

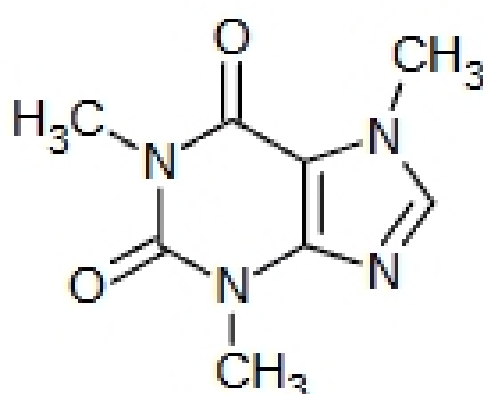
Extraction of Caffeine from Tea Leaves

Introduction

Extraction is a common technique used in organic chemistry to separate or isolate a desired compound from a mixture. The two major types of extractions used in the organic laboratory are solid-liquid extraction and liquid-liquid extraction. Solid-liquid extraction is often used to isolate a natural product from its biological source, such as the leaves or bark of a tree. For example, the anti-cancer drug paclitaxel (Taxol®) can be isolated from the bark of the Pacific yew tree by solid-liquid extraction with methanol. Liquid-liquid extraction is the most common extraction technique, and involves partitioning a solute between two immiscible liquid phases, usually an aqueous (water) phase and an organic solvent phase. The relative concentration of solute in each phase depends on the solubility of the solute in each phase and is given by the partition coefficient (K):

$$\text{Partition coefficient (K)} = \frac{[\text{g}/100 \text{ mL}] \text{ in organic phase}}{[\text{g}/100 \text{ mL}] \text{ in aqueous phase}}$$

Caffeine is an *alkaloid* (a class of naturally occurring compounds containing nitrogen and exhibiting the properties of an alkali, hence the term *alkaloid*) which is found in many sources, such as tea and coffee. In fact, the amount of caffeine in tea leaves can be as much as 5%.



Caffeine

In this experiment, you will first use an aqueous medium (sodium carbonate in water) to extract caffeine (along with tannins, gallic acid, flavinoid pigments, and some other compounds) from tea leaves by a solid-liquid extraction process, in other words, you will make tea (albeit tea that is basic). Caffeine, tannins, gallic acid, and the flavinoid pigments are soluble in this alkaline medium, but the main component of tea leaves – cellulose – is not. You will then perform a liquid-liquid extraction of the tea solution with an organic solvent, methylene chloride, to separate the caffeine from the basic tea solution and away from the tannins, gallic acid and the remainder of the tea components. Since caffeine is more soluble in the organic solvent methylene chloride (CH₂Cl₂) than in water, it partitions primarily into the methylene chloride. The tannins and gallic acid, being acidic compounds, are converted to water soluble salts by the sodium carbonate, and remain in the aqueous layer during the extraction. The other components of tea are not very soluble in methylene chloride. Therefore, the extraction of tea solution with methylene chloride removes virtually no other

compound but caffeine. The solid caffeine can be then be isolated by evaporation of the low-boiling methylene chloride.

Required Reading:

Pavia, Lampman, Kriz, and Engel:

- a) Technique 12 "Extractions, Separations, and Drying Agents", p. 669 (Parts A, C, D)
- b) "Caffeine", p. 87

Special Safety Notes:

Methylene chloride is a somewhat toxic solvent, so handle it as much as possible in a hood, and avoid spilling it on yourself.

Procedure:

Preparation of Tea

Place about 20 mL of water into a 100 mL beaker (use the beaker markings to estimate the volume). When the solution has reached a gentle boil, remove the watch glass and place a tea bag (the instructor will give you the weight of the tea in the bag) into the gently boiling water so that it is covered as completely as possible by the water. Note the approximate level of the liquid in the beaker at this point. Replace the watch glass and continue gently boiling the mixture for about 15 minutes. Water may evaporate during this step, so do not be afraid to add water to maintain the liquid level. Occasionally push down (GENTLY!) on the tea bag with the closed end of a test tube to maintain contact of the tea in the bag with the solution. After the 15 minute boiling period, remove the beaker from the hot plate and squeeze any excess liquid out of the tea bag into the beaker (use a stir rod or test tube to roll the tea bag against the side of the beaker – the tea bag will be HOT!), then add 1.1 g of sodium carbonate (Na_2CO_3) and stir the hot solution with a stir rod until the sodium carbonate is dissolved. Allow the tea solution in the beaker to cool to room temperature. While this initial tea solution is cooling, place the tea bag into a 50 mL beaker, add 5 mL of water, cover with a watch glass, and heat this to a gentle boil for about 2 minutes. Remove this beaker from the hotplate, squeeze the excess liquid out of the tea bag into beaker, then add the contents of this beaker to the tea solution in the original beaker. ***Be sure the tea solution has cooled to room temperature before you proceed to the next section!*** The tea bag from this step can be discarded.