti-bumping granules
- any gaps at the joints or a closed apparatus will be penalised

Distillation – a labelled diagram should include:

- the condenser in the sideward position
- a flask
- a side-arm connection/still head
- a thermometer (the bulb is opposite the exit to the condenser)
- a heat source
- the water flowing correctly through the condenser.
- an appropriate collecting vessel
- use of anti-bumping granules
- any gaps at the joints or a closed apparatus will be penalised.

Most organic compounds are flammable; instead of a Bunsen burner a water bath, sand bath or electric heating mantle can be used.

For the first distillation to separate the crude product from reaction mixture, collect the crude product distillate over a range.

For a final distillation, if the product is pure it should distil over a narrow range at the expected boiling point

Removal of acidic impurities using a separating funnel

- place the impure distillate in the separating funnel
- add a portion of sodium carbonate solution/sodium hydrogencarbonate solution.
- stopper and shake
- release the pressure at regular intervals by inverting and opening the tap
- allow to stand until the layers settle and separate, then run off the lower layer into a beaker
- decide which is the aqueous layer by referring to density or adding some water and observing which layer increases in size; discard the aqueous layer

Drying

- place the impure liquid product in a beaker or conical flask
- add a spatula of a drying agent, for example anhydrous magnesium sulfate/anhydrous sodium sulfate/anhydrous calcium chloride and swirl
- add more of the drying agent until the liquid is clear/no longer cloudy.
- decant/filter off the liquid

Prepare alcohols from halogenoalkanes using alkali

- place the halogenoalkane in a pear shaped/round bottomed flask
- add dilute sodium hydroxide solution
- add some anti-bumping granules
- heat under reflux
- distil off the alcohol

Investigate the relative rates of hydrolysis of halogenoalkanes

- use halogenoalkanes with the same chain length and place the chloroalkane, bromoalkane and iodoalkane (approximately 1 cm^3) in three separate test tubes.
- add an equal volume of ethanol (approximately 1 cm^3) to each test tube
- add equal volumes of silver nitrate solution (approximately 1 cm^3) to each test tube
- place the test tubes in a hot water bath at 50 °C and start the stopwatch

- Time how long it takes for each precipitate to form

Carry out the elimination of hydrogen halides from halogenoalkanes using ethanolic potassium hydroxide

When a liquid alkene is produced the following method may be used:
- place some (2 cm^3) ethanolic potassium hydroxide in a flask
- add some halogenoalkane (1 cm^3)
- shake and add a few anti-bumping granules
- reflux gently for 20 minutes
- set up for distillation and collect the product
- test the product with bromine water

If a gaseous alkene is produced the following method can be used:
- some sand is soaked with the liquid halogenoalkane, and placed in a boiling tube clamped horizontally
- some potassium hydroxide pellets are placed in ceramic wool soaked in ethanol, and placed in the middle of the boiling tube
- heat the halogenoalkane and the ethanolic potassium hydroxide and collect the gas produced over water
- test the gas produced with bromine water

Carry out test tube reactions of alcohols with sodium, hydrogen bromide and phosphorus pentachloride
- place 1 cm^3 of alcohol in a boiling tube
- cut a small 2 x 2 x 2 mm piece of sodium and use filter paper to remove excess oil from it
- add the piece of sodium to the alcohol and record observations
- place 1 cm^3 of alcohol in a boiling tube
- add a spatula measure of phosphorus pentachloride and record the observations

Prepare aldehydes, carboxylic acids and ketones from alcohols using acidified potassium dichromate(VI)

To prepare the carboxylic acid
- add acidified potassium dichromate(VI) solution (excess) to a pear shaped/round bottomed flask and set up for reflux
- add anti-bumping granules
- add a volume of primary alcohol slowly down the condenser from a dropping funnel, and cool in a water bath.
- remove the water bath and heat the mixture under reflux.
- rearrange the apparatus for distillation and distil off the acid

To prepare the aldehyde
- add acidified potassium dichromate(VI) solution (excess) to a pear shaped/round bottomed flask
- add anti-bumping granules
- add a volume of primary alcohol
- assemble the apparatus for distillation - this removes the aldehyde from the oxidising mixture immediately and prevents further oxidation

To prepare the ketone

- add acidified potassium dichromate(VI) solution(excess) to a pear shaped/round bottomed flask and set up for reflux
- add anti-bumping granules
- add a volume of secondary alcohol slowly down the condenser from a dropping funnel, and cool in a water bath.
- remove the water bath and heat the mixture under reflux.
- rearrange the apparatus for distillation and distil off the ketone

Determine the enthalpy changes for combustion and neutralisation using simple apparatus

Enthalpy of combustion

- accurately measure a volume (100 cm^3) of water into a calorimeter/beaker (100 cm^3 is the same as 100 g water as the density of water is 1 g/cm^3)
- weigh a spirit burner containing the liquid fuel to be burnt
- measure initial temperature of water using a thermometer (T_1).
- use the spirit burner to heat the water
- stop heating when there is a reasonable temperature rise (15 °C) . Stir and measure the final temperature (T_2) of the water using a thermometer
- reweigh the spirit burner
- calculate temperature change (ΔT) = $T_2 - T_1$ and the heat energy change in joules using $q = mc\Delta T$
- calculate mass of fuel used in the burner by subtraction, and calculate the number of moles of fuel used using moles = mass/RMM
- calculate the energy change per mole of fuel used

Enthalpy of neutralisation

- place a polystyrene cup in a glass beaker for support and further insulation and to prevent draughts
- use a measuring cylinder to measure a volume (25 cm^3) of a standard acid solution and transfer into the polystyrene cup
- stir the acid with a thermometer and record the temperature
- use a second measuring cylinder to measure a volume (25 cm^3) of a standard solution of sodium hydroxide
- add the sodium hydroxide solution to the acid and record the highest temperature reached
- calculate temperature change (ΔT) = $T_2 - T_1$ and the heat energy change in joules using $q = mc\Delta T$
- calculate the number of moles of acid used, the number of moles of water formed and the enthalpy of neutralisation

React Group II metals and other metals with oxygen, water and dilute acids and determine the masses of solids and volumes of gases produced.

Metals with steam

Diagram for reaction of magnesium and steam

- damp mineral wool
- Bunsen burner to heat mineral wool/metal
- delivery tube
- inverted measuring cylinder/burette under water to collect gas and measure volume
- trough/basin

Metals with water

Diagram for reaction of magnesium and calcium with water

- metal in contact with water
- inverted filter funnel
- inverted measuring cylinder/burette under water to collect and measure the volume of gas

Metals with oxygen

- measure the mass of a crucible and lid using a balance
- add some metal and measure the mass of the crucible, lid and metal
- place the crucible on a pipeclay triangle and heat strongly, raising the lid at intervals to allow air into the crucible
- allow the crucible to cool and weigh crucible, lid and contents
- repeat the heating, cooling and weighing until there is “constant mass”

Metals with dilute acid

Diagram

- acid in conical flask
- metal
- delivery tube
- gas syringe/inverted measuring cylinder or burette under water

The method for starting the reaction may vary for the reaction of metals with water or acids.

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