Extraction: Isolation Of Caffeine From Tea

Did you know that when you brew a cup of coffee or tea you are performing an extraction? This is an example of a solid-liquid extraction. The boiling water dissolves the flavor and color components away from the solid vegetable matter. Extraction is an old, well-known process. Alcohol extracts of vanilla beans and almonds are used as flavorings. Extracts of flowers and fruit peels are used as fragrances in various products from perfume to hand soap. In medicine, drugs have been isolated from plants - quinine from cinchona bark is an anti-malarial and morphine from opium poppy is a narcotic analgesic.

In this experiment you will perform two extractions. In the first, you will brew tea from tea bags with boiling water. In the second, you will extract caffeine from the brewed tea with dichloromethane (methylene chloride). This is an example of a liquid-liquid extraction. There are several criteria for choosing an extraction solvent. First, the desired compound to be isolated must be soluble in the extraction solvent. Second, the extraction solvent must be non-reactive. Third, the extraction solvent must be immiscible with the substrate solvent. If the two liquids do not separate after mixing, then all you have done is make a bigger solution. Fourth, the extraction solvent must be easily removed, usually by evaporation. Low boiling solvents are favored.

The extraction of a compound from one liquid into another is an equilibrium process governed by the solubility of the solute in the two solvents. The ratio of the solubilities is known as the *partition coefficient, k,* and is an equilibrium constant with a value for a given substance, pair of solvents, and temperature.

To a good approximation the concentration of the solute in each solvent correlates with the solubility of the solute in the pure solvent, a value found in solubility tables in reference books.

$$k = \frac{\text{concentration of solute in solvent 1}}{\text{concentration of solute in solvent 2}}$$

$$k \cong \frac{\text{solubility of solute in solvent 1}}{\text{solubility of solute in solvent 2}}$$

Consider compound A, that dissolves in t-butyl methyl ether to the extent of 12 g/100 ml and in water to the extent of 6 g/100 ml.

$$k = \frac{\text{12 g/100 ml t-butyl methyl ether}}{\text{6g/100 ml water}} = 2$$

If a solution of 6 g of A in 100 ml water is shaken with 100 ml t-butyl methyl ether, then

$$k = \frac{x \text{ grams of A/100 ml t-butyl methyl ether}}{6 - x \text{ grams of A/100 ml water}} = 2$$

therefore,

$$x = 4 \text{ g of A in ether layer}$$
$$6 - x = 2 \text{ g of A in water layer}$$

However, extraction with two 50 ml portions of t-butyl methyl ether will be more efficient than one extraction with 100 ml t-butyl methyl ether.

$$k = \frac{x \text{ grams of A/50 ml ether}}{6 - x \text{ grams of A/100 ml water}} = 2$$

then,

$$x = 3 \text{ g A in t-butyl methyl ether}$$
$$6 - x = 3 \text{ g A in water}$$

If the 3 g/100 ml water is extracted a second time with 50 ml of t-butyl methyl ether, we can calculate that 1.5 g will be in the ether layer and 1.5 g in the water layer. So, two extractions with 50 ml portions of ether yields 4.5 g of A (3 g + 1.5 g), compared to 4 g with one extraction of 100 ml. Three extractions with 33.3 ml is even more efficient (4.7 g). Usually three extractions is all that should be done manually because of diminishing returns per extraction. To obtain high yields of valuable solutes, continuous liquid-liquid extractors have been developed.

Experiment

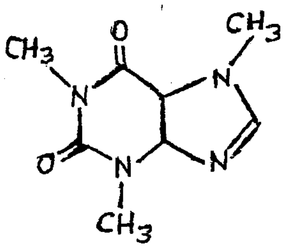
Objective:

The objective of the experiment is to isolate caffeine from tea using extraction.

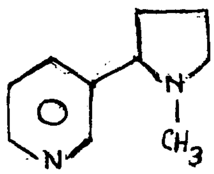
Materials and Equipment:

- tea bags
- deionized water
- sodium carbonate
- dichloromethane (methylene chloride)
- glass wool
- anhydrous sodium sulfate
- wooden boiling stick
- hot plate
- 250 ml beaker
- 400 ml beaker
- 125 ml separatory funnel/stopper
- 125 ml Erlenmeyer flasks
- long stem funnel
- 50 ml beakers or flasks
- glass stirring rod
- boiling stones
- ice bath

Caffeine is a member of a group of compounds called alkaloids (alkali-like). Alkaloids are organic bases containing nitrogen, which are characterized by their bitter taste, and often can induce physiological responses in humans. Caffeine, for example, stimulates respiration, heart rate, and the central nervous system. It is found in coffee (100 mg/cup), tea (30-75 mg/cup), chocolate(6-35 mg/oz), and cola (46 mg/12 oz). Other examples of alkaloids are nicotine, a toxic component of tobacco smoke, capsaicin, which puts the "hot" in hot peppers, and cocaine.



