Materials

Rolaids, Maalox, Mylanta, Alka-Seltzer, Baking Soda, CVS-Brand Antacid, Burets, Buret Holder, Ring Stand, Erlenmeyer Flask, Stirring Rod, Medicinal Dropper, Phenolphthalein Indicator, 0.52 M Sodium Hydroxide solution, and 0.44 M Hydrochloric Acid solution

Procedure

1. Add approximately 0.5 grams of powdered antacid to a previously weighed Erlenmeyer flask.

2. To this flask, add 25.0 mL of the 0.44 M hydrochloric acid, and stir the mixture well until all the antacid has dissolved; any undissolved antacid additives will not have any adverse effect on the experimental results.

3. To this clear solution or suspension, add 2-3 drops of phenolphthalein indicator and titrate (the colorless or colored solution depending on the antacid) with the 0.52 M sodium hydroxide.

4. Record the amount of the base (sodium hydroxide) needed to reach the end point (where the color changes from colorless to light purple).

Data Table One

Pre-Lab Questions, Experiment Data Tables, and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment One Word Document Download. Make a copy of the document in a personal Google Drive account or download the document in order to edit.

Antacids	Mass 1 (g)	Mass 2 (g)	Average Mass (g)	Neutralizing Power
Rolaids				
Mylanta				
CVS Brand Antacid				
Baking Soda				
Maalox				
Alka-Seltzer				

Data Table Two

Antacids	Initial Buret Reading (ml)	Final Buret Reading (ml)	Volume of Base used (ml)	Mmol of Base used = mmol of Acid neutralized
Rolaids				
Mylanta				
CVS Brand Antacid				
Baking Soda				
Maalox				
Alka-Seltzer				

Post-Lab Analysis

Calculate the mmol of the initial HCl amount present (mmol = molarity x volume in mL), and then calculate the mmol of NaOH used (volume of based used in titration x molarity of base); the difference between the two mmols is the amount of acid neutralized by the antacid. Use this amount and divide by the average mass of the antacid used to determine the acid neutralizing power per gram of antacid. Additionally, report on the active ingredients of each antacid and argue if one component of the antacid affects the neutralizing potential.

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Experiment Two: Extraction of Fat and Determination of Fat Content of Candy Bars

Pre-Lab Questions, Experiment Data Tables, and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment Two Word Document Download.

Pre-Lab Questions

1. Why is hexane/pentane the proposed solvent for this experiment? Why wouldn't water or any polar solvent work for the extraction?

2. When 2.788 g of Candy A was extracted with hexane, 0.884 g of total fat was obtained. Calculate the percentage of fat in Candy A.

3. Is the use of pentane or hexane expected to produce the same or different results? Explain.

4. How close do you suppose your results will be to the reported value? Explain.

5. Based on anticipated results, would you advise anyone to consume as much or as little candy as they could daily? Explain.

Introduction

Until recently, chocolate manufacturers were never held liable for revealing the fat content of their products. Contrary to popular belief and to the surprise of most consumers, the sugar content in each candy bar is not responsible for the taste; it is the fat. In fact, the more fat these manufacturers can add into their products, the better they taste. How much fat is really in each candy bar? We will extract the fat from Symphony, Hershey's, Nestle Crunch, and Dove candy bars to compute the total fat content and compare our results to their manufacturer's reported numbers.

Background

Our body needs some fats to conduct some of its functions. For instance, we need fat for healthy hair and skin, body insulation, and filler for our fat cells. Fats such as linoleic and linolenic acids are "essential fats" needed for brain development, inflammation control, and blood clotting. These fats cannot be made in the body, so we must rely on external sources to obtain them.

During exercise, our body uses calories we derive from carbohydrate sources, but that is only good enough for 15-20 minutes of exercise. Longer exercise time means that other calorie sources must be present and what better source than good old reliable fat. One gram of fat stores more calories than a gram of protein and carbohydrate combined.

Fat can be saturated or unsaturated. Saturated fat is bad for us because it raises our LDL (Low Density Lipids-bad cholesterol) level, which elevates the risk for stroke, heart attack, and other health problems. Unsaturated (mono and poly) fat, on the other hand, lowers our LDL levels.

Trans fatty acids (also called hydrogenated fats) are also unhealthy. We see them in most products because they are used to keep products fresh. Trans fats lower HDL (High Density Lipids-good cholesterol) levels in the system.

Purpose

The purpose of this work is to determine the fat content in each candy product, thus checking manufacturers' claims. In doing so, the goal is to help people make informed decisions as to which candy to consume and how much.

Materials

Dove, Nestle Crunch, Hershey’s, Symphony, magnetic stir plates, magnetic stirrers, mortar and pestle, hexane/pentane, medicinal droppers, beakers, and balance scale.

Procedure

1. Each candy bar must be frozen in a -80°C degree freezer overnight.
2. Weigh the whole candy bar initially for its total mass, then place some pieces of the candy bar in a mortar and pestle and grind to powder.
3. Weigh about 1.0-1.2 g of the resulting powder and transfer it into a 250 ml beaker.
4. Add two hundred milliliters (200 ml) of hexane/pentane to sequentially extract the fat.
5. Repeat the pentane extraction about two more times, combine the extracts, and leave under a fume hood to dry (it may take up to five days).
6. Weigh the extracted fat and complete the table of results below.

