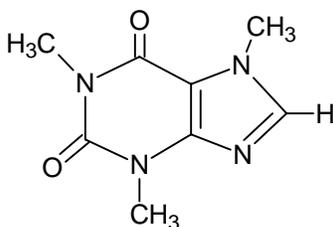


Caffeine extraction from Coffee

Introduction

First isolated from coffee in 1820, caffeine has become the most widely used legal drug in the world. It is estimated that ninety percent of those living in the United States use the drug through the consumption of coffee, tea, soda or over the counter drugs like Vivarin. Caffeine is found occurring naturally in tea leaves, coffee beans, cola nuts, maté leaves and the guarana plant. A white crystalline powder with a melting point of range of 234 to 236.5 °C, caffeine has the chemical name of 1,3,7-trimethylxanthine. Caffeine is an alkaloid, meaning that it is an organic molecule containing nitrogen which has pharmacological effects on humans and animals. The chemical structure is shown below.



Commercially, caffeine is obtained as a by-product from the decaffeinating process of coffee. Caffeine is known to have a bitter taste and is used by Barq's as a flavoring to add a sharp bitter taste to their root beer. The list below shows the amount of caffeine in a 7 oz. cup.

Brewed coffee	80-135 mg
Brewed tea	40-60 mg
Espresso	350-460 mg

Caffeine is a stimulant of the central nervous system, cardiac muscle and respiratory system. It also acts as a diuretic. An overdose of caffeine can cause headaches, muscle tremors and insomnia. Ten grams is considered to be a lethal dose.

When coffee is brewed, caffeine is dissolved into the water along with tannins. These tannins are what give the coffee its dark color. Since we are focused on isolating only caffeine, the tannins need to be removed. The addition of Na_2CO_3 reacts with the tannins to form a salt. These salts are soluble in water but are insoluble in an organic solvent such as chloroform. Even though caffeine is soluble in water to the extent of 1 g per 46 mL, caffeine is more soluble in chloroform to the extent of 1 g per 10 mL. Therefore, caffeine can be extracted by chloroform from the aqueous mixture leaving behind the tannin salts. Evaporation of the chloroform leaves behind the crude caffeine solid.

Procedure

1. Weigh 5 grams of coffee grounds. Be sure you record the mass to the nearest 0.01 grams.
2. Transfer your coffee grounds to a 100 mL beaker. Add 30 mL of deionized water and 2.0 grams of Na_2CO_3 to the coffee grounds.
3. Heat the contents of the beaker until the solution begins to boil. Be sure to stir the solution occasionally.
4. Once the solution begins to boil, continue to boil for another 15 minutes. Be careful not to allow the solution to boil over.
5. Place another 100 mL beaker with 50 mL of deionized water on the hot plate. This water will be needed in step 8.
6. While the solution is boiling, set up a vacuum filtration apparatus. Your TA will demonstrate the proper set up.
7. Once you have boiled the solution for 15 minutes, filter the solution while still hot using the vacuum apparatus.
8. Pour the hot deionized water into the empty beaker, which contained the coffee solution, and swirl. Pour this solution into the Buchner funnel and filter. This step helps to insure all the caffeine is in solution.
9. Transfer the filtered solution to a 125 mL separatory funnel and allow to cool to room temperature. (You may want to use ice to facilitate the cooling process.)
10. Once cool, add 8 mL of chloroform to the separatory funnel.
11. Stopper the separatory funnel and slosh solution back and forth. **Do not shake! Vigorous shaking will form an emulsion which will not separate.**
12. Occasionally open the stopcock to release any pressure within the funnel. Be sure funnel is pointing away from you before opening.
13. Place the separatory funnel upright in a ring stand or funnel holder, remove the stopper and allow mixture to separate. (Which layer is the organic layer? *Hint: The density of chloroform is 1.492 g/mL.*)
14. Once separated, collect the organic layer into 25 mL Erlenmeyer flask. Be sure none of the water layer is transferred.
15. Repeat steps 11 thru 14, except use only 5 mL of chloroform.
16. Combine the two organic layers and add a small scoop anhydrous Na_2SO_4 and swirl. Continue adding Na_2SO_4 until the drying agent is no longer sticking to the sides of the flask but looks fluffy or granular.
17. Using a 25 mL vacuum flask and a Hirsch funnel, filter off the drying agent.
18. Transfer solution to a pre-weighed 25 mL round bottom and use the rotovap to remove the chloroform. (Your TA will demonstrate the use of the rotovap.)
19. Once all the solvent has evaporated, reweigh your 25 mL round bottom and calculate the mass of the caffeine.
20. Determine the melting point of your caffeine and compare it to the actual value.

Questions

1. Determine the melting point of your caffeine sample.
2. What color is your caffeine sample and what color should your caffeine sample be?
3. Estimate the purity of your caffeine based on its melting point and color of the sample.
4. Calculate the percent caffeine in your coffee sample.
5. Why do you add sodium carbonate?
6. Forgetting to add anhydrous sodium sulfate would alter your melting point. Why?