EXPERIMENT FEST

HANDBOOK
CHEMISTRY

THE UNIVERSITY OF
NEWCASTLE
AUSTRALIA

newcastle.edu.au/experimentfest

INTRODUCTION

Experiment Fest is an experiment program designed to provide enriching educational experiences for senior high school students who are studying Physics, Chemistry, Biology, Earth and Environmental Science, and Food Science.

Experiment Fest is supported by the University of Newcastle's College of Engineering, Science and Environment, and takes place at both the Callaghan and Ourimbah (Central Coast) campuses of the University of Newcastle.

All experiments are complemented by notes, follow-up discussions and questions to enhance your learning experience.

CONTENTS

WELCOME

Welcome to the College of Engineering, Science and Environment at the University of Newcastle.

Experiment Fest is a wonderful chance to give you practical experience which complements your classroom learning while giving you a first-hand look at University life and facilities. Science is an exciting field of study, allowing you to move with the times and contribute actively and responsibly to society. There are many education opportunities in science after high school. Here in the College we provide study and research programs in fast-moving modern fields that make our world work.

The College staff and students who will be taking you through the experiments today are involved in contemporary science research. Please ask questions and utilise your time with them.

Take this day to enjoy being out of the classroom, exploring science with fellow students and participating in valuable experiments and discussions which will help you in your HSC and beyond.

I wish you well in your studies. I hope you apply yourselves to the learning process with enthusiasm and you enjoy your time at the University. We hope to see you studying with us in the future!

Best wishes,

Professor Craig Simmons

Pro Vice-Chancellor
College of Engineering, Science and Environment
The University of Newcastle

STUDYING CHEMISTRY

WHY STUDY CHEMISTRY?

Chemistry is truly the "central science". It is the science of the molecular scale, and it is at the molecular level where major advances are being made in many diverse areas such as medicine, drugs, nanotechnology, new materials, and the environment. A sound knowledge of chemistry is required to fully understand many other areas of science, and this is why the study of chemistry is either compulsory or recommended as part of many degree programs at the University. Chemistry opens the door for many careers because training in chemistry is essential for many positions in industry, is highly desirable for science teaching, and is useful for careers in the public service and management. Both the public and the private sectors increasingly draw their higher management echelons from chemistry graduates.

But, most importantly, it is just so fascinating! If you want to understand the workings of the world around you, then chemistry is for you!

OPPORTUNITIES FOR FURTHER STUDIES IN CHEMISTRY

The Bachelor of Science degree program at the University of Newcastle provides a foundation of knowledge, skills and attributes that allows graduates to be employable not just today but into the future and to contribute actively and responsibly to society.

Majoring in Chemistry, you have the opportunity to sample and/or specialise in any one of the following:

- Analytical Chemistry
- Environmental Chemistry
- Medicinal Chemistry
- Organic Chemistry
- Chemistry of Advanced Materials

RESEARCH IN CHEMISTRY AT THE UNIVERSITY OF NEWCASTLE

There are various groups here at the University which are committed to research in chemistry. Groups include:

- Analytical and Environmental Chemistry Electrochemical Technology
- Marine Natural Products and Chemical Ecology
- Medicinal Chemistry and Chemical Biology
- Nanostructured Materials, Polymers and Applications
- Surface and Colloid Chemistry
- Molecular Self-Assembly

CAREERS IN CHEMISTRY

The College of Engineering, Science and Environment care about our students and are interested in giving as much direction as possible to those making career choices and beyond.

The possible career paths listed below include a range of opportunities for graduates at degree, honours, and post graduate study levels.