Data and Results

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Candy Bars	Mass 1(g)	Mass 2 (g)	Mass 3 (g)	Mass of Fat Extracted (g)	Mass of Fat Extracted (g)	Mass of Fat Extracted (g)	Average Fat Extracted (g)
Nestle Crunch							
Dove							
Symphony							
Hershey's							

Candy Bars	Amt. of Fat/g	Amt. of Fat/g	Amt. of Fat/g	% Fat/ g	% Fat/g	% Fat/g	Average Fat %/ g	Mass of Fat (total) Reported (g)
Nestle Crunch								
Dove								
Symphony								
Hershey's								

Post-Lab Analysis

After one week of drying (to evaporate the solvent), weigh the extracted fat. Calculate the amount of fat per gram of candy. Report the percentage (%) of fat extracted from each candy bar, based on the initial mass of the candy bar.

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Experiment Three: Identifying Some Constituents of Bones

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Pre-Lab Questions

1. What test, if any, would you carry out to test the collagen obtained?

2. List three characteristics and/or functions of collagen.

3. A solution containing inorganic ions is known to have a pH of 5.00. How many moles of H^+ are present in 400 ml of this solution?

Introduction and Background

Bones are one of nature's many "composite materials" and have unique characteristics such as mechanical strength, hardness, and relatively low density. Bones consist of a tough, somewhat flexible matrix made of collagen (a structural protein) and fibers. This matrix acts as a support network for the inorganic salts that consist mainly of calcium phosphate and other minor constituent ions, such as Mg^{2+}, Fe^{3+}, Na^+, F-, OH^-, CO_3^2. Inorganic salts make up almost 65% of the mass of adult human bones and are responsible for their hardness.

It is possible, through the relatively simple sequence of chemical operations outlined in the flow-chart on the next page, to break apart and separate the major organic and inorganic constituents of bones. Once separated, each component can be individually identified by appropriate analytical testing.

The chemical procedure outlined in the flowchart is an example of a “qualitative analysis scheme.” Its success depends on knowledge of chemical facts, thoughtful planning, and the careful execution of each step.

Purpose

To provide the student with experience in the qualitative analysis scheme, illustrate some reactions of biologically important ions, and acquaint the student with chemical composition of bones.

Materials

Small piece of chicken bone, 250 ml beakers, glasses, 3 M hydrochloric acid, distilled water, wash bottle, hot plate, funnel, funnel support, three filter paper circles, pH paper, glass rods, spatulas, test tubes, test tube rack, 3 M sodium hydroxide, NaOH, 6 M ammonia, NH_4OH, ammonium oxalate, $(NH_4)_2C_2O_4$, Conc. nitric acid, HNO_3, potassium hexacyanoferrate(II), $K_4[Fe(CN)_6]$, potassium thiocyanate, KSCN, 0.1% copper(II) sulfate, $CuSO_4$, and ammonium molybdate reagent

Procedure

Obtain a piece of bone (break into small pieces, if possible) and scrape off any residue of meat, fat, and marrow (if present). Rinse the bone well with water and place it in a small beaker. Add about 20 mL (just enough to cover the bone pieces) of 3 M hydrochloric acid. Cover the beaker with a watch glass and place it in under the hood for one week.

1. After soaking for one week in HCl, the inorganic part of the bone should have dissolved, leaving behind a soft mass consisting of the undissolved collagen structure.

2. Pour the solution into a clean beaker for later use (the solution may be cloudy, with some floating matter); leave the collagen mass in the original beaker.

3. Wash the collagen mass by squirting enough distilled water over it, swishing it around gently, and pouring the water off into a waste collection flask. Repeat this step several times, until the wash water appears neutral when a drop of it is touched to a strip of pH paper.

4. Discard the washing liquid. By doing so, you will have separated the inorganic material (solution) from the organic material (undissolved soft mass).

5. Cover the well-washed, undissolved organic material with distilled water and place the beaker on a hot plate. Allow the water to boil gently for about 30 minutes, making sure that too much water does not evaporate. The heating causes some of the organic material to dissolve in the water.

6. Remove the beaker from the hot plate and set it down to cool.

7. Prepare a fluted filter paper in a clean funnel, wet the filter paper with distilled water, and place the funnel on a ring stand. Filter the cloudy solution from step 1, collecting the liquid in a clean beaker. After all liquid has passed down, discard the paper filter and its contents.

8. Check the acidity of the clear filtered solution by placing a drop of it on a piece of pH paper. It should cause a red (acidic) stain.

9. Gradually add a 6 M ammonia (NH_4OH) solution dropwise while stirring. After the first few drops have been added, check the pH after each addition of ammonia. As soon as a drop of solution touched on a strip of pH paper produces a blue color (basic), stop adding the ammonia.

10. Add acetic acid dropwise while stirring, until a drop of solution produces a red color on the pH paper.

11. In this step, the successive additions of ammonia (weak base) and acetic acid (weak acid) serves to raise the pH and produce a slightly acidic buffered solution. Under these conditions, Fe(III) phosphate, $FePO_4$, is insoluble and precipitates, whereas calcium phosphate remains dissolved.

12. Prepare another fluted filter paper (in a clean funnel) and filter the mixture obtained in Steps 9 and 10. Collect the clear filtered solution in a clean beaker.

13. Wash the solid collected on the filter by filling the funnel with water and allowing the water to drain into a waste-collection flask. Repeat this washing procedure three times.

14. Discard the washing liquid.

15. To the clear filtered solution from Step 12, add 5 mL of ammonium oxalate solution. A white precipitate of insoluble calcium oxalate, CaC_2O_4, should form, indicating the presence of calcium ions in the bone structure.

