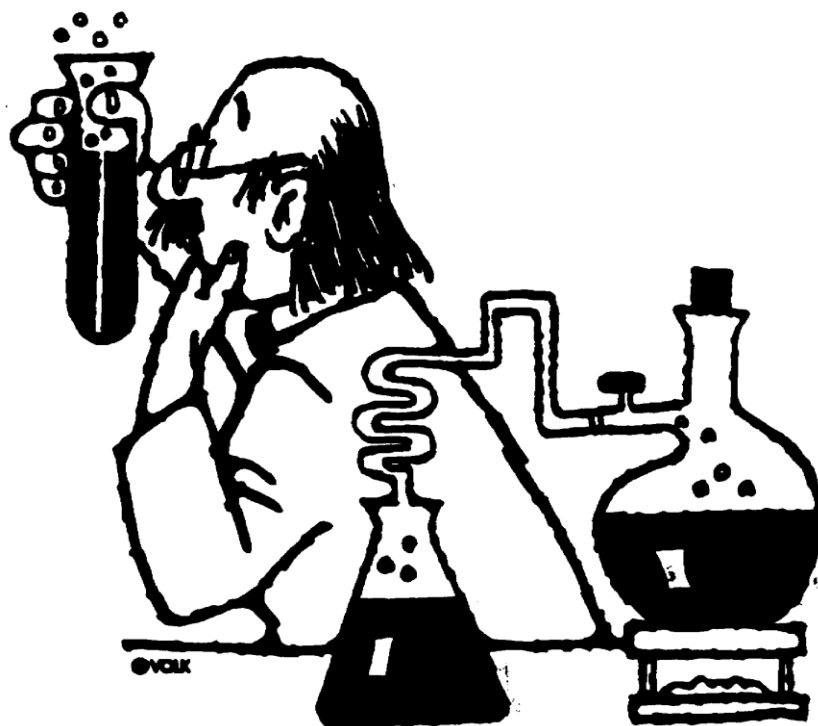


ORGANIC CHEMISTRY LABORATORY PROCEDURES

Organic Chemistry I and II
CHEM 2123 and 2125

Richard Wheet

Fourth Edition 2011



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ORGANIC CHEMISTRY LABORATORY PROCEDURES

Fourth Edition

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2011

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REFERENCES

Morrison and Boyd, *Organic Chemistry*

Hajian, *Modern Chemical Technology*

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CHEMICAL TECHNOLOGY SAFETY INSTRUCTIONS

Laboratory work in chemistry can be stimulating to students who appreciate the challenge it offers to their abilities, but it is not without certain hazards. For your safety, and for that of your classmates, a few simple regulations will have to be enforced. The observance of these safety regulations is an integral part of good laboratory technique.

1. Wear safety glasses at all times in the laboratory. This includes clean-up times and times when you yourself may not be working on an experiment, but someone else is.
2. Shoes must be worn in the laboratory. Sandals or bare feet are prohibited.
3. Shorts or cut-offs shall not be worn when working in the laboratory.
4. Light burners only when needed. Promptly extinguish any flame not being used. An open flame may ignite reagents being used by you or others near you.
5. Do not eat, drink, smoke, dip or chew tobacco in the laboratory.
6. Never look directly into the mouth of an open flask or test tube if it contains a reaction mixture.
7. Never point the open end of a test tube at yourself or at another person.
8. Avoid measuring volumes of strong acids or alkaline solutions with your graduated cylinder held at eye level. Support the graduated cylinder on your bench; add the hazardous liquids from a beaker a little at a time, inspecting after each addition.
9. Never weigh a chemical directly on a balance pan. Use a pre-weighed container, e.g. a watch glass, weighing dish or a small square of clean paper turned up on all sides.
10. Make sure all electrical equipment is safely grounded and all wires are insulated.
11. Report all accidents to your instructor.

SAFETY TEST

NAME OF STUDENT _____

ADDRESS _____

PHONE _____

IN CASE OF AN ACCIDENT NOTIFY

ADDRESS _____

PHONE _____

1. The best first aid when a chemical gets into the eyes is to
_____ a) rub the eyes.
_____ b) wash the eye with clear water.
_____ c) put on safety glasses.
2. With proper precautions, any chemical can be handled safely.
_____ a) true
_____ b) false
_____ c) no opinion
3. The best way to learn hazardous characteristics of a chemical is
_____ a) read the label on the bottle.
_____ b) ask your classmate.
_____ c) refer to your textbook.
_____ d) none of the above.
4. Safety glasses should be worn in the laboratory
_____ a) only when working with acids.
_____ b) only when working with bases.
_____ c) all of the time.
_____ d) only when heating a substance.
5. Horse play is permitted in the laboratory

- _____ a) when cooling a chemical.
_____ b) when heating a chemical.
_____ c) during an experiment.
_____ d) never.
6. Accidents should be promptly reported to
_____ a) your partner.
_____ b) your other classmates.
_____ c) your instructor.
_____ d) the school nurse.
7. Burners should be lighted
_____ a) only when needed
_____ b) all the time.
_____ c) when cooling a chemical.
_____ d) never.
8. One should use the contents of an unlabeled container.
_____ a) always
_____ b) seldom
_____ c) never
_____ d) none of the above.
9. Foods, drinks, and smoking are permitted in a laboratory.
_____ a) never
_____ b) always
_____ c) once in a while
10. Which of the following are NOT required for a fire:
_____ a) oxygen
_____ b) fuel
_____ c) heat

_____ d) carbon dioxide

11. Unauthorized experiments are NOT to be performed.

- _____ a) true
_____ b) false
_____ c) no opinion

12. Which of the following is a hazardous nature of chemicals?

- _____ a) toxic
_____ b) flammable
_____ c) irritating
_____ d) explosive
_____ e) all of the above.
_____ f) none of the above.

13. Glassware which is chipped or cracked should be

- _____ a) used right away.
_____ b) discarded
_____ c) given to your partner.

14. The proper way to dilute acid is to

- _____ a) add acid and water at the same time.
_____ b) add acid to water.
_____ c) add water to acid
_____ d) none of the above.

15. Standing on a laboratory stool is a safe practice.

- _____ a) true
_____ b) false
_____ c) no opinion

16. When dangerous gases are given off in a reaction, the experiment should be carried out

- _____ a) on your laboratory bench.
- _____ b) outdoors.
- _____ c) in a fume hood
- _____ d) in the hallways.

17. It is a good practice to read the experiment and follow the instructions carefully.

- _____ a) true
- _____ b) false
- _____ c) no opinion

18. Excess reagents should be

- _____ a) placed in a waste container.
- _____ b) should be returned to the reagent bottle.
- _____ c) consumed by the student.

19. A chemical may enter the human body through the

- _____ a) mouth.
- _____ b) skin.
- _____ c) lungs.
- _____ d) all of the above.

20. Lids and stoppers should be replaced on the corresponding reagent.

- _____ a) true
- _____ b) false
- _____ c) no opinion

THIS IS TO CERTIFY THAT _____

I have received instruction on laboratory safety and have witnessed a safety film. I certify that I understand the safety instructions and I am aware that safety glasses must be worn at all times in the laboratory.

SIGNED _____ DATE _____

Student

SIGNED _____ DATE _____

Student Witness

THIS IS TO CERTIFY THAT _____ has been given safety instructions and has satisfactorily passed a safety test.

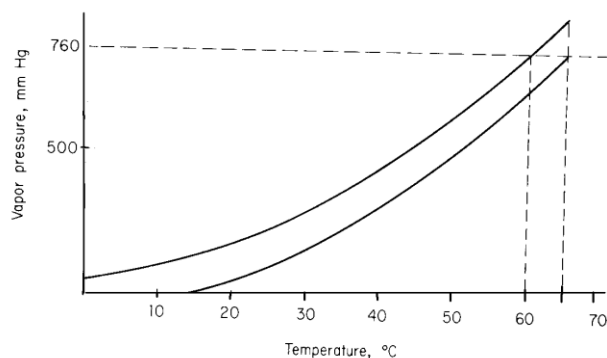
SIGNED _____ DATE _____

Instructor

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SIMPLE DISTILLATION OF A SINGLE SOLUTION

Distillation is a process where a liquid is vaporized, recondensed, and collected. Distillation is used to purify liquids and to separate one liquid from another. The liquids have different volatilities which is the relative ease with which the molecules of a liquid escape from the surface. Volatility is normally the opposite of the boiling point of a liquid. The higher the volatility of a liquid, the lower the boiling point. The lower the volatility of a liquid, the higher the boiling point. Vapour pressure is a measure of the force a liquid exerts on the surface for its molecule to escape.



The liquids will give off molecules, until the atmosphere above the liquid has a vapour pressure equal to the respective temperature. For example, if the vapour pressure of a liquid was 760 mm Hg at 60° C, molecules would escape from the surface until there was a pressure of 760 mm Hg in the atmosphere vapour exerting pressure back at the liquid. When a solvent is enclosed, the liquid will evaporate until the partial pressure of the gas above the liquid equals the vapour pressure of the liquid. If some of the gaseous vapour is removed, more liquid will evaporate in order to equalize the vapour pressure and partial pressure. This is the principle behind distillation.

A liquid is heated in a distilling flask. The temperature of the liquid will increase (specific heat) until the vapour pressure/temperature of the first liquid is reached. At this point, all the heat energy (heat of vaporization) is used to evaporate the liquid. The hot vapour

travels upward and reaches a condensing column which removes heat from the vapour. The gas recondenses back to a liquid and is collected in a receiving flask. Since this reduces the vapour pressure over the liquid in the distilling flask, more liquid is converted to vapour to equalize, which in turn recondenses. This is the general idea behind distillation. If there is only one liquid in the receiving flask, it will be separated from any nonvolatile solid. These solids would remain in the distillation flask. Thus, the liquid is purified.