- Analytical Chemist
- Clinical Research Coordinator
- Developmental Chemist
- Environmental Chemist
- Energy Technologist
- Forensic Chemist
- Geochemist
- Industrial/Production Chemist
- Laboratory Manager
- Laboratory/Research Assistant
- Meteorologist
- Organic/Synthetic Chemist
- Pharmaceutical/Medicinal Chemist
- Reproductive Medicine/IVF Chemist
- Research Scientist
- Science Information/Education Officer
- Science/Chemistry Teacher
- Sciences Technician
- Scientific Patent Attorney/Technical Advisor
- Scientific Policy Officer
- Scientific Writer

For more information on the College of Engineering, Science and Environment, check out our website:

newcastle.edu.au/college/engineering-science-environment

For more information on our degrees visit:

newcastle.edu.au/study

FOR MORE INFORMATION VISIT NEWCASTLE.EDU.AU

EXPERIMENT 1: ANALYSIS OF SODIUM IN SPORTS DRINK BY ATOMIC ABSORPTION SPECTROSCOPY

EXTRACT FROM HSC SYLLABUS

The identification and analysis of chemical is of immense importance in scientific research, medicine, environmental management, quality control, mining and many other fields.

Students investigate a range of methods used to identify and measure quantities of chemicals. They investigate and process data involving the identification and quantification of ions present in aqueous solutions. This is particularly important because of the impact of adverse water quality on the environment.

Analyse the need for monitoring the environment. Conduct investigations and/or process data to determine the concentration of coloured species and/or metal ions in aqueous solution, including atomic absorption spectroscopy.

Atomic Absorption Spectroscopy (AAS) allows the detection of very small concentrations from samples of air, water, or food. This activity depends on your ability to manipulate data and dilution factors. The absorbance values obtained using solutions of known concentration enable you to draw a calibration graph. Use the specific absorbance data provided to read off the corresponding concentration from the sample.

RISK ASSESSMENT

LONG PANTS AND ENCLOSED SHOES ARE COMPULSORY FOR ENTRY INTO THE LABORATORY.

SAFETY GLASSES AND A LABORATORY COAT MUST BE WORN AT ALL TIMES DURING THE LABORATORY SESSION. THESE WILL BE PROVIDED BY THE UNIVERSITY.

HAZARD	SUBSTANCE, APPARATUS, PROCEDURE	PRECUATION/ACTION
	Burette Filling:	Use a plastic funnel to fill burette; remove equipment from retort stand and fill over sink.
	Pipette Filling:	Do not pipette by mouth. Use a pipette bulb to fill pipette.

INTRODUCTION

Atomic Absorption Spectroscopy (AAS) is a commonly used technique for determining the concentration of metal ions. The analysis of metals is important in a range of applications from trace analysis of metals - including pollutants - in water and soil to quality control of materials such as steel and other alloys.

Quality control in products is an important commercial tool. In this instance, the presence of ions such as sodium in sports drinks is critical for their usefulness in replacing the ions loss by athletes through perspiration. Too little sodium and the product will not be effective. However, the addition of too much sodium may diminish the taste of the product, as well as slow the rate of absorption of the liquid in the stomach.

A flame is needed to atomise a sample in order to measure it. The temperature of the flame is selected based on what metal(s) are being analysed. As shown in Table 1, the less reactive metals require more energy (heat) to be atomised. The most common method for atomizing a sample is using an acetylene (C2 H2) / air mixture.

TABLE 1: FLAME GAS TEMPERATURES AND APPLICATIONS

GAS COMBINATION MAX. TEMP. (K)		ELEMENTS ATOMIZED
Natural gas/air	1800	Group 1A (Li, Na, K)
Acetylene/air	2250	Transition metals (Fe, Cu, Zn)
Acetylene/ N2O	3000	Group 2A (Ca, Mg, Ba) Group 3A (Al)

The detection limit between metals varies considerably. Under acetylene/air conditions, the detection limit for iron is 5 µg/L (or 5 parts per billion, also expressed as 5 ppb), sodium - 0.3 ppb; zinc - 1.5 ppb and mercury - 150 ppb.

The basis of this analytical method is that for a given metal ion, the absorption is directly proportional to the concentration

(A∝]), that is, the more atomised metal present, the higher the absorbance. We use this principle to prepare a calibration curve and use this to determine the concentration of the species in the unknown.