16. Scrape off the washed precipitate collected on the filter paper in Step 12. Place it into a clean container and add about 10 mL of 3 M HCl. Warm the mixture gently on a hot plate to promote dissolution.

    Use part of the solution obtained in Step 16 to carry out the following test for phosphate ions PO_4^{3-}. Place 2 mL of solution into a clean test tube. Add a few drops of concentrated nitric acid, HNO_3. ***CAUTION:*** Concentrated nitric acid is a highly corrosive material, so warm the mixture gently; do not boil. To the hot solution, add a few drops of ammonium molybdate reagent; a bright yellow precipitate is a positive test for phosphate ions.

17. Use part of the clear solution obtained in Step 16 to carry out the following tests for iron. Place 2 mL of solution in a clean test tube. Add a few drops of solution of potassium hexacyanoferrate (II), $K_4[Fe(CN)_6]$. A blue color indicates the presence of Fe^{3+} ions.

18. Place 2 mL of solution in another clean test tube, Add a few drops of solution of potassium thiocyanate, KSCN. A red color confirms the presence of Fe^{3+}ions.

Post-Lab Analysis

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Correctly list all the ions present in your assigned bone.

Bibliography/Resources

Vallarino, L. M. (1989). *Laboratory Instructions for Modern Chemistry*. Virginia Commonwealth University.

Experiment Four: Synthesis of Tylenol

Pre-Lab Questions and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment Four Word Document Download. Make a copy of the document in a personal Google Drive account or download the document in order to edit.

Pre-Lab Questions

1. If you start with 0.75 g of p-aminophenol and 2.0 ml acetic anhydride (assume this is in excess), calculate the amount of Tylenol expected.

2. Why is it important to acidify the p-aminophenol prior to its reaction with acetic anhydride? Explain.

3. How is the synthesis (making) of Tylenol similar to that of Aspirin? Which starting material is the same or different?

4. How would you estimate the dollar amount involved in the making of one tablet of Tylenol, using the same starting material proposed in this experiment?

5. State some properties of Tylenol.

Introduction and Background

Generically known as acetaminophen, Tylenol is an analgesic used in the treatment of minor aches and in fever reduction. It is the most popular painkiller in the United States, and Americans take over eight billion pills (tablets or capsules) annually. Used at recommended dosage, Tylenol possesses very minor risk but when taken too often, either intentionally or unintentionally for better and faster results, the consequences can be dire. Tylenol, when used as directed, it is safe and effective. The makers of Tylenol say this: "It's important for people to know that it's not the recommended dosage of acetaminophen that poses the risk. Rather, it is when people take more than the recommended dose”.

As with any medicine, paying attention to the dose is important. Here's what the FDA says about this: *"Acetaminophen can cause serious liver damage if you take too much. It is very important to follow your doctor's directions and the directions on the medicine label.*"

Tylenol is also a highly effective, fever reduction (anti-pyretic) agent. In fact, the use of Tylenol, instead of aspirin, to treat fevers in children has reduced the occurrence of Reye's syndrome, an often-fatal form of liver failure. Some symptoms of liver failure because of excessive consumption of Tylenol include vomiting, fatigue, loss of appetite, dark urine and stool and abdominal pain.

Purpose

Synthesis and identification of Tylenol from p-aminophenol and acetic anhydride.

Materials

p-aminophenol, acetic anhydride, Erlenmeyer flask, Buchner funnel, decolorizing charcoal (Norite), water, water aspirator or vacuum, spatula, concentrated hydrochloric acid, filter paper, and glass rod

Procedure

1. Weigh 2.1 g of p-aminophenol into a 125 mL Erlenmeyer flask.

2. Add 35 mL of water followed by 1.5 mL of concentrated hydrochloric acid.

3. Swirl the flask to dissolve the amine hydrochloride formed.

4. Add a few more drops of concentrated acid, if necessary, to dissolve the amine completely as the hydrochloride (it will be difficult to determine since the solution is very dark).

5. Add 0.3-0.4 g of decolorizing charcoal (Norite) to the solution (this is much more than usual but necessary because the crude p-aminophenol contains a lot of polymeric material), swirl the solution on a steam bath for 4-8 minutes, and periodically check to see if the solution is decolorizing (it will be difficult to determine since the solution is dark).

6. Remove the charcoal by gravity filtration into another 125 mL Erlenmeyer flask, using fluted filter paper while the solution is warm.

7. Rinse the filter paper with 1 mL of water.

8. If the charcoal comes through the filter paper, it may be necessary to refilter or to use a filter aid, Celite.

9. The filtrate may be clear or, more likely, a tea color. If the solution is deep brown, add 0.1 g of Norite, heat on the steam bath for a few minutes, and filter. ***The filtrate will darken with time!***

10. While decolorizing the p-aminophenol, prepare a buffer solution by dissolving 2.5 g of sodium acetate trihydrate in 7.5 mL of water, which will give 8.8 mL of solution. Clarify the solution by gravity filtration, if necessary.

11. Warm the filtered aqueous p-aminophenol hydrochloride solution on a steam bath, then add the buffer solution in one portion, with swirling.

12. Immediately add 2.0 mL of acetic anhydride while continuing to swirl the solution. Continue heating on the steam bath, while swirling vigorously for 10 minutes.

13. Cool the solution in an ice-water bath, stirring with a glass rod until the crude acetaminophen begins to crystallize. A little bit of rubbing/scratching with a glass rod near the surface often stimulates the crystallization.

