

VCE Chemistry

Unit 3 (2015)

Internal Assessment Guidelines & Unit Timeline

Table of Contents

General Statement, Resources and SAC Descriptions	pp.1-2
Unit Timeline	p.3
VCAA Key Knowledge for Unit 3	p.4
SAC 1 Task Description, Risk Assessment Proforma & Grading Sheets	pp.5-7
SAC 3 Grading Sheet & Sample Practical Report	pp.8-9
Practical Methods for SAC 1	pp.10-16
MSDS Information for SAC 1	pp.17-18
Practical Method for SAC 3	p.19

General Statement

- Every student is expected to keep up to date with all set exercises, practical investigations and SAC work.
- Preparation for the Unit 3 exam begins now!! The exercises that are set from the text book or from handouts will be considered to be the bare minimum – you are advised to extend your studies by using the text books in the library and by doing a variety of problems from other sources (**Study On is the prescribed revision title but you could also use Checkpoints or NEAP Smart Study**).
- The Areas of Study from the VCAA course description booklet are listed on p.3, you must make sure that you know how to address all of these in order to achieve a high grade in the 1st exam.
- You are not permitted a “cheat sheet” for Year 12 Chemistry exams but you are supplied with a VCAA Data Sheets – supplied as a separate handout and emailed as a PDF.

Resources

- You should have purchased **Study On** - you must start using it straight away. NEAP Smart Study, Cambridge Checkpoints A+ Study Notes, Exam Busters, Chem Notes, Chemistry at Work, Jacaranda Study Cards, Pearson Education Podcrammers are other possible resources.
- Various CD-ROMs should be available within the Science Department.
- Some Web Sites: www.vcaa.vic.edu.au/vce/studies/chemistry/chemindex.html (the VCAA chemistry index) <http://www.cea.asn.au/Frontpage/> (this is the address of the Chemistry Education Association and they offer a tertiary scholarship for a student who gets a study score of 44 or higher),

Unit 3 SAC Work

The internal assessment for Unit 3 Chemistry includes assessable tasks from the two learning “Outcomes” that are specific to the unit.

Outcome 1: Evaluate the suitability of techniques and instruments used in chemical analysis

SAC 1: Extended Practical Investigation

An **Extended Practical Investigation** on quantitative analysis of consumer products. You are to choose 3 practical investigations from the following:

Experiment 1: Gravimetric determination of sulphur as sulphate in fertiliser

Experiment 17: Volumetric determination of the nitrogen content of lawn fertiliser

Experiment 24: Determination of the iron (II) content of lawn fertiliser by redox titration

Experiment 32: Colorimetric determination of the phosphorous content of lawn fertiliser

Marks & Grading

50 marks are allocated and your final report will be graded according to the grading sheet as shown on p.11 of this booklet

Time Allowed

Practical Work: 180-300 minutes.

Report Write-up: Limited time out of class may be allocated to complete aspects of the final report writing but most of this work will be completed within class time.

Approach to Tasks

Practical work will be completed in pairs but the reports will be completed individually – it is not appropriate to have the same report, or a report that is too similar, to another student.

Detailed Task Description

See pp.10-11 of this booklet

Outcome 2: Identify and explain the role of functional groups in organic reactions and construct reaction pathways using organic molecules

SAC 2: analysis of 1st & 2nd hand data on reaction pathways

Marks & Grading

25 marks are allocated and they will be awarded according to a set of structured questions.

Time Allowed

Practicals: 80-100 minutes (this task will be conducted over a 2 period session in class). More specific details on the requirements for this assessment task will be supplied prior to it being conducted.

Detailed Task Description

Specific details of this task will be supplied closer to the assessment date

SAC 3: Experiment 56: Preparation of aspirin

Marks & Grading

25 marks are allocated and your final report will be graded according to the grading sheet as shown on p.12 of this booklet.

Time Allowed

Practicals: 80-100 minutes (the practical work and report write-up will both be completed in class)

Approach to Tasks

This task requires you to complete a formal practical report. An example of such a report is shown on p. of this booklet. Practical work will be completed in pairs but the reports will be completed individually – it is not appropriate to have the same report, or a report that is too similar, to another student.

Detailed Task Description

See p.18 of this booklet for the practical method. You will be supplied with a set of **Focus Questions** that relate to the theory of this practical that you must complete during class time and your formal report will also be completed during class time.

Braemar College Classroom Program Planner – 2015

Subject: Chemistry Unit 3

<i>Term 1: Friday 30 January – Friday 27 March</i>	<i>Term 2: Monday 13 April – Friday 19 June</i>
Tutorial Week – 24-28 November 2014 AS.1: Chemical Analysis Course Outline, Internal Assessment and Yearly Expectations Introduction to chemical analysis (qualitative vs quantitative), water content	13-17 April Organic Functional Groups: chloroalkanes, alkanols & carboxylic acids Systematic naming & functional groups
30 January Mole Review: $n = m/M_m$ and Balancing Chemical Equations, Significant Figures and % Errors, Stoichiometry Review: $c = n/V$, $pV = nRT$ and $n = V/V_m$ (including limiting reactants), Solubility, precipitation and Gravimetric Analysis, Gravimetric Analysis practical	20-24 April Organic Chemical reactions: combustion, substitution, addition, polymerisation Reactions of functional groups, Esters & condensation reactions
2-6 February Introduction to Volumetric Analysis: Primary Standards, Standard Solutions and acid/base indicators, Measures of Concentration, Acids and Bases, pH, Self Ionisation of Water	27 April-1 May Reaction Pathways SAC 2 – Analysis of 1st & 2nd Hand Data – Organic Reaction Pathways
9-13 February Correct Titration Procedure & Back Titration Redox Reactions: Oxidation Numbers, Half Equations, Redox Titration, Redox Titration and Practical Back Titration	4-8 May Biofuels Biomolecules: Fats & Carbohydrates
16-20 February Chromatography: Paper & Thin Layer Chromatography, Gas Liquid (GLC) & High Performance Liquid (HPLC)	11-15 May Biomolecules: Proteins & Enzymes, DNA Structure
23-27 February Spectroscopy: Atomic Absorption (AAS), UV-Visible, Calibration Curves, Colorimetry, Monochromators, Infrared (IR), Nuclear Magnetic Resonance (NMR)	18-22 May Organic Molecules to Medicine SAC 3 – Experiment 56: Preparation of aspirin
2-6 March Analytical Instruments Completion: Mass Spectrometer, choice of analytical instruments	25-29 May <p align="center">UNIT 4</p>
9-13 March SAC 1 – Extended Practical Investigation Holiday Work – Completion of Area of Study One study for AS.1 Topic Test Ecolinc Workshop: use of Analytical Instruments including GC, HPLC and AAS – collection of Instrumental Data	1-5 June <p align="center">UNIT 4</p>
16-20 March AS.2: Organic Chemical Pathways Alkanes & Alkenes: Structural Formulae and Naming of Hydrocarbons, Isomers Ethene: Properties, Production, Uses & Polymerisation	8-12 June <p align="center">VCAA Trial Examination</p> <p align="center">UNIT 4</p>
23-27 March <p align="center">EXEAT WEEK</p> <p align="center"><i>End of Term 1 – 2 weeks holiday</i></p>	15-19 June <p align="center">UNIT 4</p> <p align="center"><i>End of Term 2 – 3 weeks holiday</i></p>