EQUIPMENT & CHEMICALS

Water (H₂O)

20% Sodium Chloride (NaCl)

Organic kit

EXPERIMENTAL OBJECTIVES

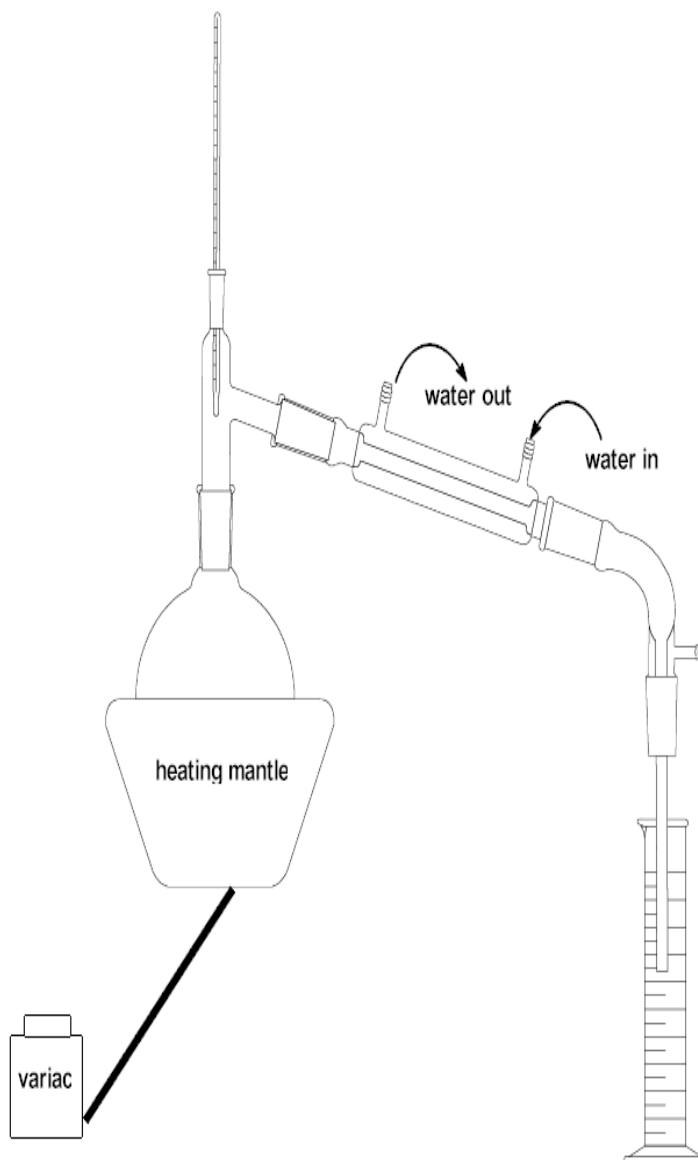
Describe the distillation process.

Perform a distillation.

PROCEDURE

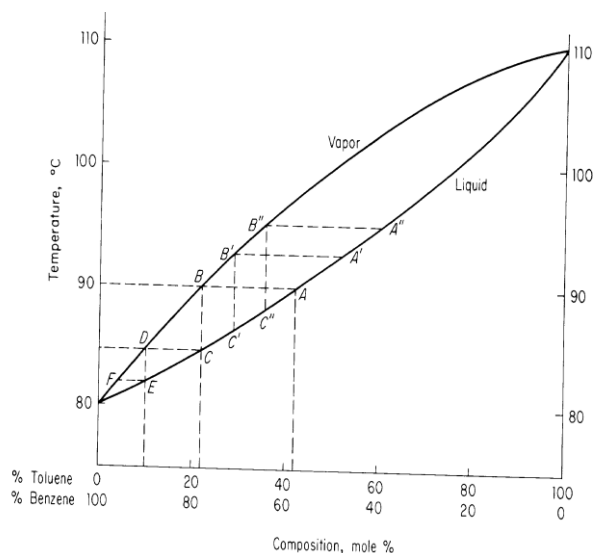
1. Add 100 ml of aqueous 20% sodium chloride (NaCl) to a 200 ml distilling flask. In picking a certain size of distilling flask, the volume of the flask should be about twice the size of the volume of liquid.
2. Add 3 or 4 boiling chips to the flask to avoid bumping.
3. Assemble the distillation apparatus as shown in Fig 1-2. Remember to put a film of silicon lubricant between the ground glass joints to prevent freezing of the joints. The thermometer bulb should be slightly below the sidearm opening.
4. The upper outlet on the condenser should be for exiting cooling water. This will prevent the accumulation of air in the condenser.
5. Turn on the cooling water.
6. Increase the heat to the distilling flask until the rate of distillate (liquid) into the receiving flask is about 2-3 drops per second.

7. Record the temperature of the vapour every 5 ml of distillate collected.
8. Continue to distill until only a small amount of residue remains in the distilled flask. Do not distill to dryness.
9. What did you notice about the temperature of the vapor?
10. What did you observe about the appearance of the distilling flask after distillation was almost complete?
11. What can you say about the relationship of the boiling point of water (theoretical) and your experimental result?



DISTILLATION OF A MIXTURE OF TWO LIQUIDS

Simple distillation of a mixture of two liquids will not effect a complete separation. If both liquids will boil, both will vaporize and be collected. The more volatile of the two liquids, will vaporize more quickly than the less volatile and form a larger proportion of the distillate in the receiving flask. When there is a large difference between the volatilities of the two liquids, most of the first portion of the distillate collected will be almost pure. The last portion will be purer in relation to the second liquid. The distillate collected in the middle will contain varying amounts of the two (figure 2-1). You can separate the two liquids by changing the receiving flask several times and numerous redistillations.



The first portion collected near the boiling point temperature of more volatile liquid will be almost pure (compound A). The last portion collected when the temperature is nearly equal to the boiling point of the less volatile will be mostly the less volatile liquid (compound B).

EQUIPMENT & CHEMICALS

Water (H₂O)

Ethyl ether

Distillation Apparatus

EXPERIMENTAL OBJECTIVES

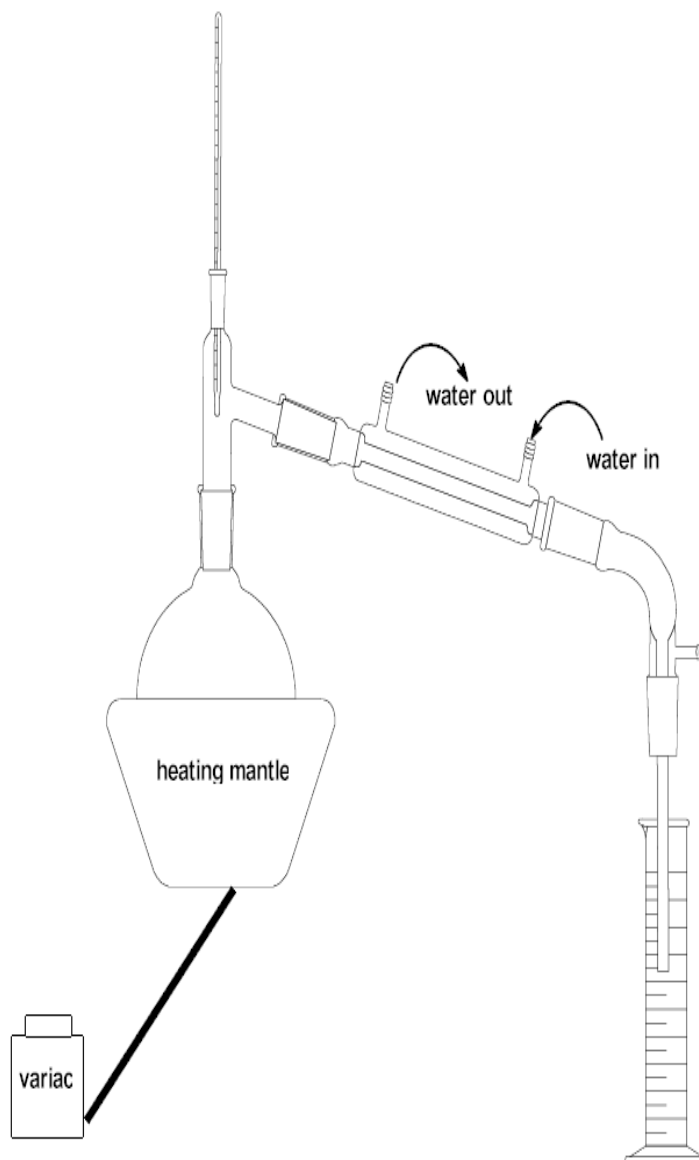
Describe the distillation process involving two liquids.

Perform a distillation.

PROCEDURE

1. Add 100 ml of a 25% ethyl ether/water mixture (v/v) to a 250 ml distilling flask. In picking a certain size of distilling flask, the volume of the flask should be about twice the size of the volume of liquid.
2. Add 3 or 4 boiling chips to the flask to avoid bumping.
3. Assemble the distillation apparatus as shown in fig 2-1. Remember to put a film of silicon lubricant between the ground glass joints to prevent freezing of the joints. The thermometer bulb should be slightly below the sidearm opening.
4. The upper outlet on the condenser should be for exiting cooling water. This will prevent the accumulation of air in the condenser.
5. Turn on the cooling water.
6. Increase the heat to the distilling flask until the rate of distillate (liquid) into the receiving flask is about 2-3 drops per second.
7. Record the temperature of the vapour every 5 ml of distillate collected.
8. Continue to distill until only about 40 ml remains in the distillation flask. Do not distill to dryness.

9. What did you notice about the temperature of the vapor?
10. What can you say about the relationship of the boiling points of water and ethanol (theoretical) and your experimental result?
11. Draw a graph of volume versus temperature and explain the findings.



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FRACTIONAL DISTILLATION

A simple distillation usually gives a crude distillation where the initial distillate has to be redistilled to reach optimum purity. The separation and purification of two or more liquids into various fractions by distillation is called fractional distillation. Fractional distillation is really the systematic redistillation of the distillates within a column with the fractions increasing in purity as the fractions move up the column. This is similar to repeatedly collecting and redistilling the various fractions. The column is filled with pieces of glass, glass beads, chips, metal wire, etc. These beads act as small condensers.

