Quantification of Caffeine and Vitamin C in Multiple Beverages

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Abstract

The purpose of this experiment involves quantifying two compounds found in coffee and fruit juices respectively: caffeine and vitamin C, or ascorbic acid respectively. This paper will detail the separation of caffeine from coffee and tea. By quantifying the caffeine contained in a comparative concentration and volume of each drink, we will be able to contrast the amounts of caffeine in each beverage and be able to determine the subsequent relative effects of each drink in terms of their caffeine concentration. Taking advantage of caffeine's strong attraction to, and thus solubility in, dichloromethane, we will be able to use a separatory funnel to separate the caffeine from the other water soluble components of both the coffee and tea.

Vitamin C, or ascorbic acid, acts as an oxidizer in the body and as a reducing agent. In this paper, we will measure the vitamin C in a given volume of freshly squeezed juice as well as a sample of boxed juice and compare their respective amounts. Because ascorbic acid readily oxidizes, we will titrate a solution of iodine into the given samples of juice as an oxidizing agent. Starch that will be added to each juice sample will indicate an excess of iodine and the endpoint of the titration.

Based on known effects of tea and coffee, we expect a lower concentration of caffeine in tea than in coffee.

Nutrient content, and thus vitamin C content is not expected to differ greatly between fresh and boxed fruit juice.

Introduction

Caffeine (1,3,7 trimethyxanthine) is a naturally occuring alkaloid found in leaves, seeds, and fruits such as cola nuts, coffee beans, cocoa beans, tea leaves, and mate leaves. Caffeine has a bitter taste and exists in tea, coffee, and many carbonated soft drinks. It is a central nervous system stimulant (stimulates alertness and reduces drowsiness) and the world's largest consumed psychoactove drug. Unlike many other psychoactive substances, it is legal in almost all parts of the world. Pure caffeine exists as a white and odorless powder. Because of it's moderate polarity, caffeine is slightly soluble in water. Its molecular weight is 194.19 grams, melting point is 236°C, and solubility in water is 2.17%. The FDA limits the amount of caffeine in carbonated beverages to a maximum of 0.02%. Thus, only 71 mg of caffeine is allowed in a 12 ounce soft drink.[(4)]

Dichloromethane (CH_2Cl_2) is a colorless, volatile, polar liquid. It is commonly used as a solvent and is miscible with many organic solvents. Caffeine is more soluble in dichloromethane than it is in water, and this difference in IMF strength is often used in the quantitative analysis of caffeine in a beverage. Water is more polar than dichloromethane, so following only the like dissolves like rule it appears that caffeine would be more soluble in water than dichloromethane.

However, polarity is only one aspect of solubility. The structure of caffeine is mostly planar. There is some polarity on the periphery of the molecule and the large plane is largely hydrocarbon. Thus, it can be said that caffeine is a polar hydrocarbon. Water is polar, but does not interact well with hydrocarbons. This can be seen in water's hatred for oils, which are basically hydrocarbons (oils usually float on the surface of water). Dichloromethane, while not as polar as water (the polarity in dichloromethane comes from the electron density being pushed toward the chlorines), has significant hydrocarbon character. Thus, dichloromethane, a hydrocarbon, dissolves caffeine, a hydrocarbon, better than water.[3]

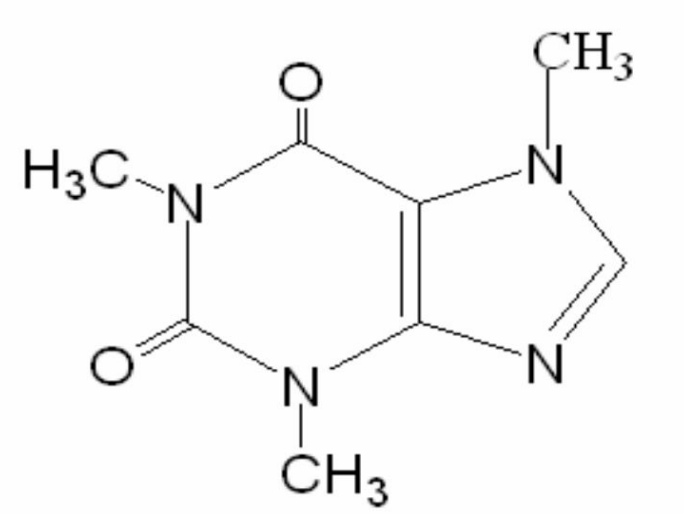

Figure 1: Caffeine (1, 3, 7 trimethylxanthine). The chemical structure of caffeine, an odorless white solid ($C_8H_{10}N_4O_2$). Slight polarity and significant hydrocarbon plane.[4]