Unit 3: Chemical pathways

Area of study 1 - Chemical Analysis

In this area of study students use a variety of analytical techniques to analyse products in the laboratory. They conduct volumetric analyses using acid-base and redox titrations and standard solutions, and carry out gravimetric analyses. They are also introduced to instrumental analytical techniques of spectroscopy and chromatography. Students review and apply their understanding of stoichiometry as they complete calculations related to their practical investigations. Students relate the operation of the analytical techniques and instruments to the chemical reactions and the chemical structures of the materials which are being analysed.

Outcome 1

On completion of this unit the student should be able to evaluate the suitability of techniques and instruments used in chemical analyses. To achieve this outcome the student will draw on key knowledge outlined in Area of Study 1 and key skills listed on page 12.

Key knowledge

- volumetric analysis including determination of excess and limiting reagents and titration curves: simple and back titrations, acid-base and redox titrations
- gravimetric analysis
- calculations including amount of solids, liquids and gases; concentration; volume, pressure and temperature of gases
- the writing of balanced chemical equations, and the application of chemical equations to volumetric and gravimetric analyses
- principles and applications of chromatographic techniques (– excluding details of instrumentation and operation –) and interpretation of qualitative and quantitative data, including use of R_f and R_t values, and the use of a calibration graph to determine amount of analyte, in thin layer chromatography (TLC), high performance liquid chromatography (HPLC) and gas chromatography (GC)
- principles and applications of spectroscopic techniques, excluding details of instrumentation, and interpretation of qualitative and quantitative data from:
 - atomic absorption spectroscopy (AAS) including electron transitions and use of calibration graph to determine amount of analyte
 - infrared spectroscopy (IR) including use of characteristic absorption bands to identify bonds
 - proton and carbon-13 nuclear magnetic resonance spectroscopy (NMR) including spin, the application of carbon-13 to determine number of equivalent carbon environments; and application of proton NMR to determine structure: chemical shift, areas under peak and peak splitting patterns, and application of $n+1$ rule to simple compounds, excluding coupling constants
 - visible and ultraviolet spectroscopy (visible-UV) including electron transitions and use of calibration graph to determine amount of analyte
 - mass spectroscopy including determination of molecular weight, molecular ion peak, and identification of simple fragments
- matching analytical technique/s to a particular task: single and combined techniques.

Area of study 2 - Organic chemical pathways

In this area of study students investigate systematic organic chemistry including production of starting materials for particular reaction pathways. Students use molecular models and conduct simple laboratory investigations to observe the properties and reactions of different homologous series and functional groups. Students investigate the use of biochemical fuels. They design reaction pathways to prepare organic compounds from given starting materials. Students investigate how forensic analysis relies on the use of organic chemicals (including DNA) and the role of organic chemicals (including proteins) in the development of medicines.

Outcome 2

On completion of this unit the student should be able to identify and explain the role of functional groups in organic reactions and construct reaction pathways using organic molecules. To achieve this outcome the student will draw on key knowledge outlined in area of study 2 and key skills listed on page 12.

Key knowledge

- structure including molecular, structural and semi-structural formulae, and International Union of Pure and Applied Chemistry (IUPAC) nomenclature of alkanes, alkenes, amines, haloalkanes, alkanols ($C_nH_{2n+1}OH$), alkanolic acids ($C_nH_{2n+1}COOH$) and esters up to C10
- common reactions of organic compounds including equations: addition reactions of alkenes (addition of hydrogen halides and water limited to symmetrical alkenes), substitution reactions of alkanes and primary haloalkanes, oxidation of primary alkanols, and esterification
- chemical bonding:
 - primary, secondary and tertiary structures of proteins
 - the role of the tertiary structure of proteins in enzyme action
 - denaturing of proteins: effect of changes in pH and temperature on bonding
- primary and secondary structure of DNA
- organic reaction pathways including appropriate equations and reagents:
 - production of esters from alkenes
 - condensation reactions that produce lipids
 - condensation and polymerisation reactions that produce large biomolecules including carbohydrates, proteins and DNA (triglycerides)
 - production of biochemical fuels including the fermentation of sugars to produce ethanol
 - function of organic molecules in the design and synthesis of medicines including the production of aspirin from salicylic acid.

Year 12 Chemistry
SAC 1: Extended Practical Investigation
Analysis of Lawn Fertiliser

Practical Investigations (methods are included in this booklet on pp.)

An **Extended Practical Investigation** on quantitative analysis of consumer products. You are to choose 3 practical investigations from the following:

Experiment 1: Gravimetric determination of sulphur as sulphate in fertiliser

Experiment 17: Volumetric determination of the nitrogen content of lawn fertiliser

Experiment 24: Determination of the iron (II) content of lawn fertiliser by redox titration

Experiment 32: Colorimetric determination of the phosphorous content of lawn fertiliser

Preparing for each practical

You need to complete risk assessments for all of your practical investigations. The proformas for these risk assessments will be supplied prior to the SAC commencing. See p.305 of Heinemann Chemistry 2 (Figure 18.15) for an example of a safety plan proforma.

Directions for each practical investigation

- You must attend the practical session with the **title, aim, method, results table** and **risk assessments** for all practical investigations pre-prepared.
- You must take “annotations” which means recording results and noting important discussion points during the practical session.