14. After crystallization begins, allow the solution to sit in the ice bath for an hour. Filter your product using a Buchner funnel and the water aspirator or house vacuum line. Wash (rinse) the crystals once with small amount of cold water (a few mL should suffice). Allow the crystals to air dry under vacuum.

15. Collect the crude crystalline product and weigh to the nearest tenth of a gram. Record the weight.

Recrystallization Procedure

1. To all but 100 mg of your crude Tylenol, dissolve the solid using the minimum amount of hot (boiling) water. Do this carefully and avoid using too much hot water.

2. Keep the solution hot and add another 2 mL of hot water. If there are no insoluble particles in the solution, you can allow it to cool slowly without having to first filter. If not, decant the hot solution or try to remove the particles with a spatula or Pasteur pipette, while keeping the solution warm.

3. After crystallization begins, cool the solution more rapidly using an ice bath. When crystallization completes (15 minutes), collect the crystals, rinsing once with a few mL of cold water, and air dry.

4. Record the weight of the dry, recrystallized Tylenol.

Post-Lab Analysis

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Calculate the percent recovery from recrystallization (for instance, if you obtain 0.62 g of recrystallized product after starting with 1.0 g crude product, you have 0.62/1.0 x 100 = 62% recovery). Calculate the theoretical yield of dry recrystallized product from the initial amount of p-aminophenol used. Use the actual recrystallized amount obtained and calculate the percent yield of Tylenol. Using the melting point apparatus, obtain the melting point of your Tylenol (Lit MP is 169-170.5°C).

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Experiment Five: Separation and Identification of Components of Combination Analgesics

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Pre-Lab Questions

1. Which component of the combination analgesic would come out first? Which solvent would you use in its extraction and why?

2. Cheysuprin, by its name contains sucrose, aspirin and one other analgesic. Can you use an organic solvent to differentiate the two analgesics?

3. Compare the structures of phenacetin and acetaminophen. What is the same about them and what is different?

4. Name all the functional groups present in acetaminophen.

5. Is phenacetin water soluble? Explain.

6. How is the synthesis (making) of Tylenol similar to that of aspirin? Which starting material is the same or different?

Introduction and Background

Among the over-the-counter pharmaceuticals with the greatest sales are the common analgesic, led by aspirin. Aspirin, or acetylsalicylic acid (ASA), was once the only choice for the relief of pain (analgesic properties) for reducing fevers (antipyretic properties) and reducing swelling, particularly the swelling associated with rheumatoid arthritis (anti-inflammatory properties). Even today, over twenty million pounds of aspirin are sold in the US annually. Aspirin is prepared from salicylic acid, which was originally derived from salicin, a compound first isolated from the bark of the white willow tree (Salix alba). The use of aqueous extracts of willow bark to relieve fever and pain was first reported in the late 1700's in England but goes much further back in history. In the middle 1800's, salicylic acid was obtained from willow bark, from the meadowsweet plant (Spirea Ulmaria), and from oil of wintergreen (from Gaultheria procumbens). It was found to be effective as an analgesic and antipyretic compound. However, physicians also found that salicylic acid was so acidic that it can cause irritation and bleeding in the mucous membranes of the digestive tract. In 1893, Felix Hofmann, a chemist working for the German dye and pharmaceutical company, Bayer, found a way to prepare the acetyl derivative of salicylic acid (ASA) in a large scale. This derivative proved to be as effective as salicylic acid in the relief of pain and fever with less irritability. Bayer marketed this drug under the trade name aspirin, and for many years, controlled a lucrative market in over-the-counter sales of this pharmaceutical.

The advent of aspirin did not stop pharmaceutical firms from investigating other potentially analgesic drugs. There are people who are allergic to aspirin or who cannot take aspirin due to aspirin's abrasive side effects of the digestive tract. Once again, Bayer was first with the next effective analgesic, phenacetin, which was prepared from p-aminophenol, a by-product of the manufacture of another company made by Bayer. For a number of years, phenacetin, which had exceptionally good analgesic and antipyretic properties, was used as a component of APC tablets, an analgesic preparation that contains aspirin, phenacetin, and caffeine. However, phenacetin has now been supplanted by acetaminophen, which is easier to produce. Acetaminophen is the sole analgesic component in over-the-counter products such as Tylenol and Datril. However, acetaminophen and phenacetin also cause a type of anemia called methemoglobinemia. While it does not seem to be a major problem, certain people are more susceptible to this anemia and should avoid using products containing acetaminophen.