The vapour leaves the surface of the liquid and travels upwards through the packed column. It condenses on the cooler beads and falls back as a liquid. The initial vapours transfer heat to the lower beads. The recondensed vapour then vaporizes again but this time move a little higher in the column and the process repeats. The less volatile vapours reach the beads which were heated by the more volatile vapour. Since the temperature of the beads are less than the boiling point of the less volatile vapour, they also recondense and fall back. But this now heats the lower beads and the process continues. The more volatile vapours pass through the column condensing and recondensing. But as the higher boiling vapours heat the lower beads, these beads revaporize the higher volatile vapours within the column. Thus the liquids do not return to the distillation flask as they move upward.

Each repeated distillation causes a greater concentration of the more volatile liquid in the rising vapour. The less volatile liquids also are in turn enriched as they pass upward through the column.

CHEMICALS AND EQUIPMENT

Ethanol

Water

Distillation Apparatus

Glass beads

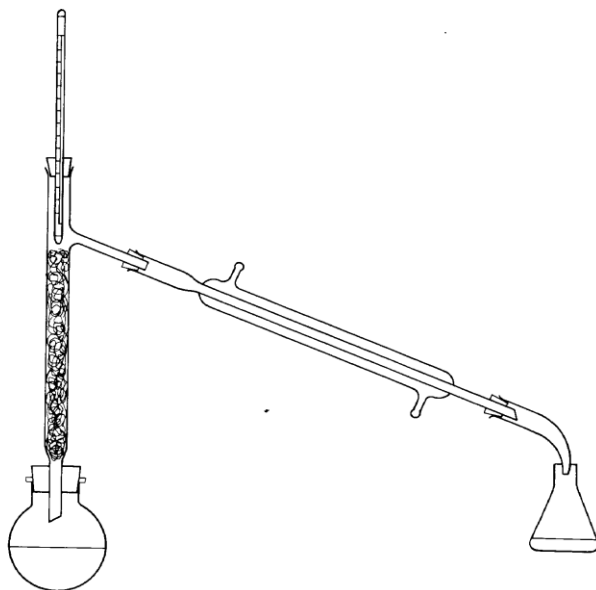
EXPERIMENTAL OBJECTIVES

The student should be able to perform a fractional distillation.

The student should be able to describe the process of fractional distillation.

PROCEDURE

1. Add 50 ml of ethanol and 100 ml of water to a 250 ml flask. Remember boiling chips.
2. Fill a condenser with glass beads. If necessary, use a small plug of glass wool to prevent the beads from falling through the column.
3. Assemble the fractionating column (Figure 1).



4. Slowly heat the distillate to a flow rate of 2-3 drops per minute.
5. Record the temperature versus the volume every 5 ml.
6. Plot a curve of volume versus temperature.
7. Explain how the results differ from a simple distillation.

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MELTING POINT

The melting point of a solid is the temperature at which the substance begins to change from a solid to a liquid. Pure organic compounds have very defined melting points. Through the use of these melting points, the purity of the compound can be determined. Contaminants normally lower the melting point and broaden the melting point range (the temperature at which melting starts and the temperature at which the substance is melted). Narrow range melting points are normally indicative of the purity of an organic compound. Extremely pure compounds have ranges of 0.1 to 0.3 °C. Commercially available compounds (technical grade) have ranges of 2-3 °C. The normal laboratory grade organic (ACS) compounds have a 1 °C range. The larger the melting point range, the less pure the compound.

The melting point range is the temperature range between where a compound starts to melt and completely melts (becomes liquid). It is highly recommended that during a melting point determination, the temperature be slowly increased. This enables one to observe the change and temperature range.

Sometimes, mixtures of two organic compounds with the same individual melting point have a much lower melting point. This temperature depression is useful for determining unknowns. When an unknown is identified as a suspected compound, this compound can be mixed with a known compound of identical melting point. The melting point depression can confirm whether the suspected organic compound is indeed the correct assumption.

Not all organic compounds melt. Some substance will decompose, discolor, soften and/or shrink as they are heated. If possible, a reference compound should be compared. If a compound decomposes, this temperature is normally a reliable indicator. The temperature is followed by the letter "d" to indicate decomposition (198 °d).

CHEMICALS AND EQUIPMENT

Paraffin Oil

Thiele tube

250 °C Thermometer

Capillary tube

Bunsen burner

Benzamide

Acetanilide

p-dichlorobenzene

Urea

Naphthalene

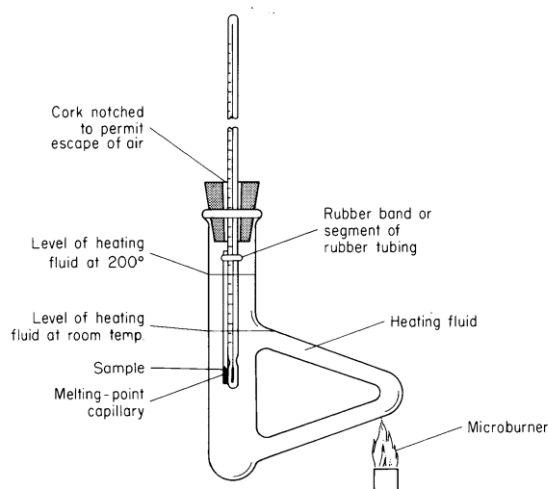
INSTRUCTIONAL OBJECTIVES

The student should be able to perform a melting point determination.

The student should be able to determine the compound and relative purity given the melting point.

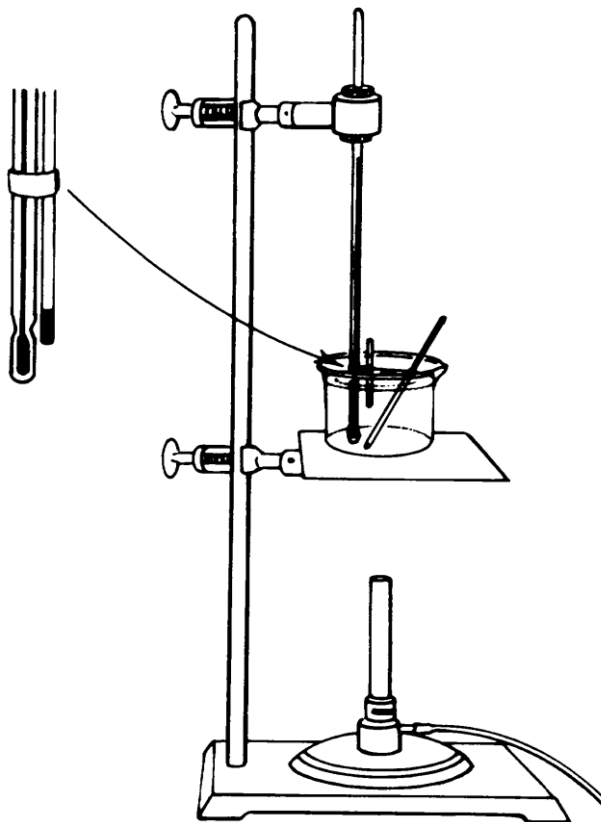
PROCEDURE

1. Assemble the melting point apparatus (Figure 1 or 2) as directed by instructor.



2. Fill a capillary tube with the compound to a height of approximately 3 - 4 mm. This can be accomplished by pressing the upper end of the capillary tube into the compound and then tapping the closed end on the table.

3. Attach the capillary tube to the thermometer using a small piece of rubber tubing or a rubber band. The bottom of the capillary tube should be even with the thermometer bulb.
4. Gradually heat the arm of the thiele tube. The rate of heating should be about 2 °C per minute.



5. When the solid in the capillary tube starts to melt, observe the melting point temperature. When the solid completely melts, observe the temperature. The two temperatures are called the melting point range.

6. Do the melting points of the following organic compounds:

Acetanilide

Benzamide

Naphthalene

p-dichlorobenzene

urea

7. Compare the experimental melting points with the theoretical melting points (found in reference books and indicate your references on your report).

EQUIPMENT & CHEMICALS

Salicylic acid
Acetic anhydride
Sodium bicarbonate (saturated)
Hydrochloric acid (conc)
200 ml beaker
125 ml Erlenmeyer flask
filtration apparatus
melting point apparatus

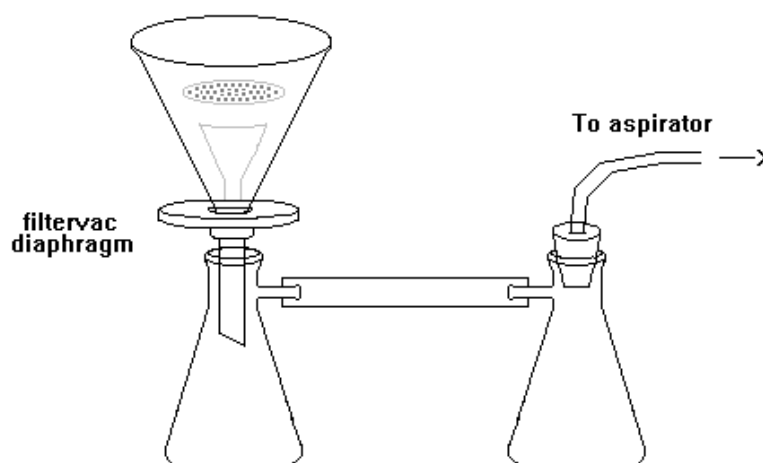
EXPERIMENTAL OBJECTIVES

1. Crystallization procedures
2. Vacuum filtration procedures
3. Acetylation reaction
4. Determination of percent yield

PROCEDURE

1. Weigh 2.0 grams of salicylic acid (0.015 moles) and transfer to a clean and dry 125 ml Erlenmeyer flask.
2. Add 5 ml of acetic anhydride (0.05 moles) to the flask.
3. Slowly add 5 drops of concentrated sulfuric acid while swirling the flask (or use a stirring bar and stirrer).
4. Gently stir until all the salicylic acid dissolves.
5. Heat the flask in a hot water bath for at least 10 minutes.
6. Remove the flask from the hot water bath and allow the mixture to cool to room temperature.
7. The acetyl salicylic acid (aspirin) that was produced in the reaction should begin to crystallize out of solution during the cooling. Note: if no precipitation occurs, scratch the walls of the flask with a glass stirring rod.