Caffeine



Nicotine

Other compounds are extracted from tea leaves when brewing tea with hot water, among which are tannins. Tannins are acidic compounds found, for example, in the skins and seeds of fruits and cause a dry, astringent feeling on the tongue. Try biting into a grape seed or the "string" from a peeled banana to experience this. If the brewed tea is extracted directly with dichloromethane, the tannins will also be extracted with the caffeine. To prevent this, the tea will be treated with base (sodium carbonate) to convert the tannins to sodium salts which are not soluble in dichloromethane. Careful extraction should give relatively pure caffeine.

Set up a hot plate and place 200 ml deionized water and several boiling stones in a 400 ml beaker. Heat to a vigorous boil. Meanwhile, place 5 tea bags and 10 g sodium carbonate in a 250 ml beaker. Pour 75 ml boiling water over the tea bags and let stand for 7 minutes. Stir occasionally with a glass stirring rod to aid extraction and dissolution of the sodium carbonate. Decant the dark, alkaline tea into a separate 125ml Ehrlenmeyer flask. Pour another 25 ml boiling water over the tea bags and let stand another 5 minutes. Combine the second extraction with the first in the flask. With a stirring rod, squeeze any remaining liquid from the tea bags and combine with the other tea in the flask. Place the flask with the hot tea into an ice bath. *The tea must be at room temperature before the next extraction.*

Set up a ring stand and clamp a small iron ring. Obtain a 125 ml separatory funnel with matching stopper (*the stopper, glass or plastic, must give a tight seal*). Place the separatory funnel in the iron ring, making sure that the ring holds the funnel. *Close the stopcock.* Pour the cold tea into the separatory funnel and then add 15 ml dichloromethane. Seal the funnel with the stopper. Using one hand to secure the stopper in place, invert the funnel and vent by opening and closing the stopcock *(do not point the funnel at anyone). Pressure can build up in a separatory funnel as you work. So, it is important to vent the funnel often.* Mix the two layers by *gently* rocking the funnel back and forth. (While it is important to intimately mix the two layers to get good extraction, too vigorous shaking will cause an emulsion to form. An emulsion occurs when small liquid droplets become dispersed in another immiscible liquid. Stable emulsions can be difficult to separate. Milk is an example of a stable emulsion.)

After mixing, place the funnel back in the ring, remove the stopper, and allow the layers to separate. This may take several minutes. If the layers separate, drain the bottom layer into a clean flask making sure not to allow any of the dark tea to drain as well. Repeat the extraction twice more with 15 ml portions of dichloromethane and combine the extracts.

If An Emulsion Forms

If an emulsion forms, place a wad of glass wool into a long stem funnel in a flask. Drain the bottom layer, including the emulsion directly into the middle of the wad of glass wool. Allow the liquid to gravity feed. Do not squeeze it through the glass wool. If done correctly, clear liquid should pass through and the dark aqueous phase should be trapped in the glass wool.

To complete the drying of the dichloromethane extract, add a little anhydrous sodium sulfate to the extract and swirl. Initially, as the drying agent pulls out the residual water, the powder will form clumps. Keep adding more drying agent with swirling until new powder is free flowing and the liquid is clear (no haze). Let stand 5 minutes.

Weigh a small, clean beaker or flask. Carefully decant the dichloromethane into the weighed container being sure that no solid transfers. Rinse the drying agent with 5 ml of fresh dichloromethane and decant again into the weighed container.

Under the hood, place a wooden boiling stick in your flask or beaker and evaporate the dichloromethane on a hot plate. *A hot plate setting of 1-2 should be sufficient.* Monitor the evaporation closely as you don't want to over heat the isolated caffeine. After complete evaporation, allow the container to cool to room temperature and reweigh to obtain the weight of isolated caffeine. If your instructor has posted the weight of tea found in one tea bag, calculate the percent caffeine in tea.