Solvent extraction

Solvent extraction is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent. This technique is used for isolating or purifying substances by adding a second, immiscible, solvent to the original mixture. If one of the components is preferentially soluble in one of the solvents then it will migrate there and thus, when the mixture is separated again, it can be isolated.

This process is normally carried out using a separating funnel. This is a funnel with a tap, like a burette tap, on the bottom. They can be made from plastic but are most commonly made of glass.

As an example, here is how it can be used in the extraction of caffeine from tea.

1. Once the tea solution has been made, you have a mixture of caffeine, tannins and all sorts of other compounds in an aqueous solution.
2. Allow it to cool.
3. Pour the (roughly) 300 cm3 of solution into a separating funnel (make sure the tap is closed!)
4. Add about 40 cm3 of dichloromethane.
5. Insert the stopper and **gently** swirl or shake the liquids. Holding the stopper but releasing it from time to time to vent the pressure.

*If you don’t vent it, there is a danger that the top will blow off spraying solvent all over the place.*

*Shaking too vigorously, as well as increasing evaporation and thus the pressure, can cause problems with the formation of emulsions that can be difficult to separate.*

1. Place the funnel in a holder (usually a ring adaptor for a clamp) and leave for the two layers to separate.
2. Open the tap and allow the bottom layer to flow out into a beaker below.

*Slow the flow when it gets close and stop the tap when all the bottom layer has come out. (It is best to leave a little of the bottom layer in the funnel rather than allow a small amount of the other layer to contaminate your solution). With a little practice you should be able to separate the two layers with almost no wastage.*

1. In this particular case, the bottom layer is the dichloromethane. Put that aside and add another portion of dichloromethane to the same tea solution.
2. Repeat the shaking, setting and decanting process as described in steps 4 – 7 above.
3. Repeat again with a third amount of dichloromethane
4. Combine the three batches of dichloromethane – these can be allowed to evaporate to leave your caffeine behind.

The exact details will vary: for instance, you may be keeping the top layer rather than the bottom one. In that case, after draining the bottom layer, you drain the top layer into a second beaker and then return the first layer to the flask.

It is important to add the solvent in three batches. Because of the way solutes partition themselves across the system, if you add them all at once then the extraction will not be as efficient.

**Partitioning and choice of solvents**

There is no such thing as a compound that is **totally** insoluble in a solvent. It is simply a matter of degree.

To get the best separation, you need to choose a pair of solvents that:

1) are immiscible

2) have the greatest possible difference in solubilities for the compounds you are trying to separate as possible.

You need to do your research and find the best solvent for your particular extraction.

## Improving the separation

**Salting out**

It is possible to enhance the differences between the solvents.

For instance, it is possible to use propan-1-ol to separate caffeine rather than the more toxic dichloromethan. Just substituting solvents, though, is not very satisfactory but this can be improved by adding NaCl to the caffeine in water solution. The water will be more attracted to the very polar NaCl and less attracted to caffeine thus “salting out” the caffeine from water solution.

**Changing pH**

Altering the pH of a solution can also affect the solubility of a compound and thus improve the separation – particularly with organic compounds.

In the case of caffeine again, adding calcium hydroxide or carbonate makest he solution basic and this causes caffeine to take its least polar form and so more readily solvated in organic solvents and less attracted to water.

In addition, the aqueous solution contains much more than just caffeine, and some of these compounds are also soluble in organic solvents. Basic Ca(OH)2 reacts with tannic acids to form insoluble tannin salts which precipitate and so can be removed from the solution before the caffeine is extracted.