As with any analytical method, the determination of metals by AAS can be affected by the presence of other species. Remember a sports drink is not just sodium ions and water! It is important that we are aware of everything else in the solution and take steps to counter their effect. Any species that changes the observed signal, while the concentration of the metal remains unchanged, is termed an interference.

TYPES OF INTERFERENCE

In this experiment we are primarily interested in determining the sodium content of sports drinks by AAS. However, potassium is also present within sports drinks, and may affect the analysis of sodium (resulting in an interference).

In AAS, it is very important that the standards we prepare are as close as possible to the sample under investigation. When using a calibration curve to calculate an unknown concentration, your results will only ever be as good as your standards. Bad standards give bad results! In this investigation, the interference due to other ions (potassium) is accounted for by adding potassium as well as sodium to the standard solutions.

The reason for adding potassium is simple. Alkali metals such as sodium have low ionisation potentials (lose electrons easily) and readily ionise in the flame. This results in sodium ions being present as well as sodium atoms, however, only sodium atoms absorb the unique radiation that is generated (via the lamp in the AAS) and measured by the instrument. When this occurs, it is called ionization interference.

The simplest way to overcome this effect is to add another alkali metal that will ionise more easily than the sodium, such as potassium. This is termed an ionisation suppressor. By having a high concentration of electrons in the flame, ionisation of the sodium is suppressed, keeping 100% of the sodium as atoms which can be measured.

To show the effect from the presence of potassium, the sports drinks will be analysed using two sets of standard solutions. One series will be made containing only sodium ions, while another series will be prepared with the same concentrations of sodium ions and will also contain a large excess of potassium ions.

PRINCIPLE

Your analysis will determine the concentration of sodium present in a range of sports drinks. A series of standard solutions of sodium will be prepared. Our selection of the range of these standards is based on our understanding of the concentration of the unknown as well as the knowledge that high concentrations of substances cause deviations from the linear relationship between absorption and concentration.

Preparation of the sample for analysis involves dilution of the initial sports drink sample. This is required to adjust the concentration of sodium into the correct range for the operation of the instrument and to ensure that the absorption reading is within the range of the standards.

PROCEDURE

PREPARATION OF STANDARDS

1. Prepare a series of sodium standards:
   Prepare a series of four solutions containing 0, 10.0, 20.0, 50.0 mg/L (ppm) of sodium. To do this, measure accurately, 0, 1, 2 & 5ml of the sodium stock solution (from the supplied burette) respectively into four separate 100ml volumetric flasks. Label these flasks as "Na stds-1, 2," etc.

2. Prepare a series of sodium standards containing a constant amount of potassium ions (as KCl):
   Prepare the same series (i.e. 0.0, 10.0, 20.0 and 50.0 mg/L) as in Step 1 above by measuring out the same volume of sodium stock solution into four new volumetric flasks. At this point, add 20ml of the 10,000 mg/L potassium (KCl) stock solution (measured with a measuring cylinder) to each volumetric flask. Label this series as "Na-K stds-1, 2;·etc.

3. Make each flask up to the mark with distilled water.

PREPARATION OF SPORTS DRINK SAMPLE

4. Pipette a 5 ml aliquot (precisely known volume - use a pipette) of Gatorade into the provided 100 ml volumetric flask. Add 20ml of the 10,000 mg/L potassium stock solution (measured with a measuring cylinder).

5. Add distilled water up to the 100.0 ml mark. (What dilution factor have you just introduced?).

6. Analyse these solutions by AAS for sodium as described below.

ANALYSIS

7. Stopper all flasks and thoroughly mix the solutions by inverting the flasks at least 6 times.

8. Measure the absorbance of the "Na stds" series (all 4 solutions). Solutions are measured from lowest to highest concentration. Record these results in the Table below. From the data, prepare a plot of Absorption Vs Concentration Na stds.

9. Measure the absorbance of the "Na-K stds" series (all 4 solutions). Solutions are measured from lowest to highest concentration. Record these results in the Table below. From the data, prepare a plot of Absorption Vs Concentration Na-K stds.