Vitamin C (ascorbic acid, ascorbate, AA) is a natural water soluble organic compound. It exists as an oderless white to slightly yellow crystalline powder with a strong acidic taste.[1] Vitamin C is a six carbon chain and is closely related to glucose.[5] It is found mostly in fruits and vegetables like citrus fruits, strawberries, peppers, tomatoes, cabbage, and spinach. It is also found in fruit juice drinks. As it can't be produced or stored by humans, vitamin C must be obtained through consumption. Vitamin C is essential to human diets to maintain connective tissue and bone, and it is considered an antioxidant that fights bacterial infections. The daily requirement of vitamin C is 100 mg per day for the average person. Vitamin C is important in biological processes such as electron transport, hydroxylation reactions, and oxidative catabolism of aromatic compounds in the metabolism of different animals. The role of vitamin C in cells is to reduce hydrogen peroxide (H_2O_2), which preserves cells against reactive oxygen species. Vitamin C content in foods can be affected by climate, harvest method, storing, and processing. As a result, quantification of vitamin C is necessary to monitor its content in agricultural and food products as well as body liquids and tissues.[7] The molecular weight of ascorbic acid is 176.12 grams, and its melting point is 190°C (with decomposition).[1]

The ascorbic acid molecule isn't symmetrical, and it has an abundance of hydroxyl groups in various orientations. These hydroxyl groups are great at forming hydrogen bonds with water molecules, thus ascorbic acid is polar. The quantification of ascorbic acid can be performed through titration methodology, commonly a redox titration with iodine. In this reaction ascorbic acid, a mild reducing agent, accepts electrons from the aqeous iodine, leaving the oxidation state of iodine at a value less than the original. The ascorbic acid in turn is oxidized to a higher oxidation state.[5]

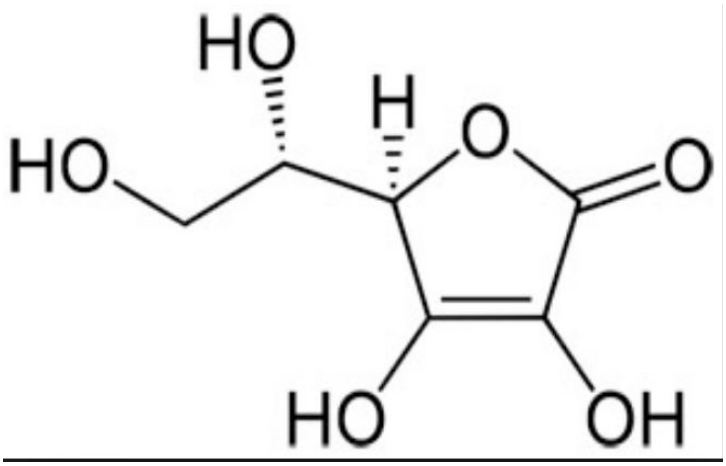

Figure 2: Ascorbic acid. The chemical structure of ascorbic acid, an odorless slightly yellow powder ($C_6H_8O_6$). Every carbon has a polar group on it (hydroxl groups).[7]

Justification

Our procedure for separating the caffeine from both the coffee and the tea revolves around the similar organic components in both: a number of natural pigments, or tannins, as well as a slew of other slightly acidic components, alongside the caffeine itself. Both the tea and coffee will be boiled with sodium carbonate because of the salt's strong ionic characteristics. Because of the presence of the ions, the acidic tannins and other components will react with the sodium carbonate to create polar or ionic particles that will be more attracted to polar water molecules than the non polar dichloromethane particles. Then, both dichloromethane and the boiled coffee or tea will be combined in a separatory funnel. In the separatory funnel, we will cap, shake, and vent. Shaking gently is important to both cause dissolution of the caffeine in the dichloromethane, while also preventing the creation of a suspension. Venting will release the pressure from vapor pressure or newly evolving gases. Then, allowing the contents of the separatory funnel to rest will leave the DCM layer on the bottom, due to its higher density than water. Draining off the DCM and evaporating it will leave us with relatively pure caffeine. Multiple rounds of DCM should be run through the coffee to dissolve as much caffeine as

possible. Part of the error in this method is that there is no realistic way to obtain all of the caffeine since some will still remain dissolved in the water, no matter how negligible.

Determining the amount of ascorbic acid utilizes titration methodology. Since our iodine solution is given in normals, we first have to standardize the solution in terms of molarity by creating a solution of known concentration of ascorbic acid. Once the iodine solution is standardized, we will fill the analyte beaker with starch and titrate the iodine solution into it, where the iodine will react with the ascorbic acid in the following reaction:

$$C_6H_8O_{6\,(aq)} + I_{2\,(aq)} \rightarrow 2I^-_{(aq)} + C_6H_6O_{6\,(aq)}$$

Iodide left as a product of the reaction will remain in solution. After the reaction is complete however, excess iodine reacts with starches to create a dark blue solution, indicating the endpoint of the titration due to the formation of a starch-iodine complex of said color. Titration always has the consistent error in that there will need to be a slight excess of titrant in order to cross the endpoint to indicate that it has passed. In this specific titration, the initial color change may be hard to initially see due to the opacity and initial color of the orange juice.