Directions for individual practical investigations

Exp.1: ♦ Answer Questions 1-7 from the practical

Exp.17: ♦ Answer Questions 1-9 from the practical

Exp.24: ♦ Answer Questions 1-4 from the practical

Exp.32: ♦ Answer Questions 1-5 from the practical

Discussion

You will write a single **Discussion** for your three practical investigations. Your **Discussion** must be able to address the grading criterion **Discussion** and it should be direct, to the point and be based upon the annotations that you have made during the practical sessions.

Conclusion

You will write a single **Conclusion** for your three practical investigations. Your **Conclusion** must be able to address the grading criterion **Conclusion**. The final section of your **Conclusion** should address the **Outcome** for **Area of Study 1: Evaluate the suitability of techniques and instruments used in chemical analysis.**

Risk Assessment Record for chemistry experiments

Activity (title/reference)				Date of activity	
Assessor (name)				Class/level	
Hazardous substance	Conc.	Amount	MSDS date	Hazards – Risk phrases	Controls –Safety phrases
Risk control for all chemical experiments	<ul style="list-style-type: none"> • Safety goggles, gloves (or wash hands after use), laboratory coats and closed shoes • Chemical labels, instructions for mixing and disposing chemicals must be followed • Obey Science Safety Rules 				
Disposal					
Other risk controls for this activity					
Assessor (signed)				Date of record	

Chemistry Unit 3
Extended Practical Investigation – Analysis of Lawn Fertiliser
 Coursework Assessment Advice

Student Name: _____

Criteria	Marks Allocated	Grading Descriptors	Grading Descriptors						Marks Awarded	Comments
			VH	H	M	L	VL	NS		
1.Laboratory Technique & Safety	10	1.Skill in obtaining appropriate experimental data								
		2.Systematic recording of experimental data in previously prepared Results tables								
		3.Appropriate application of safety procedures & completion of risk assessments								
2.Results	5	1.Processing of data into an appropriate form with suitable tables, graphs, charts and/or diagrams								
3.Interpretation & Analysis of Results	10	1.Complete and in depth responses to Practical Questions								
		2.Recognition of trends								
4.Discussion	10	1.Recognition of unreliable or invalid observations or measurements								
		2.Valid explanations made for results, including unexpected results or errors								
5.Conclusion	5	1.Relevant conclusions relating to the aim of the experiment and reflecting the results obtained								
6.Use of Scientific Language & Conventions	10	1.Accurate use of chemical terms and concepts to demonstrate understanding of qualitative observations								
		2.Accurate use of chemical symbols, equations, SI units and terminology								

Total Marks Awarded:

Grade:

Teacher's Signature: _____

Chemistry Unit 3
SAC 3: Practical Report – Preparation of aspirin
 Coursework Assessment Advice

Student Name: _____

Criterion 1 – Knowledge and application of experimental methods (8 marks)

The extent to which the report addresses the set tasks	Very High	High	Med	Low	Very Low	Not Shown
1. Skill in obtaining appropriate experimental data						
2. Appropriate application of safety procedures						

Criterion 2 – Analysis, interpretation and evaluation of experimental results and procedures (10 marks)

The extent to which the report addresses the set tasks	Very High	High	Med	Low	Very Low	Not Shown
1. Processing of data into an appropriate form with suitable tables, graphs, charts and/or diagrams						
2. Recognition of unreliable or invalid observations or measurements						
3. Accurate calculation to an appropriate number of significant figures						
4. Recognition of trends						
5. Relevant conclusions relating to the aim of the experiment						
6. Valid explanations made for results, including unexpected results or errors						

Criterion 3 – Knowledge and application of terms, concepts and relationships related to carbon chemistry in the petroleum industry or sulphuric acid production (7 marks)

The extent to which the report addresses the set tasks	Very High	High	Med	Low	Very Low	Not Shown
1. Accurate use of chemical terms and concepts to demonstrate understanding of qualitative observations						
2. Accurate use of chemical terms, concepts and equations to demonstrate understanding of quantitative aspects of reactions						
3. Accurate use of chemical symbols, SI units and terminology						

All reports should be constructed in the format shown in this sample report and they must be written in an impersonal fashion. Do not use language such as "we did ..." or "I now know ..."; take note of how this report is written! The listed **Criteria** on this page are described on the grading sheet on the next page of this handout.

Sample Practical Report in relation to *Experiment 56: Preparation of aspirin* (the method is described on the previous page of this handout)

Analysis of Aspirin Tablets

Aim: To determine the amount of active ingredient in an aspirin tablet.

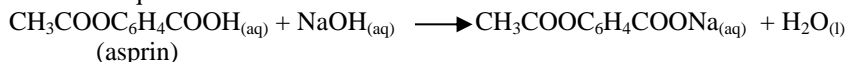
Method: As described in "Experiment 10 Analysis of Aspirin Tablets", pp.22-23, Heinemann Teacher's Resource Book, Commons et al, Heinemann.

Results: Brand: Aspro Clear, 320 mg per tablet, $c_{(\text{NaOH})}$: 0.0978 M

$V_{(\text{NaOH})}$ (titre of NaOH)

Sample	1	2	3	4
Final Burette (mL)	33.05	25.32	47.10	40.59
Initial Burette (mL)	10.93	3.47	25.32	18.76
Titre (mL)	22.12	21.85	21.78	21.83

Chemical equation:



Questions:

$$1. n_{(\text{NaOH})} = c_{(\text{NaOH})} \times V_{(\text{NaOH})}$$

Sample 2	$n_{(\text{NaOH})} = 0.0978 \text{ M} \times 0.02185 \text{ L} = 2.14 \times 10^{-3} \text{ mol}$
Sample 3	$n_{(\text{NaOH})} = 0.0978 \text{ M} \times 0.02178 \text{ L} = 2.13 \times 10^{-3} \text{ mol}$
Sample 4	$n_{(\text{NaOH})} = 0.0978 \text{ M} \times 0.02183 \text{ L} = 2.13 \times 10^{-3} \text{ mol}$

$$2. m_{(\text{aspirin})} = n_{(\text{aspirin})} \times Mm_{(\text{aspirin})} \quad n_{(\text{aspirin})} = n_{(\text{NaOH})}$$

