

EXTRACTION

Extraction is a very common laboratory procedure used when isolating or purifying a product.

Extraction is the drawing or pulling out of something from something else.

By far the most universal and ancient form of extraction is the brewing of tea or the making of coffee. Over the centuries, humans have carried out solid/liquid extraction by brewing just about every common plant leaf, fruit, or root. In the process, they have isolated a number of extracts with pharmacological activity. Many of these compounds were used for medicinal purposes.

While solid/liquid extraction is the most common technique used to brew beverages and isolate natural products, liquid/liquid extraction is a very common method used in the organic laboratory.

Organic reactions often yield a number of by-products, some inorganic, some organic. Also, since they do not go to 100% completion, some starting material is also often present at the end of an organic reaction.

Liquid/ liquid extraction is often used as the initial step in the work-up of a reaction, before final purification of the product by recrystallization, distillation, sublimation or chromatography.

Liquid-liquid extraction requires two immiscible liquids known as the organic phase and the aqueous phase. The aqueous phase is water-based and the organic phase is an organic solvent.

The basis of extractive techniques is the “like dissolves like” rule. Water typically dissolves inorganic salts (such as lithium chloride) and other ionized species, while solvents (ethyl acetate, methylene chloride, diethyl ether, etc.) dissolve neutral organic molecules.

However, some compounds (e.g., alcohols) exhibit solubility in both media. Therefore, it is important to remember that this method of separation relies on partitioning—that is, the preferential dissolution of a species into one solvent over another. For example, 2-pentanol is somewhat soluble in water (i.e., 17g/100 mL H₂O), but infinitely soluble in diethylether. Thus 2-pentanol can be preferentially partitioned into ether.

General extraction procedure: Place the solution to be extracted in the separatory funnel. As the organic solvent and water are not miscible with each other, you should be able to see the two layers (organic and aqueous layers) clearly. Now, shake the separatory funnel to increase the contact between these substances and the water. When finished, the funnel can be returned to the stand and the layers are allowed to separate. You should also have two beakers ready, one labelled "organic layer" and the other labelled "aqueous layer".



Which solvent is to be used depends on the solubility of the substance to be extracted in the solvent and whether it is suitable for subsequent crystallization or distillation.

The common solvents which are used for the extraction are diethyl ether, benzene, etc.,

*There are four extraction methods:

- 1) Solvent extraction
- 2) Extraction based on chemical reaction

3) sequential liquid–liquid extractions

4) Solid extraction

1) SOLVENT EXTRACTION

Extraction with solvents is used as a method of separation of dissolved substances from solutions. Extractions made from solvents are based on the "law of disintegration of Nerst". Distribution law or the **Nernst's distribution law** gives a generalisation which governs the distribution of a solute between two non miscible solvents.

Nernst Distribution law. "At constant temperature, a solute distributes itself between two immiscible solvents only in a particular ratio".

The Nernst distribution law permits us to determine the most favorable conditions for the extraction of substances from solutions.

*The Nernst distribution law states that, at equilibrium, the ratio of the concentrations of a third component in two liquid phases is constant. The law may be expressed in the form

$$K = \frac{\text{solubility of organic (g/100 mL)}}{\text{solubility of water (g/100 mL)}}$$

K= distribution coefficient

The Nernst distribution law permits us to determine the most favorable conditions for the extraction of substances from solutions.

**The purpose in extraction is to be able to distinguish both organic matter and not to waste too much solvent.

* When V ml aqueous solution containing M grams of compound is extracted with X ml organic solvent, M₁ g compound remains in the aqueous phase.

$$K = \frac{M_1/V}{(M_0 - M_1)/X} \quad \text{or} \quad M_1 = M_0 \frac{KV}{KV + X}$$

The same amount is subjected to a second extraction using X ml of organic solvent, the following equations can be written for the M₂ g substance remaining in the aqueous phase.

$$K = \frac{M_2/V}{(M_1 - M_2)/X} \quad \text{or} \quad M_2 = M_1 \frac{KV}{KV + X} = M_0 \left(\frac{KV}{KV + X} \right)^2$$

If the same process is done "n" times, the amount of substance remain in the water phase is M_n;

$$M_n = M_0 \left(\frac{KV}{KV + X} \right)^n$$

*A good solvent for extraction should satisfy two important conditions.

(a) The substance to be extracted should be highly soluble in the solvent.

(b) After the extraction the solvent should be easily separable from the solute.