8. After the aspirin has all precipitated and the solution is at room temperature, add 50 ml of water and place the flask in an ice bath.
9. Vacuum filter the precipitate through a Hirsch funnel and rinse the flask with cold water until all the crystals have been collected.



10. Rinse the precipitate with several portions of cold water.
11. Air dry the precipitate in the Hirsch funnel for about 10 minutes.
12. Transfer the crude aspirin to a 200 ml beaker.
13. Add 25 ml of an aqueous sodium bicarbonate (saturated).
14. Stir the mixture until all activity (bubbling) has stopped.
15. Filter the solution through a Hirsch funnel to remove any polymers.

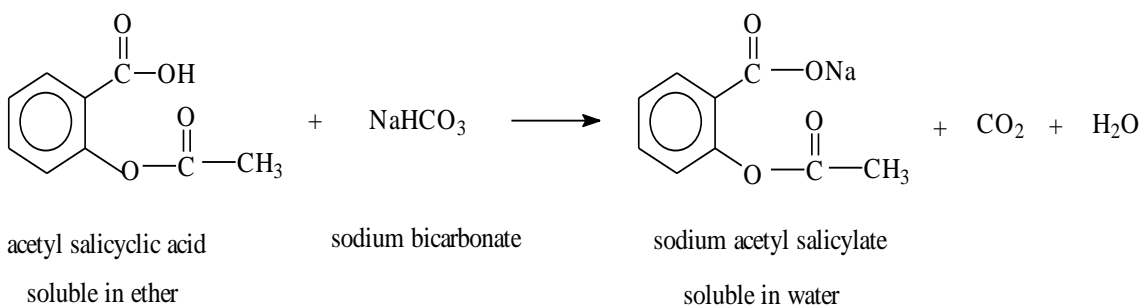
16. Rinse with about 10 ml of water.
17. Carefully add 3.5 ml of concentrated HCl to a 200 ml beaker which contains 10 ml of H₂O.
18. Carefully pour the filtrate (in small portions) into the 200 ml beaker with constant stirring.
19. The aspirin should precipitate. If no precipitate occurs, add additional HCl until the solution is acidic to litmus paper (blue to red) then proceed to the next step.
20. Cool the solution in an ice bath to precipitate all the aspirin.
21. Vacuum filter the precipitate through a Hirsch funnel and wash with ice cold water.
22. Dry the precipitate overnight.
23. Weigh the purified dried acetyl salicylic acid.
24. Calculate the percent yield using stoichiometry.
25. Determine the melting point (135-136 °C)

EXTRACTION OF A KNOWN MIXTURE

A technique called extraction will be demonstrated in this experiment. Extraction relies on the solubility of substances into solvents and the insolubility of the solvents into each other.

In this experiment, three organic compounds (aspirin, β -naphthol, and naphthalene) will be separated from each other. The three compounds are all soluble in ethyl ether (an organic solvent). By selectively reacting each organic compound, we can make it soluble in water and insoluble in ethyl ether. Since ethyl ether and water are insoluble in each other, they will form two phases and can be separated from each other using a separatory funnel. The reacted organic compound which is in the aqueous portion is then converted back into the insoluble organic compound which precipitates out of the aqueous portion.

Aspirin, β -naphthol, and naphthalene are all soluble in ether. Sodium bicarbonate (aqueous) will be added to the ether solution. Only the aspirin reacts with the sodium bicarbonate.



The aqueous layer is separated from the organic layer (extraction). The aqueous layer contains the aspirin salt. The naphthalene and β -naphthol remain in the ether layer.

The aspirin is then precipitated out of the aqueous layer by reacting it with HCl.

CHEMICALS & EQUIPMENT

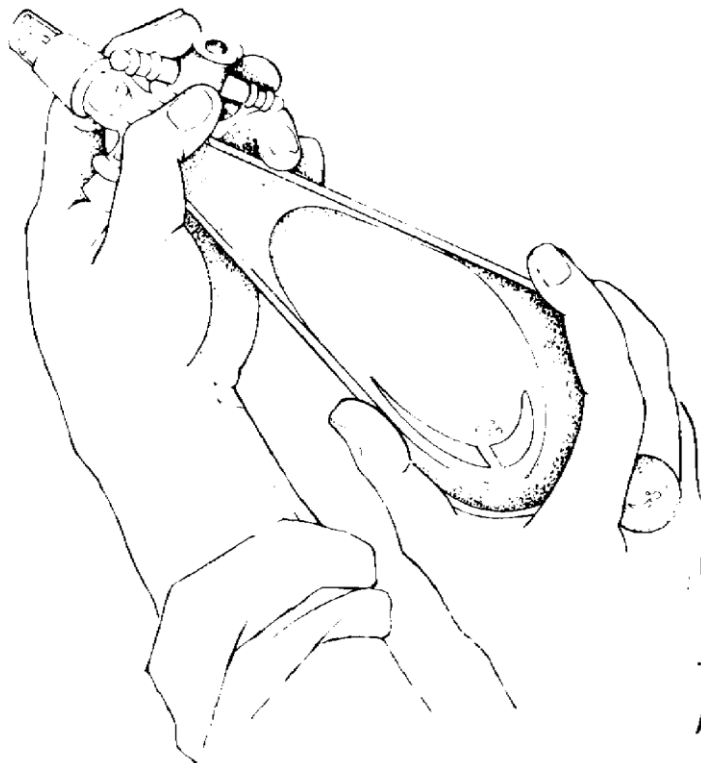
Ethyl ether
6M Hydrochloric acid (HCl)
10% Sodium hydroxide (NaOH)
5% Sodium bicarbonate (NaHCO₃)
Calcium Chloride (anhydrous)
3 - 125 ml Erlenmeyer flasks
125 ml separatory funnel
Hirsch filtration apparatus
Hot water bath

EXPERIMENTAL OBJECTIVES

Extraction procedures
Vacuum filtration procedures
Melting point
Determination of percent yield

PROCEDURE

1. Weigh out approximately 2 grams of the aspirin, 2 grams of β -naphthol, and 2 grams of naphthalene.
2. Transfer to a 125 ml Erlenmeyer flask.
3. Add 50 ml of diethyl ether (ethoxyethane) and dissolve the sample by gently swirling.
4. Pour the solution into a 250 ml separatory funnel. Make sure the stopcock is in the closed position.
5. Rinse the flask into the separating funnel with several small portions of ethyl ether.
6. Add 25 ml of 5% sodium bicarbonate (NaHCO₃) to the separatory funnel. This will react with the aspirin to form a water soluble salt.



7. Stopper the separating funnel. While holding the stopper in place with your fingers, invert the funnel and shake several times (Figure 1).
8. Point the spigot upward and away from yourself or neighbors. Slowly open the stopcock to relieve any pressure in the funnel.
9. Close the stopcock and repeat the shaking - pressure release procedure until no further pressure build up is noticed. This will indicate the aspirin/ NaHCO_3 reaction is completed.

Extraction of Aspirin

10. Place the separatory funnel into the ring stand to hold the funnel upright.
11. Remove the stopper from the funnel.
12. Open the stopcock on the separatory funnel and draw off the lower aqueous portion of the liquid into a 125 ml Erlenmeyer flask.
13. Since some of the aqueous solution is still dissolved in the ethyl ether, add an additional 20 ml portion of 5% NaHCO_3 and repeat the extraction procedure. Combine this second aqueous portion with the first.

14. Place the 125 ml flask into a warm water bath (60 °C) and gently heat. This will evaporate any ether still present in the aqueous portion. Do not overheat.
15. Cool the aqueous solution to room temperature.
16. Carefully and slowly, with constant stirring, add 6M HCl with stirring until a pH of 1-2 is indicated by pH paper. The HCl will convert the water soluble salt into the insoluble aspirin. The aspirin will start to precipitate out as the pH is reduced.
17. Vacuum filter the aspirin using a Hirsch funnel and use ice cold water to rinse.
18. Allow to air dry. Weigh, determine melting point, and calculate the percent composition of aspirin in the mixture.

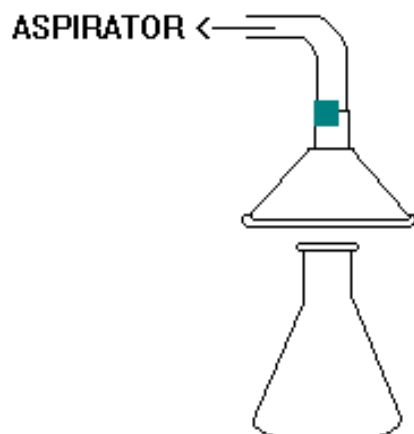
Extraction of β -naphthol

19. Add 25 ml of 10% NaOH to the separatory funnel containing the ethyl ether portion.
20. Using the extraction technique learned previously, invert, shake, and relieve pressure until no further pressure build up is noted. The NaOH is forming a sodium salt with the β -naphthol which is soluble in the aqueous phase.
21. Draw off the lower aqueous portion into a 125 ml Erlenmeyer flask.
22. Add a second 25 ml portion of 10% NaOH to the funnel and extract.
23. Combine this second aqueous portion with the first.
24. Carefully add 6M HCl to a pH of 1-2 (use pH paper). The HCl will convert the β -naphthol salt back into β -naphthol which is insoluble in water at the reduced pH. The β -naphthol will precipitate out.
25. Vacuum filter the β -naphthol using a Hirsch funnel and rinse with ice cold water.
26. Allow to air dry. Weigh, determine melting point, and calculate the percent composition of β -naphthol in the mixture.