10. Measure the absorbance of the diluted sports drink.

11. Record the absorption value in the Table below.

You must take the dilution factor into account when reporting the actual concentration of sodium in the sports drink.

RESULTS

TABLE 2: ABSORBANCE VALUES OF Na STANDARDS

CONCENTRATION OF Na STDS (mg/L)	AAS ABSORBANCE	
	Na-STDS	Na-K STDS
0.0		
10.0		
20.0		
50.0		

TABLE 3: ABSORBANCE VALUES OF Na STANDARDS

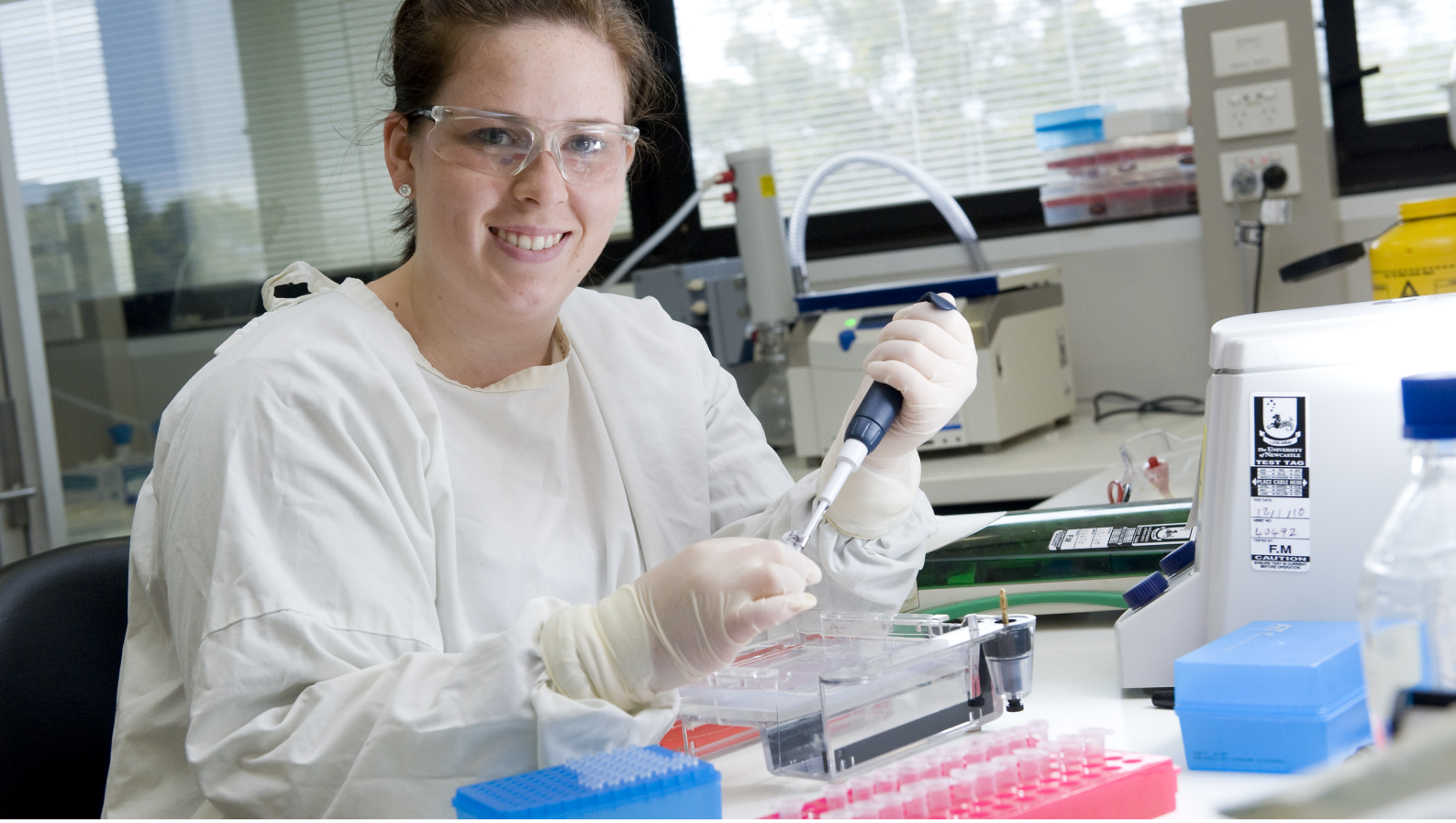
Name of sports drink	
Sodium concentration from bottle*	
AAS Absorbance	
Dilution Factor	
Concentration of sodium in sports drink determined from 'Na stds' graph	
Concentration of sodium in sports drink determined from 'Na-K stds' graph	.