The mixture of urea and benzoic acid can be separated using solvent extraction process. The mixtures are taken in a separating funnel, to the substances (Urea and benzoic acid) which is solid, diethyl ether is added. The two mixtures are shaken well to get a solution. Only benzoic acid is soluble in ether, while urea is not. Urea is at the bottom of the separating funnel, and it is collected. Now the benzoic acid in ether is left, which on heating, we get benzoic acid.

*During extraction process undesirable conditions may be encountered, such as non-separation of phases or emulsion formation.

In this case:

-The aqueous phase may be saturated with sodium chloride.

-If the solvent is suitable, a few drops of alcohol may be added.

- It may be waited for a while.

-Air may be blown through the water phase with a pipette.

-It may be heated.

2) EXTRACTION BASED ON CHEMICAL REACTION

In this type of extraction, the substance to be separated gives a chemical reaction with the extraction solution.

For example, an organic acid which dissolved in an organic solvent with a few other substances, can be pass into the aqueous phase by creating a salt form after an organic reaction with an aqueous solution of an inorganic base.

3) SEQUENTIAL LIQUID-LIQUID EXTRACTIONS

This continuous extraction is carried out when organic substances dissolve more in water than in organic solvents or in a solid phase and are less soluble in organic solvents.

In this method, even though the solubility is against the organic solvent, more substances can be recovered by using less solvent.

4) EXTRACTION FROM SOLID DRUGS

Extraction, as the term is used pharmaceutically, involves also the separation of medicinally active components by using selective solvents in standard extraction procedures. The products so obtained from plants are relatively impure liquids, semisolids or powders.

These include classes of preparations known as maceration, decoctions, infusions,

(Methods of Extraction of Medicinal Plants)

Maceration

In this process, the drug is extracted with the solvent liquid between 2-14 days. Usually the process is carried out at 15-20 ° C. The insoluble solid is removed by filtration. It is necessary to prevent evaporation of solvent during filtration.

Infusion

This is a form of maceration in which gentle heat is used during the process of extraction. It is used when moderately elevated temperature is not objectionable. The solvent efficiency of the menstruum is thereby increased.

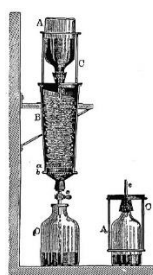
Every pot of coffee or cup of tea involves solid/liquid extraction, the extraction of organic compounds from solid ground beans or leaves using hot water as the liquid. The lower molecular weight polar molecules such as caffeine dissolve in the hot water and are removed from the high molecular weight water-insoluble cellulose, protein, and lipid materials.

Decoction

In this process, the crude drug is boiled in a specified volume of water for a defined time; it is then cooled and strained or filtered. This procedure is suitable for extracting water-soluble, heatstable constituents. The concentrated extract is filtered and used as such or processed further.

Percolation

This is the procedure used most frequently to extract active ingredients in the preparation of tinctures and fluid extracts. A percolator (a narrow, cone-shaped vessel open at both ends) is generally used. The solid ingredients are moistened with an appropriate amount of the specified menstruum and allowed to stand for approximately 4 h in a well closed container, after which the mass is packed and the top of the percolator is closed. Additional menstruum is added to form a shallow layer above the mass, and the mixture is allowed to macerate in the closed percolator for 24 h. The outlet of the percolator then is opened and the liquid contained therein is allowed to drip slowly. Additional menstruum is added as required, until the percolate measures about three-quarters of the required volume of the finished product. The marc is then pressed and the expressed liquid is added to the percolate. Sufficient menstruum is added to produce the required volume, and the mixed liquid is clarified by filtration or by standing followed by decanting.



Hot Continuous Extraction (Soxhlet)

In this method, the finely ground crude drug is placed in a porous bag or “thimble” made of strong filter paper, which is placed in chamber E of the Soxhlet apparatus (Figure 2). The extracting solvent in flask A is heated, and its vapors condense in condenser D. The condensed extractant drips into the thimble containing the crude drug, and extracts it by contact. When the level of liquid in chamber E rises to the top of siphon tube C, the liquid contents of chamber E siphon into flask A. This process is

continuous and is carried out until a drop of solvent from the siphon tube does not leave residue when evaporated. The advantage of this method, compared to previously described methods, is that large amounts of drug can be extracted with a much smaller quantity of solvent. This effects tremendous economy in terms of time, energy and consequently financial inputs. At small scale, it is employed as a batch process only, but it becomes much more economical and viable when converted into a continuous extraction procedure on medium or large scale.