Collection of Naphthalene

27. Transfer the ether remaining in the funnel to a 125 ml Erlenmeyer flask.

28. Rinse the funnel with an additional 10 ml of ethyl and combine.



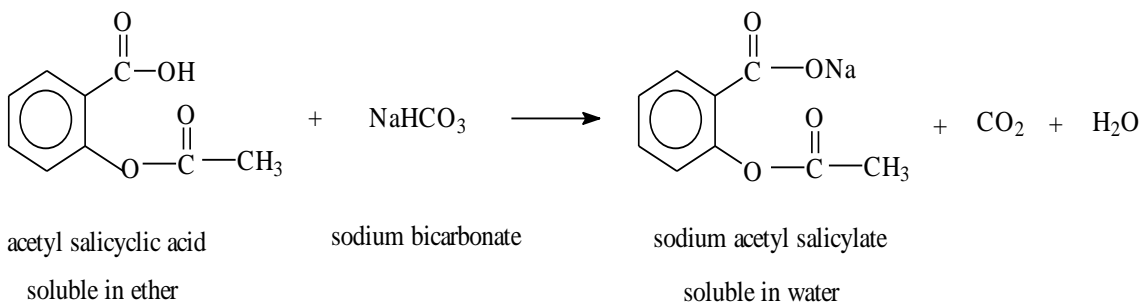
29. Add anhydrous calcium chloride (CaCl_2), about 1/10 the volume of the ethyl ether solution, to the 125 ml flask containing the ether. Since there is about 1.5% water still dissolved in the ether, the anhydrous CaCl_2 will absorb the remaining water.
30. Allow the ethyl ether solution to stand over the anhydrous CaCl_2 for 15-20 minutes with occasional swirling.
31. Decant the ethyl ether solution off the CaCl_2 into a dried 125 ml Erlenmeyer flask. Use a cotton plug in an ordinary funnel if necessary.
32. Cover the flask with paraffin wax and poke large holes in the wax. Place the flask in the hood and allow the ethyl ether to evaporate overnight. Make sure the fume hood is turned on so as to vent the ethyl ether vapour.
33. After the ether has evaporated, naphthalene should remain behind.
34. Weigh, determine melting point, and calculate the percent composition of naphthalene in the mixture.

EXTRACTION OF AN UNKNOWN MIXTURE

A technique called extraction will be demonstrated in this experiment. Extraction relies on the solubility of substances into solvents and the insolubility of the solvents into each other.

In this experiment, three organic compounds (aspirin, β -naphthol, and naphthalene) will be separated from each other. The three compounds are all soluble in ethyl ether (an organic solvent). By selectively reacting each organic compound, we can make it soluble in water and insoluble in ethyl ether. Since ethyl ether and water are insoluble in each other, they will form two phases and can be separated from each other using a separatory funnel. The reacted organic compound which is in the aqueous portion is then converted back into the insoluble organic compound which precipitates out of the aqueous portion.

Aspirin, β -naphthol, and naphthalene are all soluble in ether. Sodium bicarbonate (aqueous) will be added to the ether solution. Only the aspirin reacts with the sodium bicarbonate.



The aqueous layer is separated from the organic layer (extraction). The aqueous layer contains the aspirin salt. The naphthalene and β -naphthol remain in the ether layer.

The aspirin is then precipitated out of the aqueous layer by reacting it with HCl.

EXPERIMENTAL OBJECTIVES

- Extraction procedures
- Vacuum filtration procedures
- Melting point
- Determination of percent yield

PROCEDURE

1. Obtain a 6 gram sample of an aspirin/ β -naphthol/naphthalene mixture from your laboratory instructor. Record the sample number.
2. Weigh and transfer to a 125 ml Erlenmeyer flask.
3. Add 50 ml of diethyl ether (ethoxyethane) and dissolve the sample by gently swirling.
4. Pour the solution into a 250 ml separatory funnel. Make sure the stopcock is in the closed position.
5. Rinse the flask into the separating funnel with several small portions of ethyl ether.
6. Add 25 ml of 5% sodium bicarbonate (NaHCO_3) to the separatory funnel. This will react with the aspirin to form a water soluble salt.
7. Stopper the separating funnel. While holding the stopper in place with your fingers, invert the funnel and shake several times.
8. Point the spigot upward and away from yourself or neighbors. Slowly open the stopcock to relieve any pressure in the funnel.
9. Close the stopcock and repeat the shaking - pressure release procedure until no further pressure build up is noticed. This will indicate the aspirin/ NaHCO_3 reaction is completed.

Extraction of Aspirin

10. Place the separatory funnel into the ring stand to hold the funnel upright.
11. Remove the stopper from the funnel.

12. Open the stopcock on the separatory funnel and draw off the lower aqueous portion of the liquid into a 125 ml Erlenmeyer flask.
13. Since some of the aqueous solution is still dissolved in the ethyl ether, add an additional 20 ml portion of 5% NaHCO₃ and repeat the extraction procedure. Combine this second aqueous portion with the first.
14. Place the 125 ml flask into a warm water bath (60 °C) and gently heat. This will evaporate any ether still present in the aqueous portion. Do not overheat.
15. Cool the aqueous solution to room temperature.
16. Carefully and slowly, with constant stirring, add 6M HCl with stirring until a pH of 1-2 is indicated by pH paper. The HCl will convert the water soluble salt into the insoluble aspirin. The aspirin will start to precipitate out as the pH is reduced.
17. Vacuum filter the aspirin using a Hirsch funnel and use ice cold water to rinse.
18. Allow to air dry. Weigh, determine melting point, and calculate the percent composition of aspirin in the mixture.

Extraction of β -naphthol

19. Add 25 ml of 10% NaOH to the separatory funnel containing the ethyl ether portion.
20. Using the extraction technique learned previously, invert, shake, and relieve pressure until no further pressure build up is noted. The NaOH is forming a sodium salt with the β -naphthol which is soluble in the aqueous phase.
21. Draw off the lower aqueous portion into a 125 ml Erlenmeyer flask.
22. Add a second 25 ml portion of 10% NaOH to the funnel and extract.
23. Combine this second aqueous portion with the first.
24. Carefully add 6M HCl to a pH of 1-2. The HCl will convert the β -naphthol salt back into β -naphthol which is insoluble in water at the reduced pH. The β -naphthol will precipitate out.
25. Vacuum filter the β -naphthol using a Hirsch funnel and rinse with ice cold water.

26. Allow to air dry. Weigh, determine melting point, and calculate the percent composition of β -naphthol in the mixture.

Collection of Naphthalene

27. Transfer the ether remaining in the funnel to a 125 ml Erlenmeyer flask.
28. Rinse the funnel with an additional 10 ml of ethyl and combine.
29. Add anhydrous calcium chloride (CaCl_2), about 1/10 the volume of the ethyl ether solution, to the 125 ml flask containing the ether. Since there is about 1.5% water still dissolved in the ether, the anhydrous CaCl_2 will absorb the remaining water.
30. Allow the ethyl ether solution to stand over the anhydrous CaCl_2 for 15-20 minutes with occasional swirling.
31. Decant the ethyl ether solution off the CaCl_2 into a dried 125 ml Erlenmeyer flask. Use a cotton plug in an ordinary funnel if necessary.
32. Cover the flask with paraffin wax and poke large holes in the wax. Place the flask in the hood and allow the ethyl ether to evaporate overnight. Make sure the fume hood is turned on so as to vent the ethyl ether vapour.
33. After the ether has evaporated, naphthalene should remain behind.
34. Weigh, determine melting point, and calculate the percent composition of naphthalene in the mixture.

RECRYSTALLIZATION OF ACETANILIDE

Most organic substances are initially produced in an impure form. The substance is mixed with unreacted reagents, side products, and impurities. If the substance is a solid, a process called recrystallization can purify it.

Recrystallization is a process in which the solid of interest is dissolved in a hot solvent that is then slowly cooled. The crystals of the purified product are slowly and selectively precipitated. The impurities remain dissolved in the solution or are removed from the hot solution (before recrystallization occurs) by decolorizing carbon. The crystals are then separated from the solution by filtration.

The solvent selected is based on the solubility of the product to be recrystallized. The product should be highly soluble at high temperatures but only slightly soluble at room temperatures. If the solubility of the product at room temperature is high, the yield of product will be greatly reduced. The selection of solvent is usually on a trial and error basis unless a chemical reference can be found which recommends a solvent.

Many times a mixture of solvents is used. The product is dissolved in a small amount of solvent which it is very soluble in. While still hot, a second solvent that the product is not very soluble in is slowly added until cloudiness appears (products begin to precipitate out). The first solvent is then slowly added until the cloudiness just disappears. The mixture is then slowly cooled and the product recrystallizes.

The boiling point of the solvent should be lower than the melting point of the product. Otherwise, the product may melt in the solvent rather than dissolve. This is called "oiling out". The melted product often contains a great deal of impurities and if allowed to cool, will recrystallize in an impure state.

Decolorizing carbon is used to remove colored impurities from the solution. The carbon has a large active surface area which attracts and absorbs impurities. The carbon is added to the hot solution which prevents recrystallization of the product while the carbon is

absorbing impurities.. A process called hot filtration removes the carbon. The solution is kept hot during the filtration of the carbon to prevent recrystallization and loss of product.

Repeated recrystallization may be necessary to obtain the desired purity.

INSTRUCTIONAL OBJECTIVES

The student will be able to perform a recrystallization.

CHEMICALS AND EQUIPMENT

acetanilide

2 - erlenmeyer flask (250 ml)

filter paper

watch glass

vacuum flask

decolorizing carbon

hot plate

small stirring bar

Hirsch funnel

PROCEDURE

Dissolving the solid

1. Boil approximately 200 ml of DI water.
2. Slowly add the hot water to 5 grams of acetanilide in a 250 ml Erlenmeyer flask with constant stirring until the acetanilide is dissolved in the minimum amount of boiling solvent. If needed, place the flask in a hot water bath to keep the solution hot and prevent recrystallization. Do not place directly onto the hot plate because this might cause *oiling out*.
3. Using a minimum amount of solvent will return a greater yield of acetanilide upon cooling. Also remember that the solid may dissolve slowly. Do not add the hot solvent too rapidly.