Sample 2	$m_{(\text{aspirin})} = 2.14 \times 10^{-3} \text{ mol} \times 180 \text{ g mol}^{-1} = 0.385 \text{ g} = 385 \text{ mg}$
Sample 3	$m_{(\text{aspirin})} = 2.13 \times 10^{-3} \text{ mol} \times 180 \text{ g mol}^{-1} = 0.383 \text{ g} = 383 \text{ mg}$
Sample 4	$m_{(\text{aspirin})} = 2.13 \times 10^{-3} \text{ mol} \times 180 \text{ g mol}^{-1} = 0.383 \text{ g} = 383 \text{ mg}$

$$3. \text{Average mass per tablet} = (385\text{mg} + 383\text{mg} + 383\text{mg})/3 = 384 \text{ mg}$$

-
-
-
-
-
-
-

Discussion:

The experimentally determined value of aspirin in aspirin tablets was 384 mg per tablet. This result is significantly higher than the manufacturer's specified amount of 320 mg. Sources of error that may have led to this higher than expected value include: missing the end-point of the titration (the determination of the "pink tinge" colour change at the end-point of the titration was highly subjective and a higher $V_{(\text{NaOH})}$ would lead to a higher than expected $n_{(\text{NaOH})}$, $n_{(\text{aspirin})}$ and therefore $m_{(\text{aspirin})}$), the concentration of NaOH was lower than the specified 0.0978 M (if $c_{(\text{NaOH})}$ was lower than 0.0978 M this would lead to a higher $V_{(\text{NaOH})}$ value for the calculations of $n_{(\text{NaOH})}$, $n_{(\text{aspirin})}$ and $m_{(\text{aspirin})}$ – all would be higher than expected if the figure 0.0978 M was used during calculation), uncertainties in the instruments used ($\pm 0.002 \text{ g}$ for the electronic balance & $\pm 0.04 \text{ mL}$ for the burette) and as discussed in Q.4 the manufacturer's specified content per tablet may be a minimum value.

Conclusion:

The aspirin content of aspro clear tablets was determined through using the technique of volumetric analysis. It was found that each tablet contained 384 mg of aspirin and that this value was significantly higher than the manufacturer's specification of 320 mg per tablet. The discrepancy between the experimentally determined value and that of the manufacturer was attributed to the random experimental errors of titrating beyond the end-point and being supplied with an incorrectly labelled sample of NaOH (the concentration may have been lower than the labelled 0.0978 M), the systematic errors of the instruments used (uncertainty of $\pm 0.002 \text{ g}$ for the electronic balance & $\pm 0.04 \text{ mL}$ for the burette) and to the possibility that the manufacturer's specification is a minimum value per tablet.

Aim & Method are directly taken from the supplied method sheet. The **Method** should include as complete a reference as possible and it should have text added to it if you alter the method in any way (**Criterion 2.7**).

Results must be set out as clearly as possible (using tables can help). Note that you must also use the appropriate units of measurement for all listed values eg. mg, L etc (**Criteria 1.3, 2.1, 3.2 & 3.4**).

Calculations may appear in the **Questions** of the practical method or they may be included in the **Results**. Note that the values for n and m are listed to 3 significant figures ie. the level of accuracy is bound by the least accurate information supplied ($c_{(\text{NaOH})} = 0.978 \text{ M}$ – this value has 3 significant figures). Note that all values have the correct units of measurement listed and that they are accurate and consistent. (**Criteria 2.3, 3.2 & 3.4**)

General Questions are normally included in the practical methods to test your knowledge of the content of the unit of study ie. the Key Knowledge and Skills. (**3.1, 3.2 & 3.4**)

Discussion evaluates aspects of the practical that are significant such as "error" and recognising that the results obtained did not match what was expected. You must be specific in this section (note the example supplied), and you must not write things such as "the results were wrong because we didn't turn the burette tap off properly" – this implies that your technique is poor. (**Criteria 2.2, 2.6, 2.7, 3.1, 3.2 & 3.4**)

Conclusion addresses the **Aim**, states what the major findings of the practical were, states how the results were obtained and states the factors that introduced uncertainty into the practical and contributed to the final result. The **Conclusion** must not introduce any new material and it must be direct and concise. (**Criteria 2.4, 2.5, 3.1, 3.2 & 3.4**)

GENERAL POINTS: all reports should be easy to digest (even for a person who isn't familiar with the practical) and they should follow a logical path to a **Conclusion** that is consistent with all the information supplied. Criteria 1.1-1.4 relate to laboratory technique and they will be graded during each practical session.

1 Experiment

Gravimetric determination of sulfur as sulfate in fertiliser

This experiment is also included in *Heinemann Chemistry 2 Student Workbook* as Sample assessment task 01 Analysis of fertiliser—an extended experimental investigation, Practical activity A.

■ Purpose

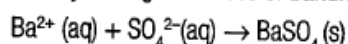
To find the percentage of sulfur (as sulfate) present in a brand of commercial fertiliser.

■ Procedure

- 1 Weigh out accurately about 1.0 g of the finely ground fertiliser into a 100 mL beaker. Record the mass and, if a commercial fertiliser is being analysed, record the brand and its sulfur content as specified by the manufacturer.
- 2 Add 50 mL de-ionised water and stir to dissolve as much of the sample as possible. Filter the mixture into a 600 mL beaker, washing the residue several times using de-ionised water.
- 3 Add about 3 mL 2 M hydrochloric acid to the filtrate and add more water so that the total volume is about 200 mL. Heat the solution until it boils.
- 4 Add 15 mL 0.5 M barium chloride solution drop by drop from a burette to the hot solution, stirring continuously. A white precipitate of barium sulfate will form.
- 5 Boil the mixture for a further minute, then remove it from the heat and allow the precipitate to settle. Ensure no sulfate ions remain in the solution by adding several more drops of barium chloride solution. If more precipitate forms, add 3 mL barium chloride solution and test again for unreacted sulfate ions. *If desired, the mixture can be left to stand overnight at this stage.*
- 6 Weigh a glass filter crucible fitted with filter paper.
- 7 Collect the precipitate in the glass filter crucible using gentle vacuum filtration. (Filtration is faster if most of the liquid is filtered before the bulk of the precipitate is collected in the crucible.) Use about 10 mL warm de-ionised water to wash any precipitate remaining in the beaker into the crucible.
- 8 Collect the last drops of filtrate in a 100 mL beaker and test for the presence of chloride ions by adding a few drops of silver nitrate solution to the filtrate. If the solution becomes cloudy, wash the precipitate with a further 10 mL warm water and repeat the test.
- 9 Place the crucible and contents in an oven heated to 100–110°C and leave overnight.
- 10 Weigh the crucible and contents and record the mass.