Salicin

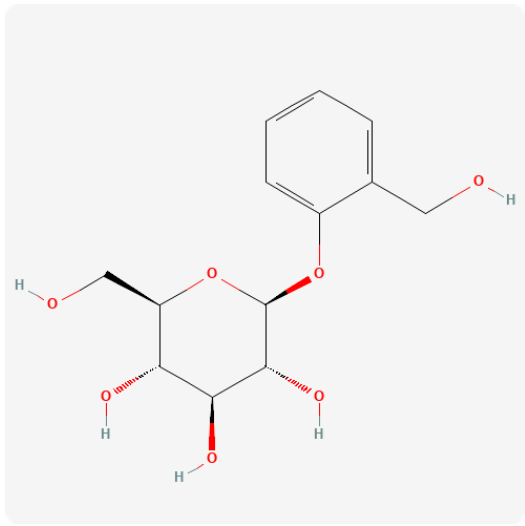


Figure 1

Salicylic Acid

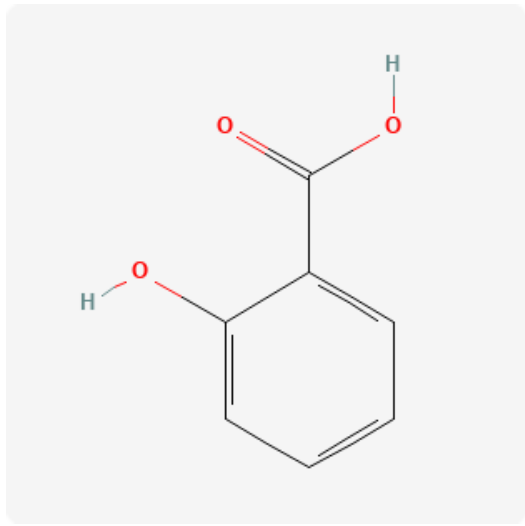


Figure 2

Acetylsalicylic Acid

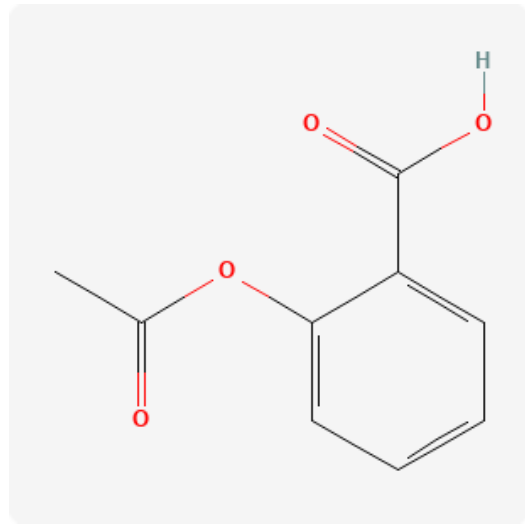


Figure 3

Phenacetin

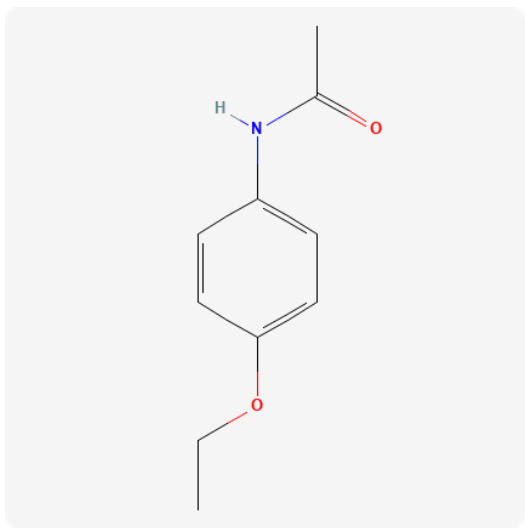


Figure 4

Acetaminophen

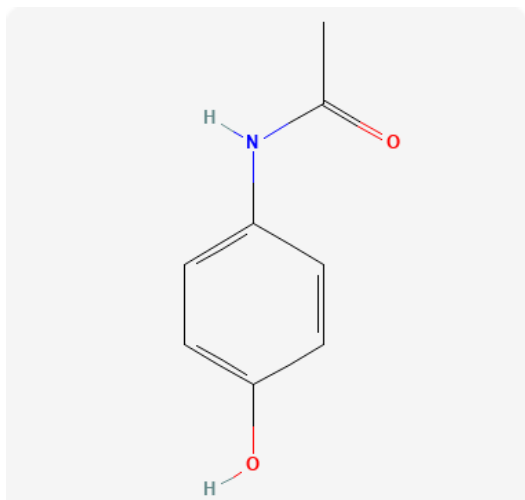


Figure 5

Caffeine

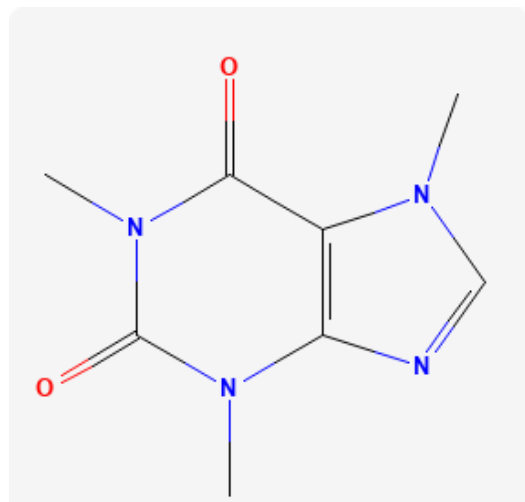


Figure 6

P-Aminophenol

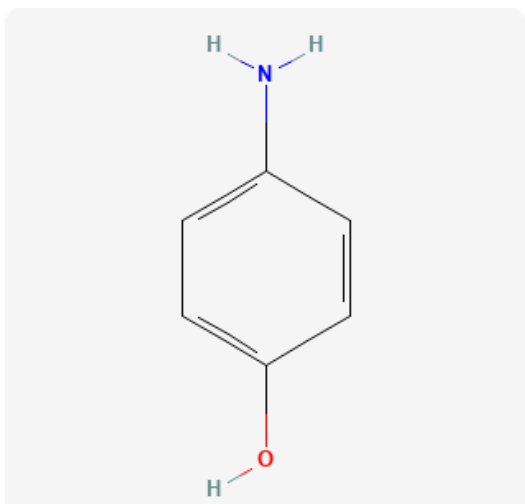


Figure 7

More recently, another compound has become popular as an over-the-counter product. This is the compound Ibuprofen, which is principally an anti-inflammatory compound. It also has analgesic and antipyretic properties and was first marketed under the trade name Motrin. It is more effective than aspirin as an analgesic and requires lower doses to achieve the same pain relief. It also has effectiveness over a wider dose range than aspirin. Like the other analgesics, Ibuprofen does have some side effects, and people who are allergic to aspirin often take ibuprofen. People with kidney disease, ulcers, hypertension, and heart disease are also advised not to take ibuprofen.