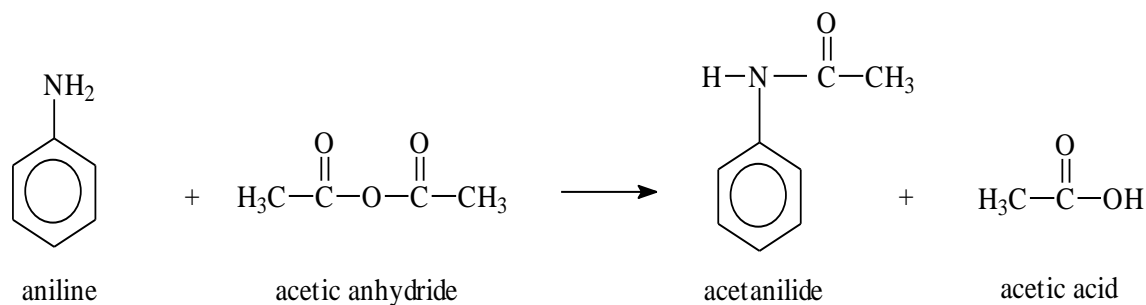
Recrystallization

4. Allow the solution to slowly cool to room temperature. Crystals will begin to form as the solution cools. It is recommended to leave the solution standing overnight. If the solvent is volatile, cover it with paraffin or a watch glass. In this experiment, we will just wait until room temperature.
5. Place the flask into an ice bath and allow cooling.
6. Filter the cold solution by vacuum filtration using a Hirsch funnel.
7. Rinse the collected crystals with COLD solvent.
8. Continue to pull air over the crystals using the filtration device.
9. Remove the crystals and filter and allow drying in a covered watch glass.
10. Weigh and calculate % yield.

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SYNTHESIS OF ACETANILIDE

Acetanilide can be prepared from the acetylation of aniline by the following reaction:



EQUIPMENT & CHEMICALS

aniline (10 grams)
acetic anhydride (10 ml)
zinc dust (pinch)
erlenmeyer flask (500 ml)

graduated cylinder (10 ml)
hot plate
vacuum distillation apparatus

EXPERIMENTAL OBJECTIVES

Synthesize and purify acetanilide.

PROCEDURE

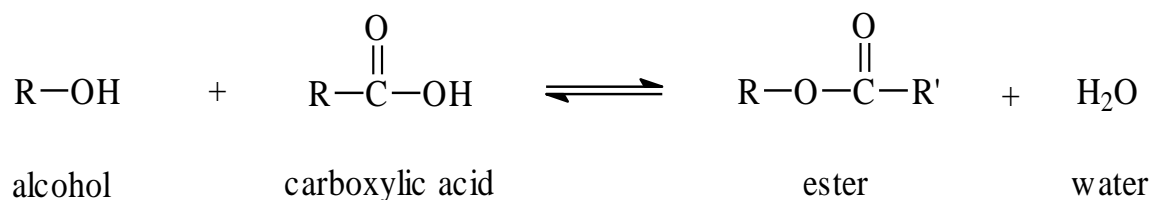
1. Using a 10 ml graduated cylinder, add 4.0 ml of aniline (0.04 moles) to a 500 ml Erlenmeyer flask.
2. Add 30 ml of DI water and a pinch of zinc dust to the flask.
3. With constant stirring, slowly add 6 ml of acetic anhydride (0.06 moles). This should be done in several small portions.
4. Crude acetanilide should slowly begin to precipitate. Continue to stir the solution for at least 20 minutes.
5. Dissolve the precipitant with a minimal amount of hot water. Remember that the zinc dust will not dissolve.

6. Remove the zinc by performing a hot filtration.
7. Recrystallize the acetanilide.
8. Calculate the % yield using aniline as the limiting reagent.
9. Compare the melting point with the literature value. What does this indicate?

THE SYNTHESIS OF ESTERS

The flavor of almost any fruit and many foods is due primarily to a class of organic compounds called esters. Since esters can be easily produced in the laboratory by a process called esterification, the food and beverage industries are very interested in these synthetic esters. While naturally obtained esters are preferable, they are not often suitable for the food and drink industry. Natural esters can produce "off-flavors" when heated or may have a short shelf life. Since the demand for flavors has increased dramatically over the years, the availability of natural flavors cannot meet the demand, thus, the widespread use of synthetic flavorings. In recent years, the use of synthetic flavors is more common than the use of natural flavoring. In fact, many of the flavors you associate with fruits, have nothing to do with the natural flavor. These synthetic flavors have been "enhanced" by the flavoring industry.

Esterification is the reaction of an alcohol and a carboxylic acid. The reaction is reversible and uses an acid as a catalyst.



To force the reaction to the right (ester half) an excess of either alcohol or carboxylic acid is added. The crude product is washed with cold water in a separating funnel to remove most of the acid and some of the unreacted alcohol and carboxylic acid. The product is then washed with sodium bicarbonate to convert any remaining acid and carboxylic acid to a water soluble salt. The partially purified ester is then dried using an anhydrous drying agent and distilled.

EQUIPMENT & CHEMICALS

One of the carboxylic acid and alcohols in list

Sulfuric acid (conc)

Sodium bicarbonate

Sodium sulfate (anhydrous)

Separating funnel

Blue litmus paper

Distillation apparatus

EXPERIMENTAL OBJECTIVES

- Describe the esterification reaction.
- Perform a reflux.

PROCEDURE

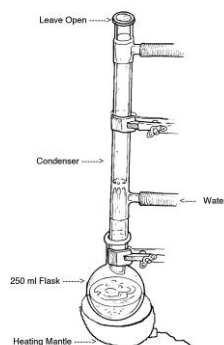
1. Using Table 1, choose a flavor. The flavor is matched with the carboxylic acid and alcohol.

Favor	Alcohol	Carboxylic Acid
Banana	Isoamyl alcohol (0.25 moles)	Acetic acid (0.60 moles)
Peach	Benzyl alcohol (0.20 moles)	Acetic acid (0.60 moles)
Pear	Propanol (0.35 moles)	Acetic acid (1.0 moles)
Pineapple	Ethanol (0.35 moles)	Butyric acid (0.70 moles)
Raspberry	Isobutanol (0.30 moles)	Formic acid (0.60 moles)
Wintergreen	Methanol (1.5 moles)	Salicylic acid (0.1 moles)

2. Fill in the following table:

Favor	Ester	Alcohol	Carboxylic acid
Name			
Molecular weight			
Boiling point			
Density			
moles needed			
grams needed			
ml needed			

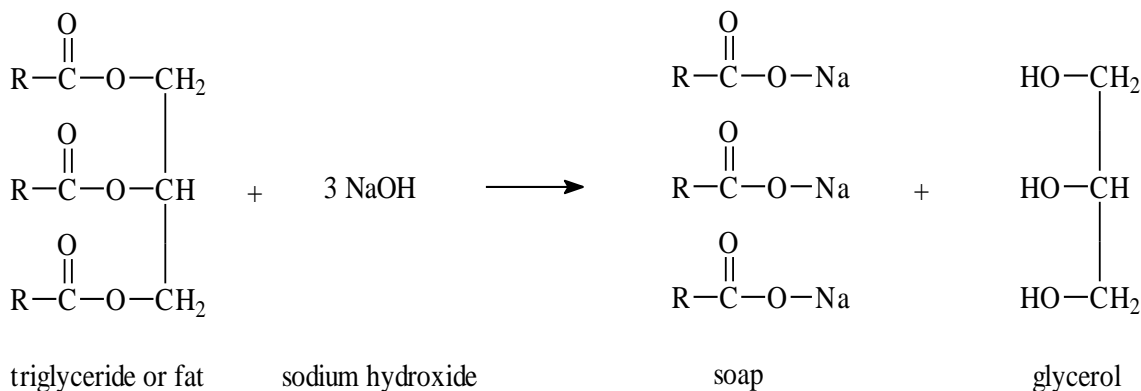
- In a 250 ml ground glass flask mix the alcohol and carboxylic acid in the amounts indicated. *Remember: grams = moles x mol wt and ml = grams/density.*
- Many of the carboxylic acids have disagreeable odors and should be recapped immediately and spills prevented. Butyric acid smells like rancid butter and should be done in a hood.
- Slowly add 5 ml of concentrated sulfuric acid (H_2SO_4) and swirl.
- Add a couple of boiling chips and reflux for 2 hours (Figure 1).
- The reflux will allow the mixture to heat without loss of components through evaporation. The vertical condenser returns the evaporated liquids to the boiling flask.
- Cool the mixture to room temperature and pour into a separating funnel.



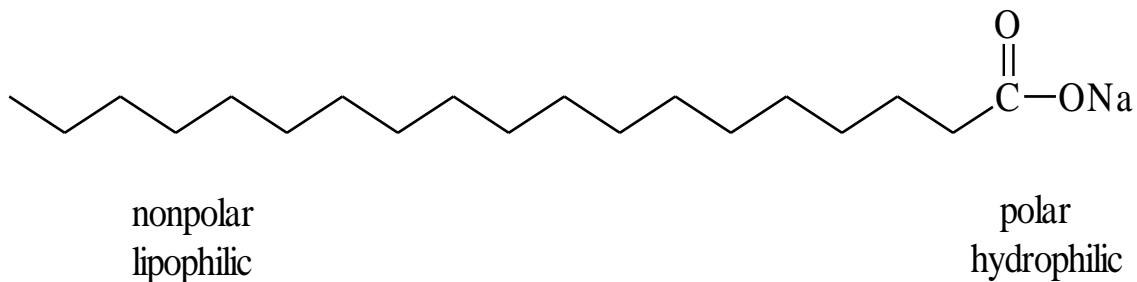
9. Rinse the flask with several portions water and carefully add to the separatory funnel. Caution is needed because the separating funnel contains concentrated acid from the reaction.
10. Carefully add ice cold water to the funnel until the aqueous layer is about twice the volume of the organic layer. This will remove excess acid.
11. Extract and separate the organic layer from the aqueous layer. Make sure you know which layer is which. If the ester and water layer do not separate because of similar densities, add about 30 ml of methylene chloride. The lower layer will be the ester and methylene chloride. The methylene chloride can later be removed in the final distillation by slowly heating with a warm water bath.
12. Extract the organic layer with an equal volume of cold 10% sodium bicarbonate (NaHCO_3) solution. This will neutralize excess acid and convert any remaining carboxylic acid to a soluble salt. Remember to frequently release the pressure in the separatory funnel.
13. Test the organic layer with blue litmus and repeat the extractions with fresh sodium bicarbonate until all the acid is removed.
14. Transfer the organic layer to a 125 ml erlenmeyer flask and add enough anhydrous sodium sulfate to cover the bottom of the flask.
15. Allow the crude ester to stand until the liquid clears. Add additional sodium sulfate (Na_2SO_4) if necessary.
16. Decant the dried ester into a 250 ml flask and distill.
17. Collect the boiling fraction in the boiling point range listed for your particular flavor.
18. Using stoichiometry, determine the percent yield of your product and the boiling point.