FOLLOW UP ACTIVITY

1. Which of the two values you have reported in Table 3 above would be expected to be closer to the "real" value? Justify your choice.

2. Draw a block diagram. ofan AAS unit and use this to briefly explain. how the system operates.
   https://www.youtube.com/watch?v=rv9ge_lw6nk

3. What else could be analysed using AAS?



EXPERIMENT 2: SYNTHESIS & DECOMPOSITION OF ASPIRIN

EXTRACT FROM HSC SYLLABUS

Analyse the structure of, and predict reactions involving, carbon compounds.

Explore the many different types of chemical reactions and the factors that affect the rate of chemical reactions.

Conduct investigations and/or process data to determine the concentration of coloured species and/or metal ions in aqueous solution, including atomic absorption spectroscopy.

Conduct qualitative investigations to test for the presence in organic molecules of the various functional groups.

Investigate the processes used to analyse the structure of simple organic compounds addressed in the course, including but not limited to: proton and carbon-13 NMR, mass spectrometry, and infrared spectroscopy.

RISK ASSESSMENT

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HAZARD	SUBSTANCE, APPARATUS, PROCEDURE	PRECAUTION/ACTION
	Steambath / Hotplate	Take care in handling all equipment and glassware while hot. Use rubber 'fingers' or tongs. Use boiling chips when boiling liquids to prevent bumping.
	Phosphoric acid	WEAR GLOVES. Contact with combustible material may cause fire. Toxic by inhalation. Cause severe burns. Risk of serious damage to eyes. Reacts violently with water. Use only in fumehood with dropper bottle.
	Acetic anhyrdide	WEAR GLOVES. Contact with combustible material may cause fire. Toxic by inhalation. Cause severe burns. Risk of serious damage to eyes. Reacts violently with water. Use only in fumehood with dropper bottle.

INTRODUCTION

While medicines based on plants rich in salicylate, such as willow, stretch back to antiquity, pure acetylsalicylic acid (also known as aspirin) was first manufactured and marketed for medicinal use in 1899. Used as a pain-reliever to reduce fever and as an anti-inflammatory drug, aspirin is still one of the most common over-the-counter medications used today.

Long term, low-dose aspirin is also used to help prevent heart attacks, strokes and blood clots due to its inhibition of platelet binding in the blood.

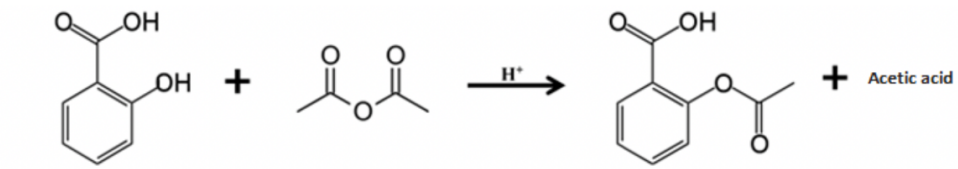
SYNTHESIS

You will be synthesising aspirin starting from the salicylic acid, a natural product that can be isolated from the leaves or bark of a range of plants, including the white willow (salix alba). Salicylic acid is the common name for 2-Hydroxybenzoic acid.

