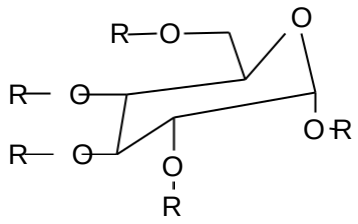
Extraction

Extraction is separation technique used to isolate one component of a mixture. In teaching labs an extraction involves the movement of the desired compound from the mixture into a solvent. There are two basic types of extractions that are seen in teaching laboratories, a liquid/solid extraction and a liquid/liquid extraction. A liquid/solid extraction involves placing a solid mixture in a flask along with a solvent, and it is usually heated to speed up the process. An example is the extraction of caffeine from either coffee grinds or tea leaves with water.

Because of its stimulating effects, caffeine has become the most popularly consumed drug in the world. Tea and coffee are the two most popular beverages that contain caffeine. Tea comes from the plant *Camellia sinesis*, and its consumption dates back as early as 4,700 years ago in China. Today, it is mainly grown in China and India, and it is the most consumed beverage in the world. Coffee has origins in an Arabian legend around the 10th century A.D., though there is evidence that the Ethiopians were chewing coffee berries as early as the 6th century A.D. Arabian goat herders reportedly noticed increased activity of their herd after the goats consumed the berries of a certain plant. Arabian coffee comes from the berry of the plant *Coffea arabica*. Today coffee is cultivated in South America, Indonesia and Africa.

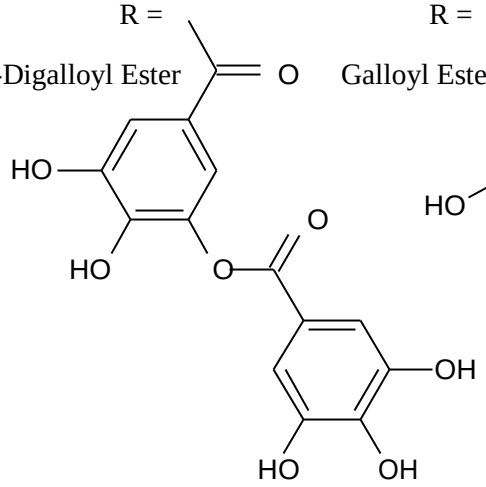
A problem with extracting caffeine from either coffee or tea is that there are other substances that must be separated from the caffeine. The primary impurities that need to be separated during caffeine extraction are called tannins. The term tannins refers to a class of phenolic compounds that can be grouped into two classes: those that can be hydrolyzed and those than cannot. The hydrolyzable tannins are generally esters of glucose and galloyl or digalloyl group. The non-hydrolyzable tannins in tea consist of polymers of catechin.

Hydrolyzable Tannin



R =

meta-Digalloyl Ester



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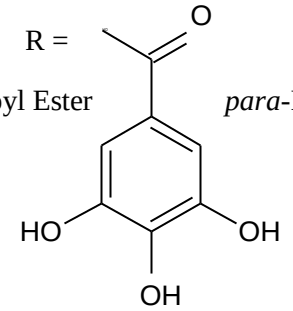
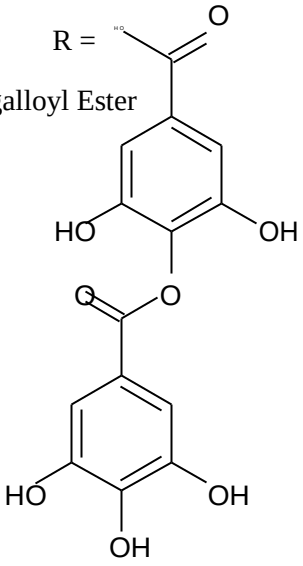
Galloyl Ester



R =

para-Digalloyl Ester



The isolation of compounds like caffeine from natural sources usually requires multiple steps to produce pure product. Often one of the other steps used is a liquid/liquid extraction. In this type of extraction, the mixture is dissolved in one solvent (original solvent), and the desired compound (C) is extracted into a second solvent (extraction solvent). When conducting a liquid/liquid extraction, three areas that need to be addressed to ensure a successful result.