SYNTHESIS OF SOAP

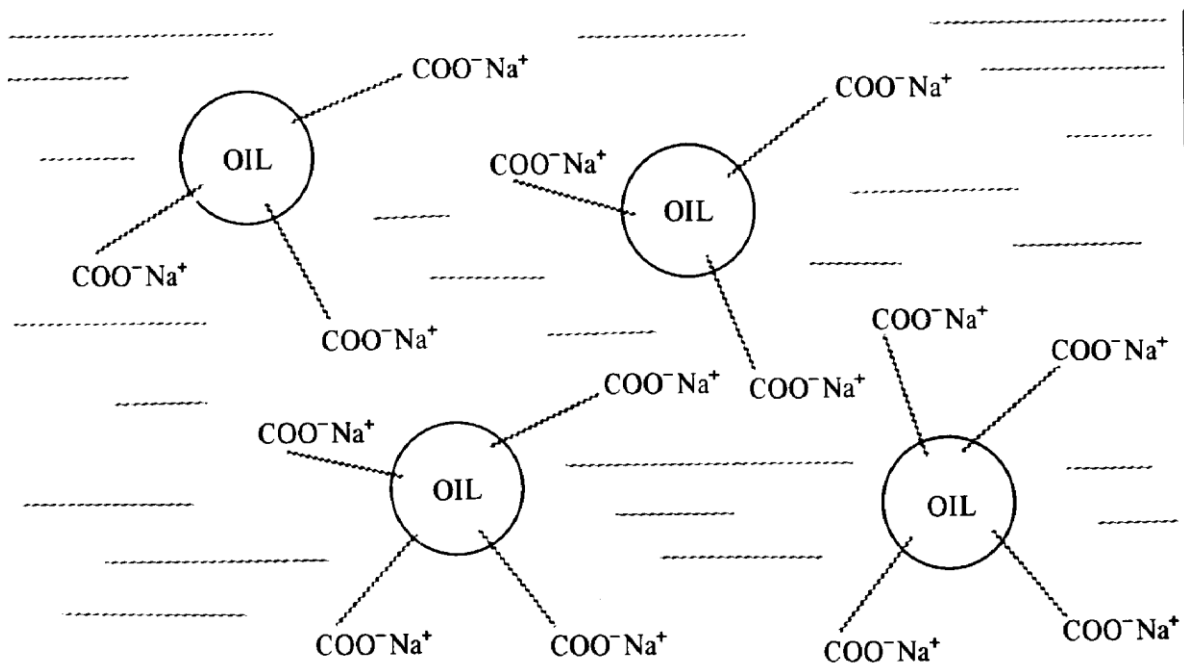
The production of soap is one of the oldest chemical procedures known to mankind. The production of soap is through the alkaline hydrolysis (saponification) of a fat or oil to yield soap and glycerol. Fats consist of a mixture of triglycerides, which are long chain fatty acid esters usually from chains of 12 to 18 carbons.



Soap can clean by using its particular chemical structures.

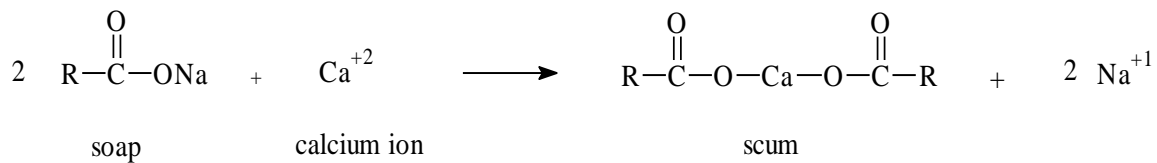


The soap molecule has a polar head which is soluble in water and a non-polar head which is oil soluble. The water soluble head will dissolve in the water (hydrophilic) while the oil soluble head will dissolve in the oil (lipophilic). Each droplet of oil becomes surrounded by soap molecules. Repulsion by the positive water soluble head keeps the oil droplets isolated from each other. When the water portion is removed, the oil portion is removed with it. (Figure 1)

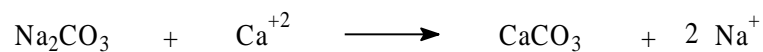


Since oil is what normally holds the dirt in place, removing the oil frees the dirt.

The type of water in which the soap dissolves is very important. Hard water (high mineral content) which contains dissolved Mg and Na salts, will cause soap to precipitate out of solution. The Mg and Ca ions replace the Na on the polar (hydrophilic) end. These salts are insoluble in water (hydrophobic) and the soap precipitates as soap scum.



Sodium carbonate is often added to soaps to act as a water softener. The Na_2CO_3 precipitates out the Ca and Mg ions before they can react with the soap



EQUIPMENT & CHEMICALS

5% MgCl_2	(ethanol)
5% Na_2CO_3	NaOH pellets
Fat or oil	250 ml round bottom flask
Reflux apparatus	Vacuum filtration apparatus

PROCEDURE

1. Add 20 ml of water, 20 ml of 95% ethanol and 6 grams of NaOH to a 200 ml round bottomed flask.
2. Add 6 grams of a fat or oil (lard, crisco, corn oil, etc.) to the flask.
3. Reflux for 30 minutes.
4. Pour the soap solution into 200 ml of a 10% NaCl solution.
5. Cool to room temperature using an ice bath.
6. Collect the precipitant by vacuum filtration.
7. Dry and record the percent yield based on the grams of fat and grams of soap.
8. Dissolve 1.0 grams of your soap in 50 ml of water.
9. Place 10 ml into a test tube and shake. Observe the foam.
10. Add 10 drops of a 5% MgCl_2 solution, shake and observe. What forms? Base this on theoretical knowledge.
11. Repeat step 10, but add 10 drops of 5% Na_2CO_3 first. State your observation and explain.

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ESSENTIAL OILS OF PLANTS

Many plants have compounds within them that produce familiar odours or flavors. These aromas are the result of volatile essential oils. These essential oils are often isolated from the respective plant to produce concentrated natural flavorings. These essential oils may be a combination of organic compounds.

Spices are well known for their flavor and odour. By using a process of steam distillation, one can separate the essential oil from the organic bulk. One requirement of steam distillation is that the compound to be removed is insoluble in water. This results in both compounds distilling at a lower boiling point than the single pure compound.

Each immiscible liquid will exert its own vapour pressure over the liquid, independent of the other liquid. The total pressure is the sum of the individual vapour pressures (Dalton's Law of Partial Pressures). The total pressure must be equal to the atmospheric pressure. Thus, the two compounds will distill at a lower temperature than their normal boiling points.

The oil is then separated from the condensed water using solvent extraction.

EQUIPMENT & CHEMICALS

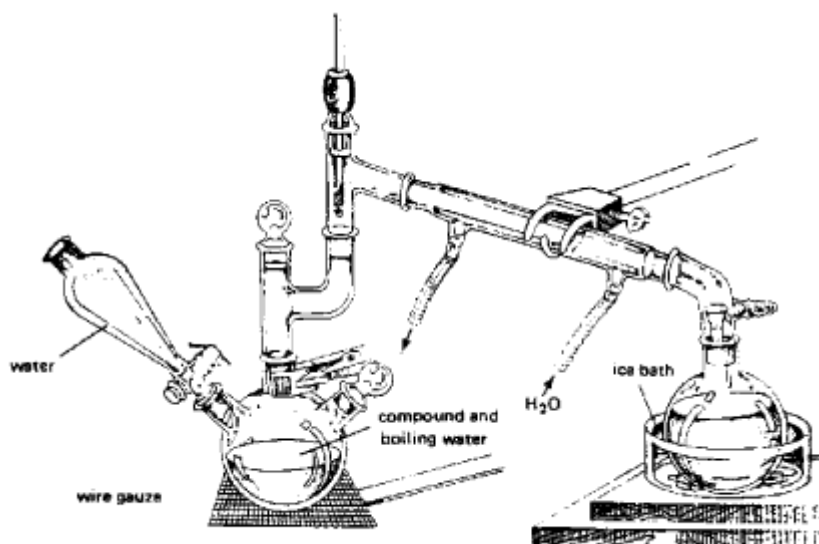
- 125 ml dropping funnel
- 500 ml 3-neck flask
- 250 separating funnel
- heating mantel
- ground spice
- methylene chloride
- anhydrous sodium sulfate