This type of reaction is a specific type of condensation reaction known as esterification. The addition of acetic anhydride converts the phenol group of salicylic acid into an ester, producing acetic (ethanoic) acid [vinegar] as a by-product. The reaction scheme for this transformation is available below in **diagram 1.1.**

DIAGRAM 1.1

White Willow (*salix alba*)



Salicylic acid
(MW = 138.12 g/mol)

Acetic anhydride

Acetylsalicylic acid
(MW = 180.16 g/mol)

SPECTROSCOPY

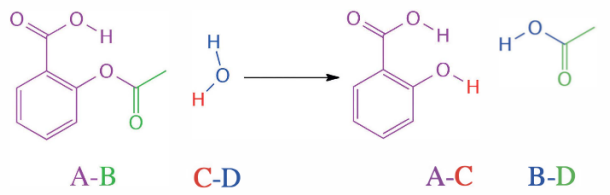
The individual atoms contained in molecules are far too small to be directly observed. The elucidation of the structure of molecules (i.e. the bonding arrangements between atoms and the types of atoms) in chemistry is carried out using spectroscopic and spectrometric techniques, including Infrared (IR) Spectroscopy, Nuclear Magnetic Resonance (NMR) Spectroscopy and Mass Spectrometry (MS).

Spectroscopy is the study of the interaction between electromagnetic radiation and molecules and how these interactions can be quantified, analysed and interpreted to gain information about molecular structure. IR spectroscopy is a non-destructive spectroscopic technique that uses radiation in the infrared region of the electromagnetic radiation spectrum. The energy of molecular vibrations corresponds to that of the IR region of the electromagnetic spectrum, with organic functional groups displaying characteristic vibration frequencies within well-defined regions of the IR spectrum. You will use infrared spectroscopy to assess the success of your synthesis, based on changes to the molecules functional groups and the result IR spectra.

DECOMPOSITION

Following their synthesis, many drugs are susceptible to some form of chemical decomposition as a result of improper use or storage. This leads to a reduction in the therapeutic value of the drug, and in the likely formation of toxic or harmful compounds. Examples of chemical decomposition include hydrolysis, oxidation, photochemical decomposition and polymerisation.

In this experiment you will also induce and observe the chemical decomposition of aspirin via a hydrolysis reaction. During hydrolysis, a molecule of water (H-O-H) is chemically added to a substance (the starting material), in such a way as to cause both the starting material molecule and the water molecule to split into two parts and then cross-combine. The two products formed gain respectively a proton (H+) and a hydroxide ion (OH-) from the water molecule (see scheme below). Note the bonds are colour-coded to show where the electron-pairs forming the bonds end up.



The reformation of salicylic acid can be monitored by the addition of iron (111) chloride which results in a purple coloured complex. Using a colorimeter the amount of light absorbed by this iron-salicylate complex can be measured. By measuring theabsorbance of solutions of known concentrations, a calibration curve can be produced which shows the direct relationship between absorbance and concentration.
This calibration curve can then be used to determine the concentration of salicylic acid in your sample over time.

The hydrolysis of acetylsalicylic acid is a pseudo-first order reaction. This means that the rate of decomposition is primarily affected by the concentration of one reactant, in this case the acetylsalicylic acid itself. The general rate law for first order reactions is:

$$\frac{-\Delta[A]}{\Delta t} = k\,[A]$$

Where t is the time (in seconds), [A] is the concentration of the reactant and k is the rate constant. By integrating the rate law we get the following:

$$\ln [A] = -kt + \ln[A]_0$$

Thus a plot of ln[A] vs time gives a straight line with slope of -k. An additional useful formula can also be derived from this equation, where the half-life of a first-order reaction is given by the following and is independent of the initial concentration:

$$t_{\frac{1}{2}} = \frac{0.693}{k}$$

PROCEDURE

DECOMPOSITION (HYDROLYSIS) OF ASPIRIN

1. This experiment will utilise an unltraiolet visible spectrophotometer. Instructions for using the spectrophotometer can be found on your bench, and a walk-through of the experiment will be discussed by your demonstrator

2. Measure the absorbance of the provided iron-salicylate standards using the UV-Vis Spectrophotometer at 535nm.

3. Add 5 drop of the provided iron chloride (FeCl3) solution to a measuring cylinder and make up to 19 ml with water. Transfer this solution to a 50 ml beaker.