■ Theory

This practical activity determines the amount of sulfur present in a fertiliser in the form of sulfate ions. The sulfate is precipitated as barium sulfate from a solution containing a known mass of the fertiliser by adding an excess of barium chloride solution:



The proportion of sulfate ions and therefore of sulfur in the fertiliser is determined by collecting and weighing the precipitate that is formed during the above reaction.

This exercise is an example of gravimetric analysis, a technique which is described further in *Heinemann Chemistry 2*, Chapter 2.

■ Duration


70 minutes, over two or three days

■ Materials

- 1.0 g fertiliser*, finely ground using a mortar and pestle
- 3 mL 2 M hydrochloric acid
- 5 mL 0.1 M silver nitrate solution
- 20 mL 0.5 M barium chloride solution
- 200 mL de-ionised water
- 20 mL warm de-ionised water
- 2 × 100 mL beakers
- 600 mL beaker
- 10 mL measuring cylinder
- burette and stand
- filter funnel
- filter paper
- vacuum flask and vacuum pump (water-jet type)
- glass filter crucible (No. 4 porosity) and rubber adaptor**
- stirring rod
- wash bottle containing de-ionised water
- mortar and pestle
- Bunsen burner, tripod stand and gauze mat
- bench mat
- electronic balance
- oven
- safety glasses

* 100 g of a suitable lawn food fertiliser for all analyses in this experimental investigation can be made by mixing 100 g NH_4SO_4 , 5.4 g KH_2PO_4 and 1.5 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. *Yates Thrive Granular Lawn Food* or *Yates Gro-Plus Lawn Food* are suitable commercial alternatives.

** A Gooch crucible, rubber adaptor and filter paper to suit may be used instead. Although the Gooch crucible is less expensive, filtering often takes longer. Filtration using a glass filter crucible usually takes only minutes.



Safety

- Wear safety glasses and a laboratory coat for this experiment.
- Barium chloride solution is harmful if swallowed. Wash hands after use.
- Silver nitrate solution stains skin and clothing.
- Heated solution and hot crucible may cause burns.
- Hydrochloric acid is corrosive.

Fertilisers provide plants with a source of a number of elements that are necessary for them to grow, including nitrogen, phosphorus, potassium and sulfur. Plants do not require all these elements to the same extent and the needs of different plants vary. The composition of different fertilisers is therefore of interest to farmers and other consumers who wish to ensure that the fertilisers they purchase represent good value and are suitable for particular applications.

■ Questions

Calculations

- 1 Determine the mass of the barium sulfate precipitate obtained.
- 2 Find the mass of sulfate ions present in the barium sulfate precipitate.
- 3 Determine the percentage by mass of sulfate in the fertiliser.
- 4 Determine the percentage by mass of sulfur (as sulfate) in the fertiliser.

General questions

- 5 How could the results obtained be affected if:
 - a the mixture in step 5 of the procedure were not tested with more barium chloride solution?
 - b the filtrate were not tested with silver nitrate solution when the precipitate was washed in step 8?
- 6 What sources of error might arise in a gravimetric analysis such as this?
- 7 If you analysed a commercial fertiliser, how well does your result for the percentage of sulfur (as sulfate) present agree with the manufacturer's specification? Try to account for any difference.

Volumetric determination of the nitrogen content of lawn fertiliser

This experiment is also included in *Heinemann Chemistry 2 Student Workbook* as Sample assessment task 01 Analysis of fertiliser—an extended experimental investigation, Practical activity B.

Purpose

To find the percentage by mass of available nitrogen, present as the ammonium ion, in a lawn fertiliser.

Procedure

- Record the brand of fertiliser to be analysed and its nitrogen content as specified by the manufacturer.
- Accurately weigh about 1.3 g of fertiliser using a weighing bottle.
- Using a small funnel, carefully transfer the sample of fertiliser to a 250 mL volumetric flask. Rinse the weighing bottle with a little de-ionised water and add this to the contents of the volumetric flask. Add more water until the flask is almost half full, stopper, and swirl the contents to dissolve the ammonium compounds in the fertiliser. (Some compounds with low solubilities may also be present in the fertiliser.)
Add more water to make up the volume of solution in the volumetric flask to the calibration line. Restopper the flask and shake to mix thoroughly.
- Using a 20 mL pipette, dispense aliquots of the fertiliser solution into each of three 250 mL conical flasks.
- Thoroughly wash the pipette, and rinse it with a small volume of standard sodium hydroxide solution. Place a 20.00 mL aliquot of sodium hydroxide solution in each of the flasks containing fertiliser solution. Record the concentration of the standard solution of sodium hydroxide.
- To one of the flasks, add 50 mL de-ionised water. Boil the mixture for about ten minutes. If necessary, add more water to maintain a constant volume of solution while boiling. Test the vapour at the neck of the flask with a moist strip of red litmus paper. If ammonia is present the paper will turn blue and heating should be continued until the point where litmus paper does not detect the presence of ammonia. When this point has been reached, cool the flask under running water.
- Repeat step 6 with each of the other two conical flasks.
- Rinse and fill a burette with standard hydrochloric acid. Record the concentration of the acid and the initial burette reading.
- Add two to three drops of methyl red indicator to each conical flask containing the fertiliser mixture, and titrate. The end point occurs when the initially yellow solution becomes orange. For each flask, record the burette reading once the end point has been reached.

Theory

The NH_4^+ ion present in fertilisers is too weak an acid to give a sharp end point in direct titrations. The NH_4^+ ion is therefore reacted with an excess of sodium hydroxide solution and the amount of unreacted sodium hydroxide found by titration with hydrochloric acid solution. The amount of NH_4^+ ion present can then be deduced from the amount of sodium hydroxide that reacted with it. A titration of this type is known as a back titration. Back titrations are described further in *Heinemann Chemistry 2*, Chapter 4.

Nitrogen for plant growth is supplied in most common fertilisers in the form of both NH_4^+ and NO_3^- ions. In this experiment only the NH_4^+ ion content of a fertiliser is determined.