EXPERIMENTAL OBJECTIVES

1. steam distillation
2. solvent extraction

PROCEDURE

1. Add 15 grams of a ground spice to a 500 ml 3-neck round bottom flask.
2. Add 150 ml of DI water to the flask.

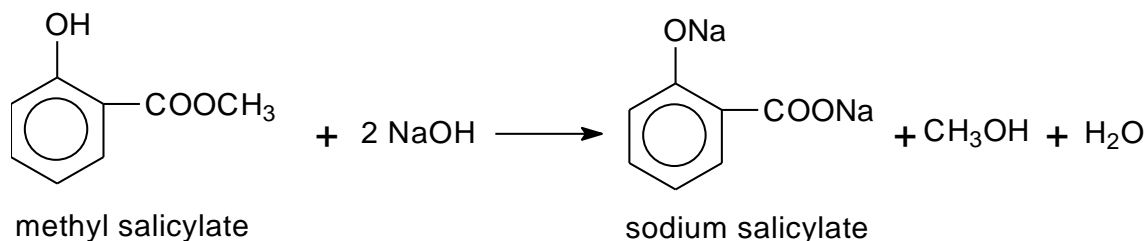


(Figure 1)

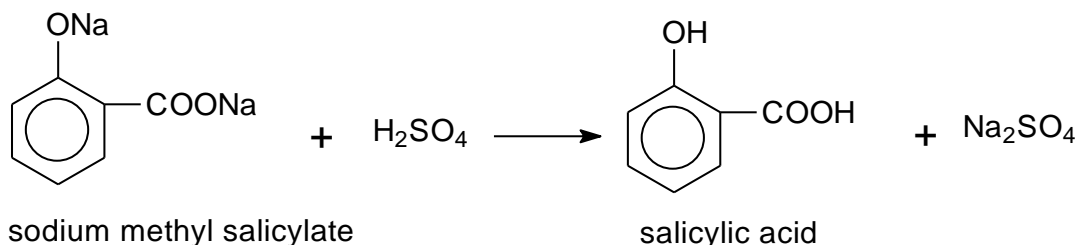
3. Add 125 ml DI to a dropping funnel and assemble the apparatus (Figure 1).
4. Distill the mixture and add sufficient water from the dropping funnel to maintain the original water level in the 3-neck flask.
5. Continue the distillation until no further droplets of oil can be seen distilling over into the condenser. This is normally after 100-150 ml of water has been collected.
6. Extract the distillate in a 250 ml separating funnel twice using 15 ml of methylene chloride each time. Combine the two methylene chloride extractions into a 50 ml flask.
7. Dry the methylene chloride over a small portion of anhydrous sodium sulfate.
8. Decant of the methylene chloride into a pre-weighed dry 50 ml flask.
9. Remove the methylene chloride by evaporating over a hot water bath. Do this in the fume hood.
10. Weigh the dried flask and determine the % yield compared to the original sample of spice.

SYNTHESIS OF ACETYL SALICYLIC ACID FROM OIL OF WINTERGREEN

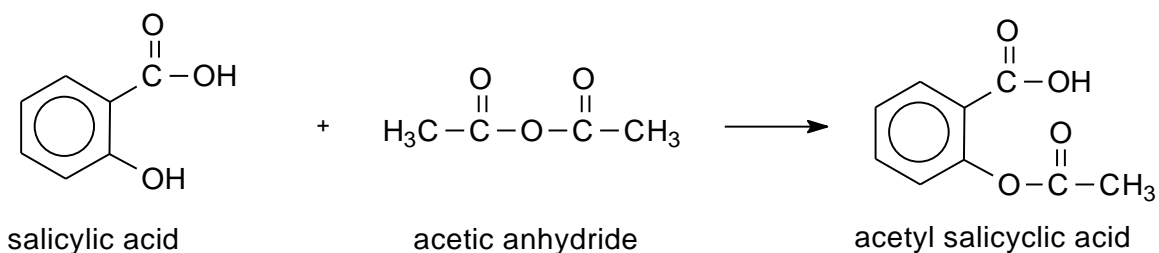
Methyl Salicylate (oil of wintergreen) will be reacted with sodium hydroxide to form sodium salicylate.



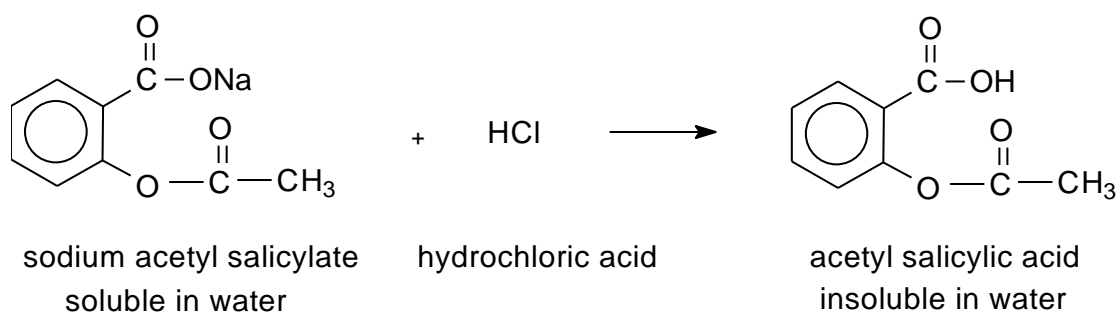
Sodium methyl salicylate is reacted with sulfuric acid to remove the sodium salts and produce salicylic acid.



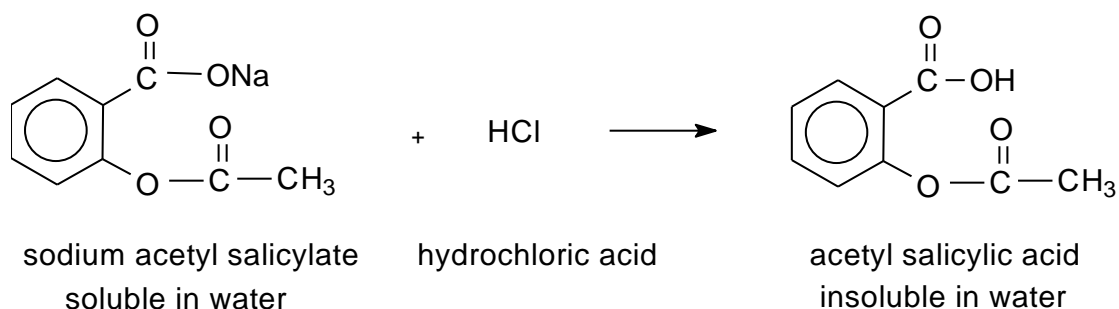
Salicylic acid (o-hydroxy benzoic acid) will be reacted with acetic anhydride to form acetyl salicylic acid (aspirin) that is insoluble in water.



This reaction will also produce a polymer type by-product that needs to be removed. The aspirin is reacted with sodium bicarbonate to form a soluble salt. The polymer that is insoluble is then filtered off.



The pH of the solution is then lowered with HCl. The addition of HCl converts the soluble salt back into the water insoluble aspirin. The precipitated aspirin is then filtered and collected.



EQUIPMENT & CHEMICALS

Methyl salicylate
 Acetic anhydride
 Sodium bicarbonate (saturated)
 Hydrochloric acid (conc)
 Sulfuric acid (conc)
 Sulfuric acid (3M)
 Sodium hydroxide (6M)
 200 ml beaker
 125 ml Erlenmeyer flask
 Vacuum filtration apparatus
 200 ml reflux apparatus

PROCEDURE

Synthesis of Salicylic Acid from Oil of Wintergreen

1. Add 3 ml of methyl salicylate (density = 1.184) to a 200 ml round bottom flask.
2. Add 50 ml of 6M NaOH to the round bottom flask.
3. Add a couple of boiling chips.

4. Reflux for 45 minutes from the point the mixture starts to boil.
5. If the after 45 minutes the reaction mixture still has oil or is cloudy, continue to reflux.
6. Once the mixture is clear and homogeneous, check to make sure that it no longer smells of oil of wintergreen. If the smell still exists, continue to reflux.
7. Remove the boiling chips and transfer to a 250 ml beaker.
8. Slowly add 6M H_2SO_4 until the pH is 2.0.
9. Cool the mixture in an ice water bath for 10 minutes.
10. Vacuum filter and rinse with ice cold water.
11. Dry and weigh.
12. Using stoichiometry, calculate the percent yield.

Synthesis of Aspirin from Salicylic Acid

1. Using the salicylic acid produced above, transfer to a clean and dry 125 ml Erlenmeyer flask.
2. Add a 3X molar excess of acetic anhydride to the flask.
3. Slowly add 5 drops of concentrated sulfuric acid while swirling the flask (or use a stirring bar and stirrer).
4. Gently stir until all the salicylic acid dissolves.
5. Heat the flask in a hot water bath for at least 15 minutes.
6. Remove the flask from the hot water bath and allow the mixture to cool to room temperature.
7. The acetyl salicylic acid (aspirin) that was produced in the reaction should begin to crystallize out of solution during the cooling. Note: if no precipitation occurs, scratch the walls of the flask with a glass-stirring rod.
8. After the aspirin has all precipitated and the solution is at room temperature, add 50 ml of water and place the flask in an ice bath.
9. Vacuum filter the precipitate and rinse the flask with cold water until all the crystals have been collected.
10. Rinse the precipitate with several portions of cold water.

11. Air-dry the precipitate in the Hirsch funnel for about 10 minutes.
12. Transfer the crude aspirin to a 200 ml beaker.
13. Add 25 ml of an aqueous sodium bicarbonate (saturated).
14. Stir the mixture until all activity (bubbling) has stopped.
15. Filter the solution through a Hirsch funnel to remove any polymers.
16. Rinse with about 10 ml of water.
17. Carefully add 3.5 ml of concentrated HCl to a 200 ml beaker that contains 10 ml of H₂O.
18. Carefully pour the filtrate (in small portions) into the 200 ml beaker with constant stirring.
19. The aspirin should precipitate. If no precipitate occurs, add additional HCl until the solution is acidic to litmus paper (blue to red) then proceed to the next step.
20. Cool the solution in an ice bath to precipitate all the aspirin.
21. Vacuum filter the precipitate and wash with ice cold water.
22. Dry the precipitate overnight.
23. Weigh the purified dried acetyl salicylic acid.
24. Calculate the percent yield of aspirin from salicylic acid using stoichiometry.
25. Calculate the overall percent yield based on the original methyl salicylate.