Ibuprofen

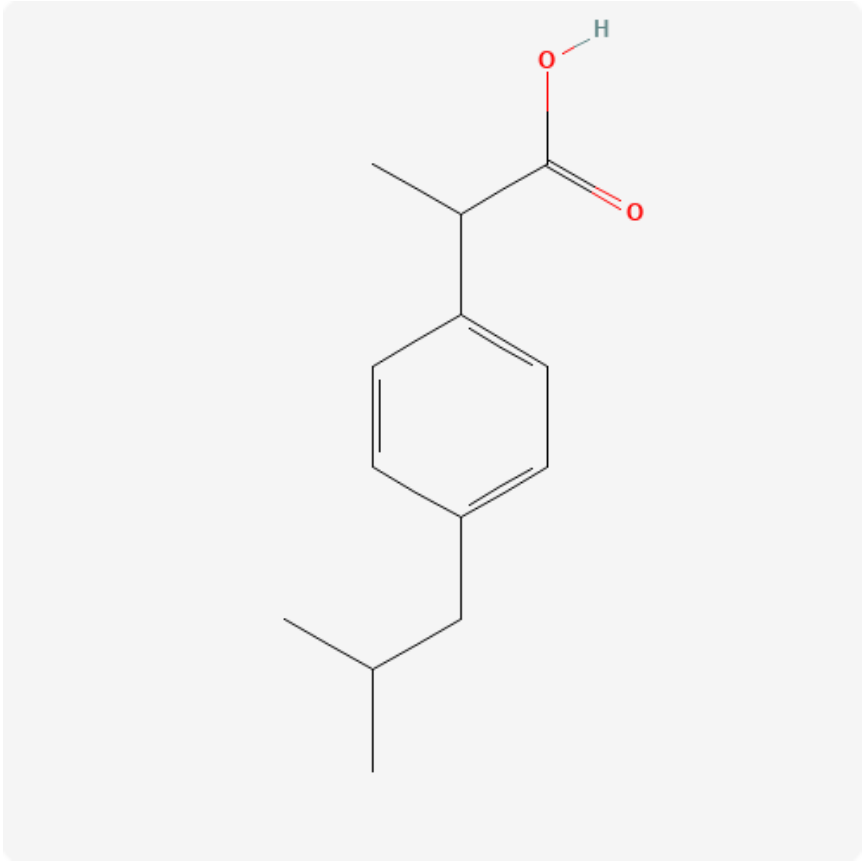


Figure 8

Each tablet or capsule of a commercial analgesic product contains only a small amount of the actual drug. For example, each 5-gram Bayer Aspirin tablet contains only 325 mg of aspirin. Each Extra-Strength Excedrin tablet contains 250 mg of acetaminophen, 250 mg of aspirin, and 65 mg of caffeine. The remainder of the tablet is made of inert filters, such as starch and binders to hold everything together. This is typical of many over-the-counter and prescription pharmaceuticals. Consequently, if one were to analyze a pharmaceutical preparation, it is often necessary to separate the individual components from one other. One way to accomplish this is to take advantage of the different solubilities of the individual components. For example, many of the fillers and binders are soluble in water but not in organic solvents. On the other hand, the active ingredients are often more soluble in organic solvents than in water. Alternatively, different functional groups within the mixture of organic compounds will undergo characteristic reactions. These differences can be used to achieve a separation.

Objective

In this experiment, you will separate the individual components of a special Cheyney-made analgesic product called Cheysuprin. There are two versions of the analgesic mixtures: Cheysuprin-A and Cheysuprin-B. Both products contain aspirin as one analgesic component and sucrose as a filler. One contains phenacetin as the second analgesic component, while the other contains acetaminophen. You will separate the individual components of one of the Cheysuprins, as assigned by your instructor, based upon the different solubilities and reactivities of each component, then determine the amounts of each component in the Cheysuprin.

