

- **EXTRACTION**

In a chemical sense, extraction is the general term for recovery of a substance from a crude solid or a solution by bringing it into contact with a solvent that preferentially dissolves the desired material. In the isolation of organic compounds from a plant source, extraction of the dried leaf, bark or wood is commonly the first step. In synthetic organic chemistry, a reaction product is frequently obtained as a solution or a suspension in water, along with inorganic and organic by-products. By shaking the aqueous mixture with a water-immiscible organic solvent such as ether or dichloromethane, the product is transferred to the organic layer and can then be recovered by evaporation of the solvent.

The extraction of a compound from an aqueous solution using an organic solvent, or *vice versa*, is an equilibrium process governed by the solubilities of the substance in the two phases. The ratio of solubilities in the two solvents is called the distribution coefficient, $K_D = C_1/C_2$, which is an equilibrium constant with a characteristic value for any compound at a given temperature.

Let us consider the extraction of a compound whose solubilities in ether and water are 10 g/100 mL and 2 g/100 mL, respectively. If a solution of 1 g of the compound in 100 mL of water is extracted with 100 mL of ether, and the amount that transfers to the ether layer is x g, then the fraction of the compound transferred to the ether phase can be calculated as follows:

$$\text{Distribution coefficient, } K_{e/w} = \frac{C_{\text{ether}}}{C_{\text{water}}} = \frac{10/100}{2/100} = 5$$

where C_{solvent} = concentration of the compound in the solvent at equilibrium. If, originally, there was 1 g compound in 100 mL water, and the amount of material in ether in g at equilibrium = x , then there must be $1 - x = g$ of compound in the water at equilibrium. Using this, the distribution coefficient equation can be written as:

$$K_{e/w} = \frac{x/100}{(1-x)/100} = 5$$

Rearranging this gives: $x = 5 - 5x$

Which solves as: $x = 0.83$ g in ether and $1 - x = 0.17$ g in water

If the extraction is carried out with the same amount of ether in two equal portions of 50 mL each then we have for the first extraction:

$$\frac{x/50}{(1-x)/100} = 5; \quad \begin{array}{l} x = 0.71 \text{ g in ether} \\ 1 - x = 0.29 \text{ g in water} \end{array}$$

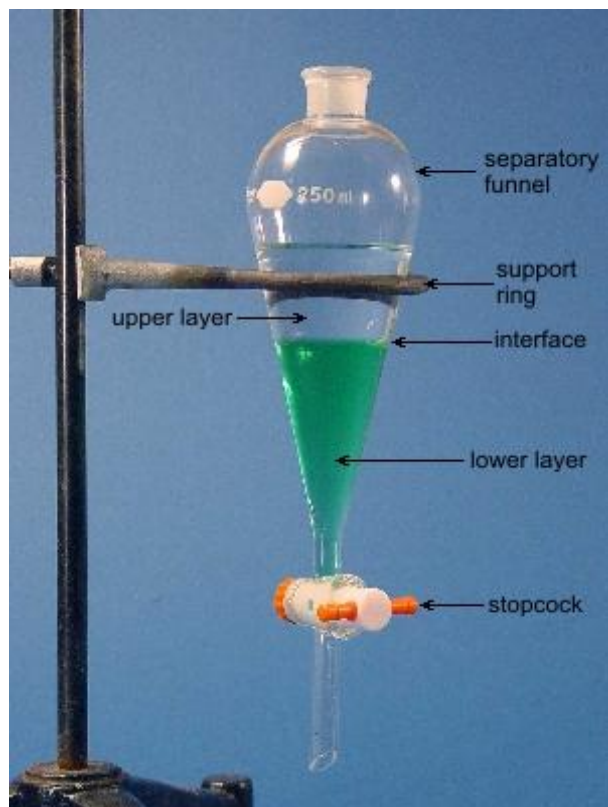
and for the second extraction (x' = g in ether at equilibrium):

$$\frac{x'/50}{(0.29-x')/100} = 5; \quad \begin{array}{l} x' = 0.21 \text{ g in ether} \\ x + x' = 0.92 \end{array}$$

The total amount extracted by 100 mL as 2 x 50 mL portions of ether is thus 0.92 g. (compared to only 0.83 g using 1 portion of 100 mL.)

These values show that virtually complete removal of the compound can be effected, even if the distribution coefficient is very low, by repeated extractions with small volumes of solvent. In practice this is accomplished by use of an apparatus in which the solution to be extracted is continuously treated with fresh solvent. Most applications of liquid-liquid extraction in the laboratory require only a few contacts with fresh portions of solvent, using a simple separatory funnel.

Use of a Separatory Funnel (quick link to video : [extraction](#))



The separatory funnel is a tapered vessel with a stopcock at the bottom which permits a sharp separation of two liquid layers in a liquid-liquid extraction or in any situation requiring the separation of an organic liquid from the aqueous layer. A separatory funnel is expensive and fragile, and when full, it is top heavy. The funnel should be supported on a ring of the proper size at a convenient height; don't prop it up on its stem. Before each use, check that the stopcock is seated and rotates freely. A clip or leash should be used to prevent the stopcock from falling out if it is accidentally loosened. A very light film of stopcock lubricant should be applied around the stopcock in bands on each side of the hole (Teflon stopcocks require no lubricant). Excess grease will be washed away by organic solvents and contaminate the solution.

The separatory funnel should be filled to no more than about three-quarters of the total depth, so that thorough mixing is possible. After filling (check first that stopcock is closed!), stopper securely with a properly fitting glass, plastic or rubber stopper. Before using the funnel for the first time, it is a good idea to shake with a few milliliters of solvent to make sure that stopcock and stopper are tight.



Hold the funnel with the stopcock end tilted up; the stopper is kept in place securely with the heel of one hand and the stopcock end is supported in the other hand. Now gently invert the funnel. As soon as the funnel is inverted, open the stopcock to release any pressure. Then close the stopcock and shake in a horizontal position for about 1-minute. Stop, and slowly open the stopcock a few times to vent any pressure. Replace the funnel in the ring and remove the stopper. When the phases have completely separated, draw off the lower layer through the stopcock.

In the extraction of an aqueous solution, the solvent may be either lighter than water (e.g. ether or hexane) or heavier than water (e.g. chloroform or dichloromethane). In the first case, if several portions of solvent are used, the aqueous layer must be drained into a receiver (usually the flask in which it was originally contained), and the ether solution is transferred to a second flask. The aqueous phase is then returned to the separatory funnel for further extraction as needed. With a solvent denser than water, the aqueous solution is simply retained in the funnel and shaken with successive portions of solvent.

It is sometimes not immediately obvious which layer in the separatory funnel is the organic phase and which is the aqueous. If in doubt, withdraw a small sample of the lower phase in a test tube and add a few drops of water to see whether or not two layers form. If you see two layers then this indicates that the lower phase in the separatory funnel is the organic layer. Alternatively add a few drops of water to the top of the separatory funnel and watch to see which layer the drops end up in. If they sink through the upper layer into the bottom layer, then the lower layer is the water layer.

In many cases the separatory funnel is used simply as a means of recovering an organic product from a large amount of water with minimum mechanical loss. A small volume of ether or other solvent is added to the mixture to permit sharp separation of layers. Even though the compound may have negligible solubility in water, a second portion of solvent should be used to rinse the aqueous layer and the separatory funnel.

A common application of extraction is the removal of water-soluble impurities from an organic solution, e.g. an ether solution may contain dissolved hydrogen chloride. This is removed by 'washing' the ether in a separatory funnel with aqueous carbonate or hydroxide solution and then water.