4. Using a pipette, add 1 ml of the provided 20 mM acetylsalicylic acid (aspirin) solution to the beaker.

5. Fill the provided sample cuvette with the aspirin solution, and place into the UV-Vis Spectrophotometer. Plot the change in absorbance vs time for the next 30 minutes. Recording an absorbance value every 30 seconds.

6. Determine the reaction rate for the decomposition and the reaction half-life using your results on the spreadsheet provided. Record in the table below;

SYNTHESIS OF ASPIRIN

7. Accurately weigh approximately 1.5 g of salicylic acid into a 125 ml quick-fit conical flask.

8. In a fumehood, add 3ml of acetic anhydride to the flask, followed by 3 drops of concentrated phosphoric acid.

9. Stopper the flask and heat on a steambath for 5 minutes.

10. Remove from the steambath and immediately add 20 ml of R.O. water.

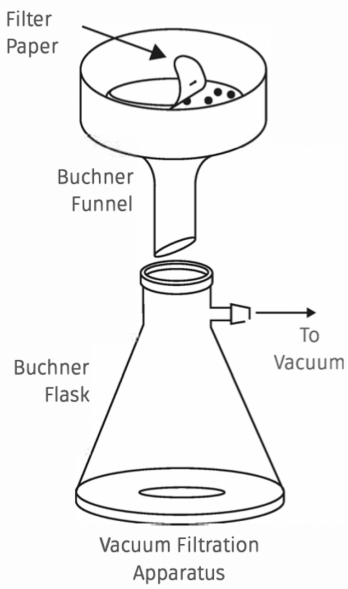
11. Allow to cool. Then add an additional 20 ml of water. Stand at room temperature until crystals begin to form.

12. Add an additional 10ml of water, swirl and place in an icebath.

13. Isolate product via vacuum apparatus using the instructions and **Diagram 1.2** below.

- Slide the rubber tubing provided onto the side arm of the Buchner flask.
- Clamp the Buchner flask firmly to the metal frame available at your bench.
- Attach the other end of the rubber tubing to the in-line vacuum system.
- Place a Buchner funnel on top of the secured Buchner flask.
- Place an appropriately sized piece of filter paper flat into the Buchner funnel.
- Wet it with a small amount of water.
- Turn the vacuum on (not to full power) and slowly pour the solution into the funnel.
- Rinse the flask and funnel with a small amount of ice cold R.O. water.
- Filter for 10 minutes to dry the product.

DIAGRAM 1.2



Vacuum Filtration
Apparatus

14. Collect and weigh the product. This compound is acetylsalicylic acid (aspirin).

Note the appearance and colour, record the mass.

15. Check the efficiency of the reaction by placing a small amount of salicylic acid and your synthesised aspirin into separate test tubes.

Add 1-2 ml of iron chloride ($FeCl_3$) solution and observe any colour change.

16. Measure the IR spectra of the salicylic acid and your synthesised aspirin. Note any differences between the spectra.

Use the material provided in the lab to correlate these differences to the structural changes which have occurred during synthesis.

17. Review the provided Nuclear Magnetic Resonance (NMR) spectra and Mass Spectrometry (MS) data for your starting material and products. Note any difference between the spectra.

Use the material provided in the lab to correlate these differences to the structural changes which have occurred during synthesis.

RESULTS

TABLE 1: ASPIRIN SYNTHESIS YIELDS

Mass of Salicylic Acid Used (g)	
TheoreticalYield of Aspirin (g)	
Mass of Aspirin Obtained (g)	
% Yield	.

TABLE 2: HYDROLYSIS KINETICS

Rate Constant	
Half-Life (t½)	.

FOLLOW-UP ACTIVITY

1. What are the recommended storage conditions for aspirin and why are they important?

1. What is the purpose of the phosphoric acid added after the acetic anhydride?

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