Sucrose

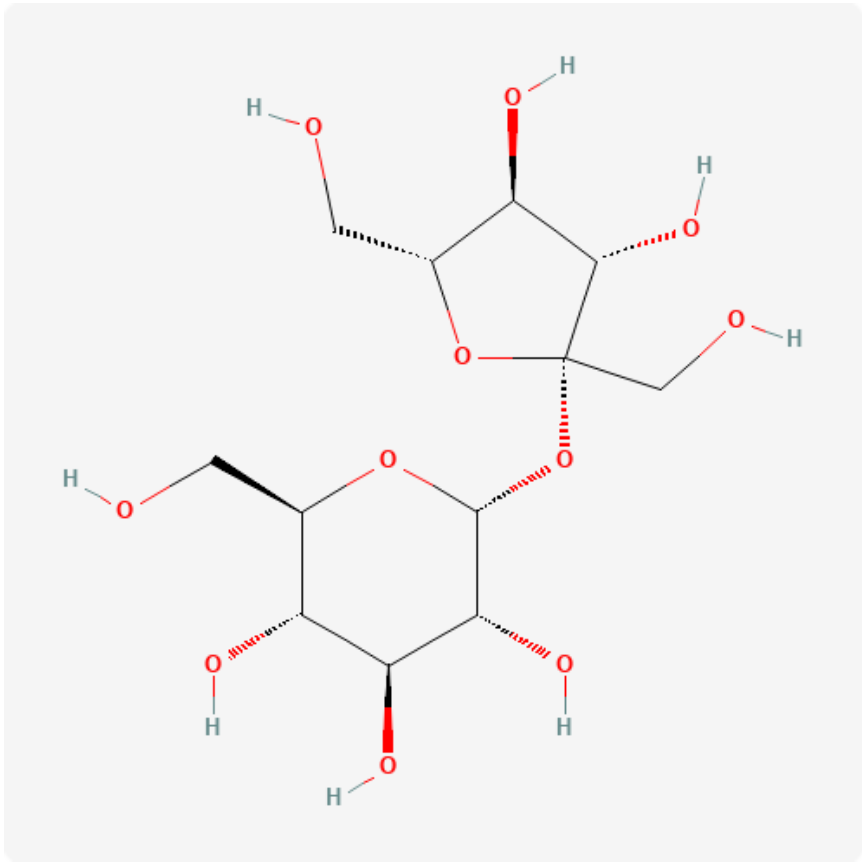
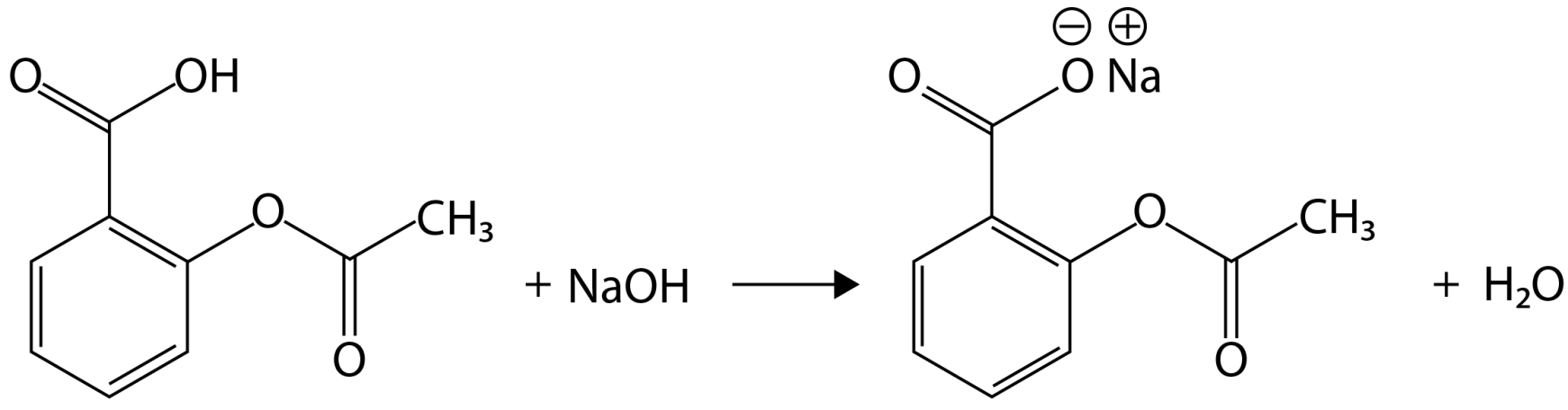


Figure 9

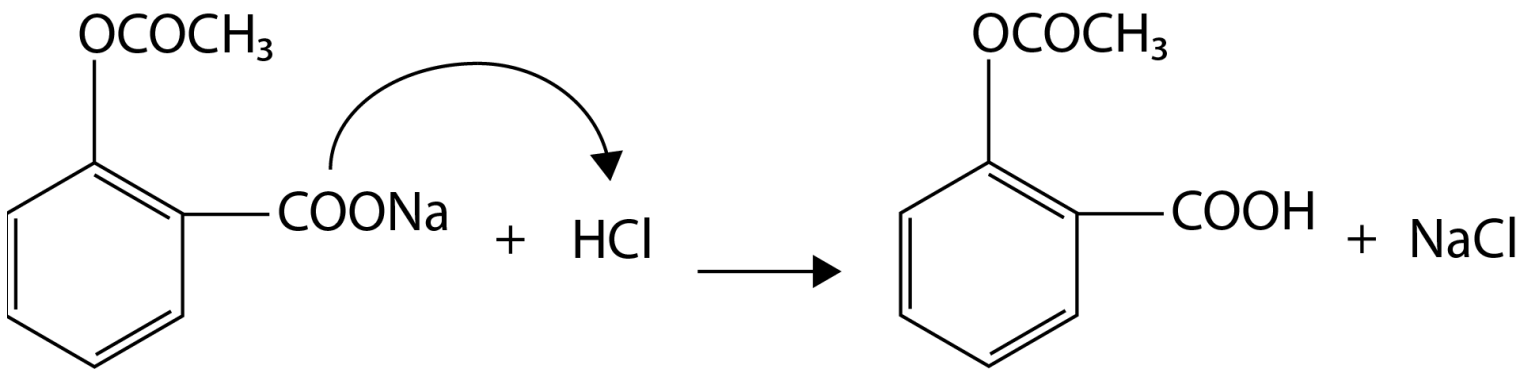
The first step of the separation will be based on different solubilities. Sucrose, or table sugar, is very water soluble, but acetylsalicylic acid, acetaminophen, and phenacetin are not particularly soluble in water. On the other hand, acetylsalicylic acid, acetaminophen, and phenacetin are all soluble in dichloromethane, while sucrose is not. By first mixing the Cheysuprin with dichloromethane, the sucrose can be removed by simple filtration. This is an example of a separation based on solubility. Next, the dichloromethane is extracted with a sodium hydroxide solution. The acetylsalicylic acid, as the name implies, is an acid and sodium hydroxide is a base. They react, as shown below, to form a salt that is now more soluble in water than dichloromethane. The acetaminophen or phenacetin is not affected by treatment with the base unless heat is applied, so the third component remains dissolved in the dichloromethane. Since water and dichloromethane are not miscible, the layers can be separated, thus separating the two analgesic components. The acetylsalicylic acid can then be recovered by converting the salt back to the acid with concentrated hydrochloric acid.