The first area to address for a successful liquid/liquid extraction is the extraction act itself. These types of extractions are normally done in separatory funnels (see figure 1). Both solvents are added to the funnel and the stopper is put in place. The movement of the compound from one solvent to the next occurs at the interface between the two solvents. In order to maximize the movement of the compound, it is important to maximize the surface area contact between the two solvents. This is achieved by shaking the funnel. It is important to shake the funnel correctly. If the funnel is shaken too vigorously, then emulsions may form. An emulsion is a colloidal mixture of two or more liquids that are immiscible with each other If the funnel is shaken too lightly, then not enough surface area contact will occur, which can result in little or no separation. It is also important to vent the funnel periodically. Because the extraction solvents often have high vapor pressures, they will vaporize during the extraction process which builds up pressure. This pressure must be release, and it is achieved by venting. To vent the separatory funnel, invert the funnel so that the stopcock is pointed upwards and away from anyone in the lab. Open the stopcock slowly to release any pressure, then close it and continue shaking. Venting should be performed often during the extraction.

Figure 1 Separatory funnel

The second area to address for a successful liquid/liquid extraction is the choice of the two solvents. When choosing the original solvent (S_O) and the extraction solvent (S_X), care must be taken to ensure optimal results. Four criteria need to be met for a liquid/liquid extraction to be successful.

I. The two solvents, S_X and S_0, must be immiscible. The compound (C) moves from S_O to S_X during the extraction. For the process to succeed, the two solvents will need to be separated from each other after the extraction is completed. This separation will not be possible if the two solvents do not form two separate layers.

II. There cannot be an irreversible reaction between the extraction solvent and the mixture, especially the desired compound. If the result of the extraction is the conversion of C to another compound, then the extraction has failed to isolate C. Thus, this extraction solvent cannot be used. However, if a reaction occurs that can readily be reversed, then the solvent can be considered.

III. The extraction solvent is as specific towards the desired compound as possible. It is important to minimize the number of separation or purification steps as possible because each step leads to loss of some of the product. Therefore, it is important to minimize the number steps necessary to maximize the overall yield.

IV. The extraction solvent should be easily separated from the desired compound once the extraction is complete. Since an extraction results in a solid dissolved in liquid, simple distillation is often used to remove S_X. It is generally beneficial to select extraction solvents that are volatile which makes the distillation easier.

The third area to address for a successful liquid/liquid extraction is to maximize the movement of the compound from the original solvent to the extraction solvent. One measure used to determine if the extraction solvent will result in maximum movement is the partition coefficient (K). The partition coefficient is equal to the concentration of the compound in the extraction solvent (C_X) divided by the concentration of the compound in the original solvent (C_O) as shown below in equation 1. To achieve the best result, a large partition coefficient is preferred.

Equation 1

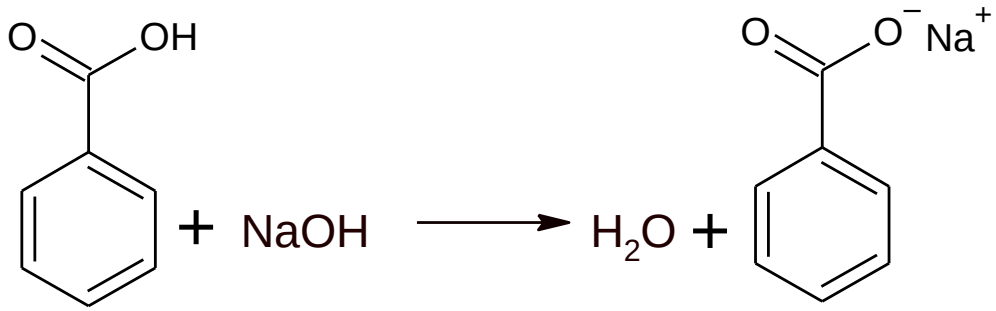
$$K = \frac{[C_X]}{[C_O]}$$

One method to obtain a large partition coefficient is to manipulate the solubility of the compound during the extraction. There are several ways to manipulate the solubility of a compound, such as changing temperature, pressure, ionic strength or pH. An example of how these manipulations effect the extraction is the extraction of benzoic acid from ether into water. If neutral water is used in the extraction, very little if any benzoic acid will be isolated in water because the solubility of benzoic acid in neutral water is small. Thus, the partition coefficient of this extraction is small. However, if a base like NaOH is added to the water, the sodium hydroxide

reacts with the benzoic acid converting it to the conjugate base (see figure 2). The conjugate base (sodium benzoate) is very soluble in water, thus the partition coefficient is much larger, and a large amount of the benzoic acid will be isolated in the water.

A small problem that occurs with using NaOH for the extraction is that the product isolated in water is not benzoic acid, rather the conjugate base. Therefore, the NaOH must be neutralized to reverse the acid/base reaction. This reversal is easily accomplished with the addition of a strong acid, such as HCl. Once the NaOH is neutralized, the benzoic acid will precipitate out of solution. The precipitated benzoic acid can then be isolated by filtration or dissolving it into diethyl ether.

Figure 2 Benzoic Acid reaction with NaOH



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