Duration

90 minutes

Materials

- 1.3 g lawn fertiliser, finely ground
- 100 mL standard sodium hydroxide solution (approximately 0.1 M)
- 100 mL standard hydrochloric acid (approximately 0.1 M)
- methyl red indicator
- 500 mL de-ionised water
- wash bottle containing de-ionised water
- red litmus paper
- 2 × small funnels
- 250 mL volumetric flask
- 3 × 250 mL conical flasks
- 20 mL pipette
- pipette filler
- 50 mL measuring cylinder
- burette and stand
- weighing bottle or watch glass
- spatula
- white tile
- Bunsen burner, tripod stand and gauze mat or hot plate
- bench mat
- safety glasses

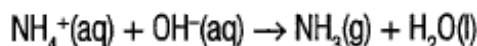
Safety

- Wear safety glasses and a laboratory coat for this experiment.
- Sodium hydroxide solution is an irritant to skin and eyes.
- Methyl orange may cause irritation to the skin and eyes. Avoid contact.
- Boiling solutions may cause burns.

■ Questions

Calculations

- 1 Using the concentration of the standard solution of sodium hydroxide, calculate the amount of sodium hydroxide, in mol, in each 20.00 mL aliquot added to the fertiliser before heating.
- 2 Ammonium compounds in the fertiliser react with the sodium hydroxide according to the equation:



After heating, all of the ammonium ions will have reacted and the amount of unreacted hydroxide ions can be determined by titrating the solution with standard hydrochloric acid.

- a Write an equation for the reaction of sodium hydroxide with hydrochloric acid.
 - b Calculate the average titre of hydrochloric acid.
 - c Use the concentration of the hydrochloric acid and the average titre to calculate the amount of hydrochloric acid consumed in the titration.
 - d Find the amount, in mol, of sodium hydroxide that did not react with fertiliser solution in each conical flask.
- 3 Calculate the amount, in mol, of sodium hydroxide that reacted with the fertiliser solution in each flask.
 - 4 Find the amount of NH_4^+ ions present in each 20.00 mL aliquot of fertiliser solution.
 - 5 Calculate the amount, in mol, of NH_4^+ ions that were originally present in the 250 mL volumetric flask.
 - 6 Find the mass of nitrogen present in the sample of fertiliser.
 - 7 Calculate the percentage by mass of nitrogen in the sample of fertiliser.

General questions

- 8 How well does your result for the percentage of nitrogen available as an ammonium compound agree with the manufacturer's specification? Account for any difference.
- 9 Inspect the label of a commercial fertiliser packet. What ammonium compounds are present in the fertiliser?

24 Experiment

Determination of the iron(II) content of lawn fertiliser by redox titration

This experiment is also included in *Heinemann Chemistry 2 Student Workbook* as Sample assessment task 01 Analysis of fertiliser—an extended experimental investigation, Practical activity C.



Purpose

To design and perform a redox titration analysis to determine the iron content (as water-soluble iron(II) ions) of a lawn fertiliser.

Duration

50 minutes

1 For the reaction of iron(II) with permanganate ions, write:

- a half equation for the oxidation reaction
- a half equation for the reduction reaction
- the overall equation.

2 Approximately 20 mL 1 M sulfuric acid should be added to the fertiliser solution before it is titrated with the potassium permanganate solution. Explain why the acid is added.

3 Suppose you are supplied with a standard solution of approximately 0.01 M potassium permanganate solution. If the fertiliser contains about 0.3% iron by mass in the form of a soluble iron(II) compound, calculate the mass of fertiliser that would have been used if a titre of about 10 mL potassium permanganate solution is required to reach the end point.

4 Inspect a solution of potassium permanganate.

- Explain why an indicator is not required when a redox titration is performed using potassium permanganate solution in the burette.
- Suggest why it is usual practice to record the volume of potassium permanganate solution in a burette by reading from the top, rather than from the bottom, of the meniscus.

Procedure

- Weigh approximately 10.000 g of fertilizer into a 100 mL beaker, record the exact mass, add 20 mL of 1 M H₂SO₄ and dissolve the fertiliser.
- Filter the solution into a 100 mL volumetric flask, wash the 100 mL beaker and solid residue in the filter paper with de-ionised water.
- Discard the solid waste.
- Add de-ionised water and make up to 100 mL calibration line in the volumetric flask.
- Rinse a 250 mL beaker with a small sample of the fertilizer solution and then pour the fertiliser solution into a 250 mL beaker.
- Rinse the pipette with a small amount of the fertilizer solution and then pipette 25.00 mL of the fertilizer solution into 3 100 mL conical flasks. **Note: no indicator is necessary because MnO₄⁻ (purple) —→ Mn²⁺ (colourless). The end point of this titration occurs when there is excess MnO₄⁻ and the mixture in the conical flask first retains a purple tinge.**
- Rinse and fill the burette with 0.01 M KMnO₄.
- Titrate the 3 fertiliser solution samples.

Theory

Iron is one of the minerals required by living things. In this experiment the iron is present as iron(II) ions. The concentration of iron(II) ions in a solution can be determined by titration against standard potassium permanganate (KMnO₄) solution. In this reaction, the iron(II) ions are oxidised to iron(III) ions and the permanganate ions are reduced to manganese(II) ions.

Questions

- Calculate the iron content of the fertiliser as a percentage by mass. Clearly set out your working.
- How could the results obtained be affected if the fertiliser contained other chemicals that can be oxidised by permanganate ions?
- What sources of error might arise in a redox analysis such as this?
- If you analysed a commercial fertiliser, how well does your result for the percentage of iron present agree with the manufacturer's specification? Try to account for any difference.

Materials

- Fertiliser containing iron(II)
- 0.01 M potassium permanganate solution
- 1 M sulfuric acid

Safety

- Wear a laboratory coat, safety glasses and gloves.
- Fertiliser is harmful if swallowed.
- Potassium permanganate solution is harmful if swallowed
- Dilute sulfuric acid is corrosive to eyes and skin. Avoid contact.

32 Experiment

Colorimetric determination of the phosphorus content of lawn fertiliser

This experiment is also included in *Heinemann Chemistry 2 Student Workbook* as Sample assessment task 01 Analysis of fertiliser—an extended experimental investigation, Practical activity D.

Purpose

To determine the phosphorus content of a lawn fertiliser by colorimetric analysis.

Procedure

Part A—Preparation of solutions

If the fertiliser contains about 1.3% phosphorus in the form of water-soluble phosphate, calculate the approximate mass of fertiliser required to prepare the 250 mL solution.