Acetylsalicylic Acid + NaOH conversion to Sodium Acetylsalicylate + H_2O

Chemical Hazards/Caution

- **Acetylsalicylic Acid:** poison, irritant
- **Acetanilide:** toxic, irritant
- **Phenacetin:** cancer suspect agent, mutagen
- **Dichloromethane:** carcinogen, toxic, irritant
- **Sodium Hydroxide:** toxic, corrosive agent
- **Hydrochloric Acid:** poison, toxic by inhalation, corrosive irritant



Sodium Acetylsalicylate + HCl conversion to Aspirin + NaCl

Materials and Supplies

Cheysuprin-A, Cheysuprin-B, 125 mL Erlenmeyer flasks, dichloromethane, spatula, weighing paper, balance, fluted filter paper, funnel, ring stand, O-rings, distilled water, separatory funnel, 1 M sodium hydroxide, NaOH, ice bath, conc. hydrochloric acid, HCl, glass rod, and pH paper

Procedure

1. Weigh approximately 2.5 g of the assigned Cheysuprin (unknown analgesic) sample into a 125 mL Erlenmeyer flask.

2. Add 50 mL of dichloromethane and vigorously swirl the mixture until no more of the solid appears to dissolve.

3. Make sure that any clumps of the solid are broken up during the stirring. Weigh a fluted piece of filter paper.

4. Filter the mixture through the pre-weighed filter paper by gravity filtration.

5. Wash the collected solid (the sucrose) with 5 mL of dichloromethane.

6. Place the filter paper containing the collected sucrose on a watch glass and allow it to dry completely (it may take a couple of days).

7. When the filter paper and sucrose are completely dry, weigh the filter paper plus the solid, and calculate the amount of sucrose recovered.

8. Report the results as a weight and percent of the total weight of the Cheysuprin sample (percent composition).

9. Add the dichloromethane solution to a separatory funnel and extract it with two 25 mL portions of 1M sodium hydroxide solution.

10. Add one portion of the sodium hydroxide solution to the separatory funnel containing the dichloromethane solution, stopper the funnel, and place a finger over the stopper to hold it in place.

11. Shake the funnel back and forth gently for 1-2 minutes (release the stopper every 30 seconds to release fumes. Point away from your face and others!). Keep shaking after venting.

12. When the extraction period is finished, release the pressure one final time, close the stopcock, and place the funnel on a ring stand/clamp.

13. Remove the stopper and drain the lower dichloromethane layer into a flask.

14. When the layer is removed, close the stopcock, and pour the aqueous layer from the funnel into a different Erlenmeyer flask.

15. Return the dichloromethane layer into the funnel and repeat the separation procedure with a second portion of sodium hydroxide.

16. Combine the aqueous layer in the same flask and save the dichloromethane layer in a separate flask for the last step.

17. Cool the combined aqueous layers in an ice bath, and slowly add 10 mL of concentrated hydrochloric acid, with stirring.

18. When the addition is complete, test the pH of the solution by inserting a glass rod, then touching the solution at the tip of the rod on pH paper. (If pH is 2 or lower, add additional HCl until the pH is 2 or below.)

19. Continue to cool the aqueous solution in the ice bath until all the solid acetylsalicylic acid forms.

20. Collect the solid by gravity filtration through a piece of pre-weighed filter paper and wash the solid with 5 mL of cold water.

21. Allow the water to pass through the filter paper in the funnel.

22. Open the filter paper on a watch glass and place under the hood for the collected solid to dry (it may take a couple of days).

Post-Lab Analysis

Pre-Lab Questions and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment Five Word Document Download. Make a copy of the document in a personal Google Drive account or download the document in order to edit.

List the components of Cheysuprin-A or Cheysuprin-B. Calculate the amount of each component, relative to the initial mass of your assigned analgesic.

Bibliography/Resources

Sneden, A. T. (1995) *Organic Chemistry Laboratory Manual*, Virginia Commonwealth University.

Figure References (Return to Experiment 5)

Figure 1: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 439503, Salicin. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Salicin.

Figure 2: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 338, Salicylic Acid. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Salicylic-Acid.

Figure 3: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 2244, Aspirin. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Aspirin.

Figure 4: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 4754, Phenacetin. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Phenacetin.

Figure 5: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 1983, Acetaminophen. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Acetaminophen.

Figure 6: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 2519, Caffeine. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Caffeine.

Figure 7: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 403, 4-Aminophenol. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/4-Aminophenol.

Figure 8: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 3672, Ibuprofen. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Ibuprofen.

Figure 9: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 5988, Sucrose. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Sucrose.