Materials

- Dichloromethane
- Separatory funnels
- Lab balances
- Watch glasses
- Sodium Carbonate
- Iodine solution
- Starch
- Coffee
- Tea
- Fruit juice
- Ascorbic acid
- Ring stand
- Assorted glassware
- Hot plate

Protocol

Caffeine Separation:

1. Mass about 35g of coffee powder, record mass, and place in 250 mL beaker
2. Add about 4 grams of Na_2CO_3, to make water soluble constituents more soluble in water. Place in beaker
3. Add 100 mL of hot distilled water into beaker until coffee and sodium carbonate are fully dissolved
4. Place solution into separatory funnel

5. Into separatory funnel, add 30 mL dicholoromethane
6. Place finger over the top of the separatory funnel and gently mix solution for about 2 minutes by hand. Every 2 swirls, open separatory funnel to vent any built up pressure
7. Place separatory funnel on ring stand and allow DCM to separate from water portion
8. Mass 125 mL beaker
9. Using separatory funnel, separate bottom DCM layer into 125 mL beaker
10. Repeat steps 5-9 two more times
11. If separated DCM layer is cloudy, empty and wash separatory funnel and prepare a salt water solution
12. Add both DCM fluid and 100 mL salt water solution and mix and vent for 1 minute
13. Empty off bottom DCM layer into 125 mL beaker again
14. Gently boil off DCM at about 50 degrees C
15. Mass beaker after all DCM has been evaporated

Ascorbic Acid Separation:

1. Using graduated cylinder, measure and record the volume of about 30 mL given juice
    a. Alternatively, squeeze a fresh orange or citrus fruit into a beaker until about 30 mL of fresh juice is obtained. Be sure to filter off pulp
2. Pour juice/solution into 250 mL beaker
3. Standardize iodine solution
    a. Mass 1.7612g ascorbic acid and place in 100 mL volumetric flask
    b. Fill 100 mL volumetric flask to the line with distilled water
    c. Swirl to dissolve ascorbic acid
    d. Measure 10 mL iodine solution into 125 mL beaker
    e. Titrate ascorbic acid solution into iodine until solution is completely colorless
    f. Calculate molar concentration of iodine solution
4. Create starch indicator solution
    a. Add 5 g of starch into 50 mL volumetric flask
    b. Fill 50 mL volumetric flask to line with distilled water
    c. Swirl until starch is dissolved
5. Wash a buret and pour iodine solution into buret
6. Titrate by drop the iodine solution into the juice until the juice solution turns a slight blue
7. Record the volume of iodine solution required to neutralize the ascorbic acid

Safety Analysis

General Precautions:

- Wear appropriate eye (safety goggles/glasses) and skin (lab coats and pants) protection
- Immediately wash eyes or skin if contacted
- Remove clothing that becomes wet or significantly contaminated

- Seek medical attention if eyes, skin, or nose are irritated, substance is swallowed, tears are discharged, headache is induced, chest tightens, skin burns, or rash appears
- Immediately remove to fresh air if feeling light-headed
- Always handle materials with labeled containers

Sodium Carbonate (Na_2CO_3): Safety Sheet[6]

- Harmful if inhaled
- Causes eye, skin, and possibly respiratory tract irritation
- Minimize dust generation and accumulation and avoid excess heat or moist air
- Store in a cool, dry, tightly closed container and keep away from acids

Ascorbic Acid ($C_6H_8O_6$): Safety Sheet[2]

- Sensitive to moisture
- May cause irritation of the skin, eyes and respiratory tract
- There have been cases of allergic reaction with eczema, urticaria, and asthma
- When heated to decomposition it emits acrid smoke and irritating fumes

Iodine Solution (I_2): Safety Sheet[2]

- May decompose upon heating to produce corrosive and or toxic fumes
- If contacted will cause irritation to eyes, skin, or nose
- Discharge of tears, headache, chest tightness, skin burns, rash, and cutaneous hypersensitivity are all hazardous symptoms
- Reacts vigorously with reducing materials

Dichloromethane (CH_2Cl_2): Safety Sheet[2]

- If exposed to high temperatures may emit toxic chloride fumes
- Vapors are narcotic in high concentrations
- Inhalation may lead to anesthetic effects, nausea, and drunkenness
- Contact will cause skin, eyes, and nose irritation

Works Cited

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