- 1 If a commercial fertiliser is to be analysed, record the brand and its phosphorus content (as water-soluble phosphate) as specified by the manufacturer. Accurately weigh about 0.0310 g of finely ground fertiliser into a 100 mL beaker and record the mass.
- 2 Add about 20 mL hot water and stir to dissolve the powder.
- 3 Transfer the mixture to a 250 mL measuring cylinder, using water to rinse into the cylinder traces of fertiliser solution left in the beaker. Add water to the cylinder to make 250 mL of solution. Transfer the solution to a 600 mL beaker and stir to ensure the concentration is uniform.
- 4 Dilute the fertiliser solution by a factor of 10 by pouring 25 mL of the fertiliser solution into a clean 250 mL measuring cylinder and adding water to make 250 mL of solution. Mix well. Discard the remainder of the fertiliser solution.
- 5 Label six test-tubes as follows: '10.0 mg L⁻¹', '7.5 mg L⁻¹', '5.0 mg L⁻¹', '2.5 mg L⁻¹', '0.0 mg L⁻¹' and 'unknown'.
- 6 Place 20 mL of the diluted fertiliser solution prepared in step 4 into the test-tube labelled 'unknown'.
- 7 Use a 10 mL measuring cylinder to add standard phosphate solution and water to the labelled test-tubes according to Table 32.1.

Table 32.1 Phosphate solution standard dilutions

Test-tube label (mg L ⁻¹)	Volume of 10 mg L ⁻¹ standard phosphate solution (mL)	Volume of water (mL)
10.0	20	0
7.5	15	5
5.0	10	10
2.5	5	15
0.0	0	20

- 8 Add 2 mL ammonium molybdate reagent and a few crystals of ascorbic acid to each of the six test-tubes. Stir each test-tube to dissolve the crystals.
- 9 Place the six test-tubes in a 600 mL beaker containing 200 mL boiling water. Heat for five minutes. On heating, the solutions should turn blue. Remove all test-tubes at the same time and allow to cool. (Heating time affects the intensity of colour obtained, so it is important that each tube be heated in exactly the same way for the same length of time.)

Duration

40 minutes

Materials

- fertiliser powder, finely ground
- 12 mL ammonium molybdate reagent (see *Part 3 Teacher's Guide* for preparation details)
- 50 mL 10.0 mg L⁻¹ standard phosphate solution (see *Part 3 Teacher's Guide* for preparation details)
- ascorbic acid (Do not use the calcium salt.)
- 20 mL hot water
- 2 × 10 mL measuring cylinders
- 25 mL measuring cylinder
- 2 × 250 mL measuring cylinders
- 100 mL beaker
- 2 × 600 mL beakers
- 6 × large test-tubes
- test-tube rack
- test-tube holder
- stirring rod
- marking pen
- Bunsen burner
- bench mat
- electronic balance
- colorimeter and data collection system (optional)
- safety glasses
- gloves

Safety

- Wear safety glasses and a laboratory coat for this experiment.
- Ammonium molybdate reagent causes severe burns. Harmful in contact with skin or if swallowed. Irritant to respiratory system, eyes and skin.

Weigh approx. 0.0500 g and accurately record the mass.

No dilution is necessary – ignore step 4.

- 10 Compare the colour of the solution in the test-tube labelled 'unknown' with the colour of the standards and so estimate the concentration of phosphate in the diluted fertiliser solution.

Part B —Use of colorimeter and electronic data collection equipment

A colorimeter (and electronic data collection device if available) can be used to improve the accuracy of this analysis.

- 1 Fill a cell to three-quarters of its volume with the 0 mg L⁻¹ solution and wipe the outside of the cell with a tissue. By following the manufacturer's instructions, calibrate the colorimeter to read zero transmittance when no light passes through the cell and 100% transmittance when yellow light (565 nm) passes through the cell. Use yellow light for the remainder of this experiment.
- 2 Discard the liquid from the cell, rinse the cell twice with the 2.5 mg L⁻¹ solution, and fill the cell to three-quarters of its volume with this solution. Measure the absorbance and record the results in a table similar to Table 32.2. In a similar fashion, measure the absorbance of the other standard solutions.
- 3 Construct a graph of absorbance against concentration (using data collection software if possible). Because absorbance is directly proportional to concentration, you can draw a straight line of best fit through the data points and passing through the origin.
- 4 Measure the absorbance of the solution of unknown concentration and determine, using the graph, the concentration of phosphate ions in this sample.

Table 32.2

Standard (mg L ⁻¹)	Absorbance
10.0	
7.5	
5.0	
2.5	
0.0	

Theory

When a solution containing phosphate ions is added to a solution of ammonium molybdate reagent, an intense blue compound is formed. Ammonium molybdate causes phosphates to be converted to phosphomolybdate. Phosphomolybdate is then reduced by addition of ascorbic acid. The intensity of colour of the blue compound that develops is dependent on the concentration of the phosphate in the original solution. The reaction is not instantaneous. The blue reaches its most intense shade after about 10 minutes, after which time it gradually fades.

By comparing the intensity of the colour of a sample solution that has an unknown phosphate concentration with the colours of a series of standard solutions, the concentration of an unknown solution can be estimated. This analytical technique is known as colorimetry.

Questions

- 1 The initial 250 mL of fertiliser solution was diluted 10 times before being analysed. What is the concentration of phosphorus ions, in mg L⁻¹, in the initial 250 mL of fertiliser solution?
- 2 Use your answer to Question 1 to find the mass of phosphate ions in the initial 250 mL solution. This is the same as the mass of phosphorus in the sample of fertiliser.
- 3 Calculate the percentage of phosphorus, by mass, in the fertiliser sample.
- 4 If you analysed a commercial fertiliser, how well does your result for the percentage of phosphorus present agree with the manufacturer's specification? Try to account for any difference.
- 5 If this analysis was performed using a colorimeter, yellow light would be used to measure the absorbance of solutions. Why is blue light not used?

No dilution so $C_{(\text{PO}_4^{3-})}$ is the same as the result on the bottom of the computer readout.

phosphate

phosphate

MSDS Information & Safety Information**Experiment 1: Gravimetric determination of sulphur as sulphate in fertiliser**

Chemical	Risk Phrases	Safety Phrases	Disposal
2M HCl	R23 Toxic by inhalation R35 Causes burns	S1/2 Keep locked up/ Keep out of reach of children. S9 Keep container in a well ventilated place S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S36/37/39 Wear suitable protective clothing./ Wear suitable gloves/ Wear eye/face protection S45 In case of accident or if you feel unwell, seek medical advice immediately (show label whenever possible).	Return to lab technicians
0.1M AgNO ₃	Not Hazardous	Not Hazardous	Return to lab technicians
0.5M BaCl ₂	R22 Harmful if swallowed.	S1/2 Keep locked up/ Keep out of reach of children. S45 In case of accident or if you feel unwell, seek medical advice immediately (show the container label where possible)	Return to lab technicians
BaSO _{4(s)}	No information	No information	Return to lab technicians
Solid Fertiliser Waste	Not Hazardous	Not Hazardous	Discard solid waste into rubbish bin

Experiment 17: Volumetric determination of the nitrogen content of lawn fertiliser

Chemical	Risk Phrases	Safety Phrases	Disposal
0.1M HCl	R34 Causes burns	S1/2 Keep locked up/ Keep out of reach of children. S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S36/37/39 Wear suitable protective clothing./ Wear suitable gloves/ Wear eye/face protection S45 In case of accident or if you feel unwell, seek medical advice immediately (show label whenever possible).	Wash down the sink with a large amount of water
0.1M NaOH	Not Hazardous	Not Hazardous	Wash down the sink with a large amount of water
Solid Fertiliser Waste	Not Hazardous	Not Hazardous	Discard solid waste into rubbish bin

Experiment 24: Determination of the iron (II) content of lawn fertiliser by redox titration

Chemical	Risk Phrases	Safety Phrases	Disposal
0.1M KMnO ₄	Not Hazardous	Not Hazardous	Return to lab technicians
1M H ₂ SO ₄	R36/38 irritating to eyes & skin	S1/2 Keep locked up/ Keep out of reach of children. S9 Keep container in a well ventilated place S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S30 Never add water to this product S45 In case of accident or if you feel unwell, seek medical advice immediately (show label whenever possible).	Wash down the sink with a large amount of water
Solid Fertiliser Waste	Not Hazardous	Not Hazardous	Discard solid waste into rubbish bin

Experiment 32: Colorimetric determination of the phosphorous content of lawn fertiliser

Chemical	Risk Phrases	Safety Phrases	Disposal
Ammonium molybdate (H ₂₄ Mo ₇ N ₆ O ₂₄)	R22- Harmful if swallowed R36/37/38 Irritating to eyes/Irritating to respiratory system/ Irritating to skin R41- Risk of serious damage to eyes.	S2- Keep out of the reach of children. S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S39- Wear eye/face protection. S46- If swallowed, seek medical advice immediately and show this container or label.	Return to lab technicians
KH ₂ PO ₄	No info	No info	Return to lab technicians
Ascorbic acid	Not hazardous	Not hazardous	Return to lab technicians
Solid Fertiliser Waste	Not Hazardous	Not Hazardous	Discard solid waste into rubbish bin

Preparation of aspirin

This experiment is also included in *Heinemann Chemistry 2 Student Workbook* as Sample assessment task 01 Preparation of aspirin—written report of a practical activity.

Purpose

To prepare aspirin, which is an ester of pharmaceutical importance, and determine the percentage yield.

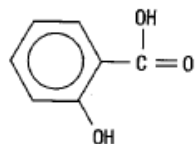
Procedure

- 1 Place a weighed mass of about 3 g salicylic acid in a conical flask.
- 2 Record the exact mass.
- 3 In a fume cupboard, add 10 mL acetic anhydride and 1 mL concentrated sulfuric acid to the flask. Swirl the mixture, without external heating, until all the solid has dissolved.
- 4 Allow the mixture to cool and then add 100 mL ice-water. The resulting oil should be 'scratched' with a glass rod to induce crystallisation. (Alternatively, a seed crystal of aspirin could be added.)
- 5 When crystallisation is complete, collect the solid by vacuum filtration in a Buchner funnel and wash with ice-water.
- 6 Allow the crystals to dry and then weigh them, recording your result.

Theory

In the chemical preparation of aspirin, an ester functional group is formed when salicylic acid reacts with acetic anhydride $\text{CH}_3\text{COOCOCH}_3$. Ethanoic acid is a by-product of this reaction (Figure 56.1). Aspirin (acetylsalicylic acid) is considered to be the world's most widely used pain killer. Refer to *Heinemann Chemistry 2*, Chapter 14 for more information about the synthesis of aspirin.

Salicylic acid



Acetylsalicylic acid (aspirin)

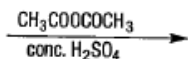
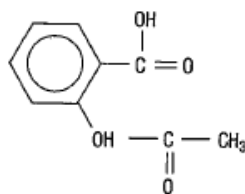


Figure 56.1 Preparation of aspirin

Questions

- 1 Sulfuric acid is a catalyst in the reaction of salicylic acid with acetic anhydride.
 - a How would the sulfuric acid affect the yield of aspirin at equilibrium?
 - b How might you prove that sulfuric acid acts as a catalyst, rather than a reactant, in this reaction?
- 2 Draw the structural formula of aspirin. Indicate and name two functional groups present in the molecule.
- 3 A commercial *Aspro* tablet contains 300 mg of aspirin. Calculate the equivalent number of tablets that you could make from the aspirin produced in this experiment.
- 4 Assuming the salicylic acid is the limiting reactant in your synthesis of aspirin, calculate the maximum mass of aspirin that could be theoretically obtained using the original 3 g salicylic acid.
- 5 Use your answer to Question 4 to calculate the percentage yield of aspirin for this experiment (the mass actually obtained, expressed as a percentage of the maximum possible mass).

Duration

40 minutes, plus 5 minutes to weigh the sample the following day

Materials

- 3 g salicylic acid
- 10 mL acetic anhydride
- 1 mL conc. sulfuric acid
- 250 mL conical flask
- 10 mL measuring cylinder
- filter flask
- large watch glass
- glass rod
- Buchner funnel
- filter paper
- 130 mL ice-water
- electronic balance
- disposable gloves
- safety glasses

Safety

- Wear safety glasses, a laboratory coat and gloves for this experiment.
- Concentrated sulfuric acid. Extremely corrosive; causes severe burns. Highly toxic. Harmful by inhalation, ingestion and skin contact.
- Acetic anhydride: corrosive; causes burns.
- Salicylic acid. Harmful by inhalation and skin absorption. Irritant.
- Dilute solution of ethanoic acid is an irritant to skin, eyes and respiratory system. Concentrated solution is corrosive and causes severe burns.
- Ethanol and ethanoic acid are flammable. Do not use near a naked flame.
- **Do not ingest the aspirin prepared in this experiment.**